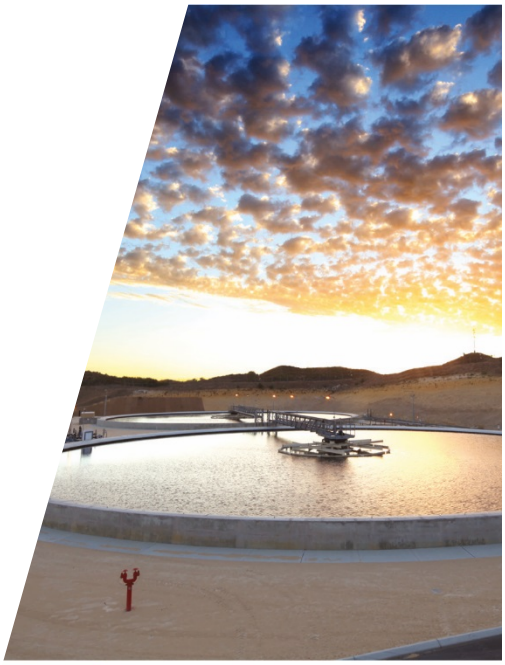
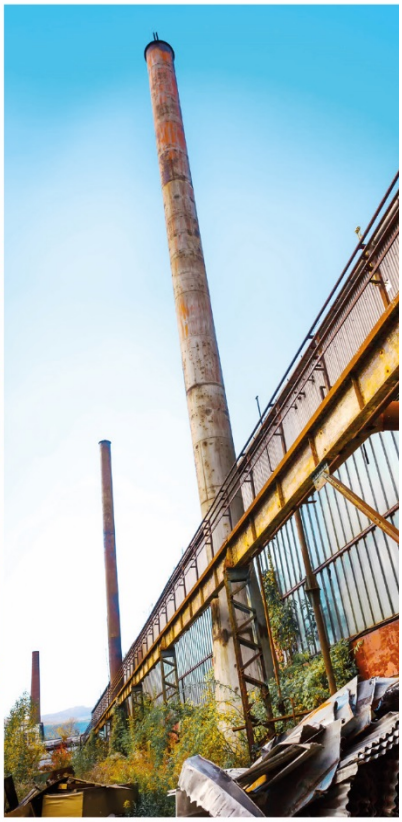


RCRA Facility Investigation Work Plan

Former Cities Refinery
East Chicago, Indiana

Oxy USA, Inc.



RCRA Facility Investigation Work Plan
Former Cities Refinery, East Chicago, Indiana
OXY USA, Inc.
Revised: June 2020

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Appendix Index

1. Introduction

GHD Services, Inc. (GHD) has prepared this Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan (Work Plan) on behalf of OXY USA, Inc. (OXY) for the Former Cities Service Oil Company Refinery (Former Cities Refinery or Facility or Site) in East Chicago, Indiana (U.S. EPA RCRA I.D. # INR000123927, formerly part of IND095267381). The Work Plan has been prepared in accordance with Administrative Order on Consent (AOC) between the United States Environmental Protection Agency – Region 5 (U.S. EPA) and OXY, with an effective date of January 24, 2020. The Work Plan was originally submitted on February 25, 2020, and U.S. EPA comments we received on May 29, 2020; the comments have been addressed and incorporated in this revised Work Plan.

The Former Cities Refinery property is presented on Figure 1.1. The Former Cities Refinery encompasses approximately 93.5 acres. The property is located to the south of the CITGO Terminal.

In accordance with the AOC and Corrective Action Framework (CAF), the purpose of the RFI investigation is to:

1. Describe the nature and extent of any releases of hazardous waste and hazardous constituents at or from the Facility that may pose an unacceptable risk to human health and the environment
2. Explain whether each release poses an unacceptable risk to human health and the environment
3. Provide the basis for those conclusions, including an evaluation of the risks
4. Provide a basis for developing the final corrective measures for the Facility

Additional phases of investigation may be required, as necessary. The need for further data collection will be based on the investigation results, and will be completed in consultation with the U.S. EPA. Separate work plans will be prepared for additional phases of investigation, as necessary, which will include investigation locations, vertical extent, density, methodology, Contaminants of Potential Concern (COPCs), screening data, and corresponding rationale. Separate work plans will be reviewed and approved by U.S. EPA consistent with the approved CAF systematic planning process.

2. Background

Prior to the execution of their respective AOCs, CITGO and OXY jointly coordinated efforts to conduct a Site Perimeter Investigation, covering both the Former Cities Refinery and the CITGO Terminal. The Site Perimeter Investigation was completed in accordance with the "Site Perimeter Investigation Work Plan – Phase I" submitted to the U.S. EPA on March 13, 2019. The U.S. EPA commented on the submission and responses were provided to the U.S. EPA on April 14, 2019. The U.S. EPA approved the Work Plan, as modified by the responses, on April 19, 2019. The primary goal of the Site Perimeter Investigation was to investigate the perimeter of the Former Cities

Refinery and the CITGO Terminal to identify potential for off-Facility impacts. The results of the Site Perimeter Investigation were presented to U.S. EPA during a September 24, 2019 meeting which included the conceptual scope of work for the next phase of investigation (the basis of this Work Plan). The Investigation Results Report was submitted to U.S. EPA on October 31, 2019.

OXY and CITGO may continue to coordinate efforts to increase efficiency and avoid duplication of elements of the corrective action of relevance to both parties. OXY and CITGO are proceeding under separate AOCs and CAFs for their respective portions of the contiguous property that was once owned by Cities Service Oil Company.

3. RFI Scope of Work

The soil and groundwater sampling design and procedures shall be consistent with applicable guidance, including but not limited to: Soil Screening Guidance (U.S. EPA 1996, 2002); Guidance on Choosing a Sampling Design for Environmental Data Collection (U.S. EPA 2002); and Incremental Sampling Methodology (ITRC 2012).

3.1 Monitoring Well Installation

Twelve new monitoring wells are proposed for this phase of the RFI investigation. The proposed monitoring well locations are presented on Figure 3.1. In general, the RFI investigation locations were selected for one or more of the following reasons:

- Evaluate locations with elevated LIF or MIP response from the Site Perimeter Investigation
- Evaluate locations on or near the Facility perimeter with potential soil or groundwater impacts
- Evaluate former operational areas, historical investigation areas, utilities
- Evaluate groundwater flow across the Facility, and evaluate the potential for utility interference/preferential pathways

Monitoring wells will be constructed from 2-inch diameter PVC. A 5-foot screen is proposed for monitoring wells that are installed to collect a sample from a targeted depth based on the Site Perimeter Investigation membrane interface probe (MIP) results responses and a 10-foot screen across the water table is proposed for all other monitoring wells. Due to the high groundwater table, typical monitoring well completion details may need to be modified, or the ground surface may need be raised, where required (to ensure a minimum 2 feet of bentonite sealing, etc.). Either stickup or flush mount protective casings are proposed, as appropriate based on the location of the well.

Following installation, the monitoring wells will be surveyed in Indiana West State Plane NAD83 horizontal coordinate system and the NAVD88 vertical datum for location and reference point elevations to support the evaluation of groundwater flow directions. The groundwater monitoring wells will be developed and gauged with a dual phase probe to determine groundwater elevation and to identify the presence/absence of light non-aqueous phase liquid (LNAPL). Investigation derived waste generated from the installation of the monitoring wells and development of the monitoring wells will be managed in accordance with applicable laws.

Monitoring wells will be installed and developed in accordance with GHD's Standard Operating Procedures (SOPs) which are included in the Sampling and Analysis Plan (SAP) presented in Appendix A.

Note, there is an existing monitoring well/piezometer network at the Site; however, OXY does not propose to use these locations to collect samples since the wells are not properly constructed (1-inch diameter, do not have a protective casing, and in many cases the screen is located below the water table).

Additional wells, if needed, to characterize groundwater quality downgradient of locations reporting LNAPL, will be approved by U.S. EPA prior to installation.

3.2 Soil Sampling

GHD proposes to collect two soil samples (1 sample in the 0-2 ft bgs interval and 1 sample immediately above the water table), plus applicable Quality Assurance/Quality Control (QA/QC) samples, as specified in the Quality Assurance Project Plan (QAPP) which is presented in Appendix B, during the installation of each monitoring well to evaluate potential exposures. In accordance with the CAF, soil samples will be analyzed for target compound list (TCL) volatile organic compounds (VOCs), TCL semi-volatile organic compounds (SVOCs),target analyte list (TAL) inorganics, and 1,4(p)-dioxane. All detected compounds will be reported.

Samples will be collected and analyzed in accordance with the procedures outlined in the SAP (Appendix A) and QAPP (Appendix B).

3.3 Groundwater Sampling

Quarterly groundwater sampling events and quarterly gauging events for one year or four events are proposed as part of the scope of work. Investigation derived waste generated from the quarterly groundwater sampling events will be managed in accordance with applicable laws.

Prior to the collection of the first round of groundwater samples, a full round of static groundwater elevations will be collected from new monitoring wells (12), existing groundwater monitoring wells (21), and existing piezometers (6). Groundwater elevations will be measured to the nearest 0.01 foot using a dual phase probe. If LNAPL is present, a correction to the measured groundwater level will be made based on the specific gravity and thickness of the LNAPL. Static groundwater elevations will be collected in accordance with GHD's SOPs which are included in the SAP provided in Appendix A. Following the first round of water levels, the existing monitoring well network will be evaluated for use in future rounds of static groundwater elevation monitoring events.

One representative groundwater sample will be collected from each monitoring well using low-flow procedures (e.g., low flow sampling at a rate between 0.1 and 0.5 liter per minute [L/min]), plus applicable QA/QC. In accordance with the CAF, groundwater samples will be analyzed for TCL VOCs, TCL SVOCs, dissolved TAL inorganics, and 1,4(p)-dioxane. All detected compounds will be reported.

Samples will be collected and analyzed in accordance with the procedures outlined in the SAP (Appendix A) and QAPP (Appendix B).

3.4 LNAPL Sampling

If measurable LNAPL thickness is reported in any of the monitoring wells, a groundwater sample will not be collected from the location reporting LNAPL, but from a downgradient location with no LNAPL. The LNAPL sample will be collected (if sufficient LNAPL can be recovered), and analyzed for TCL VOCs, TCL SVOCs, TAL inorganics, and 1,4(p)-dioxane. LNAPL samples will also be evaluated for physical properties (e.g., specific gravity, viscosity), as applicable. All detected compounds will be reported.

Groundwater will be sampled downgradient of locations of reported LNAPL, based on groundwater contours. This may require the installation of additional wells. Following each event, the need for additional wells will be evaluated. A scope of work with proposed monitoring wells will be submitted to EPA for review and approval, with the intent of having new monitoring wells installed prior to the next monitoring event. If off-Site monitoring locations are required, appropriate access will be obtained prior to completing well installations.

It is important to note that collecting a groundwater sample from a location with LNAPL results in significant risk in producing erroneous/non-representative results. The potential for contamination of the groundwater sample will be high, and the possibility that the reported results will be biased high will therefore also be significant. (Zemo 2006), concluded that: "…ground water samples collected within the smear zone should be critically evaluated and not be assumed to represent only the dissolved phase or be acceptably reproducible. This evaluation shows that a significant percentage of ground water monitoring data collected from the smear zone is unreliable for characterizing dissolved concentrations of petroleum constituents." It is notable that this article considered the effect of turbidity (and NAPL adsorbed to sediment) and did not even address the significant added complication and potential impact of actual NAPL presence (sheen or measurable thickness).

Samples will be collected and analyzed in accordance with the procedures outlined in the SAP (Appendix A) and QAPP (Appendix B).

3.4.1 LNAPL Mobility Evaluation

Evaluation of LNAPL mobility, if reported in wells, will be based on an assessment of LNAPL transmissivity (T_n). The evaluation of T_n provides a standardized science-based method to quantify the potential mobility and recoverability of LNAPL at a given site. Results can be compared against widely accepted de minimis criteria to assess whether LNAPL may be considered to be sufficiently mobile such that hydraulic recovery may be feasible and/or provide some technical benefit in terms of reducing LNAPL saturations (or mass) in the interest of mitigating LNAPL migration potential. Where T_n is found to be of de minimis magnitude, LNAPL is considered to be largely present at residual levels and hydraulically immobile and unrecoverable. Where this is the case, LNAPL mass recovery efforts will not provide a technical benefit in terms of mitigating mobility potential since the LNAPL body as a whole will already be largely immobile.

The testing will be performed pursuant to the methodology contained in ASTM International (ASTM) Standard E2856-13 *Standard Guide for Estimation of LNAPL Transmissivity* (May 2013) using the baildown technique at selected wells with in-well LNAPL thickness greater than 0.5 feet. The test involves the rapid removal of LNAPL from the well (via bailer), followed by the monitoring of LNAPL recharge into the well using an oil-water interface probe. The LNAPL monitoring continues until the

observed in-well LNAPL recharge data provides sufficient information to estimate T_n. Wells exhibiting no LNAPL or less than 0.5-feet in-well LNAPL thickness will be assumed to represent at de minimis LNAPL mobility/recoverability condition by default.

LNAPL transmissivity will be estimated based on the observed LNAPL recharge rates and/or LNAPL drawdown recovery (depending on the analytical solution) using the American Petroleum Institute (API) *LNAPL Transmissivity Workbook: Calculation of LNAPL Transmissivity from Baildown Test Data* (September 2012). The API workbook uses the field data from a baildown test to estimate LNAPL transmissivities using three different solutions for unconfined conditions: Bouwer & Rice; Cooper & Jacob; and Cooper-Bredehoeft-Papadopulos. The detailed field methodology and data treatment techniques associated with LNAPL transmissivity estimations are detailed in ASTM E2856-13.

The results will be compared against a guideline de minimis criterion of 0.8 ft^2/day (0.08 m^2/day) recommended by the Interstate Technology & Regulatory Council (ITRC Publication No. LNAPL-3, 2018). Values of T_n greater than the de minimis criterion indicate hydraulic LNAPL recovery is technically feasible and may be beneficial in providing a meaningful reduction in LNAPL saturation and mobility. Where T_n is less than the de minimis criterion, it can be assumed that most of the LNAPL exists as unrecoverable residual. When this is the case, ongoing LNAPL recovery efforts are not expected to provide a meaningful reduction in LNAPL saturation; and therefore, will not provide a beneficial change in subsurface conditions.

For wells exhibiting elevated T_n values, additional work may be required to quantify the potential benefit of LNAPL recovery. While T_n describes whether a minimum LNAPL recharge rate can be sustained such that there may be a mobility/recoverability concern, it does not quantify what fraction of the LNAPL in place might be considered mobile and/or recoverable. Additional work in this regard may be based on a targeted soil core sampling and petrophysical laboratory testing program that will allow an additional quantification of LNAPL mobility and an assessment of the need for and expected value of LNAPL recovery in terms of the realistic potential change in subsurface conditions. If necessary, additional work related to LNAPL mobility will be detailed in future phases of work.

3.5 Screening Levels/Risk Assessment

RFI investigations will continue until there is sufficient data to define the vertical and horizontal extent of COPC-impacted soil and groundwater.

Data will be initially screened against IDEM published background levels for metals. Data below the IDEM published background levels will not be evaluated further. Remaining data will be screened against the 2019 IDEM screening and closure tables, which are based on the 2018 U.S. EPA Regional Screening Levels (RSLs) (residential at property boundary and industrial at the Former Refinery). For chemicals with maximum contaminant levels (MCLs), they will be used in lieu of IDEM screening levels for drinking water. COPC impacts will be delineated to residential land use criteria at the property boundary, but any corrective actions will consider actual land use (i.e., industrial, on site) and may incorporate institutional/engineering controls to eliminate potential exposure pathways. LNAPL analytical data is for informational purposes and will not be screened against published levels.

After the first two groundwater sampling events, the COPC parameter list will be evaluated and reductions to parameters, if appropriate, will be proposed to U.S. EPA for approval.

Risk assessments will estimate human health and ecological risk under reasonable maximum exposure for both current and reasonably expected future land use scenarios. In conducting the risk assessments, Respondent will consider the Risk Assessment Guidance for Superfund (RAGS) or other appropriate U.S. EPA guidance. Respondent will use appropriate conservative screening values when screening to determine whether further investigation is required. Appropriate screening values, which will be determined by U.S. EPA after consultation between U.S. EPA and OXY, and may include those derived from Federal Maximum Contaminant Levels, U.S. EPA Regional Screening Levels for Chemical Contaminants, U.S. EPA Region 5 Ecological Screening Levels, RAGS, Indiana Screening Levels, and U.S. EPA technical documents and tools.

3.6 RFI Scope of Work Summary

The following presents a summary of the proposed RFI investigation field activities:

RFI Scope of Work	Quantity
Monitoring Well Installation	12 wells
Soil Sampling Event	1 event Up to 24 investigative soil samples, plus QA/QC
Groundwater Sampling Events	Quarterly events – 12 groundwater samples, plus QA/QC, per event
Gauging	Quarterly events - new monitoring wells (12)*
LNAPL Sampling	If LNAPL is encountered and sufficient LNAPL can be recovered, LNAPL will be analyzed
LNAPL Mobility	If the in-well thickness of LNAPL is greater than 0.5 ft, conduct transmissivity testing
* The first round of water levels will also include gauging at existing monitoring wells (21) and existing piezometers (6). Following the first round of water levels, the existing monitoring well network will be evaluated for use in future rounds of static groundwater elevation monitoring events.	

The proposed locations of the 12 monitoring wells are presented on Figure 3.1.

A breakdown of each proposed investigation location, the associated Site Perimeter Investigation location with LIF/MIP data (if applicable), the proposed screen length and target depths, and a brief description of the rationale for each well is presented in Table 3.1.

The RFI investigation is intended to be iterative and adaptive based on conditions encountered in the field. Pending the results of the initial 12 investigation locations, additional phases may be identified during or after the quarterly monitoring program, as needed, consistent with the scope and objectives outlined in Section 1 above.

A visual Site inspection will be conducted of on-Site structures for groundwater/oil during each quarterly groundwater event. Removal of oil from the structures will be completed as necessary and documented in the quarterly progress reports.

4. Deliverables

An interim RFI data report summarizing the results of this Work Plan and an updated Conceptual Site Model (CSM), including proposed additional activities, if any, will be prepared for submittal to the U.S. EPA following two rounds of quarterly groundwater sampling. Subsequent quarterly groundwater results will be included in either the quarterly progress reports or in a subsequent interim RFI data report(s) (if applicable).

Once it has been determined that sufficient data has been obtained to describe the nature and extent of any releases of hazardous waste and hazardous constituents at or from the Facility that may pose an unacceptable risk to human health and the environment, a final RFI Report will be developed and submitted to the U.S. EPA by no later than October 1, 2021 unless a revised date is agreed to by both U.S. EPA and OXY. The RFI report will describe the nature and extent of any releases of hazardous waste or hazardous constituents at or from the Facility that do or do not pose an unacceptable risk to human health and the environment, and provide a basis for those conclusions, including an evaluation of the risks. The investigation shall include a consensus driven balance between qualitative and quantitative high-resolution investigation techniques.

5. Schedule

The following presents a tentative schedule for the implementation of this Work Plan, following U.S. EPA approval.

Field activities related to this Work Plan will be initiated within 60 days of U.S. EPA approval of this Work Plan. CITGO and OXY may continue to coordinate efforts to increase efficiency and avoid duplication of elements of the corrective action of relevance to both parties.

Within 60 days of U.S. EPA approval of the RFI Work Plan, an initial cost estimate for the scope of work included in the RFI Work Plan will be provided.

An interim RFI data report, as described in Section 4 above, will be submitted following the completion of two quarterly rounds of groundwater sampling.

The current conceptual project schedule as identified in the CAF is presented in Appendix C.

6. Administrative Requirements

The AOC provides for the following Reporting and Administrative requirements under Section VII. Paragraph 22.

6.1 Job Creation

Job creation has been considered in the development of this Work Plan. Local vendors will be utilized where practicable to perform field activities.

During the Site Perimeter Investigation in 2019, local vendors were commissioned to complete surveying, utility clearance, hydro-excavation, and fencing. Drillers with specialized equipment were

commissioned from out of state (Ohio). Local GHD staff conducted field oversight activities. The project utilized approximately 1,200 hours of on-Site labor, or approximately 6.92 full-time equivalents (FTE) for the duration of field activities (approximately 1 month or 0.58 FTE averaged over the year).

The project utilized approximately 1,000 hours of off-Site labor, or approximately 0.48 FTE averaged over the year, which included technical/professional labor facilitating project management, coordination, work plans, reports, visuals, etc.

Therefore in 2019, the project created several temporary professional, management, technical, and skilled trade jobs (on-Site and off-Site labor) accounting for a FTE of 1.06.

A similar FTE value is expected for work proposed in 2020.

6.2 Public Repository

A public repository was established at a local East Chicago, IN (2401 E Columbus Drive) library for information regarding Facility activities and to conduct public outreach and involvement activities, as needed. Information can also be found on the public EPA webpage at:

https://www.epa.gov/hwcorrectiveactionsites/hazardous-waste-cleanup-former-cities-service-refinery-east-chicago-indiana.

6.3 Green Remediation Best Management Practices

Green Remediation Best Management Practices (BMPs) will be considered when completing the RFI for the Facility in accordance with U.S. EPA 542-F-16-002 "Green Remediation Best Management Practices" to evaluate and minimize the environmental footprint of activities involved in cleaning up contaminated sites.

Activities proposed in this Work Plan include investigation and monitoring. The following BMPs will be considered when implementing this Work Plan:

- Work has been scheduled for Spring 2020, thus reducing the amount of fuel needed for heating or cooling equipment and supplies
- Wherever practicable, local vendors, product suppliers, and laboratories, will be utilized
- Identify local sources of trucks and machinery equipped with advanced emission controls and clean alternative fuels
- Identify the nearest acceptable facility to be used for disposing of wastes
- Establish electronic networks for data transfers, team decisions, and document preparation, and select electronic products through tools such as the Electronic Product Environmental Assessment Tool (EPEAT)
- Reduce travel through teleconferencing and compressed work hours
- Select facilities with green policies for worker accommodations and meetings

When developing interim measures and remedial alternatives to implement Green Remediation Best Management Practices will continue to be evaluated.

6.4 Quarterly Progress Reports

In accordance with the AOC, quarterly progress reports will be submitted to U.S. EPA by the fifteenth day of the month after the end of each quarter. The report will list work performed to date, data collected, problems encountered, project schedule, and percent project completed.

6.5 Semi-Annual Meetings

The parties (OXY and U.S. EPA) will communicate frequently and in good faith and will meet (either by phone or in-person) on at least a semi-annual basis to discuss the work proposed and performed as part of the RFI.

7. References

1. Zemo, D.M. 2006, 'Sampling in the Smear Zone: Evaluation of Nondissolved Bias and Associated BTEX, MTBE, and TPH Concentrations in Ground Water Samples', Groundwater Monitoring & Remediation, vol 26, no 3, pp 125 – 133.

LEGEND

CITGO TERMINAL (228.5 ACRES)

FORMER CITIES REFINERY (93.2 ACRES)

0 300 600ft

Coordinate System: IN83-WF

N

GHD

FORMER CITIES REFINERY
EAST CHICAGO, INDIANA

FACILITY LOCATIONS

Project No. **11208111**
Date **February 2020**

FIGURE 1.1

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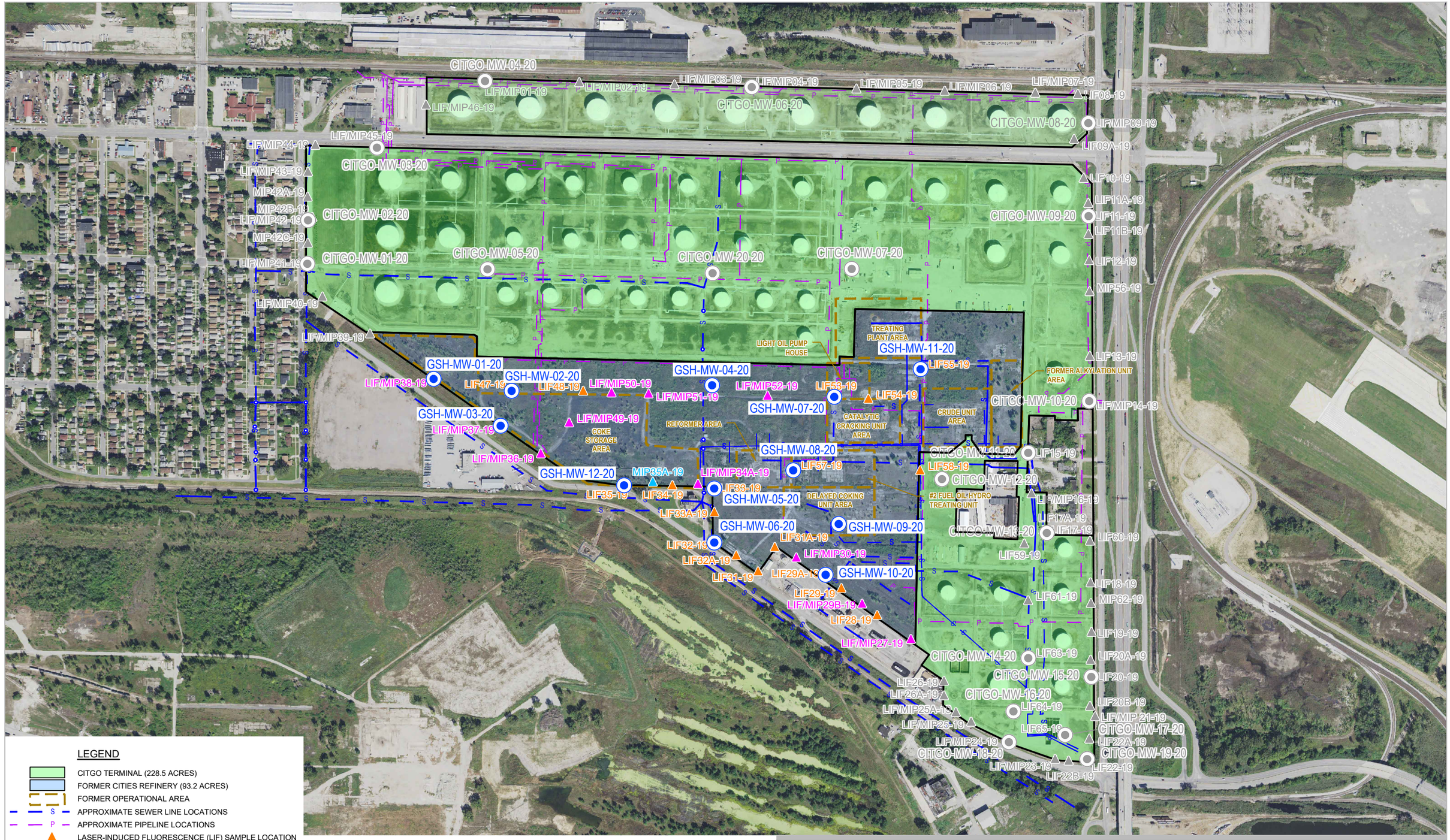

LEGEND

- CITGO TERMINAL (228.5 ACRES)
- FORMER CITIES REFINERY (93.2 ACRES)
- FORMER OPERATIONAL AREA
- S — APPROXIMATE SEWER LINE LOCATIONS
- P — APPROXIMATE PIPELINE LOCATIONS
- LASER-INDUCED FLUORESCENCE (LIF) SAMPLE LOCATION
- MEMBRANE INTERFACE PROBE (MIP) SAMPLE LOCATION
- LASER-INDUCED FLUORESCENCE (LIF) AND MEMBRANE INTERFACE PROBE (MIP) SAMPLE LOCATION
- PROPOSED SOIL SAMPLING/MONITORING WELL LOCATION
- EXISTING CITGO INVESTIGATION LOCATION
- PROPOSED CITGO INVESTIGATION LOCATION

NOTE: FORMER ASBESTOS-CONTAMINATED SOIL AREA NOT IDENTIFIED (AOC 2).

0 300 600ft

Coordinate System: IN83-WF

N

GHD

FORMER CITIES REFINERY
EAST CHICAGO, INDIANA

PROPOSED INVESTIGATION LOCATIONS

Project No. **11208111**
Date **June 2020**

FIGURE 3.1

Source: 2016 AERIAL IMAGE FOR LAKE COUNTY, INDIANA IMAGE PROVIDED BY THE UNITED STATES DEPARTMENT OF AGRICULTURE (USDA) AS PART OF THE NORTH AMERICAN IMAGE PROGRAM (NAIP).

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Plot Date: 03 June 2020 11:12 AM

Table 3.1

Proposed RFI Investigation Rationale
Former Cities Refinery
East Chicago, IN

Proposed MW Location	Associated Site Perimeter Investigation Location	Max LIF Response (%RE)	LIF Response Above Baseline (Y or N)	Max PID ($uVx10^5$) (approx.)	Max FID ($uVx10^5$) (approx.)	MIP Response Above Baseline (Y or N)	Proposed Screen Length (ft) (1)	Target Screen Depth (ft bgs)	Well Location (perimeter or internal)	Purpose/Rationale
GSH Property										
GSH-MW01-20	LIF/MIP38-19	3.4	N	0.17	0.15	**Y**	5	2-7	Perimeter	MIP Response/Groundwater contouring/Neighboring Properties (Residential)
GSH-MW02-20	LIF47-19	66.3	**Y**	na	na	na	10	Water Table	Internal	LIF Response/Groundwater contouring/Former Operations (Tanks/Light oil lines)/Active Pipelines (Explorer, Wolverine)
	LIF48-19	40.3	**Y**	na	na	na				
GSH-MW03-20	LIF/MIP37-19	2.1	N	0.17	0.07	N	10	Water Table	Perimeter	Groundwater contouring
GSH-MW04-20	none	-	-	-	-	-	10	Water Table	Internal	Groundwater contouring (Spacing)/Utility evaluation (City Water)
GSH-MW05-20	LIF33-19	261.6	**Y**	na	na	na	10	Water Table	Perimeter	LIF Response/Utility evaluation (City Water)/Former Operations (Rail/Truck loading area, Tanks)
	LIF33A-19	203.4	**Y**	na	na	na				
GSH-MW06-20	LIF32-19	225.2	**Y**	na	na	na	10	Water Table	Perimeter	LIF Response/Utility evaluation (City Water)/Former Operations (Rail/Truck loading area, Tanks)
	LIF32A-19	292.2	**Y**	na	na	na				
GSH-MW07-20	LIF53-19	130.4	**Y**	na	na	na	10	Water Table	Internal	LIF Response/Utility evaluation (Industrial Sewer System)/Former Operations (Light oil pump house)
	LIF54-19	153.1	**Y**	na	na	na				
GSH-MW08-20	LIF57-19	79.3	**Y**	na	na	na	10	Water Table	Internal	LIF Response/Former Operational Area (#2 Fuel oil hydro treating unit/Reformer Area)
GSH-MW09-20	none	-	-	-	-	-	10	Water Table	Internal	Utility evaluation (Industrial Sewer System)/Former Operational Area (Delayed Coking Unit Area)/ Historical investigations identified LNAPL and benzene in this area
GSH-MW10-20	LIF29-19	13.2	**Y**	na	na	na	10	Water Table	Perimeter	LIF Response/Neighboring Property (Asphalt Plant)
	LIF29A-19	225.8	**Y**	na	na	na				
GSH-MW11-20	LIF55-19	80.1	**Y**	na	na	na	10	Water Table	Internal	LIF Response/Groundwater contouring/Utility evaluation (Industrial Sewer System)/Former Operations (Treatment Plant Area/Crude Unit Area)
GSH-MW12-20	LIF35-19	2.0	N	na	na	na	10	Water Table	Perimeter	Fill gap on southern boundary per EPA comment. Groundwater contouring

Notes:

(1) 5-foot screen used where there is a MIP response at the perimeter. All other monitoring well locations will have 10-foot screens and will straddle the water table.
na - not analyzed
- Shallow soil samples (0-2') are to be collected from each monitoring well location and analyzed for TCL VOCs, TCL SVOCs, and TAL inorganics
- Shallow groundwater samples are to be collected using low-flow sampling procedures and analyzed for TCL VOCs, TCLS SVOCs, and dissolved TAL inorganics

Appendices

Appendix A
Sampling and Analysis Plan (SAP)

Sampling Analysis Plan (SAP)

Former Cities Refinery
East Chicago, Indiana

OXY USA, Inc.

Table of Contents

Appendix Index

1. Introduction

This Sampling Analysis Plan (SAP) is submitted and forms part of the work plans prepared to complete the Resource Conservation and Recovery Act (RCRA) Facility Investigation. This SAP has been prepared by GHD Services, Inc. (GHD) on behalf of OXY USA, Inc. (OXY) for the Former Cities Refinery (Facility) in East Chicago, Indiana (United States Environmental Protection Agency [U.S. EPA] Identification Number INR 000 123 927, formerly part of IND092567381).

The SAP describes procedures for the collection of surface, subsurface soil samples, and groundwater samples. Field Method Guidelines (FMGs) are included in Appendix 1. The SAP provides additional detail on procedures where a FMG is unavailable. If no FMG is available for the particular field method, a complete description of the method will be provided in this text.

2. General Sampling Protocols

2.1 Sampling

Samples will be collected at the locations and frequencies specified in the work plans. As discussed in Quality Assurance Project Plan (QAPP), samples will be collected in order of decreasing analyte volatility (i.e., volatile organic compounds (VOCs) first, semi-volatile organic compounds (SVOCs) second, which are followed by containers for the remaining analysis). A summary of the investigation plan including sample collection and analysis is provided in the RFI Work Plan.

The following protocols will be employed during all sampling conducted during the RFI:

1. All sampling instruments and equipment will be cleaned in accordance with the protocol presented herein prior to collecting samples for chemical analyses at each location (refer to FMG 9.0).
2. A double pair of disposable nitrile gloves will be used at each sample location for chemical analyses. A new overpair will be used for each sample. Additional glove changes at each sample location will be made for conditions such as: if the gloves are observed to be torn, or the gloves are suspected of being soiled from a source other than the sample media itself.
3. Quality assurance samples will be collected as outlined in the QAPP.
4. All sampling generated wastes such as gloves, tyvek, etc. will be collected and containerized for proper disposal (refer to FMG 10.0).
5. Samples will be labelled noting the project name, Facility, unique sample number, sample location, sample interval (if appropriate), analysis required, preservative added, date, time and sampler's initials, in accordance with the QAPP. All sample preservation protocols will be followed in adherence with the QAPP. A hard cover bound field book and/or field forms will be maintained to record all samples and sampling events (refer to FMG 1.4).
6. Containers for sample collection and preservation requirements will be determined as required by the analytical parameters (refer to the QAPP). All sample bottles will be provided

by the laboratory and will be prepared using a standard laboratory validated washing procedure.

7. All collected sample shipments for chemical analysis will be iced in supplied coolers after collection and labelling. Any remaining space will be filled with packing to cushion the containers within the shipment coolers. Each cooler will be sealed with two seals comprised of Chain-of-Custody tape (or custody seal) and the sampler's name. The cooler will then be sealed with packing tape (refer to FMG 6.10).

   All samples will be delivered to the laboratory by a commercial courier.

8. Samples will be shipped under chain-of-custody procedures as outlined in the QAPP and FMG 6.10.

2.2 Equipment Cleaning

Upon mobilization of the drill rig to the Facility, and prior to commencing work, the drill rig and all associated equipment will be thoroughly cleaned in accordance with FMG 9.0. Cleaning of non-dedicated downhole equipment will be completed prior to use and between each borehole.

2.3 Waste Handling

All soil cuttings, development/purge water, excess sampling water, and decontamination water/fluids will be placed in containers and stored in GSH's designated disposal area (as required) until they can be properly characterized, in accordance with FMG 10.0. All soil cuttings and/or water collected during well purging and development will be tested using the Toxicity Characteristics Leaching Procedure (TCLP) to determine appropriate disposal options. This waste will be properly stored, characterized, and disposed according to appropriate regulations.

2.4 Utility Clearance

Prior to commencing work at the Facility, all utilities will be cleared in accordance with FMG 1.3.

For this project, the following state utility clearance service is:

- Indiana 811: 800-382-5544 or 811 when in Indiana

All locations on-Facility must be cleared through the Facility contact:

- Mr. Rick Passmore 859-221-7616

If Facility utility maps and a Facility-specific knowledge are insufficient to locate utilities, a private utility located may also be used.

2.5 Field Instrument Use and Calibration

All field instruments utilized during the field implementation will be used and calibrated in accordance with FMG 8.0.

3. Soil Sampling Protocols

3.1 Subsurface Soil Sampling

Soil samples will be collected continuously during drilling to identify and classify soil materials (refer to FMGs 2.2, 2.3, 2.6, and 6.1). Drilling techniques for subsurface soil sampling will include hollow-stem auger and/or direct-push methods. Soil samples will be collected using the standard penetration test method and/or Shelby tubes, or alternately, continuous sampling techniques such as direct push methodologies (e.g., Geoprobe). All collected soil samples will be described and classified according to the Unified Soil Classification System (USCS), as described in FMG 2.6).

The soil samples selected for chemical analyses will be collected in accordance with FMG 6.1 and Section 4 of the QAPP. Additional details (not included in the FMGs) for collecting VOC samples are provided below:

- VOC analysis will utilize the methanol field preservation kit as follows:
  - Using the 10-gram sampling device, transfer one 10-gram soil plug from the sample point to the 40-ml VOA vial containing methanol. Quickly cap and tighten the lid.
  - For soils being preserved for volatile analyses, one vial is needed per test. For each analysis, place 10 grams of soil into a 40-ml, tared vial containing 10 milliliters of methanol.
  - Put soil from the sample point in the dry weight jar, and screw the cap tight.

Dry weight analysis must be performed from an unpreserved jar. This container can be from another test being performed or a separate 2-oz plastic container can be used.

The quality assurance/quality control (QA/QC) procedures for the analyses are outlined in the QAPP.

3.2 Surface Soil Sampling

Surface soil samples will be collected in accordance with the following protocols:

1. Surficial soil samples will be collected using a pre-cleaned stainless steel trowel or other appropriate tool (e.g., hand auger, SPT, Shelby tube, etc.). Each sample will consist of soil from the 0-2-foot interval (excluding pavement). Any surficial debris (i.e., grass cover) should be removed from the area where the sample is to be collected using a separate pre-cleaned device.
2. A double pair of disposable nitrile gloves will be used at each sample location (the outer gloves will be changed for each sample).
3. Prior to use, for each sample, all sampling equipment will be pre-cleaned using the prescribed decontamination procedure (refer to FMG 9.0).
4. Samples for VOC analysis will be collected using methanol preservation kit as follows:
   - Using the 10-gram sampling device, transfer one 10-gram soil plug from the sample point to the 40-ml VOA vial containing methanol. Quickly cap and tighten the lid.

- For sols being preserved for volatile analyses, one vial is needed per test. For each analysis, place 10 grams of soil into a 40-ml, tared vial containing 10 milliliters of methanol.
- Put soil from the sample point in the dry weight jar, and screw the cap tight.

5. Samples for chemical analysis will be in accordance with FMG 6.1.
6. The sample locations will be surveyed upon completion.

3.3 Borehole Abandonment

Upon completion of soil boring/sampling, the boreholes will be abandoned/sealed in accordance with FMG 2.5.

4. Hydrogeologic Investigation

4.1 Monitoring Well Installation Procedures

Monitoring well installations will be completed in accordance with U.S. EPA technical guidance and referenced FMGs, where available. Soil samples for stratigraphic definition will be collected continuously in accordance with FMGs 2.3 and 6.1. All cuttings will be containerized and disposed of in accordance with FMG 10.0.

The overburden monitoring wells will be completed in accordance with FMGs 3.1 and 3.2.

4.2 Water Level Measurements

Following installation of the monitoring wells, a ground elevation and a top of casing (TOC) measuring point elevation survey will be conducted for all recently completed wells. The groundwater elevation at each well will be measured in accordance with FMG 5.1 and will be completed within a 24-hour period in order to avoid readings over potential changes in weather. All data will be reduced to water elevations and contoured, if applicable.

4.3 Slug Injection Tests

Slug injection tests will be completed in accordance with FMG 5.2.

4.4 Well Development

All monitoring wells will be developed prior to sampling in accordance with FMG 3.6.

4.5 Well Decommissioning

All well abandonment/decommissioning will be completed in accordance with FMG 3.7, as necessary.

5. Groundwater Quality Sampling Protocols

All wells used for groundwater quality monitoring will be sampled utilizing low-flow sampling techniques according to FMG 6.4. Monitoring and sample collection for non-aqueous phase liquid will be completed in accordance with FMG 6.5, if applicable. Wells will not be sampled for groundwater analyses if NAPL is present in the well.

6. Field Log and Field Forms

The written field data will be recorded on standardized, pre-printed field forms or in a field log book, in accordance with FMG 1.4.

7. Sample Shipment and Containers

All samples collected and submitted to the laboratory for analysis will be handled and shipped in accordance with FMG 6.10 and Section 2 of this SAP.

Appendix 1
Field Method Guidelines

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LIST OF FORMS
(Following Text)

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 1.3
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 0	REVISION DATE:

UTILITY CLEARANCE

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

Invasive field investigation activities such as drilling, soil gas surveys, test excavation or remedial construction activities require location of underground utilities prior to initiating work. Such clearance is sound practice in that it minimizes the potential for damage to underground facilities and more importantly, is protective of the health and safety of personnel. Under no circumstances will invasive activities be allowed to proceed without obtaining proper utility clearance by the appropriate public agencies and/or private entities. **This clearance requirement applies to all work on both public and private property, whether located in a dense urban area or a seemingly out-of-the-way rural location.**

The responsibility of obtaining this clearance lies with the Consultant or Contractor performing the work.

In most states such utility clearance is required by law, and obtaining clearance includes contacting a public or private central clearance agency via a "one-call telephone service and providing them with proposed exploration location information. This is discussed in more detail herein. It is important to note that public utility agencies may not, and usually don't have information regarding utility locations on private property. As such, utility clearance on OXY property must be cleared using available site drawings, and written approval must be obtained from personnel with appropriate knowledge of existing utilities. In the event the utility clearance is required for an active facility, the Facility Area Manager (FAM) will be the single source of contact for utility clearances.

PROCEDURES REFERENCED

- FMG 1.1 - Interior and Exterior Inspections
- FMG 1.2 - Soil-Gas Surveys (Passive and Active)

- FMG 2.0 - Subsurface Investigations
- FMG 4.1 - EM Survey

PROCEDURAL GUIDELINES

- Before marking any proposed exploration or underground construction locations, it is critical that all readily available information on underground utilities and structures be obtained. This includes publicly available information as well as information in the possession of private landowners. Any drawings obtained must be reviewed in detail for information pertaining to underground utilities. The FAM will be the single point of contact for utility clearances at active facilities. At active facilities Form FMG 1.3-02 - Utility Clearance Checklist (Active Facilities) will be completed.
- Using the information obtained, the site should be viewed in detail for physical evidence of buried lines or structures, including pavement cuts and patches, variation in or lack of vegetation, variations in grading, etc. (Care must also be taken to avoid overhead utilities as well.) Presence of surface elements of buried utilities should be documented, such as manholes, gas or water service valves, catch basins, monuments, or other evidence.
- Overhead utility lines must be taken into account when choosing exploration and excavation locations. Most states require a minimum of 10 feet of clearance between equipment and energized wires. Such separation requirements may also be voltage-based and may vary depending on state or municipality regulations.
- In evaluating clearance from overhead lines, the same restrictions may apply to "drops", or wires on a utility pole connecting overhead and underground lines.
- Using the information obtained and observations made, proposed exploration or construction locations should be marked in the field. Marking locations can be accomplished using spray paint on the ground, stakes, or other means. **All markings of proposed locations should be made in white**, in accordance with the generally accepted universal color code for facilities identification (AWMA 4/99):
  - White: Proposed Excavation or Drilling Location.
  - Pink: Temporary Survey Markings.
  - Red: Electrical Power Lines, Cables, Conduit, and Lighting Cables.
  - Yellow: Gas, Oil, Steam, Petroleum, or Gaseous Materials.
  - Orange: Communication, Alarm or Signal Lines, Cables, or Conduits.
  - Blue: Potable Water.
  - Purple: Reclaimed Water, Irrigation, and Slurry Lines.
  - Green: Sewers and Drain Lines.

- In order to effectively evaluate the proposed locations with these entities, detailed, accurate measurements between the proposed locations and existing surface features should be obtained. Such features can be buildings, street intersections, utility poles, guardrails, etc.
- Obtaining the utility clearance generally involves two entities:
  - The designated "one-call" underground facilities protection organization for the area; and
  - The landowner.

  Both entities must be contacted and the proposed locations evaluated in light of information available for existing underground facilities. The detailed measurement information described above will be required by the "one call" agency. The owners of the applicable, participating, underground utilities are obligated mark their respective facilities at the site in the colors described above. Utility stake-out activities will typically not commence for approximately 72 hours after the initial request is made.
- The public and private utility entities generally only mark the locations of their respective underground facilities within public rights-of-way. Determination of the locations of these facilities on private property will be the responsibility of the project Consultant or Contractor. If available information does not contain sufficient detail to locate underground facilities with a reasonable amount of confidence, alternate measures may be appropriate, as described below. In some cases, the memory of a long-time employee of a facility on private property may be the best or only source of information. It is incumbent on the Consultant or Contractor to exercise caution and use good judgement when faced with uncertainty.

  Note: It is important to note that not all utilities are participants in the "one-call" agency or process. As such, inquiries must be made with the "one-call" agency to determine which entities do not participate, so they can be contacted independently.

  Most utility stake-outs have a limited time period for which they remain valid, typically 2 to 3 weeks. It is critical that this time period be taken into account to prevent expiration of clearance prior to completion of the invasive activities, and the need to repeat the stake-out process.
- Care must be exercised to document receipt of notice from the involved agencies of the presence or absence of utilities in the vicinity of the proposed locations. FMG 1.3-01 - Property Access/Utility Clearance Data Sheet can be used to record contacts made and responses received from each entity.

  Most agencies will generally provide a telephone or fax communication indicating the lack of facilities in the project area. **If contact is not made by all of the agencies identified by the "one-call" process, do not assume that such utilities are not present. Re-contact the "one-call" agency to determine the status.**
- For complicated sites with multiple proposed locations and multiple utilities, it is advisable to arrange an on-site meeting with utility representatives. This will minimize the potential for miscommunication amongst the involved parties.
- Completion of the utility stake out process is not a guarantee that underground facilities will not be encountered in excavations or boreholes; in fact, most "one-call" agencies and

individual utilities do not offer guarantees, nor do they accept liability for damage that might occur. Accordingly, it is advisable that any invasive activities proceed with extreme caution in the upper 4 to 5 feet in the event the clearance has failed to identify an existing facility, or possibly deeper at a facility that has built new surfaces on fill dirt on top of old slabs. This may necessitate hand-excavation or probing to confirm potential presence of shallow utilities. If uncertainty exists for any given utility, extra activities can be initiated to solve utility clearance concerns. These options include:

- Hand digging, augering or probing to expose or reveal shallow utilities and confirm presence and location. In northern climates this may require advancing to below frost line, typically at least 4 feet.
- Screening the proposed work areas with utility locating devices, and/or hiring a utility locating service to perform this task. The private utility locating service is a growing industry that has formed a national organization. The National Utility Locate Contractors Association (NULCA) can be reached at 715-635-6004.

EQUIPMENT/MATERIALS

- White spray paint.
- Wooden stakes, painted white or containing white flagging.
- Color-code key.
- Available drawings.
- Form FMG 1.3-01 - Property Access/Utility Clearance Data Sheet and Form FMG 1.3-02 - Utility Clearance Checklist (Active Facilities).

REFERENCES

American Public Works Association, April 1999, Uniform Color Code (http://www.apwa.net/).

PROPERTY ACCESS/UTILITY CLEARANCE DATA SHEET

PROJECT NAME: _____ PROJECT NUMBER: _____

OXY REPRESENTATIVE: _____

CLIENT: _____ CLIENT REPRESENTATIVE: _____ PHONE: _____

ON-SITE PROPERTY ACCESS APPROVAL _____ (OWNER OR AUTHORIZED AGENT SIGNATURE)

OFF-SITE PROPERTY ACCESS APPROVAL (if applicable) _____ (OWNER OR AUTHORIZED AGENT SIGNATURE)

UTILITY CLEARANCE APPROVAL _____ (OWNER OR AUTHORIZED AGENT SIGNATURE)

CONTRACTOR VERIFICATION APPROVAL _____ (OWNER OR AUTHORIZED AGENT SIGNATURE)

UTILITIES (INDICATE THAT LOCATION/UTILITY PRESENCE WAS CHECKED) *												
Borehole/ Excavation Location	Date (m/d/y)	Telephone	Water	Storm Sewer	Sanitary Sewer	Process Sewer	Gas	Electrical	Cable	Overhead Utilities	Other	Comments/Warnings

Additional Comments:

_____

_____

_____

White: Field Office
Yellow: Field File
Pink: Owner/Client/Agent

* Note as appropriate, Contractor, Client or Owner, or Agent to sign, indicating no utilities are at the selected borehole/excavation locations.

UTILITY CLEARANCE CHECKLIST (ACTIVE FACILITIES)

Prior to excavating or penetrating (i.e., soil boring, wells, Geoprobe, etc.) soil on OXY property, it is necessary to check for underground utilities that may be routed through the area in question. The following process is recommended:

1. Process is intended only for locating utilities on OXY property.
2. Utilities of concern include, but are not limited to, the following:
    - storm and sanitary sewers, drain tiles, gas, electric, telephone, city water, steam, condensate, process waste, and other process piping.
3. Underground structures such as foundations, tunnels, etc. also require location and identification.
4. Review all site utility, mechanical, electrical, and foundation drawings for the area in question.
5. Interview site personnel familiar with the area.
6. Visually inspect area in question.
7. Look for disruptions in pavement or flooring indicating a trench.
8. Look for manholes, pipe risers, catch basins, fire hydrants, post indicator valves, etc. (PIVs and hydrants are located above a pipe, but don't assume it is the main line. Most often they are above a lateral line.)
9. Check adjacent buildings or structures for locations of utilities entering the ground. In buildings look for services adjacent to machines.
10. Compare the drawings with observations in the field.
11. A pipe and cable locator device should be used to help verify physical location of utilities shown on drawings.
12. Call the following if uncertain of the location of a public utility. They will have maps of storm, sanitary, water, etc. They will also mark the utility's location:
    - Local electric, gas, or telephone companies, contract service, municipal or city water departments and/or engineering departments.
13. Evaluate safety needs and develop a safe operating procedure (SOP).
14. Dig a test pit when utility location cannot be absolutely determined.
15. Identify the utility(s) and mark location.
16. Update record drawings when discrepancy identified in the field.

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DATA RECORDING – FIELD BOOKS/DIGITAL RECORDING

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 1.4
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 0	REVISION DATE:

DATA RECORDING – FIELD BOOKS/DIGITAL RECORDING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure describes protocol for documenting standard investigation activities in the field. Field data serves as the cornerstone for an environmental project, not only for site characterization but for additional phases of investigation or remedial design. Inaccurate or incomplete field data may create significant problems and additional project costs. In addition, recorded field data becomes a legal record of project work, and should be approached with that in mind. Producing legally defensible data includes proper and appropriate recording of field data as it is obtained in a manner that will preserve it for future use.

This procedure provides guidelines for accurate, thorough collection and preservation of written and electronic field data.

PROCEDURES REFERENCED

- FMG 8.0 - Field Instruments – Use/Calibration

PROCEDURAL GUIDELINES

Typical field data to be recorded generally includes, but is not limited to, the following:

- General field observations.
- Numeric field measurements and instrument readings.
- Quantity estimates.
- Sample locations and corresponding sample numbers.
- Relevant comments and details pertaining to the samples collected.

- Documentation of activities, procedures, and progress achieved.
- Contractor pay item quantities.
- Weather conditions.
- A listing of personnel involved in site-related activities.
- A log of conversations, site meetings, and other communications.
- Field decisions made and pertinent information associated with the decisions.

Written Field Data

Written field data is generally recorded on one of two media: a standardized, pre-printed field log form, or a bound field log book. In general, use of a field log form is preferable as it prompts field personnel to make appropriate observations and record data in a standardized format. This promotes completeness and consistency from one person to the next. Most of the OXY FMGs include standardized field log forms.

In the absence of an appropriate pre-printed form, the data should be recorded in an organized and structured manner in a dedicated project field log book. Log books must be hard-cover, bound so that pages cannot be added or removed, and should be made from high-grade 50 percent rag paper with a water-resistant surface.

The following are guidelines for use of field log forms and log books:

1. Information must be factual and complete. Do not abbreviate.
2. All entries will be made in black indelible ink with a ballpoint pen and will be written legibly. Do not use "rollerball" or felt tip-style pens, since the water-soluble ink can run or smear in the presence of moisture.
3. All pages in a log book must be consecutively numbered. Field log forms should also be consecutively numbered.
4. Each day's work must start a new log book page.
5. At the end of each day, the current log book page must be signed and dated by the field personnel making the entries.
6. When using field log forms, they must also be signed and dated.
7. Make data entries immediately upon obtaining the data. Do not make temporary notes in other locations for later transfer to log forms or log books; this only increases the potential for error or loss of data.
8. Entry errors are to be crossed out with a single line, dated, and initialed by the person making the correction.
9. Do not leave blanks on log forms, if no entry is applicable for a given data field, indicate so with "NA" or a dash ("--").

10. At the earliest practical time, photocopies of log forms and log book pages should be made and placed in the project file as a backup in the event the book or forms are lost or damaged.
11. Log books should be dedicated to one project only, i.e., do not record data from multiple projects in one log book.

Electronic Data

Electronic data recording is widely used in environmental investigation and remediation projects. In general, it involves electronic measurement of field information through the use of monitoring instruments, sensors, gauges, and equipment controls. The following is a list of guidelines for proper recording and management of electronic field data:

1. Field data management should follow requirements of a project-specific data management plan (DMP), if one exists.
2. Use only instruments that have been calibrated in accordance with manufacturer's recommendations.
3. Usage of instruments, controls, and computers for the purpose of obtaining field data should only be performed by personnel properly trained and experienced in the use of the equipment and software.
4. Use only fully licensed software on PCs and laptops. Software piracy, even if unintentional, is a felony and exposes OXY, its contractors, and their employees to severe criminal and financial penalties.
5. Loss of electronic files may mean loss of irreplaceable data. Every effort should be made to back up electronic files obtained in the field as soon as practical. A backup file placed on a disk and kept in a separate location from the original will minimize the potential for loss.
6. Electronic files, once transferred from field instruments or laptops to office computers, should be protected if possible to prevent unwanted or inadvertent manipulation or modification of data. Several levels of protection are usually available for spreadsheets, including making a file "read-only" or assigning a password to access the file.
7. Protect floppy disks from exposure to moisture, excessive heat or cold, magnetic fields, or other potentially damaging conditions.
8. Remote monitoring is often used to obtain stored electronic data from site environmental systems. A thorough discussion of this type of electronic field data recording is beyond the scope of this FMG. Such on-site systems are generally capable of storing a limited amount of data as a comma-delimited or spreadsheet file. Users must remotely access the monitoring equipment files via modem or other access, and download the data. In order to minimize the potential for loss of data, access and downloading of data should be performed frequently enough to insure the data storage capacity of the remote equipment is not exceeded.

EQUIPMENT/MATERIALS

- Five by seven-inch National 407 Field Book, with high-grade 50 percent rag paper with water-resistant surface, hard-cover, or equivalent.
- Appropriate field log forms.
- Indelible ball point pen (do not use "rollerball" or felt-tip style pens).
- Straight edge.
- Pocket calculator.
- Laptop computer (if required).

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DRILLING TECHNIQUES

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 2.2
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

DRILLING TECHNIQUES

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This section will provide a brief description of common methods for conducting subsurface investigations. It should be noted that every drilling technology has its advantages and limitations.

PROCEDURES REFERENCED

- FMG 2.3 - Soil Borings
- FMG 2.4 - Bedrock Coring
- FMG 2.6 - Soil Classification
- FMG 2.7 - Rock Classification
- FMG 3.2 - Overburden and Top of Rock Wells
- FMG 3.3 - Deep Bedrock Wells
- FMG 3.5 - Piezometers
- FMG 3.6 – Well Development

PROCEDURAL GUIDELINES

It is important that the drilling method or methods used minimize disturbance of subsurface materials and not contaminate the subsurface and groundwater. The actual drilling method would be dependent upon site-specific geologic conditions and project requirements. It is important to note that the drilling equipment selected be decontaminated before and between borehole locations to prevent cross contamination (see FMG 9.0 - Equipment Decontamination).

Where possible drilling methods that minimize waste generation (soil cuttings), and wastewater generation (decontamination water), should be selected for OXY Remediation Team investigation/remedial tasks.

In other settings it may be desirable to dictate drilling procedures that minimize turbidity/maximize the ability to achieve sediment-free groundwater. Generally, rotosonic techniques or rotary spun casing techniques achieve these objectives, or oversizing the borehole/sand pack may be considered, as well.

A brief description of each drilling method, listed in the order most commonly used at OXY sites, is presented below.

Rotosonic Drilling

This method consists of a combination of rotation with high frequency vibration to advance a core barrel and outer casing to a desired depth. Typically, the core barrel is advanced in 10-foot intervals and then the outer casing is advanced to the core barrel depth and usually requires the injection of small quantities of water. 5-foot and 20-foot intervals are also used, depending on project requirements. Once the vibration is stopped, the core barrel is retrieved, and the sample is vibrated or hydraulically extracted into plastic sleeves or sample trays. The soil materials between the inner and outer casings are displaced into the sidewall of the geologic unit. Usually little to no soil or water is returned to the surface during drilling. The rotosonic method can usually drill easily through formations such as gravel, sand, clay, or glacial till. However, rotosonic drilling is slow in hard overburden formations (e.g., dense glacial till where displacement of soils into the sidewall are difficult), and can be very difficult in bedrock formations.

Monitoring wells shall be installed through the outer casing with minimal formation disturbance and mixing of formation materials. Rotosonic drilling generally requires less time than more traditional methods and minimizes soil mixing and soil disturbance (preferred for well locations where low turbidity is an important objective). Continuous, relatively undisturbed samples can be obtained through virtually any formation. Conventional sampling tools can be employed as attachments (i.e., hydropunch, split spoon, Shelby tube, etc.). No mud, air, water, or other circulating medium is required, although water is injected during advancement of the outer casing. The rotosonic method can drill easily through formations such as gravel, sand, clay, or glacial till. The main limitation of this method is the availability of equipment, the large area required (i.e., drill units are quite large), and costs. In addition, in some soils (e.g., silty sands, clayey sands) extra well development may be required due to displacement and compaction of soil cuttings into the borehole wall.

Hollow-Stem Auger

The hollow-stem continuous-flight auger is among the most frequently used in the drilling of monitoring wells (overburden wells) or for placement of overburden casings for bedrock wells.

The primary advantages of hollow-stem augering are that:

- Generally, no additional drilling fluids are introduced into the formation.
- It is a common drilling method and easy to find drilling companies with that capability.
- Representative geologic soil samples can be easily obtained using split-spoon samplers in conjunction with the hollow-stem augers.
- Monitoring wells can be installed through the augers eliminating the need for temporary borehole casings.

Disadvantages of hollow-stem augering are:

- Creates problems for select parameters.
- May not be possible in environments with strong upward gradients in granular environments.
- Large volumes of cuttings are typically generated.
- Decontamination is fairly time consuming/labor intensive.
- Relatively slow when compared to direct-push methods (soil sampling tasks).

Installing monitoring wells through hollow-stem augers is a relatively simple process although precautions need to be taken to ensure that the well is properly backfilled. This can be particularly problematic in cases where flowing/heaving sand is present.

Hollow-stem augers are available with inside diameters of 2.5, 3.25, 4.0, 4.25, 6.25, 8.25, 10.25, and 12.25 inches. The most commonly used are 4.25 inches for 2-inch (5 cm) monitoring wells and 6.25 inches for 4-inch (10 cm) monitoring wells. Boreholes can usually be drilled with hollow-stem augers to depths up to 100 feet (30 m) in unconsolidated clays, silts, and sands. Removing augers in flowing sand conditions while installing monitoring wells may be difficult since the augers have to be removed without being rotated. A bottom plug or pilot bit assembly should be utilized to keep out soils and/or water that have a tendency to plug the bottom of the augers during drilling. If flowing sands are encountered, potable water (analyzed once for contaminants of concern) may be poured into the augers to equalize the pressure to keep the formation materials and water from coming up into the auger once the bottom plug is removed.

Direct-Push (Geoprobe™)

Direct-push refers to the sampler being "pushed" into the soil material without the use of drilling to remove the soil. This method relies on the amount of the drill weight combined with rapid percussion for advancement of the tool string. Discrete soil samples are continuously obtained. Groundwater and vapor samples can also be collected utilizing this method. Subsurface investigations typically probe to depths of 30 feet or more, depths will vary based on site-specific

geology. The direct-push equipment typically advances either 4-feet long or 5-feet long samplers and drill rods.

Direct-push method is widely used for underground storage tank (UST) investigations and property investigations. This method is used extensively for initial site screening activities to delineate vertical and horizontal plume presence and can significantly reduce investigative costs.

This method is more popular due to the limited cuttings that are produced during the sampling process and the rapid sampling process speed. However, due to compaction of soils into the narrow diameter soil sampler, it is common that full recovery of the sampled interval is not obtained. The soil sampler tip displaces some soil into the borehole sidewall.

Depending on the diameter of the soil sampler tubing used, pre-pack well screens and riser pipe can be installed directly through the drill rods. Alternatively, most direct-push drill rigs can advance hollow-stem augers to limited depths, with some machines able to advance large diameter augers in certain conditions.

Rotary Method

This method consists of a drill rod attached to a drill bit (soils: tricone, drag; rock: button studded, diamond studded) that rotates and cuts through the soils and rock. The cuttings produced are forced to the surface between the borehole wall and the drill rod by drilling fluids which generally consist of water, drilling mud (mixed with water), or air. The drilling fluids not only force the cuttings to the surface but also keep the drilling bit cool. Using rotary methods for well installations can be difficult as it usually requires several steps to complete the installation. First, the borehole is drilled; then temporarily cased; then the well is installed; and then the temporary casing is removed. In some cases, the borehole may remain open without installing a casing, but this will only occur in limited instances (i.e., cohesive soils).

i) Water Rotary

When using water rotary, the potable water supply should be analyzed for contaminants of concern. Water rotary is the preferred rotary method since the potable water is the only fluid introduced into the borehole during drilling. However, the use of water as a fluid is generally only successful when drilling in cohesive soils. The use of potable water (only) also reduces well development time, when compared to mud rotary.

ii) Air Rotary (typically used in rock)

When using air rotary, the air compressor must have an in-line oil filter system assembly to filter the oil mixed with the air coming from the compressor. This will help eliminate contaminant introduction into the formation. The oil filter system should be regularly inspected. Air compressors not having an in-line oil filter system are not acceptable for air rotary drilling. A cyclone velocity dissipater or similar air containment system should also be used to funnel the cuttings and produced water to one location rather than letting the cuttings blow uncontrolled out of the borehole. Air rotary may not be an acceptable

method for well installation where certain contaminants are present in the formation. Alternatively, it may be necessary to provide treatment for the air being exhausted from the borehole during the installation process.

iii) Mud Rotary

Mud rotary is the least preferred rotary method because contamination can be introduced into the borehole from the constituents in the drilling mud (i.e., Ohio, Michigan). The drilling muds are generally non-toxic and do not introduce contaminants into the borehole, however, it is possible for mud to infiltrate permeable zones and can affect water quality by sorbing metals and polar organic compounds (Aller et al., 1991). Chemical composition and priority pollutants analysis may be obtained from the manufacturer. Mud rotary shall utilize only potable water and pure (no additives) bentonite drilling muds. The viscosity of the drilling mud shall be kept as low as possible in order to expedite well development. Proper well development is essential to ensure the removal of all the drilling mud and to return the formation to its previously undisturbed state. This usually requires significant surging and purging, jetting, airlifting, or a combination of these well development methods. Simply pumping is not sufficient.

Dual-Wall Reverse Circulation Air Method of Drilling

This method consists of two concentric strings of drill pipe (an outer casing and a slightly smaller inner casing). The outer drill pipe is advanced using rotary drilling with a donut-shaped bit attached to the dual casing string. The drill bit cuts an area only the width of the two casings and annulus between. Compressed air is continually forced down the annulus between the inner casing and outer casing carrying the drill cuttings and groundwater to surface. At the surface, the inner casing is connected to a cyclone hopper where the drill cuttings and groundwater fall out the bottom of the hopper, and air is dispersed out the top. The dual wall provides a fully cased borehole in which to install a monitoring well. The only soil or groundwater materials exposed at any time are those at the drill bit, providing depth-discrete soil sample cuttings in the drill returns. The potential for carrying contamination from one stratum to another is minimal. Depth-specific groundwater samples can be collected during drilling; however, since the groundwater is aerated, analysis for volatile compounds may not be valid, or additional purging with a pump may be required.

Well Points

In some limited cases, well points (sand points) are driven into place without the use of augers. This method provides no information on the geologic condition (other than the difficulty of driving which may be related to formation density). Well points are most often used simply to provide dewatering of a geologic unit prior to excavation in the area. Well points are also used in monitoring shallow hydrogeologic conditions such as in stream beds or adjacent to streams/ponds for monitoring hydraulic head and geochemical conditions. Well points are typically less than 1.25-inch diameter, which may restrict available well development or sampling methods.

REFERENCES

Numerous publications are available describing current monitoring well design and construction procedures.

Driscoll, F.G., 1986. Groundwater and Wells, 2nd Edition. Johnson Division.

EPA/625/6-90/0166 (July 1991), Handbook Ground Water Volume II: Methodology.

Freeze, R.A. and Cherry, J.A., 1979. Groundwater. Prentice Hall, Inc.

National Water Well Association, 1989. Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells.

Environmental Protection Agency (1986), RCRA Groundwater Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

In addition, the following ASTM publications apply:

ASTM D5474	Guide for Selection of Data Elements for Ground-Water Investigations
ASTM D5787	Practice for Monitoring Well Protection
ASTM D5521	Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers
ASTM D5978	Guide for Maintenance and Rehabilitation of Ground-Water Monitoring Wells
ASTM D5299	Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and Other Devices for Environmental Activities
ASTM D5092	Standard Practice for Design and Installation of Ground Water Monitoring Wells in an Aquifer.

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FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 2.3
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

SOIL BORINGS

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

The following presents a description of the methods generally employed for the advancement of boreholes and the collection of subsurface soil samples. Boreholes are typically installed to define geologic conditions for hydrogeologic and geotechnical evaluation; to allow the installation of monitoring wells and piezometers; and to allow the collection of subsurface soil samples (generally above the water table) for chemical analysis.

Several manual methods are available for the collection of shallow subsurface soil samples (e.g., hand augers, post-hole augers, vibratory hammers). However, the most common methods used by OXY to advance boreholes are rotosonic drilling techniques, hollow-stem augers (HSA), or the use of a direct-push equipment. Rotosonic drilling and direct-push techniques are preferred boring approaches at OXY Facilities. FMG 2.2 - Drilling Techniques, provides insight into the advantages/disadvantages of these drilling methods.

PROCEDURES REFERENCED

- FMG 1.3 - Utility Clearance
- FMG 2.2 - Drilling Techniques
- FMG 2.6 - Soil Classification
- FMG 2.7 - Rock Classification
- FMG 6.1 - Surficial Soil Sampling
- FMG 6.15 - PFAS/POFA Sampling

PROCEDURAL GUIDELINES

The following activities must be undertaken prior to undertaking a borehole installation and subsurface soil sampling program.

i) Assemble all equipment and supplies required per the Work Plan.

ii) Obtain a site plan and any previous stratigraphic logs. Determine the appropriate number and location of boreholes to be installed and the depths of samples for chemical analysis.

iii) Contact the analytical group to arrange/determine:

- Laboratory;
- Glassware/sample jars;
- Cooler;
- Shipping details;
- Start date;
- Expected duration; and
- Arrange bids if appropriate between the OXY Lab program accepted labs for best cost.

iv) Establish borehole locations in field using available permanent landmarks and conducting swing ties or by surveying methods if necessary.

v) Arrange for utility clearance of franchised utilities and site utilities.

vi) Arrange for hydrovac/air knifing services to daylight utilities/clear locations, if required by the project and clear locations with onsite personnel.

vii) Prepare FMEAs to be reviewed prior to drilling.

viii) Determine notification needs with the Project Manager. Confirm all appropriate groups have been notified of the planned sampling event like the regulatory groups, landowner, OXY facility personnel, and laboratory.

ix) Determine the methods for handling and disposal of drill cuttings, wash waters, and spent decontamination fluids.

Once the prior planning and preparation activities are completed, the borehole installation and subsurface soil sampling program can proceed. The typical series of events which takes place is:

- Locating and marking of borehole locations (if not already completed).
- Equipment decontamination.
- Final visual examination of proposed drilling area for utility conflicts/final hand auger or post-hole check to verify utility absence.
- Daylighting of utilities, if required
- Advancement of borehole and collection of the soil sample.

- Field screening of soil sample.
- Description of soil sample. [Form FMG 2.6-01 - Stratigraphy Log - Overburden (Page 1/Page 2) will be used to record data.]
- Sample preparation and packaging.
- Abandonment of boreholes or installation of monitoring well.
- Surveying of borehole locations and elevations.
- Field note completion and review.
- Double check all equipment, personal protective equipment (PPE), field notebooks for possible contaminants especially if sampling for per- and polyfluoroalkyl substances (PFAS)/ perfluorooctanoic acid (PFOA) (see FMG 6.15-PFAS/POFA Sampling for special considerations if sampling for PFAS/PFOA).

i) Locating and Marking of Boreholes/Final Visual Check

The proposed borehole locations marked on the site plan are located in the field and staked. On most sites, this will likely be done several days in advance of the drill rig arriving on site. Unless boreholes are to be installed on a fixed grid, the proposed locations are usually strategically placed to assess site conditions.

Once the final location for the proposed boring has been selected and utility clearances are complete (FMG 1.3 – Utility Clearance), one last visual check of the immediate area should be performed before drilling proceeds. This last visual check should confirm the locations of any adjacent utilities (subsurface or overhead) and verification of adequate clearance. If gravity sewers or conduits exist in the area, any access manholes or chambers should be opened and the conduit/sewer alignments confirmed. Do not enter manholes unless confined space procedures are followed.

ii) Borehole Advancement

If possible, it is prudent to use a hand auger or post-hole digging equipment to a sufficient depth to verify the absence of buried utilities and pipelines. Alternatively, hydrovac/air knifing can be used to daylight the hole prior to drilling. This procedure should clear the area to the full diameter of the drilling equipment which will follow.

If it is necessary to relocate any proposed borehole due to terrain, utilities, access, refusal, etc., the Project Manager must be notified and an alternate location will be selected using previous methods.

Prior to use and between each borehole location at an environmental site, the drilling and sampling equipment must be decontaminated. All decontamination must be conducted in accordance with the project-specific plans or the methods presented in FMG 9.0 - Equipment Decontamination.

The clean augers/tooling are covered with clean plastic sheeting (check for PFAS/PFOA in sheeting materials if sampling for PFAS/PFOA, see FMG 6.15-PFAS/POFA Sampling for special considerations) to prevent contact with foreign materials. For geotechnical, geologic, or hydrogeologic studies where contaminants will not be tested, it is sufficient to clean the drilling equipment simply by removing the excess soils.

Collection of soil samples is one of the most important considerations in selecting drilling methods. Therefore, the need for reviewing drilling techniques (FMG 2.2 - Drilling Techniques) and the site objectives must first be considered. Soil Classification will be completed in accordance with FMG 2.6 - Soil Classification. Sections iii) and iv) describe borehole soil sampling procedures using direct-push tooling and HSA/split-spoon sampling (Standard Penetration Testing - SPT), respectively.

iii) Subsurface Sample Collection Methods

Any drilling procedure that provides a suitably clean and stable hole before insertion of the sampler and assures that the penetration test or other sampling technique is performed on essentially undisturbed soil is acceptable. The drilling method is to be selected based on the subsurface conditions. Each of the following procedures have proven to be acceptable for specific subsurface conditions:

- Conventional drilling with continuous flight hollow-stem auger (HSA) method (with inside diameter between 2.2 and 6.5 inches) using split-spoon samplers (Standard Penetration Test – STP) or Shelby tube samplers; Direct-push samplers, advanced using a percussion/vibratory hammer (Geoprobe™ or equivalent);
- Rotosonic (sonic) drilling, advanced using a 5-6" diameter, 5-10 foot long steel core barrel by an oscillator within the drill head that generates a high-frequency, resonant energy and is combined with rotational movement;
- Hand–held/driven split-spoon sampling equipment, portable hammer and split-spoon sampling equipment (final depth will be limited).

Several drilling methods are not acceptable. These include: jetting through an open tube sampler and then sampling when the desired depth is reached; use of continuous flight solid auger equipment below the groundwater table in non-cohesive soils; casing driven below the sampling depth prior to sampling; and advancing a borehole with bottom discharge bits.

The following subsections describe the specific methods for completing direct-push sampling, core barrel sampling, split-spoon sampling and Standard Penetration Testing (SPT), and Shelby tube sampling. The following section, Soil Core Chemical Sample Collection Procedure describes the soil sampling procedure for chemical analysis, once a soil core is recovered from any of the above sample collection devices.

Direct-Push/Macro-Core™ Soil Sample Method

The operation of the direct-push soil sampler (e.g., Macro-core™, Dual Tube™, or equivalent) consists of "pushing" the sampler into the subsurface and then retrieving it using a direct-push soil probing machine. The collected soil core is contained within an internal soil liner (acetate, polyethylene, or Teflon) (check soil liner material if sampling for PFAS/PFOA, see FMG 6.15-PFAS/POFA Sampling for special considerations) and removed from the sampler once returned to the ground surface. Sampler length is variable depending on equipment available (2 feet, 4 feet, 5 feet). Once the soil liner has been removed and the outer sampler decontaminated, a new liner is inserted and the sampler reassembled. The clean sampler is then driven back down the same hole to collect the next soil sample.

Once driven to the required depth, the sampler body/soil liner and soil core is removed from the borehole for inspection and sample collection. Once above grade the sampler is opened by the probe operator and the liner removed and cut open (opened with a dual blade cutting tool), to expose the soil for inspection and sampling.

The sampler body and ends are decontaminated, a new liner is inserted, and the sampler reassembled for collection of the next interval (ensure the liner is free of PFAS/PFOA if sampling for PFAS/PFOA, see FMG 6.15-PFAS/PFOA Sampling for special considerations). The clean sampler is then advanced back down the same hole to collect the next soil sample. The Macro-Core™ sampler can be used in either the open-tube or closed-point sampling mode. The open-tube is most commonly used method, typically employed in stable soil conditions when the borehole does not collapse. The closed-point system seals the cutting shoe opening until the sampler is at the next sample interval, this prevents collapsed soil from entering the sampler as it is advanced back down the hole. Once at the sample depth, the closed-point is unthreaded and released from the cutting shoe area, such that it rides on top of the soil core as it is being driven into the next interval.

Soil Core Chemical Sample Collection Procedure, presented below, describes the soil sampling procedure for chemical analysis, once a soil core is recovered from the direct-push sampler.

Sonic Core Barrel Sample Method

Once the core barrels are advanced to the required depth, the inner core barrel is pulled from the ground, and the soil sample is extruded using vibration from the drill head. Soil Core Chemical Sample Collection Procedure, presented below, describes the soil sampling procedure for chemical analysis, once a soil core is recovered from the sampler.

Split Spoon Sampling and Standard Penetration Testing (SPT) Sampling and Testing Procedure

This method is used to obtain representative samples of subsurface soil materials and to determine a measure of the in situ relative density of the subsurface soils. The test methods described below must be followed to obtain accurate SPT values.

SPT sampling is performed by using a split barrel sampler in accordance with ASTM D1586. The split barrel sampler, or split spoon, consists of an 18- or 24-inch long, 2-inch outside diameter tube, which comes apart length wise into two halves. The split spoon is typically driven in advance of an HSA string which allows collection of the disturbed but representative soil sample.

Once the borehole is advanced to the target depth and the borehole cleaned of cuttings, representative soil samples are collected in the following manner:

- The split-spoon sampler should be inspected to ensure it is properly cleaned and decontaminated (if sampling for PFAS/PFOA, ensure that the cleaner is free of PFAS/PFOA, see FMG 6.15-PFAS/PFOA Sampling for special considerations). The driving shoe (tip) should be relatively sharp and free of severe dents and distortions.
- The cleaned split-spoon sampler is attached to the drill rods and lowered into the borehole. Do not allow the sampler to drop onto the soil.
- After the sampler has been lowered to the bottom of the hole, it is given a single blow to seat it and make sure that it is in undisturbed soil. If there still appear to be excessive cuttings in the bottom of the borehole, remove the sampler from the borehole and remove the cuttings.
- Mark the drill rods in three or four successive 6-inch (0.15 m) increments, depending on sampler length, so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-inch (0.15 m) increment.

The sampler is then driven continuously for either 18 or 24 inches (0.45 or 0.60 m) by use of a 140-pound (63.5 kg) hammer. The hammer may be lifted and dropped by either the cathead and rope method, or by using a trip, automatic, or semi-automatic drop system. The hammer should free-fall a distance of 30 inches (±1 inches) (760 mm, ±25 mm) per blow. Measure the drop at least daily to ensure that the drop is correct. To ensure a free-falling hammer, no more than 2 1/4 turns of the rope may be wound around the cathead (see ASTM D1586). The number of blows applied in each 6-inch (0.15 m) increment is counted until one of the following occurs:

- A total of 50 blows have been applied during any one of the 6-inch (0.15 m) increments described above;
- A total of 100 blows have been applied;
- There is no advancement of the sampler during the application of ten successive blows of the hammer (i.e., the spoon is "bouncing" on a stone or bedrock); or
- The sampler has advanced the complete 18 or 24 inches (0.45 or 0.60 m) without the limiting blow counts occurring as described above.

In some cases where the limiting number of blow counts has been exceeded, the field supervisor may direct the driller to attempt to drive the sampler more if collection of a greater sample length is essential.

On the field form, record the number of blows required to drive each 6-inch (0.15 m) increment of penetration. The first 6 inches is considered to be a seating drive. The sum of the number of blows required for the second and third 6 inches (0.15 m) of penetration is termed the "standard penetration resistance" or the "N-value".

Note: If the borehole has sloughed and there is caved material in the bottom, the split spoon may push through this under its own weight, but now the spoon is partially "pre-filled". When the spoon is driven the 18 or 24 inches representing its supposedly empty length, the spoon fills completely before the end of the drive interval. Two problems arise:

1. *The top part of the sample is not representative of the in-place soil at that depth; and*
2. *The SPT value will be artificially higher toward the bottom of the drive interval since the spoon was packed full. These conditions should be noted on the field log.*

The sampler is then removed from the borehole and unthreaded from the drill rods. The open shoe (cutting end) and head of the sampler are partially unthreaded by the drill crew and the sampler is transferred to the geologist/engineer work surface.

Note: A table made out of two sawhorses and a piece of plywood is appropriate, or a drum, both covered with plastic sheeting.

The open shoe and head are removed by hand, and the sampler is tapped so that the tube separates.

Note: Handle each split spoon with clean disposable gloves if environmental issues are being investigated.

Measure and record the length of sample recovered making sure to discount any sloughed material that is present on top of the sample core. Note that surficial or shallow soils may be lodged in the auger borehole, thus split spoon samples from depth may contain sloughed material including topsoil/grass or fill materials previously encountered.

Caution must be used when conducting SPT sampling below the groundwater table, particularly in sand or silt soils. These soils tend to heave or "blow back" up the borehole due to the difference in hydraulic pressures between the inside of the HSA and the undisturbed soil, and the syringe-like effect of pulling the center plug from the augers for sampling. To equalize the hydraulic pressure, the inside of the HSA must be filled with water (preferred) or drilling mud. The drilling fluid level within the boring or HSAs needs to be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling. Since heave or blow back is not always obvious to the driller, it is essential that the water level in the borehole always be maintained at or above the groundwater level.

Heaving conditions and the use of water or mud should be noted on the field logs.

Soil Core Chemical Sample Collection Procedure, presented below, describes the soil sampling procedure for chemical analysis, once a soil core is recovered from a split-spoon sampler.

SPT sampling below the water table in sands and silt occasionally results in low SPT values being obtained due to the heaving effect disturbing the soil especially if the water level in the hole has not been maintained at the in-situ water level. Suspect low N values should be noted on the field logs. If it is critical to have accurate N values below the water table, other methods can be employed, such as conducting a dynamic cone penetration test. This quick and easy test involves attaching a cone shaped tip to the end of the drill rods, and driving the tip into the ground similar to the SPT method, except that the borehole is not pre-augered. Cones may be driven 20 to 40 feet through a formation without augering. Blow counts are recorded for each foot (0.3 m) of advancement.

A variation of split barrel sampling involves the use of a longer barrel (continuous sampler) in conjunction with HSAs. The sampling barrel is installed inside the auger with a swivel attachment to limit rotation of the barrel. After completion of a 5-foot auger penetration, the auger is left in place and the continuous sampler barrel retrieved from the borehole. The sampler should be handled and the sample retrieved in the same way as described above for SPT sampling.

Thin-Walled Samplers (Shelby Tubes)

Thin-walled samplers are used to collect relatively undisturbed samples (as compared to split-spoon samples) of soft to stiff clayey soils. Shelby tubes are commonly used. The Shelby Tube has an outside diameter of 2 or 3 inches and is 3 feet long. These undisturbed samples are used for certain laboratory tests of structural properties (consolidation, hydraulic conductivity, shear strength) or other tests that might be influenced by sample disturbance. Procedures for conducting thin-walled tube sampling are provided in ASTM D1587, and are briefly described below.

- The soil deposit being sampled must be cohesive in nature, and relatively free of sand, gravel, and cobble materials, as contact with these materials will damage/collapse the sampler.
- Clean out the borehole to the sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above groundwater level during the sampling operation.
- Bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted. Remove loose material from the center of a casing or HSA as carefully as possible to avoid disturbance of the material to be sampled.
- Place the sample tube so that its bottom rests on the bottom of the hole. Advance the sampler into the formation without rotation by a continuous and relatively rapid motion; usually hydraulic pressure is applied to the top of the drill rods.

- Determine the length of advance by the resistance and condition of the formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays.
- In no case should the length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 inches for cuttings.
- The tube may be rotated to shear the bottom of the sample 2 to 3 minutes after pressing in, and prior to retrieval to ensure the sample does not slide out of the tube. Lift the weight of the rods off of the tube prior to rotating.
- Withdraw the sampler from the formation as carefully as possible in order to minimize disturbance of the sample.

On occasion it may be required that extraction of the sample from the tube be conducted in the field for chemical sample collection. The following procedure should be followed.

- A sample extruder, which consists of a clamp arrangement to hold the tube and a hydraulic ram to push the sample through the tube, is usually mounted on the side of the rig. To prevent cross-contamination, be certain that the extruder is field cleaned between each sample.
- The sample is then extruded into a carrying tray; these are often made from a piece of 4-inch or 6-inch diameter PVC pipe cut lengthwise. Be certain that the carrying tray is field cleaned between each sample. The sample is carried to the work station to describe the sample, trim the potentially cross-contaminated exterior, and select the area for sample collection (see Section 2.4 - Soil Core Chemical Sample Collection Procedure). Form FMG 2.3 -01 - Soil Sample Selection Details shows the method for obtaining a soil sample from a Shelby tube soil core.
- The Shelby tube may then be thoroughly field cleaned and decontaminated for reuse. Since they are thin-walled, the tubes are easily damaged, crimped, or otherwise distorted during handling or pushing. The Shelby tube should be inspected before use and any which are significantly damaged should be rejected.

Soil Core Chemical Sample Collection Procedure, presented below, describes the soil sampling procedure for chemical analysis, once a soil core is recovered from a Shelby tube sampler.

iv) Soil Core Chemical Sample Collection Procedure

The following describes the collection of soil samples for chemical analysis from a split-spoon soil core, Shelby tube soil core, direct-push sample core, or sonic core barrel. Form FMG 2.3-01 - Soil Sample Selection Details shows the soil sample selection details. Sample preparation and selection is as follows:

- Record soil core recovery and soil stratigraphy data.
- Discard upper and lower ends of sample core (± 3 inches).

- If clayey soils are present use a pre-cleaned stainless steel knife to cut the remaining core longitudinally, alternatively if sandy soils are present, use a clean stainless-steel spoon to scrape away the soil surface.
- Screen the exposed soil surface with a photoionization detector (PID) to monitor for the presence of volatile organics.
- With a sample knife or spoon, remove soil from the center portion of the core and place in the sample jar (when only one aliquot is required), or when more than one aliquot is required place soils in a pre-cleaned stainless steel bowl for homogenization.
- Do not sample large stones and natural vegetative debris.
- Homogenize the soil and place directly into the sample jars. Do not homogenize soil for VOC analyses.
- Place collected samples on ice or cooler packs in laboratory-supplied shipping coolers.
- Package and transport the sample in accordance with FMG 6.10 - Sample Handling and Shipping.

When only one sample container is required, the collected soil will be placed directly into the clean, pre-labeled sample jar. When more than one sample container requires filling or samples will be split for duplicate analyses; the soils will first be homogenized in a pre-cleaned stainless steel bowl; and then placed into the respective sample containers. It is important that soil samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. When round bowls are used for sample mixing, mixing is achieved by stirring the material in a circular motion and occasionally turning the material over. Soil samples collected for volatile organic compounds (VOCs) analyses shall not be mixed.

Exception is noted for the collection of VOCs which require special sample collection methods and is usually collected first to minimize VOC loss. VOCs are collected directly into a sample vial (triplicate volume typically required) without headspace, or collected in triplicate using an EnCore Sampler™, or equivalent sampler, (triplicate samples collected per manufacturer's instructions). Samples for VOCs are typically collected first, without homogenization or extra handling to limit the loss of volatile constituents.

The VOC sample collection methodology will be identified in the Work Plan, which will dictate the sample method. The methodology for VOC sampling varies from area to area, so careful review of this issue in advance of the field efforts is required.

If PFAS/PFOA is being analyzed, specialized collection procedures, containers and equipment should be utilized (see 6.15-PFAS/POFA Sampling).

v) Borehole Completion

At the completion of the soil boring, once the soil/groundwater samples have been collected, the borehole annulus is then abandoned. Borehole abandonment options are identified in FMG 2.5 -

Borehole Abandonment/Sealing. Each boring will be surveyed to establish vertical/horizontal information; field ties (i.e., swing ties) will also be collected to document the boring location. Once completed, a stratigraphic log will be prepared for reporting purposes.

EQUIPMENT/MATERIALS

- Drilling equipment.
- Form 2.6-01 - Stratigraphy Log - Overburden (Page 1/Page 2).
- Tape measure.
- Cutting Instrument.
- Plastic sheeting (free from PFAS/PFOA if sampling for PFAS/PFOA).

REFERENCES

ASTM D420-93 - Guide to Site Characterization for Engineering, Design, and Construction Purposes.

ASTM D1452-80 - Practice for Soil Investigation and Sampling by Auger Borings.

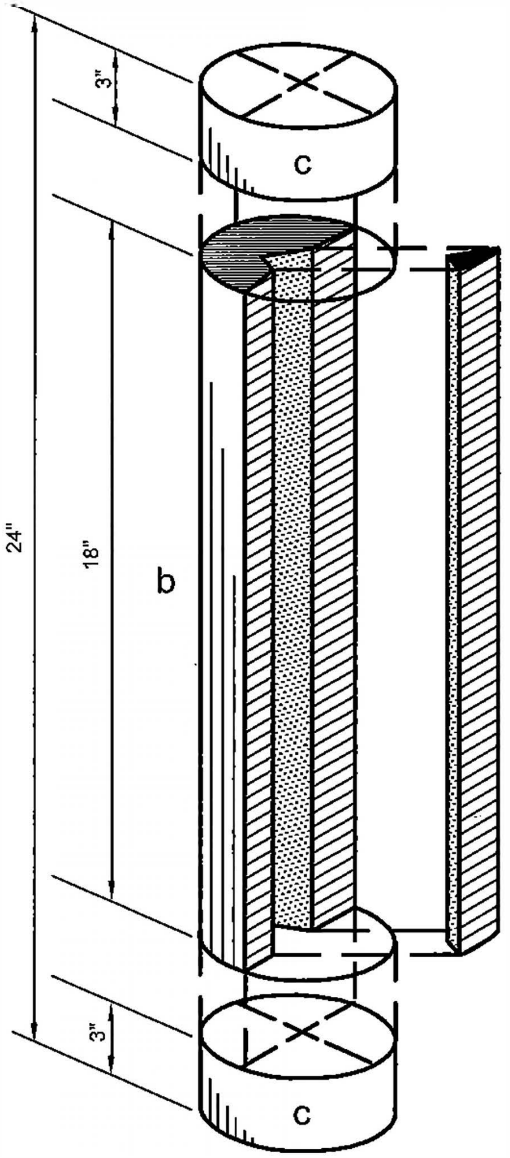
ASTM D1586-84 - Test Method for Penetration Test and Split-Barrel Sampling of Soils.

ASTM D1587-94 - Practice for Thin-Walled Tube Geotechnical Sampling of Soils.

ASTM D2488-93 - Practice for Description and Identification of Soils (Visual-Manual Procedure).

EPA OSWER-9950.1, 1986. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document.

National Water Well Association, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells. 1989.



TYPICAL SOIL CORE

a

PORTION OF SAMPLE FOR CHEMICAL ANALYSIS

-CONTACT WITH UNSTERILIZED MATERIALS IS NOT ACCEPTABLE

-STORAGE - REFRIGERATED (4°C)

-SHIPPING - ON ICE BY COURIER TO DESIGNATED LAB

b

PORTION OF SAMPLE TO BE RETAINED FOR GEOLOGIC RECORDS

-CONTACT WITH UNSTERILIZED MATERIALS IS NOT A PROBLEM

-CONTAINER: - CLEAN GLASS JAR
- CLEAR GLASS IS SUITABLE

-STORAGE - IN STANDARD SHIPPING CARTON
- NO REFRIGERATION REQUIRED

c

PORTION OF SAMPLE TO BE DISCARDED

-DISCARDED WITHIN 55 GALLON DRUM MAINTAINED ON-SITE

figure 2.3

SOIL SAMPLE SELECTION DETAILS

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BOREHOLE ABANDONMENT/SEALING

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 2.5
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

BOREHOLE ABANDONMENT/SEALING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

The following procedure describes common techniques for the abandonment/sealing of overburden boreholes. Borehole completion may have been performed by a rotosonic drilling technique, direct push sampling device, hollow-stem augering/split-spoon sampling, solid-stem augering, or other soil sample collection techniques. The method of borehole abandonment selected for a program will be dependent on a number of factors such as: depth to groundwater, presence of contamination [and degree of contamination i.e., light or dense non-aqueous phase liquids (NAPL)], confining layer presence and/or physical setting (i.e., open field/vacant land, vs. facility setting). The Work Plan guiding these activities (soil boring/boring closure) will dictate which method of borehole abandonment/sealing is required. The borehole abandonment/sealing techniques reviewed in the following consist of:

- Soil cutting backfill;
- Bentonite chip backfill;
- Cement/bentonite grout backfill using tremie techniques; or
- Bentonite slurry using tremie techniques.

Boreholes need to be abandoned and sealed properly to prevent surface water entry to the groundwater regime, to eliminate any physical hazard, and to prevent/protect groundwater movement from one aquifer to another.

PROCEDURES REFERENCED

- FMG 2.3 - Soil Borings
- FMG 3.1 – Well Construction Materials

PROCEDURAL GUIDELINES

Soil Cutting Backfill

Typically employed when working above groundwater table and at shallow depths (maximum depth 2 feet).

- The final depth of borehole will be measured and recorded.
- Cuttings are dropped into borehole after sample equipment is removed.
- Drill rod and/or probe rodding is used to compact/compress cuttings to allow return of all cuttings back into borehole.
- Mound final surface of cuttings above ground surface to allow settlement and promote surface water runoff away from boring. Final restoration will be completed in accordance with needs of the OXY Facilities representative and/or the OXY Project Manager.
- Borehole abandonment will be documented in field records/notes.

Bentonite Chip Backfill

Typically employed when working above or just into the groundwater table.

- Excess cuttings have been drummed for disposal or excess cuttings have been spread at ground surface.
- The depth of the borehole will be measured and recorded.
- Bentonite chips (bentonite gravel) will be dropped into borehole as hollow-stem augers are removed, or after the boring equipment has been removed from the borehole (solid-stem auger, probing tools, split-spoon samplers).
- Sufficient water will be needed to hydrate bentonite chips as they are placed.
- The bentonite chip backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow settlement and promote surface water runoff away from the boring. Alternatively, the borehole cuttings may be mixed with bentonite to complete the abandonment/sealing task. Final restoration will be completed in accordance with needs of the OXY Facilities representative and/or the OXY Project Manager.
- Borehole abandonment will be documented in field records/notes.

Cement/Bentonite Grout Backfill

Typically employed when working below the groundwater table, or in an area where a confining layer exists and the potential for groundwater/NAPL movement along a preferential pathway (i.e., former borehole) must be eliminated. Cement/bentonite grout sets up hard, like a soft

concrete. If future site development is planned or excessive surface water may be present, neat bentonite grout may be preferred.

- The final depth of borehole will be measured and recorded.
- The volume of grout required will be calculated from the above measurements.
- A grout mix of one bag (94 pounds) of Portland cement and 3 pounds of bentonite with approximately 7.5 gallons of clean water will be prepared.
- Using a tremie tube placed at the base of the borehole the grout will be pumped until observed at the required elevation. The tremie tube will be raised as the grout level rises (positive displacement technique).
- The bentonite/grout backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow settlement and promote surface water runoff away from boring. Final restoration will be completed in accordance with the OXY Facility representative and/or the OXY Project Manager.
- Borehole abandonment will be documented, noting depth of borehole, volume of grout used and mix ratio.
- Groundwater displaced from the borehole may or may not require containment depending on borehole setting and/or water quality.

Note: At the completion of borehole abandonment/sealing activities (regardless of methodology employed) it is necessary to check for surface settlement a few days after work completion to determine if the borehole area requires "topping off".

Final Restoration

The area around the borehole and the borehole surface shall be restored as directed by the OXY Facility representative (e.g., asphalt, concrete, vegetation). Time for borehole settlement may be permitted, then final restoration performed; or alternatively final restoration may be required immediately in active interior work areas.

Cleanup

The area around the borehole shall be completely cleaned up of any investigation related materials (litter, etc.).

EQUIPMENT/MATERIALS

- Grout pump/mixing equipment.
- Form FMG 2.6-01 - Stratigraphic Log (Overburden) (Page 1/Page 2).

REFERENCES

ASTM D5299 "Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and Other Devices for Environmental Activities.

United States Environmental Protection Agency (1992) "Guide to Management of Investigation-Derived Wastes", Quick Reference Fact Sheet.

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SOIL CLASSIFICATION

LIST OF FORMS
(Following Text)

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SOIL CLASSIFICATION

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

The stratigraphic log is a factual description of the soil at the borehole location and is relied upon to interpret the soil characteristics, and their influence and significance in the subsurface environment. The accuracy of the stratigraphic log is to be verified by the person responsible for interpreting subsurface conditions. An accurate description of the soil stratigraphy is essential for a reasonable understanding of the subsurface conditions. Confirmation of the field description by examination of representative soil samples by the project geologist, hydrogeologist, or geotechnical engineer (whenever practicable) is recommended.

The ability to describe and classify soil correctly is a skill that is learned from a person with experience and by systematic training and comparison of laboratory results to field descriptions.

It is OXY Remediation Team's Policy to log soils according to the Unified Soil Classification System (USCS) described in the following.

PROCEDURES REFERENCED

- FMG 2.1 - Test Pits
- FMG 2.3 - Soil Borings

PROCEDURAL GUIDELINES

Several methods for classifying and describing soils or unconsolidated sediments are in relatively widespread use. The Unified Soil Classification System (USCS) is the most common. With the USCS, a soil is first classified according to whether it is predominantly coarse-grained or fine-grained.

The description of fill soil is similar to that of natural undisturbed soil except that it is identified as fill and not classified by USCS group, relative density, or consistency. Those logging soils must attempt to distinguish between soils that have been placed (i.e., fill) and not naturally present; or soils that have been naturally present but disturbed (i.e., disturbed native).

It is necessary to identify and group soil samples consistently to determine the subsurface pattern or changes and non-conformities in soil stratigraphy in the field at the time of drilling. The stratigraphy in each borehole during drilling is to be compared to the stratigraphy found at the previously completed boreholes to ensure that pattern or changes in soil stratigraphy are noted and that consistent terminology is used.

Visual examination, physical observations and manual tests (adapted from ASTM D2488, visual-manual procedures) are used to classify and group soil samples in the field and are summarized in this subsection. ASTM D2488 should be reviewed for detailed explanations of the procedures. Visual-manual procedures used for soil identification and classification include:

- Visual determination of grain size, soil gradation, and percentage fines.
- Dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) tests for identification of inorganic fine-grained soil (e.g., CL, CH, ML, or MH).
- Soil compressive strength and consistency estimates based on thumb indent and pocket penetrometer (preferred) methods.

The three main soil divisions are: coarse-grained soil (e.g., sand and gravel), fine-grained soil (e.g., silt and clay), and soil with high natural organic matter content (e.g., peat and marl).

Coarse-grained Soil

The USCS group symbols for coarse-grained soil are primarily based on grain or particle size, grain size distribution (gradation), and percent fines (silt and clay content).

Coarse-grained soils are then further subdivided according to the predominance of sand and gravel. Coarse-grained soil is made up of more than 50 percent, by weight, sand size, or larger (75 μm diameter, No. 200 sieve size or larger). It is noted that there are other definitions for coarse-grained or coarse textured soil and for sand size in other soil classification systems, such as soil having greater than 70 percent particles equal to or greater than 50 μm diameter.

Descriptions for grain size distribution of soil include; poorly graded (i.e., soil having a uniform grain size or missing grain size fractions (gap graded), SP and GP) and well graded (i.e., poorly sorted; having wide range of particle sizes with substantial intermediate sizes, SW and GW).

Coarse-grained soils are further classified based on the percentage of silt and clay it contains (fines content). Coarse-grained soils containing greater than 12 percent fines is commonly described as dirty. This description arises from the soil particles that adhere when the soil is rubbed between the hands or adhere to the sides of the jar after shaking or rolling the soil in the jar. The jar shake

test which results in segregation of the sand and gravel particles is also used as a visual aid in determining gravel and sand percentages.

Examples of the group symbol, name, and adjectives used to describe the primary, secondary, and minor components of soil are; GW - Sandy Gravel (e.g., 70 percent gravel and 30 percent sand) or Sandy Gravel trace silt (less than 10 percent silt), and SP - Sand, uniform.

Relative density is an important parameter in establishing the engineering properties and behavior of coarse-grained soil. Relative density of non-cohesive (granular) soil is determined from standard penetration test (SPT) blow counts (N values) (after ASTM Method D1586).

The SPT gives a reliable indication of relative density in sand and fine gravel. N values in coarse-grained soil are influenced by a number of factors that can result in overestimates of relative density (e.g., in coarse gravel and dilatent silty fine sand) and can be conservative and underestimate the relative density (e.g., sand below the groundwater table and uniform coarse sand). These effects will be assessed by the project geotechnical engineer, if required, and need not be taken into account by field personnel.

Other dynamic methods, such as modified SPT and cone penetration tests, are used on occasion to supplement or replace the SPT method for certain site-specific conditions. The details of all modifications to the SPT or substitute methods should be recorded as they are required to interpret test results and correlate to relative density.

Fine-grained Soil

A soil is fine-grained if it is made up of half or more of clay and silt [i.e., fines greater than 50 percent by weight passing the 75 μm (No. 200) sieve size]. A description of visual-manual field methods and criteria (after ASTM D2488) that are used to further characterize and group fine-grained soil (e.g., CL, CH, ML, or MH) including dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) follows. Fine-grained soils are subdivided on a basis of the liquid limit and the degree of plasticity.

The accurate identification of silts and clays can be aided by the use of some single field tests. Clay is sticky, will smear readily, and can be rolled into a thin thread even when the moisture content is low. When it is dry, clay forms hard lumps. Silt on the other hand, has a low dry strength, can be rolled into threads only at high moisture content, and a wet silt sample will puddle when it is tapped.

Criteria for Describing Dry Strength

Description	***Criteria***
None	The dry specimen crumbles into powder with mere pressure of handling.
Low	The dry specimen crumbles into powder with some finger pressure.

Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure.
High	The dry specimen crumbles into powder with finger pressure. Specimen will break into pieces between thumb and a hard surface.
Very High	The dry specimen cannot be broken between the thumb and a hard surface.

Criteria for Describing Dilatancy

Description	***Criteria***
None	No visible change in small wetted specimen when rapidly shaken in palm of hand.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing or stretching.

Criteria for Describing Toughness

Description	***Criteria***
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.

Criteria for Describing Plasticity

Description	***Criteria***
Nonplastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be re-rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Examples of group symbol identification based on visual-manual procedures and criteria for describing fine-grained soil are:

Group Symbol	*Dry Strength Plasticity*	*Dilatency*	*Toughness*
ML	None to low Slight	Slow to rapid	Low or thread cannot be formed
CL	Medium to high Low	None to slow	Medium
MH	Low to medium Low	None to slow	Low to medium
CH	High to very high High	None	High

A requirement for positive classification by USCS group symbols (as described in Test Method ASTM D2487) is laboratory determination of particle size characteristics, liquid limit and plasticity index. The need for this type of testing will be determined by the project geologist, hydrogeologist, or geotechnical engineer.

Examples of name terminology that accompanies the group symbols are ML - Sandy Silt (e.g., 30 percent sand) and CL - Lean Clay with sand (e.g., 15 to 29 percent sand).

The correlation between N value and consistency for clays is rather unreliable. It is preferable to determine consistency using more appropriate static test methods, particularly for very soft to stiff clay soil. N value estimates of consistency are more reasonable for hard clay.

Unconfined compressive strength (Su) may be estimated in the field from the pocket penetrometer test method. To obtain a pocket penetrometer estimate of consistency and compressive strength, the soil core is cut perpendicular to the core length, the length of core (minimum 4 inches) is held in the hand and a moderate confining pressure is applied to the core (not sufficient to deform the core); the penetrometer piston tip is slowly inserted into the perpendicular face of the core until the penetrometer indents into the soil core to the mark indicated on the tip of the penetrometer piston; the penetrometer estimate of soil compressive strength (Su) is the direct reading of the value mark on the graduated shaft (in tons per square foot or other unit of pressure as indicated) indicated by the shaft ring marker, or in some models, by the graduated piston reading at the shaft body. To obtain an average estimate, this procedure is completed several times on both ends and mid cross-section of the core. For Shelby tube (or thin wall sampler) samples the pocket penetrometer tip is applied to the exposed bottom of the sample at several locations.

Estimates of compressive strength for clay soil of very soft to stiff consistency are better established by in situ shear vane tests or other static test methods.

The description of consistency (or strength) is an important element in determining the engineering properties and strength characteristics of fine-grained cohesive soil. Consistency terms (e.g., soft,

hard) are based on the unconfined compressive strength (Su) and shear strength or cohesion (cu) of the soil.

The ease and pattern of soil vapor and groundwater movement in the subsurface is influenced by the natural structure of the soil. Soil structure, for the most part, depends on the depositional environment and, to a lesser extent, climate.

Visual Appearance/Other Features

Those logging soils should also note the presence, depth and components of fill soils (if evident) and note the distinction between disturbed native soils (i.e., excavation likely performed) vs. undisturbed native soils.

Other features such as color, root presence/structure, and soil fractures should also be recorded. Soil fractures should be described noting fracture orientation (i.e., horizontal/vertical), length/aperture and appearance of soil infilling, oxidation and/or weathering (if present).

Field Sample Screening

Upon the collection of soil samples, the soil is screened with a photoionization detector (PID) for the presence of organic vapor. This is accomplished by running the PID across the soil sample. Record the highest reading and sustained readings.

Note: The PID measurement must be done upwind of the excavating equipment or any running engines so that exhaust fumes will not affect the measurements.

Another method of field screening is head space measurements. This consists of placing a portion of the soil sample in a sealable glass jar, placing aluminum foil over the jar top, and tightening the lid. Alternatively, plastic sealable bags maybe utilized for field screen in lieu of glass containers. The jar should only be partially filled. Shake the jar and set aside for at least 30 minutes. After the sample has equilibrated, the lid of the jar can be opened; the foil is punctured with the PID probe and the air (headspace) above the soil sample is monitored. Record this headspace reading on the field form or in the field book. The selection of samples for chemical analysis may be specified in the Work Plan or be dependent in part on the PID responses.

Note: Perform all headspace readings in an area that is not subject to wind. Also, in the winter, it is necessary to allow the samples to equilibrate in a warm area (e.g., site trailer, van, etc.). This requirement is dictated by the Work Plan.

All head space measurements must be completed under similar conditions to allow comparability of results.

NAPL Detection

During soil examination and logging, the sampler shall carefully check for the presence of light or dense non-aqueous phase liquid (NAPL). NAPL may be present in gross amounts or present in small/minute quantities. The adjectives and corresponding quantities used when describing NAPL within a soil matrix are as follows:

Visual Description	***Fraction of Soil Pore Volume Containing NAPL***
Saturated	>0.5
Some	0.5 to 0.25
Trace	<0.25

A complete description of NAPL, must describe the following:

- Color.
- Quantity.
- Density (compared to water i.e., light/floats or heavy/sinks).
- Odor (if observed).
- Viscosity (i.e., mobile/flowable, non-mobile/highly viscous-tar like).

The presence of an "iridescent sheen" by itself does not constitute "NAPL presence", but may be an indicator that NAPL is close to the area.

NAPL presence within a soil matrix may be confirmed by placing a small soil sample within water, shaking, and observing for NAPL separation (i.e., light or dense), from the soil matrix.

Trace amounts of NAPL are identified/confirmed by a close visual examination of the soil matrix, [i.e., separate soil by hand (wearing disposable gloves)] and perform a careful inspection of the soil separation planes/soil grains for NAPL presence.

Often during the sample examination with a knife, an iridescent sheen will be noted on the soil surface (i.e., clay/silts) if the knife has passed through an area of NAPL.

There are several more sophisticated tests available to confirm/identify NAPL presence, these are:

- UV fluorescent analysis.
- Hydrophobic dyes.
- Centrifugation.
- Chemical analysis.

Typically consultants will utilize organic vapor detection results, visual examination, soil/water shake testing, and chemical analysis, to confirm NAPL presence. The more complex techniques

described may be incorporated on sites where clear colorless NAPL is present and its field identification is critical to the program.

Note: When describing the presence of vegetative matter in the soil sample, do not use the term "organic" as this often leads to confusion with regards to the presence of organic chemicals (i.e., NAPL).

EQUIPMENT/MATERIALS

- Pocket knife or small spatula.
- Small handheld lens.
- Form FMG 2.6-01 - Stratigraphic Log - Overburden (Page 1/Page 2).
- Tape measure.

REFERENCES

American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", "Annual Book of ASTM Standard", Section 4, Volume 04.08.

ASTM Standards on Environmental Sampling (1995), Standard D2488-93, "Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)"

ASTM Standards on Environmental Sampling (1995), Standard D4700-91, "Guide for Soil Sampling from the Vadose Zone".

ASTM Standards on Environmental Sampling (1995), Standard D1586-92, "Test Method for Penetration Test and Split-Barrel Sampling of Soils".

ASTM Standard D2487, "Classification of Soils for Engineering Purposes (Unified Soil Classification System)".

Geotechnical Gauge, Manufactured by W.F. McCollough, Beltsville, MD.

Sand Grading Chart, by Geological Specialty Company, Northport, Alabama.

Stratigraphy Log (Overburden)

Project name		**Drilling contractor**		**Hole designation**	
Project number		**Driller**		**Date/Time started**	
OXY Site		**Surface elevation**		**Date/Time completed**	
Location		**Weather (A.M.)**		**Drilling method**	
		(P.M.)		**Supervisor**	

Stratigraphic Intervals (Depths in ft/m BGS)			**Sample Description**	**Sample Details**											
			Order of descriptors: **Soil type symbol(s) - primary component(s), (nature of deposit), secondary components, relative density/consistency, grain size/plasticity, gradation/structure, colour, moisture content, supplementary descriptors.**			**Penetration Record Split Spoon Blows (Record N-Values & Recoveries)**									
From	**At**	**To**	**Note: Plasticity determination requires the addition of moisture if the sample is too dry to roll (indicate if moisture was added or not).**	**Sample Number**	**Sampling Method**	**6"**	**6"**	**6"**	**6"**	**N**	**R**	**Sample Interval**	**PID/FID (ppm)**	**Chemical Analysis**	**Grain Size/ Other Analysis**

Notes and Comments

Depth of borehole caving _____ Depth of first groundwater encounter _____ Topsoil thickness _____

Water level in open borehole on completion _____ After _____ Hours _____

Notes: _____

Soil Classification System (U.S.C.S.)
(ASTM D2488 Visual-Manual Procedure)

Major Divisions			Group Symbol	Typical Description
Highly Organic Soils (see note below)			PT	Peat and other highly organic soils
Coarse-Grained Soils (more than half by weight larger than no. 200 sieve size)	Gravels more than half of coarse fraction larger than no. 4 sieve size	"Clean" Gravels	GW	Well graded gravel, gravel-sand mixtures, ≤ 5% fines
			GP	Poorly graded gravels and gravel-sand mixtures, ≤ 5% fines
		"Dirty" Gravels	GM	Silty gravels, gravel-sand-silt mixtures, ≥ 15% fines
			GC	Clayey gravels, gravel-sand-clay mixtures, ≥ 15% fines
	Sands more than half of coarse fraction smaller than no. 4 sieve size	"Clean" Sands	SW	Well graded sands, gravelly sands, ≤ 5% fines
			SP	Poorly graded sands, or gravelly sands, ≤ 5% fines
		"Dirty" Sands	SM	Silty sands, sand-silt mixtures, ≥ 15% fines
			SC	Clayey sands, sand-clay mixtures, ≥ 15% fines
Fine-Grained Soils (more than half by weight passes no. 200 sieve size)	Silts below "A" line on plasticity chart; negligible organic content		ML	Inorganic silts and very fine sand, rock flour, silty sands of slight plasticity
			MH	Inorganic silts, micaceous or diatomaceous, fine sandy or silty soils
	Clays above "A" line on plasticity chart; negligible organic content		CL	Inorganic clays of low to medium plasticity, gravelly, sandy, or silty clays, lean clays
			CH	Inorganic clays of high plasticity, fat clays
	Organic silts & organic clays below "A" line on plasticity chart		OL	Organic silts and organic silty clays of low plasticity
			OH	Organic clays of high plasticity

Note:

Use dual symbols for coarse-grained soils if soil is estimated to contain 5% to 15% fines (equals "with").

Non-Cohesive (Granular) Soil

Relative Density	Blows Per Foot (N-Value)
Very Loose	Less than 5
Loose	5 to 9
Compact	10 to 29
Dense	30 to 50
Very Dense	Greater than 50

Cohesive (Clayey) Soil

Consistency	Blows Per Foot (N-Value)
Very Soft	0 to 2
Soft	3 to 4
Firm	5 to 8
Stiff	9 to 15
Very Stiff	16 to 30
Hard	Greater than 30

Grain Size Classification
(based on standard sieve sizes)

Cobbles	Greater than 3 inches (76 mm)
Gravel	3 in. to No. 4 (4.76 mm)
Coarse Gravel	3 in. to 3/4 in.
Fine Gravel	3/4 in. to No. 4 (4.76 mm)
Sand	No. 4 (4.76 mm) to No. 200 (0.074 mm)
Coarse Sand	No. 4 (4.76 mm) to No. 10 (2.0 mm)
Medium Sand	No. 10 (2.0 mm) to No. 40 (0.42 mm)
Fine Sand	No. 40 (0.42 mm) to No. 200 (0.074 mm)
Silt	No. 200 (0.074 mm) to 0.002 mm
Clay	Less than 0.002 mm

Component Percentage Descriptors
(estimate to nearest 5%)

Coarse Grained Soils

Noun(s) (e.g., sand, gravel)	Major Component
Adjective (e.g., silty, clayey)	Greater than 15%
With (e.g., with silt, with clay)	5% to 15%
Trace (e.g., trace silt, trace clay)	Less than 5%

Fine Grained Soils

Noun(s) (e.g., silt, clay)	Major Component
Adjective (e.g., sandy, gravelly)	Greater than 30%
With (e.g., with sand)	15% to 30%
Few (e.g., few sand)	5% to 15%
Trace (e.g., trace sand)	Less than 5%

Soil Structure Terms

Stratified	Blocky
Laminated	Lenses/Seams
Fissured	Homogeneous

Moisture

Dry
Moist
Wet

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WELL CONSTRUCTION MATERIALS

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WELL CONSTRUCTION MATERIALS

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INTRODUCTION

In environmental subsurface investigations, the information used to evaluate subsurface conditions often relies heavily on the installation of quality groundwater monitoring wells. The application and use of the proper well construction materials to the specific well installation is crucial to obtaining representative and reliable groundwater samples.

The two general types of wells are groundwater monitoring wells and pumping (also referred to as recovery, extraction, or withdrawal) wells. The specific use of a groundwater well dictates the types of materials used to construct it.

This FMG outlines the general types and use of well construction materials and considerations involved in selecting appropriate materials for specific well installation applications. Installation of these materials are described in detail in the specific well installation FMGs listed below.

PROCEDURES REFERENCED

- FMG 3.2 - Overburden and Top of Rock Wells
- FMG 3.3 - Deep Bedrock Wells
- FMG 3.4 - Pump Wells
- FMG 3.5 - Piezometers

EQUIPMENT DESCRIPTIONS

Well Screen

Well screen is the portion of the well pipe that contains appropriately sized openings and allows groundwater to enter the well. The screen materials used in groundwater monitoring wells are crucial to ensuring the installation of an efficient, productive, and durable groundwater well.

The diameter of the well screen is generally dependent upon the application of the well. For monitoring wells used in groundwater level measurements and groundwater sampling, screen diameter will generally be 2.0-inch inner diameter (ID) flush-threaded screen segments (piezometers are typically 1.0-inch ID but may be 2-inch also). These screen segments are typically available in 10-foot lengths. Four-inch diameter or larger well screens are usually reserved for recovery or production well applications where larger diameters permit greater groundwater withdrawal rates. Larger diameter wells also allow a well to serve additional functions such as housing oil recovery systems.

Screen material will be either thermoplastic Schedule 40 polyvinyl chloride (PVC) (ASTM D1785, ASTM D2665, ASTM F480) or Schedule 5 Type 316 stainless steel, depending primarily on the depth of the well and the groundwater quality (degree and nature of contamination). Shallower depths and generally low levels of contaminants in groundwater allow for PVC applications, whereas greater depths and severely degraded groundwater quality, or the presence of free-phase oils or solvents, may necessitate stainless steel due to its greater strength and resistance to chemical degradation. It should be noted that PVC and stainless steel are appropriate for the vast majority of environmental applications and are generally accepted by regulatory agencies. Well materials other than PVC or stainless steel should be used only in certain instances, to be determined and approved by the Project Manager on a case-by-case basis.

Certain applications such as investigation of inorganic (metals) concentrations in groundwater, or the presence of low pH (acidic) conditions, may preclude the use of stainless steel wells. Stainless steel, which contains molybdenum in addition to its iron content, may leach out metal compounds which could lead to misleading groundwater analysis results.

PVC may likewise leach out or degrade specific thermoplastic elements of its composition which may compromise the well integrity or groundwater analyses. PVC generally performs well in acidic groundwater conditions; however, it may degrade in the presence of certain organic compounds such as ketones, aldehydes, or chlorinated compounds in high concentrations. Certain additives to the PVC may also affect groundwater quality.

Well screen slot sizes and well screen type will also be consistent for groundwater monitoring wells. Screen slot size is typically 0.010 inches; 0.020-inch slot size may be more appropriate for coarser formation materials or where the well may serve as a recovery well for free-phase oils. For monitoring applications, slot type should be either factory machine-slotted or continuous-wrap

slotted. Perforated, bridge-slotted or louver-slotted well screens are generally not acceptable for most environmental applications and should be avoided.

Screen slot sizes may vary from these two sizes when used in production or recovery (pumping) well applications where the need to maximize groundwater withdrawal is essential. In such cases, screen slot sizes can be manufactured to exact specifications for a particular well based on particle size analysis results and formation transmissivity or permeability.

Well Riser Pipes and Casings

Well riser pipe is a solid extension of the well screen that extends from the screen up to the surface. The riser pipe protects the well screen, prevents outside groundwater from entering the well, and allows groundwater pumped from down in the open interval to be routed up through the well to the surface.

Well riser pipe should be of the same material and size as the well screen described above. In instances to be determined and approved by the Project Manager on a case-by-case basis only, differing materials may be approved for use in the same well (e.g., stainless steel well screen connected to PVC riser). Well risers should extend to the surface and should either be cut at grade in flush-mount completions or as an approximately 3-foot stickup to be covered with a steel protective casing.

Well riser pipe sections shall be flush-threaded and fitted with neoprene, rubber, or other appropriately constructed, durable o-rings to properly seal the threaded pipe joints. Glues or cements are not to be used in well construction.

In installations of bedrock monitoring wells, which have an open rock monitoring interval and a permanent well casing that extends from bedrock to the surface, the permanent casing (or casings in telescoping wells) shall be made of carbon steel or low-carbon steel (greater than 0.8 percent carbon and less than 0.8 percent carbon, respectively). The well casing should be a minimum of 4 inches in diameter (at least 4 inches diameter for the innermost casing).

On sites wells where dense, non-aqueous phase liquid (DNAPL) is present or may be a concern, in screened wells it is advisable to install a collection sump on the base of the well below the well screen to collect infiltrated DNAPL for possible measurement and/or sampling. Sumps should be installed as a 1- to 5-foot section of solid riser material with a sealed bottom placed below the well screen.

Sand Packs

The filter pack, or sand pack, installed in a well replaces formation material immediately around a well with a more permeable material (sand). The sand pack separates the well screen from the formation, increases the hydraulic diameter of the well, and prevents fines (silt or clay) from entering or clogging the well screen.

Sand pack of an appropriate size shall be utilized based on the well screen slot size being used. Sand pack size should be chosen so that the majority of the sand (sand pack has inherent variation in its particle grain size distribution) is larger than the screen slot size while sized small enough to prevent deleterious amounts of formation fines from entering the well through the sand pack. Screen slot sizes of 0.010-inch and 0.020-inch typically use a sand pack such as Morie or U.S. Silica No. 1, No. 0, No. 00N, or equivalent.

Sand pack shall be washed silica sand with a silica content of at least 95 percent. Sands should meet one or more of the following requirements: NSF 61, AWWA B-100, ANSI, or equivalent standards for uniformity and chemical inertness. In cases to be determined and approved by the Project Manager on a case-by-case basis only, differing sand pack materials may be approved for use in a well. Sand packs used for production and recovery wells with larger screen slot sizes will use larger particle sized sand packs of the same type and quality. The slot size and sand pack size for recovery wells should be chosen based on results of formation grain size distribution analysis.

Seals

Bentonite and grout seals are installed above the sand pack to isolate the monitoring interval and prevent groundwater from infiltrating into the well screen from other water-bearing zones. Seals also prevent migration of backfill or formation materials downward into the sand pack.

Bentonite is the generic name for a group of a naturally occurring clay minerals (montmorillonites) that come in a variety of forms: pellets, chips, granulated, or powdered. This material is commercially available as "Wyoming Bentonite". When hydrated it swells to many times its original volume and forms an ultra-low permeability clay seal.

Bentonite chips or pellets are generally used to create a seal immediately above the sand pack. The chips/pellets are dropped inside the augers or well casing by hand down through the water column onto the top of the sand pack. Care must be taken to prevent "bridging" of the bentonite particles in the casing above the target zone. Measurements of the depth to the top of the seal must be obtained during installation of the seal to ensure its proper position and thickness. In the absence of significant water in a casing or borehole, potable water must be added to hydrate the bentonite. The bentonite seal will be allowed to set for a minimum of one-half hour, in order to hydrate properly, before additional seals (grout) are applied. Once the bentonite has set for one-half hour the grout seal may be placed, as described below.

In saline groundwater environments, such as where ocean water may infiltrate the monitoring interval, a zeolite-based seal material may be used, as saline conditions may hamper the performance of bentonite pellets.

Portland cement grout (grout) forms a concrete-like seal that can be more manageable than bentonite (e.g., able to be pumped through a water pump). Grout is generally placed on top of the hydrated bentonite seal to form a solid cement seal around the well riser up to the surface. In certain circumstances, only under approval of the OXY Project Manager, soil cuttings may be used to backfill the borehole in lieu of grout.

The grout mixture will consist of one 94-pound bag of Portland cement and 3 to 5 pounds of powdered bentonite added per sack of cement. Two pounds of calcium chloride may also be added (under certain conditions, e.g., very cold days) to accelerate the setting time of the grout, as well as to increase the dry strength of the grout. The grout will be thoroughly mixed with 6.5 gallons of potable water per sack of cement. Grout is generally placed using either the tremie or Halliburton grouting methods. These are described in the specific well installation FMGs.

Protective Casings and Surface Seals

Once the well screen, riser, and all seals have been placed to ground surface, the well riser must be protected. This includes protection from vehicles, damage, surface water infiltration, and weather. This is typically accomplished using either a flush-mount roadbox or a stickup casing.

Flush-mount roadboxes are circular steel casing segments with a heavy-duty steel lid with locking bolts. These units are widely available and come in a number of diameters and lengths, depending on the well diameter. A stickup protective casing is generally a length of carbon or stainless-steel pipe with a locking top.

For a typical 2-inch monitoring well, the roadbox should be at least 6 inches in diameter; a stickup casing should be at least 4 inches in diameter. A roadbox should be at least 12 inches in length (they are typically 16 to 18 inches long) and is installed flush with the ground surface. A stickup casing should be at least 5 to 6 feet long such that approximately 2.5 to 3 feet is below ground surface and 2.5 to 3 feet is protruding above grade. In wells where a permanent steel casing is installed (serves as the well riser pipe) and brought to the ground surface, it may be used as the protective casing provided it is equipped with a semi-permanent, metal, locking cap or cover that can be affixed to the steel casing.

Flush-mount installations should have at least the last 18 inches of the open borehole filled with coarse sand, placed up to ground surface to allow drainage of surface water infiltration down through and out of the roadbox. This also prevents infiltrating surface water from accumulating up over the top of the well riser and draining down into the well. This sand drain is not necessary in the locking cap stickup casings.

Both roadbox and stickup casings must be secured in the ground with concrete, which also serves as a surface seal.

In areas of high vehicle traffic activity, protective steel bollards should be installed. This is typically a vertically oriented, concrete-filled, steel pipe (minimum 4 inches diameter) cemented at least 3 feet into the ground, acting as a "guard rail" for the well casing and preventing it from being damaged by vehicles. Three bollards should be placed around a well to provide adequate protection.

EQUIPMENT/MATERIALS

- Drilling equipment.
- Well screen and riser materials.
- Sand pack.
- Bentonite pellets/chips.
- Powdered bentonite.
- Portland cement.

REFERENCES

ASTM D1785-99, Standard Specification for Poly(Vinyl Chloride) (PVC) Plastic Pipe, Schedules 40, 80, and 120.

ASTM D2665-00, Standard Specification for Poly(Vinyl Chloride) (PVC) Plastic Drain, Waste, and Vent Pipe and Fittings.

ASTM F480-00, Standard Specification for Thermoplastic Well Casing Pipe and Couplings Made in Standard Dimension Ratios (SDR), Schedule 40 and Schedule 80.

ASTM A53/A53M-01, Standard Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated, Welded and Seamless for Ordinary Uses.

Campbell, M.D., and Lehr, J.H., Water Well Technology, McGraw Hill, 1973.

Cold Weather Concreting, ACI Committee 306, Materials Journal, Volume 85, Issue 4, July 1, 1988.

Driscoll, Fletcher G., Groundwater and Wells, Johnson Filtration Systems, Inc., 1986.

Freeze, R. Allen, and Cherry, John A., Groundwater, Prentice-Hall, 1979.

USEPA, 1986, RCRA Groundwater Monitoring Technical Enforcement Guidance Document, Office of Solid Waste and Emergency Response, 1986.

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OVERBURDEN AND TOP OF ROCK MONITORING WELL INSTALLATION

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure describes procedures for the installation of overburden groundwater monitoring wells.

PROCEDURES REFERENCED

- FMG 1.3 – Utility Clearance
- FMG 2.2 – Drilling Techniques
- FMG 3.1 – Well Construction Materials
- FMG 3.7 – Well Development
- FMG 6.15 – PFAS/PFOA Sampling
- FMG 9.0 – Equipment Decontamination
- FMG 10.0 – Waste Characterization

EQUIPMENT/MATERIALS

The following lists the equipment and materials used for the installation of overburden wells.

1. Site Plan, Field Sampling Plan, and/or Work Plan, with proposed soil boring/monitoring well locations.
2. Personal protective equipment (PPE) as required by the site-specific Health and Safety

Plan (HASP).

3. Drilling equipment appropriate for the site and investigation objectives.
4. Well construction materials appropriate for the intended use of the groundwater monitoring well. FMG 3.1 – Well Construction Materials outlines the general types and use of well construction materials, and considerations involved in selecting materials for specific well applications.
5. Water level meter.
6. Weighted tape measure, graduated in tenths of a foot.
7. Electronic water level probe.
8. Locks and keys for locking the completed groundwater monitoring wells.
9. A heavy-duty folding ruler for measuring soil sample recovery and noting stratigraphic changes.
10. Permanent marker for labeling the well cover or casing.

DRILLING PROCEDURES

FMG 2.2 – Drilling Techniques presents descriptions of various drilling methods that are available, including rotosonic, direct-push, hollow-stem auger, rotary spun casing, and dual-wall reverse circulation air techniques. Regardless of the method chosen, the following procedures will be followed:

- Construct a temporary decontamination pad from plywood sheets, 2 X 6 boards and 6-millimeter (minimum thickness) plastic capable of fitting the drill rig. An alternate containment structure may be used as long as it is suitable to contain the decontamination waste material.
- Drilling and sampling equipment will be decontaminated prior to drilling, between samples that are being collected for laboratory analysis, and prior to leaving the site in accordance with the FMG 9.0 – Equipment Decontamination.
- No oils or grease will be used on equipment introduced into the borehole.
- Environmental grade grease may be used to lubricate drill threads (for per- and polyfluroalkyl substances (PFAS)/perfluorooctanoic acid (PFOA) use restrictions if sampling for PFAS/PFOA, see FMG 6.15 – PFAS/PFOA Sampling).
- Drilling-generated waste materials will be characterized in accordance with FMG 10.0 - Waste Characterization.
- The depth to the target interval may be determined from an existing adjacent monitoring well/boring or from information obtained from sampling the borehole. The criteria for determining the target interval to be monitored will be presented in the project Work Plan. Typically, an 8-inch diameter borehole will be advanced to the target interval, although a larger- or smaller-sized borehole may be necessary based on the objectives of the

groundwater monitoring program. For example, a larger diameter sand pack may be desirable to limit the mobilization of particulates from the soil column in response to sampling activities, or a smaller diameter well and sand pack may be practical due to access limitations.

- Unless otherwise approved, a minimum annular space of one inch should be maintained between the well casing and the borehole casing or augers to facilitate proper placement of the sand pack and seal materials and to minimize the chance for "bridging" of the materials.
- In instances where the borehole is advanced deeper than the target interval, a hydrated bentonite pellet seal will be installed to bring the bottom of the boring to within 6 inches of the target interval. Six inches of filter sand will then be placed above the bentonite seal prior to installing the well to prevent the introduction of clay particles into the well.
- In some areas where the water table is known to be at or near the top of bedrock, the base of the overburden well may be installed at the top of bedrock.

WELL INSTALLATION

The well installation procedures presented below are the recommended guidelines. Due to variations in subsurface conditions, changes in these well installation guidelines may be necessary (e.g., to accommodate installation of the protective casing in instances where the water table is very shallow, or to properly monitor a thin water bearing unit).

Well construction materials are discussed in FMG 3.1 – Well Construction Materials. Well screen lengths of 5 or 10 feet are typically used; however, other screen lengths may be applicable depending on subsurface conditions. Water table monitoring wells will be constructed with the screen straddling the water table, and with approximately 7 feet of a 10-foot well screen or 3-feet of a 5-foot well screen extending below the water table. The screen placement should allow for fluctuation in groundwater levels, and well screen lengths may need to be increased if groundwater is known to fluctuate more than a few feet. Monitoring wells installed in confined aquifers should center on the permeable confined unit without overlapping across impermeable unit and possibly cross-connecting vertical aquifers.

Top of Rock (TOR) monitoring wells should be constructed in such a fashion that the bottom of the well screen is placed on the top of the bedrock. Depending on project requirements, a sump consisting of blank well casing material, may be installed to the bottom of the well screen and sumped a couple of feet into the rock to measure and possible collect suspected DNAPL. When installing TOR monitoring wells, a temporary casing should be utilized during the installation process to minimize contamination drawdown and seal off overlying aquifers.

Once the target well depth is reached, a pad of sand is placed below the base of the well screen and the well materials are placed in the borehole. As the augers or drill casing are slowly removed, sand filter pack is placed in the annular space around the well screen and casing from the base of the screen to approximately 2 feet above the screen. A shallow water table may necessitate a shorter sand pack. The filter pack shall consist of clean, uniform, well-rounded

silica sand of an appropriate size based on the screen slot size being used and the soil particle size in the screened interval, as specified in the Work Plan and/or dictated by site conditions. The types of sand used as filter pack are discussed in detail in FMG 3.1 – Well Construction Materials.

A hydrated bentonite seal with a minimum thickness of 2 feet is placed above the sand pack. If the water table elevation is at least several feet above the top of the sand pack, a 2-foot thick (minimum) layer of bentonite pellets will be placed above the sand pack using a tremie pipe. No coated bentonite pellets will be used in monitoring well drilling or construction, due to the potential for cross-contamination. The seal will be hydrated and allowed to set for approximately 45 minutes. Granular or flaked pH-neutral bentonite will be hydrated and used for seals placed above the water table. The types of sealing and grouting materials are discussed in detail in FMG 3.1 – Well Construction Materials. Grout should not be used directly above or below the sand pack without a hydrated bentonite seal.

During the placement of the sand pack and bentonite seal, a weighted tape will be employed to provide constant measurements and help prevent bridging. Above the bentonite seal, Portland cement grout containing three to five percent bentonite will be tremied into place. If the total well depth is 20 feet or less, the bentonite seal may be extended to the base of the surface seal. The augers or drill casing will be gradually pulled during the addition of the filter pack, bentonite seal and cement-bentonite grout seal.

Accurate measurements of the material depths will be made during installation. The volume of materials needed will be calculated and compared to the actual volume used. Materials used, and depths of placement will be recorded on FMG 3.2-01 – Overburden Well Installation Report.

The well casing will be secured with a vented lockable cap. If the well is located in a high traffic area, the casing will be protected by a flush-mounted roadway box installed with a sand drain and set in a concrete seal. It is recommended that the surface seal extend a minimum of three inches outside the well casing, to allow for a proper seal and to resist damage from frost. A lockable gripper plug must be installed at the top the inner well casing. In cases where the well is in a flowing artesian condition, an inflatable packer may be used to prevent the groundwater from discharging to the surface. Alternatively, in low traffic areas, the well casing may be cut above grade and completed with 4- or 6-inch diameter steel protective, lockable, casing with approximately 3 ft of stick up, set in a concrete surface seal. Details regarding the type of appropriate well covers and concrete surface seals are contained in FMG 3.1 – Well Construction Materials.

After installation, the monitoring well will be labeled with the well identification and a reference point for water level and depth measurements will be marked on the inner well casing. The well will also be locked unless deemed unnecessary by the OXY Project Manager. Locks placed on site monitoring wells should be keyed alike and made of material that is resistant to corrosion such as heavy-duty aluminum alloy with a chrome-plated hardened steel shackle, brass tumbler, and double steel locking mechanism (e.g., American Lock® brand locks or similar). The well will be allowed to sit for at least 24 hours prior to well development to allow grout to harden, in

accordance with FMG 3.7 – Well Development. Following installation, tie-in measurements to a minimum of two nearby site features will be made and recorded. Monitoring wells will generally be surveyed following their installation.

DOCUMENTATION OF WELL DESIGN AND CONSTRUCTION

The following information regarding the design and construction of each well will be recorded on the form FMG 3.2-01 – Overburden Monitoring Well Installation Report, or equivalent:

- Date/time of installation;
- Drilling method;
- Surveyed well location;
- Borehole diameter and well diameter;
- Well depth;
- Screened Interval;
- Casing materials;
- Screen materials and design;
- Screen slot size/length;
- Filter pack material/grain size;
- Sealant materials (percent bentonite);
- Sealant materials (lbs./gallon of cement);
- Sealant placement method;
- Surface seal design/construction;
- Type of protective well cap; and
- Detailed drawing of well.

EQUIPMENT CLEANING

Drilling equipment and well materials (casing and screen) will be cleaned using high-pressure steam-cleaning equipment and potable water, in accordance with FMG 9.0 – Equipment Decontamination. Drilling equipment will be cleaned prior to use on the site, between monitoring well locations, and at the completion of the drilling program, prior to leaving the site.

DISPOSAL METHODS

All Investigation-Derived Waste (IDW), including water generated during decontamination

procedures will be handled in accordance with the site waste disposal plan in coordination with the Client and the site Resource Manager (RM), and FMG – 10.0 – Waste Characterization.

REFERENCES

American Society for Testing and Materials (ASTM) (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

American Society for Testing and Materials (1991), Standard D5092, "Practices for Design and Installation of Ground Water Monitoring Wells in Aquifers", Annual Book of ASTM Standard, Section 4, Volume 04.08.

Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

Driscoll, Fletcher G., Groundwater and Wells, Johnson Filtration Systems, Inc., 1986.

Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

Freeze, R. Allen, and Cherry, John A., Groundwater, Prentice-Hall, 1979.

Form FMG 3.2 - 01	**OVERBURDEN MONITORING WELL INSTALLATION REPORT**	Well No.
		Boring No.

PROJECT _____

LOCATION _____ **PROJECT MANAGER** _____

CLIENT _____ **FIELD REP.** _____

CONTRACTOR _____ **DATE INSTALLED** _____

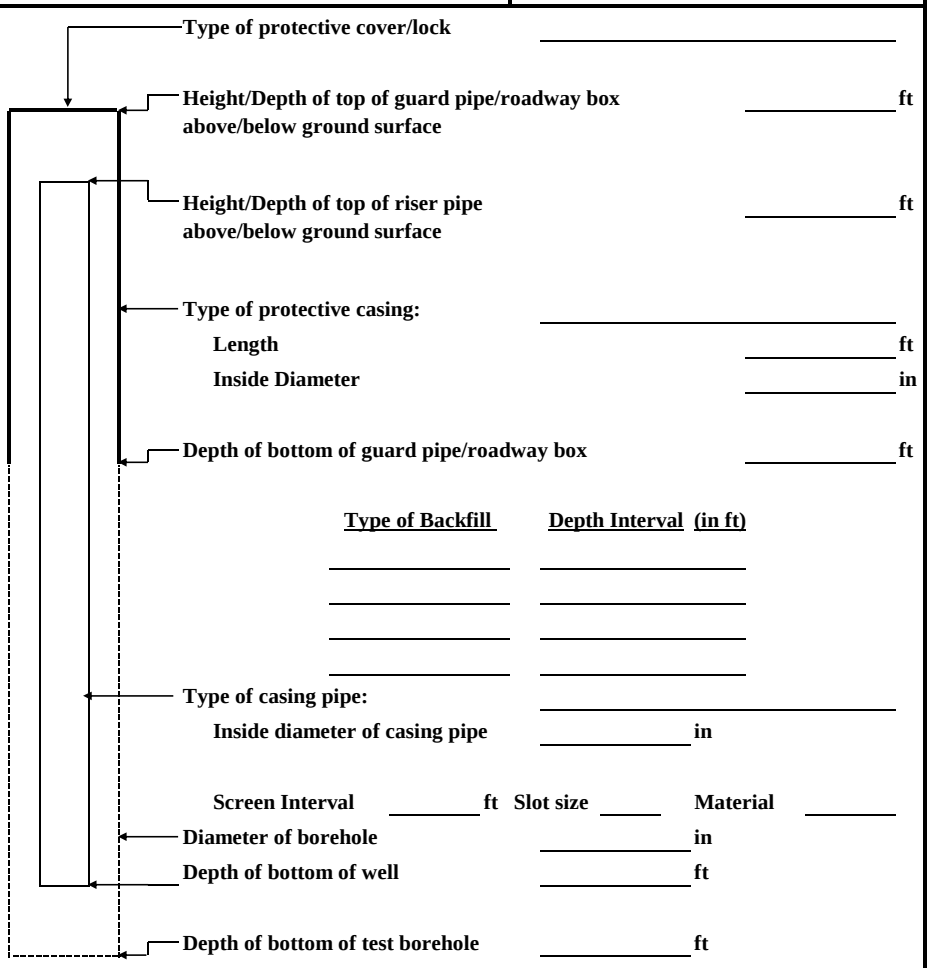
DRILLER _____ **WATER LEVEL** _____

Ground Elev. _____ ft **Location** _____ ☐ **Guard Pipe**

Top of Casing Elev. _____ ☐ **Roadway Box**

SOIL CONDITIONS	**BOREHOLE BACKFILL**

(Bottom of Exploration)
(Numbers refer to depth from ground surface in feet)



(Not to Scale)

* - Elevation Datum = _____

COMMENTS: _____

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WELL DEVELOPMENT

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure is for the development of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Before a newly constructed well can be used for water quality sampling, measuring water levels, or aquifer testing, it must be developed. Well development refers to the procedure used to clear the well and formation around the screen of fine-grained materials (sands, silts, and clays) produced during drilling or naturally occurring in the formation. Sampling should not be done for 1-2 weeks after well development to allow the well to return to normal groundwater conditions.

Well development is completed to remove fine grained materials from the well but in such a manner as to not introduce fines from the formation into the sand pack. Well development continues until the well responds to water level changes in the formation (i.e., a good hydraulic connection is established between the well and formation) and the well produces clear, sediment-free water to the extent practical.

PROCEDURES REFERENCED

- FMG 3.2 - Overburden and Top of Rock Wells
- FMG 3.3 – Deep Bedrock Wells
- FMG 6.15 – PFAS/PFOA Sampling
- FMG 10.0 - Waste Characterization

PROCEDURAL GUIDELINES

The well development procedures presented below are the recommended standards. However, due to variations in conditions, changes in these standards may be necessary in order to facilitate successful monitoring well development.

Well development can be accomplished by using in-place pumps or by using portable equipment; either peristaltic, bladder, or other appropriate pumps depending on well depth. In the case of developing wells installed utilizing the mud rotary methods (least preferred method) it would be beneficial to surge the well prior to and during development to help break down the filter cake that may have built up on the well screen.

- Don appropriate safety equipment.
- All equipment used for development purposes entering each monitoring well will be cleaned using a soapy wash [laboratory grade, confirm no presence of per- and polyfluroalkyl substances (PFAS) or perfluorooctanoic acid (PFOA) if sampling for PFAS/PFOA (see FMG 6.15 – PFAS/PFOA Sampling for further details)], tap water rinse, isopropyl alcohol rinse (or other rinse agent that is appropriate for site-specific conditions), and distilled/deionized water rinse.
- Uncap well and allow water level to stabilize. Attach appropriate pump and lower tubing into well.
- Turn on pump. If well runs dry, shut off pump and allow to recover.
- Collect the groundwater sample in a glass jar to determine relative turbidity, and measure and record the temperature, pH, turbidity, and specific electrical conductance.
- The above steps will be repeated until groundwater is relatively silt-free; no further change is noted; the temperature, pH, turbidity, and specific conductance readings have stabilized to within 10 percent.
- The time period between development and groundwater sampling will be dependent upon the project objectives, and the chemicals of concern (COCs). When sampling for COCs sensitive to turbidity presence (i.e., semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), metals), an extended time period between the development activity and the sampling event will be observed. On OXY sites, sampling will be conducted in accordance with the following:

Primary COC	***Time Period Between Development and Sampling***
General Chemistry	1 week
Volatile organic compounds (VOCs)	1 week
SVOCs, PCBs, Metals	2 weeks

Waste Disposal

- All waste generated will be disposed in accordance to the methods and procedures contained in FMG 10.0 - Waste Characterization through the onsite Resource Manager, if the site is active.
- All water generated during cleaning and development procedures will be collected and contained in accordance to the site-specific disposal requirements.
- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from soil sampling and handling activities, will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

EQUIPMENT/MATERIALS

- Appropriate health and safety equipment.
- Knife.
- Power source (e.g., generator, battery).
- Field book.
- Form FMG 3.6-01 - Well Development and Stabilization Form.
- Well keys.
- Graduated pails.
- Surge block
- Form FMG 3.6-01 - Well Development and Stabilization Form.
- Well keys.
- Graduated pails.
- Pump and tubing.
- Cleaning supplies (including non-phosphate soap, buckets, brushes, laboratory-supplied distilled/deionized water, tap water, isopropyl alcohol or other site-specific rinse agent (e.g., nitric acid solution), aluminum foil, plastic sheeting, etc.).
- Water level meter.
- pH/temperature/conductivity meter.
- Turbidity meter.
- Clear glass jars (e.g., drillers' jars).

REFERENCES

Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

WELL DEVELOPMENT AND STABILIZATION FORM

PROJECT NAME: _____ **PROJECT NO.:** _____

DATE OF WELL DEVELOPMENT: _____

DEVELOPMENT CREW MEMBERS: _____

PURGING METHOD: _____

SAMPLE NO.: _____

SAMPLE TIME: _____

WELL INFORMATION

WELL NUMBER: _____

WELL TYPE (diameter/material) _____

MEASURING POINT ELEVATION: _____

STATIC WATER DEPTH: _____ **ELEVATION:** _____

BOTTOM DEPTH: _____ **ELEVATION:** _____

WATER COLUMN LENGTH: _____

SCREENED INTERVAL: _____

WELL VOLUME: _____

Note: For 2-inch diameter well: 1 foot = 0.14 gallons (Imp) or 0.16 gallons (US)
1 meter = 2 liters

	UNITS	*1*	*2*	*3*	*4*	*5*	*TOTAL/ AVERAGE*
VOLUME PURGED (volume/total volume):							
FIELD pH:							
FIELD TEMPERATURE:							
FIELD CONDUCTIVITY:							
CLARITY/TURBIDITY VALUES:							
COLOR:							
ODOR:							
COMMENTS:							

COPIES TO: _____

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WELL DECOMMISSIONING

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 3.7
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

WELL DECOMMISSIONING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure is for the decommissioning/abandonment of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Well decommissioning refers to the procedure used to properly abandon or remove the monitoring well from the formation while taking the proper precautions to help eliminate cross-contamination.

The proper methods for properly abandoning monitoring wells are either by leaving the well materials in place and pressure grouting with a cement/bentonite slurry directly into the well or by over-drilling with augers, removing the well material, and backfilling with a cement-bentonite slurry. Individual state regulations must be reviewed and followed prior to and during well abandonment procedures.

PROCEDURES REFERENCED

- FMG 3.2 - Overburden and Top of Rock Wells
- FMG 3.3 - Deep Bedrock Wells
- FMG 9.0 - Equipment Decontamination
- FMG 10.0 - Waste Characterization

PROCEDURAL GUIDELINES

Pressure Grouting

- The borehole log from the monitoring well needs to be obtained to determine the well construction in order to prepare the proper materials and calculate the quantity of cement/ bentonite slurry that will be required.
- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately 1 to 2 feet below ground surface.
- A tremie pipe will be placed into the well completely to the bottom. A cement/bentonite slurry will then be pressure grouted in to the monitoring well backfilling completely to the surface. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water. The grout will be allowed to sit for approximately 1 hour to allow any settlement of the cement/bentonite slurry and then augment if needed.

Overdrilling

- Based on the diameter of the monitoring well, this information can be obtained from the well completion diagram, the proper sized augers need to be specified.
- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately 1 to 1 feet below ground surface.
- The augers are then placed over the riser pipe of the monitoring well and then drilling commences. The drilling continues until the final depth to which the monitoring well was installed is reached. The well materials are then removed (pulled) from the augers.
- A cement/bentonite grout will be placed from the bottom of the borehole to the top of the augers. As each flight of augers is removed from the ground, the cement/bentonite grout will continue to be placed in the augers, to the top. This will continue until all the augers have been removed from the borehole. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water.
- The area final restoration will be completed in accordance with the directions of the OXY Facility representative (e.g., asphalt, concrete, vegetation). In active work areas final restoration maybe necessary immediately; or time to allow settlement of the abandoned well area may be permitted prior to final restoration being performed.
- Documentation/Notification requirements include modification of the well log to reflect closure and if necessary notification to the appropriate regulatory agency.

Waste Disposal

- All waste generated will be disposed of in accordance with the methods and procedures contained in FMG 10.0 - Waste Characterization.
- All material generated during well decommissioning procedures will be collected and contained on site in roll-off boxes or 55-gallon drums for future analysis and appropriate disposal.
- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from well closure activities, will be placed in plastic bags. These bags will be handled in accordance with the Work Plan.

EQUIPMENT/MATERIALS

- Drilling equipment.
- Well supplies.
- Subsurface boring log.
- Tape measure.

REFERENCES

Michigan Department of Public Health, Ground Water Quality Control Section – Division of Water Supply (1988), Michigan Water Well Grouting Manual, MDPH GW-3-302.

ASTM D5229 "Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and other Devices for Environmental Activities".

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 5.1
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WATER LEVEL MEASUREMENTS

LIST OF FORMS
(Following Text)

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OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

WATER LEVEL MEASUREMENTS

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure describes measurement of water levels in groundwater monitoring and extraction wells, piezometers and boreholes. This procedure does not cover automated measurement of water levels with a transducer/datalogger and does not cover measurement of phase-separated liquids.

Water levels in monitoring wells will be measured prior to each sampling event and at other times as indicated in the project Work Plan. Water levels will be acquired in a manner that provide accurate data that can be used to calculate vertical and horizontal hydraulic gradients and other hydrogeologic parameters. Accuracy in obtaining the measurements is critical to insure the useability of the data.

PROCEDURES REFERENCED

- FMG 6.5 - Non-Aqueous Phase Liquid (NAPL)
- FMG 8.0 - Field Instruments – Use/Calibration
- FMG 9.0 - Equipment Decontamination

PROCEDURAL GUIDELINES

In order to provide reliable data, water levels must be collected over as short a period of time as practical. Barometric pressure can affect groundwater levels and, therefore, observation of significant weather changes during the period of water level measurements must be noted. Tidal fluctuations, navigation controls on rivers, rainfall events, and groundwater pumping can also affect groundwater level measurements. Personnel collecting water level data must note if any of these controls are in effect during the groundwater level collection period. Due to possible changes

during the groundwater level collection period, it is imperative that the time of data collection at each station be accurately recorded.

In conjunction with groundwater level measurements, surface water (e.g., ponds, lakes, rivers, and lagoons) often are monitored as well. This information is very helpful (and can be critical) in understanding the hydrogeologic setting of the site and most importantly how contaminants may move beneath the site.

The depth to groundwater will be measured with an electronic depth-indicating probe. Prior to obtaining a measurement, a fixed reference point on the well casing shall be established for each well to be measured. Unless otherwise established, the reference point is typically established and marked on the north side of the well casing. Avoid using protective casings or flush-mounted road boxes for reference, due to the greater potential for damage or settlement.

If provided for in the project Work Plan, the elevation of the reference point shall be obtained by accepted surveying methods, to the nearest 0.01 foot.

The water level probe will be lowered into the well until the meter indicates (via indicator light or tone) the water is reached. The probe will be raised above water level and slowly lowered again until water is indicated. The cable will be held against the side of the inner protective casing at the point designated for water level measurements and a depth reading taken. This procedure will be followed three times or until a consistent value is obtained. The value will be recorded to the nearest 0.01 foot on Form FMG 5.1-01 - Groundwater Level Monitoring Report or other designated data recording location if specified in the project Work Plan.

Upon completion, the probe will be raised to the surface and together with the amount of cable that entered the well casing, will be decontaminated in accordance with methods described in FMG 9.0 - Equipment Decontamination.

EQUIPMENT/MATERIALS

- Battery-operated, non-stretch electronic water level probe with permanent markings at 0.01-foot increments (traceable to national measurement standards), such as the Solinst Model 101 or equivalent.
- The calibrated cable on the depth indicator will be checked against a surveyor's steel tape once per quarter year. A new cable will be installed if the cable has changed by more than 0.01 percent (0.01 foot for a 100-foot cable). See also FMG 8.0 - Field Instruments – Use/Calibration.

REFERENCES

ASTM D4750 - Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well).

ASTM D6000 - Guide for Presentation of Water-Level Information from Ground-Water Sites.

Form FMG 5.1-01

GROUNDWATER LEVEL MONITORING REPORT

WELL NUMBER

Page of

PROJECT ______________________ **PROJECT MANAGER** ______________

LOCATION ______________________ **FIELD REP.** ______________

ELEVATION REFERENCED TO: ______________________ **DATE** ______________

Date	Time	Elapsed Time (days)	Depth of Water from ( ) in ft	Elevation of Water	Remarks	Read By

FMG MODIFICATIONS MUST BE ACCOMPANIED BY A REVISION REQUEST FORM APPROVED BY THE PROJECT MANAGER

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 5.2
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IN SITU HYDRAULIC CONDUCTIVITY (SLUG TEST) PROCEDURE

LIST OF FORMS
(Following Text)

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EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

IN SITU HYDRAULIC CONDUCTIVITY (SLUG TEST) PROCEDURE

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure describes the protocol for performing in situ hydraulic conductivity (slug) tests, including preparation, collection of valid field data, and preliminary evaluation of the data.

A slug test is performed to assess the horizontal hydraulic conductivity of a water-bearing zone. Slug tests are accomplished by stressing the screened water-bearing zone through an instantaneous displacement (with a slug) (or removal of water with a bailer) and subsequently measuring and recording the water level response in the well versus time. If the removal of the slug or bailer does not result in the well recovering more than 5 percent of the "90 percent recovery time", then it is considered an "instantaneous" displacement.

Slug testing in select monitoring wells will be performed after the wells have been installed and developed as covered in the Work Plan. Slug testing data will be acquired in a manner that provides valid data that can be used to calculate the horizontal hydraulic conductivity of the formation tested.

There are two types of slug tests: falling-head tests and rising-head tests. It is generally preferable to do a rising-head slug test due to a number of potential problems that can arise with falling-head tests (some of these may lead to inaccurate hydraulic conductivity estimates). It is strongly recommended that water level measurements should be collected automatically using a datalogger/ pressure transducer system if at all possible, but they may be collected manually using a battery-operated water level measurement probe if necessary.

PROCEDURES REFERENCED

- FMG 3.0 - Monitoring Wells, Pump Wells, and Piezometers

- FMG 5.1 - Water Level Measurements
- FMG 8.0 - Field Instruments – Use/Calibration
- FMG 9.0 - Equipment Decontamination
- FMG 10.0 - Waste Characterization

PROCEDURAL GUIDELINES

A slug test involves rapidly changing the water level in a well and then measuring the water-level response over time. A very quick change in the water level in a well should be effected at the beginning of a slug test using one of several methods:

- Preferably by inserting or withdrawing a solid or sealed object with an appropriate overall density.
- By changing the air pressure in a well, or pneumatic slug testing (only when a pressure transducer is used).
- Only if absolutely necessary, adding or removing a slug of water (bailer).

The method chosen will depend on project needs, equipment availability, water disposal/ treatment options, pertinent laws and regulations, and operator experience.

The protocols that follow assume that a person can effectively perform one of the above methods for rapidly changing the water level in a well at the start of a slug test and can then use either a manual or automatic procedure for measuring water level response over time.

Considerations

Certain activities should be avoided in slug testing. In general, a person should **not** conduct any type of slug testing in a well if:

- The well contains a pipe, a tube, or an obstruction in a depth range where the water level would change.
- The casing diameter in a well varies in the depth range where the water level would change.
- The water level in a well has not yet recovered to nearly static conditions (e.g., 95 percent or more) after a prior disturbance (e.g., drilling, purging, development, previous well testing, etc.).
- Non-aqueous phase liquid (NAPL) is present in a well.

A ***rising-head*** test should generally **not** be conducted:

- By bailing multiple times, rather than creating an instantaneous water level change.

- By pumping to remove water, unless the amount of water to be removed by the pump can be removed nearly instantaneously and any backflush can be eliminated.
- By using bailers. If bailers must be used, avoid:
  - using a bailer that has a leaky check valve, or
  - using a bailer with a diameter so close to that of the casing that groundwater is suctioned into the well while the bailer is raised.
- If the slug cannot be removed nearly instantaneously (e.g., if removal takes over 5 percent of the 90 percent recovery time).

Falling-head tests are generally **not** recommended due to inherent problems associated with reproducibility, the introduction of fluids, and general application restrictions. They are recommended in circumstances when no other option is available. Consult with the Project Manager or an experienced hydrogeologist before undertaking a falling-head test program. Note: under no circumstances should a falling-head test be performed in a well where the static water level is within the screened section of the well.

Pneumatic slug tests (using pressurized air or nitrogen to effect displacement) do not add noise to the data or disturb equipment in the well like other methods. The amount of displacement can be adjusted prior to beginning the test by varying the application of pressure to the riser. A pneumatic slug test can reduce cross-contamination of wells, reduces equipment contact with water that may be hazardous and allows testing where a traditional slug test may be prohibited. While initial equilibration can take time in a pneumatic test, the displacement event is nearly instantaneous.

Field Documentation

The following data should be obtained prior to heading into the field and/or in the field during slug testing and recorded appropriately (e.g., on Form FMG 5.2-01 - Slug Test Data Report), in a field book, and/or onto an electronic form copied to computer disk):

- Client name.
- Site name.
- Testing company.
- Name of tester.
- Date and time of test.
- Well number.
- Well location.
- Well casing, screen and borehole diameters.
- Well open hole section diameter.
- Total depth of well.
- Any unusual well, weather, or hydrologic features or conditions.

- Top-of-riser distance above ground surface.
- Test procedure used (slug, pneumatic, etc.).
- Transport and disposal methods for any water removed.
- Well drilling method (hollow-stem auger, mud rotary, etc.).
- Decontamination procedures.
- Problems and solutions to problems encountered during testing.
- Static water level.

Other information needed for proper slug-test data interpretation includes:

- Depth interval of screen or open section in well.
- Sandpack porosity (if water levels intersect screen).
- Sandpack diameter (if water levels intersect screen).
- Details of stratigraphic profile including soil/rock types and elevations of contacts.
- Hydraulic conductivity of bounding low hydraulic conductivity units, if present (helpful, but not essential).
- Ground surface elevation.

Testing

The steps for conducting a slug test are as follows. Dataloggers should be used to collect water level measurements if at all possible. Manual measurements should only be used if absolutely necessary but can, and should, be used to collect backup data. The steps for conducting a slug test using automatic water level measurements are as follows:

1. Conduct a review of the Work Plan and the Health and Safety Plan with the project field supervisor, and plan, as needed, for notifications to responsible parties and for site access.
2. Gather equipment needed and inspect for operation.
3. Decontaminate all necessary equipment before entering a site and between each well or as required in the Work Plan or in accordance with FMG 9.0 - Equipment Decontamination, if different.
4. Measure and record the static water level (SWL) in the well to be tested, the depth to bottom, and record whether the bottom is a hard or soft (silty) base. Calculate the depth from the SWL to the top of the well screen.
5. Test the pressure transducer and data logger, and obtain well-bottom and SWL pressures, using the following steps:
    - Place the pressure transducer at least several feet below the top of water as well as below the projected depth of the lowest part of the slug to be used.
    - Make pressure readings until three uniform values are read consecutively.

- Raise the datalogger 1 foot from its original position. View the pressure reading to confirm that the change in position was accurately reported by the transducer. Repeat the procedure, if required, lowering the transducer a greater distance and again confirming the readings.
- Return the transducer to its original position and secure the suspension cable to the well casing. Again, make pressure readings until three uniform values are read consecutively. Compare with the original readings to make sure no drift occurs.

6. Perform the following pre-test activities if a rising-head test is to be performed:
   - Allow the slug that will be used to move slowly down into the groundwater. If possible, fully immerse the slug. If there is not enough water in the well for the slug to be fully immersed, then let the bottom of the slug gently come to rest on the well bottom if a hard base can be confirmed, or in the case of a soft well base, enough above the well bottom to avoid immersion in silt. For bailers, prevent agitation of sediment on the bottom of the well as sediment in the bailer may keep the check valve from properly sealing. Ensure that the slug will not bind with the transducer cable and cause the transducer to move.
   - Measure falling pressures during recovery using the pressure transducer until the water level in the well re-equilibrates to near-static conditions (95 percent recovery).
   - Set the pressure transducer below the base of the immersed slug.
7. Perform the following pre-test activities if a rising-head test is conducted utilizing pneumatic slug testing:
   - Once the depth to water and depth to well screen have been determined, the amount of pressure to use during the pneumatic slug testing can be determined. It is important to not depress the SWL to the top of the screen as this can inadvertently inject air/gas into the formation and cause the hydraulic conductivity value to be much lower than the actual value.
8. Start the slug test by creating a nearly instantaneous displacement in water level:
   - For a ***rising-head*** test:
     - Pull the slug rapidly upwards and either remove it from the well (preferred), or secure/suspend it within the well several feet above the SWL if conditions prohibit removing it (for example, depths to water are significant and manual water level measurements must be collected). When using a bailer ensure, upon retrieving the bailer to the surface, that it is not leaking and contains the appropriate volume of water (full if entirely immersed, etc.).
     - Simultaneously pull slug and initiate the datalogger, beginning the measuring/ recording of rising water levels in the well at the predetermined time frequencies (a logarithmic time scale is usually employed).
     - If a bailer is used, listen for cascading water while the bailer is being raised or is suspended, a sign of check valve failure; if failure occurs, clean and repair the valve and start over.
     - If a bailer is used, measure the volume of water removed by the bailer after retrieval.

- For ***pneumatic rising-head*** tests:
  - Install the well-head assembly.
  - Program the pressure transducer using software specific to the transducer. Ensure the transducer is recording prior to pressurization to capture the SWL.
  - Begin pressurizing the well, a pressure equivalent of 2 to 8 feet is recommended. Allow the pressure in the well to stabilize as determined by the pressure gauge on the well-head assembly.
  - Once equilibrium has been reached, release the ball valve and monitor the rise in water level.
- For ***falling-head*** tests, if employed, prepare the test in the same manner as for the rising-head test, but instead add a solid slug or a known volume of water as opposed to removing a slug or bailer of water.

9. Continue measuring the water levels as they change over time until the water in the well rises or falls to the limit specified in the Work Plan (if not specified then usually 90 percent recovery or 1 hour, whichever comes first -- check with Project Manager to be sure). A preset logarithmic sampling interval, with increasing intervals of time, is ideal, usually predetermined by the datalogger's default setup. Check the datalogger to ensure data were collected.

10. Compare the volume of groundwater recovered in the bailer, if one is used, with the volume of groundwater estimated to have been removed from the well (V) based on the initial recorded water level displacement (H) and borehole radius (r), e.g., $V=H\pi r^2$. If, for a rising-head test, the static water level lies within the screened section of the well, then the sandpack porosity (n) and radius (R) must accounted for also in the volume calculation, e.g., $V=H\pi r^2 + nH\pi(R-r)^2$. A similar comparison can be performed if a slug is used. If the volume recovered and the calculated volume do not reasonably correlate, re-test under site-specific conditions, the test should be performed again.

11. Record all general data in a field book and all pertinent testing data on Form FMG 5.2-01 - Slug Test Data Report.

12. Decontaminate all necessary equipment in accordance with the Work Plan or methods described in FMG 9.0 - Equipment Decontamination.

13. Properly containerize and label spent decontamination fluid or groundwater removed from the well in accordance with the Work Plan or methods described in FMG 10.0 - Waste Characterization.

14. Lock all well caps and secure the site as needed.

15. Submit the slug test data to a qualified scientist or engineer assigned by the Project Manager for interpretation. The data should be interpreted by an experienced hydrogeologist. Calculations should be based on an appropriate model for the known hydrogeologic conditions in the field. Evaluation of slug test data should be performed using an acceptable analytical method; OXY preference is that slug tests be evaluated using either the Bouwer and Rice (1976) or Hvorslev (1951) method.

Any variations from these procedures should first be approved by the project field supervisor and/or Project Manager.

EQUIPMENT/MATERIALS

- A battery-operated water level measurement probe, marked in 0.01-foot increments.
- Form FMG 5.2-01 - Slug Test Data Report.
- Data logger and laptop computer with fully charged battery (if required).
- A solid or sealed slug (or a clean bailer).
- Clean rope or string for raising and lowering a slug.
- Appropriate container for withdrawn groundwater and/or decontamination fluids.
- If snow or soil removal from over a well might be required, a shovel.
- Site-access and well-cap keys, as needed.
- Site maps (including property lines, wells, topography, etc.), as needed.
- If a well to be slug tested is an artesian flowing well, duct tape, couplings, and extra casing of appropriate diameter for increasing casing height so as to enable measurement of a static water level.
- Pressure transducer of appropriate pressure range for the depths of water to be tested, if needed.

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SLUG TEST DATA REPORT

WELL NO.

PROJECT _____ **PROJECT MGR.** _____

LOCATION _____ **FIELD REP.** _____

CLIENT _____ **TEST DATE** _____

Well Type (overburden, bedrock, etc.) _____

Installation Date _____

Displacement method (slug, bailer, pneumatic) _____

Test Section Length, L (ft.) _____

Borehole Diameter (in.) _____

Casing/Riser Diameter (in.) _____

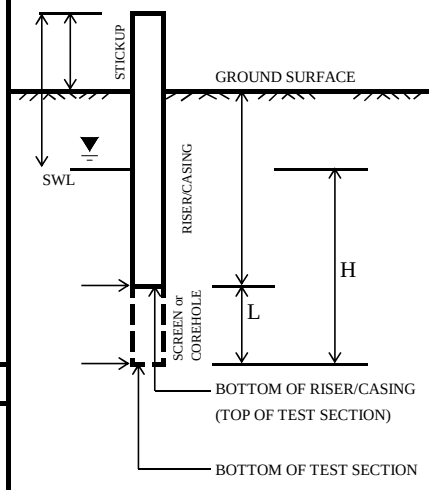
Interval type (screen, open rock, etc.) _____

Saturated Thickness, H (ft.) f _____

Soil Description, depths _____

_____

SKETCH/REMARKS



ALL DIMENSIONS IN FEET - Not To Scale

DEPTH/HEIGHT/ELEVATION:

Stick-Up/Stick-Down (ft.) _____ Static Water Level (ft.) _____

Top Of Test Interval (ft.) _____ Depth to Bottom (ft.) _____

Bottom Of Test Interval (ft.) _____

WATER LEVEL MEASUREMENT DATA

Clock Time	Time (min:sec)	Elapsed Time (min)	Depth To Water From _____ (ft)	Water Elevation (ft)	Comments

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SURFICIAL SOIL SAMPLING

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 6.1
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OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

SURFICIAL SOIL SAMPLING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

The following procedure describes typical surficial soil sample collection methods for submission of samples to a laboratory for chemical analysis. See FMG 2.3 - Soil Borings for subsurface sampling procedures. See FMG 2.1 - Test Pits for test pit soil sampling procedures.

Soil sampling procedures may vary from project to project due to different parameters of concern, different guidance provided by the state/province where the site is located, or the specific objectives for the project. Therefore, it is essential that the sampling team members carefully review the Work Plan requirements and the rationale behind the program. The primary goal of soil sampling is to collect representative samples for examination and chemical analysis (if required). **Any questions regarding whether a sample should be collected or additional samples or different depths should be collected should be directed to the Client while in the field to limit need for remobilization.**

Grab Versus Composite Samples

A grab sample is collected to identify and quantify compounds at a specific location or interval. The sample shall be comprised of no more than the minimum amount of soil necessary to make up the volume of sample dictated by the required sample analyses. Composite samples are a mixture of a given number of sub-samples and are collected to characterize the average chemical composition in a given surface area or vertical horizon.

PROCEDURES REFERENCED

- FMG 2.1 - Test Pits
- FMG 2.2 - Drilling Techniques

- FMG 2.3 - Soil Borings
- FMG 2.6 - Soil Classification
- FMG 6.14 – Incremental Soil Sampling
- FMG 6.15 – PFAS/PFOA Sampling
- FMG 6.10 - Sample Handling and Shipping
- FMG 9.0 - Equipment Decontamination

PROCEDURAL GUIDELINES

1. Sample Strategy -Random, Biased, and Grid-Based Sampling

Random Sampling: Random sampling involves selecting locations for sampling in advance using a randomizing method. Therefore, all locations have an equal chance of being sampled for any sample location. Unless there is a strong indication of contaminant presence, such as staining, then soil sample locations may be randomly selected from several areas within the site.

Biased Sampling: Biased sampling involves preferentially selecting locations for sampling based the parameters of interest. If any areas show evidence of contamination, such as staining or vegetative stress, biased samples are normally collected from each area to characterize the contamination present in each area. Biased sampling may reduce sampling variability and the number of samples required. Background and control samples are also biased, since they are collected in locations typical of non-site-impacted conditions.

Grid-Based Sampling: Grid-based sampling involves collecting samples in a regular (grid) pattern. When soil sampling investigations involve large areas, a grid-based soil sampling program can be used. There is no single grid size that is appropriate for all sites. Common grid sizes are developed on 50-foot and 100-foot centers. It is acceptable to integrate several different grid sizes in a single investigation.

See FMG 6.14-Incremental Soil Sampling for further details on collecting samples under the Incremental Sampling Methodology (ISM).

For surficial soil sampling programs, it is also important to consider the presence of structures and drainage pathways that might affect contaminant migration. It is sometimes desirable to select sampling locations in low lying areas which can retain some surface water flow since these areas could provide samples which are representative of historic site conditions (worst-case scenario if surface water flow was a concern).

2. Sample Interval

Surficial soils are generally considered to be soil between ground surface and 6 to 12 inches below ground surface. However, for risk assessment purposes, regulatory authorities often consider soil from ground surface to 2 feet below ground surface to be surficial soil. The exact interval to be considered as surficial soil is often a matter of discussion with the regulatory authorities that review the Work Plan. The sample interval is important to the manner in which the data are ultimately interpreted. Another important factor is the type of soil. If there are different types of soil present at the site, this may have a bearing on the sample interval. For example, it may be important to separately sample a layer of material with high organic carbon content which overlies a layer of fine grained soil.

3. Sampling Procedure

Soil sampling techniques are dependent upon the sample interval of interest, the type of soil material to be sampled, and the requirements for handling the sample after retrieval. The most common method for collection of surficial soil samples involves the use of a stainless-steel trowel or hand auger. Soil samples may also be collected with spoons and push tubes. The sampling equipment is cleaned between sample locations. A typical surficial soil sampling protocol is outlined below:

- Surficial soil samples will be collected using a pre-cleaned stainless steel trowel or other appropriate tool. Each sample will consist of soil from the surface to the depth specified within the Work Plan.
- A new pair of disposable gloves will be used at each sample location. If sampling for per- and polyfluoroalkyl substances (PFAS)/perfluorooctanoic acid (PFOA), special considerations apply, see FMG 6.15-PFAS/PFOA Sampling for further details.

  - Any surficial debris (i.e., grass cover, gravel) should be removed from the area where the sample is to be collected using a separate pre-cleaned device. Gravel presents difficulties for the laboratory in terms of sample preparation and is typically not representative of contaminant concentrations in nearby soil.
  - A pre-cleaned sampling tool will be used to remove the sample from the layer of exposed soil.
  - When only one sample container is required, the collected soil will be placed directly into the clean, pre-labeled sample jar. When more than one sample container requires filling or samples will be split for duplicate analyses; the soils will first be homogenized in a pre-cleaned stainless steel bowl; and then placed into the respective sample containers. It is important that soil samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. When round bowls are used for sample mixing, mixing is achieved by stirring the material in a circular motion

and occasionally turning the material over. Soil samples collected for volatile organic compounds analyses shall not be mixed.

- Samples will be placed on ice or cooler packs in laboratory supplied shipping coolers after collection.

Exception is noted for the collection of volatile organic compounds (VOCs) which require special sample collection methods. VOCs are collected directly into a sample vial (triplicate volume typically required) or collected using an EnCore Sampler™, or equivalent sampler (triplicate samples collected in accordance with manufacturer's instructions). Some VOC analyses require preservation of the sample immediately upon collection (i.e. methanol). Samples for VOCs are typically collected first, without homogenization or extra handling to limit the loss of volatile constituents. Please note that PFAS/PFOA samples will require special handing and containers, see FMG 6.15-PFAS/PFOA Sampling for further details.

The VOC sample collection methodology will be identified in the Work Plan, which will dictate the sample method. The methodology for VOC sampling varies from area to area, so careful review of this issue in advance of the field efforts is required.

EQUIPMENT/MATERIALS

- Drilling equipment and soil sampling tools
- Decontamination fluids and rinse water
- Subsurface boring log
- Tape measure
- Water level probe

REFERENCES

ASTM D1452-80 - Practice for Soil Investigation and Sampling by Auger Borings.

ASTM D1586-84 - Test Method for Penetration Test and Split-Barrel Sampling of Soils.

ASTM D1587-94 - Practice for Thin Walled Tube Geotechnical Sampling of Soils.

ASTM D2488-93 - Practice for Description and Identification of Soils (Visual-Manual Procedure).

ASTM D4700-91 - Guide for Soil Sampling from the Vadose Zone.

Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

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GROUNDWATER SAMPLING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure is for the collection of groundwater samples for laboratory analysis.

The objective of most groundwater quality monitoring programs is to obtain samples that are representative of existing groundwater conditions, or samples that retain the physical and chemical properties of the groundwater within an aquifer.

One of the most important aspects of groundwater sampling is acquiring samples that are free of suspended silt, sediment, or other fine-grained particulates. Fine-grained particulates may be comprised of naturally occurring inorganic constituents or adsorbed anthropogenic chemicals and may bias the aqueous phase concentration.

Constituents that may adsorb to fine-grained materials suspended in the groundwater include: polychlorinated biphenyls (PCBs), semi-volatile organic compounds (SVOCs), and Non-Aqueous Phase Liquids (LNAPL). Monitoring programs where these constituents are suspected or known to be prevalent must employ sampling methods that minimize the entrainment of fine-grained particulates.

The sampling method of "preference" for OXY sites where fine-grained particulates may be an issue is the "low stress/low flow/very low flow" sampling techniques described within this FMG. Experience has shown that the "low stress/low flow" technique typically achieves representative groundwater samples with minimal fine-grained particulates. In addition to the "low stress/low flow/very low flow" technique, a "typical sample method" has been presented for the collection of constituents less sensitive to particulates presence (i.e., VOCs), or "direct-push sample methods" generally employed as a "pre-screening tool" to evaluate the presence or absence of VOC. Direct-push sample procedures will result in groundwater samples with particulates present. The goal would be to have flow at rates which mimic the natural flow in the aquifer itself.

Lastly, in "extreme" cases "ultra-low flow" techniques have been employed at select sites where "low stress/low flow" methods were used, yet particulate-sensitive constituents continue to bias the analytical results. Ultra-low flow techniques are conducted at purging rates below 100 mL per minute and should only be utilized after careful review and a procedural variance has been approved.

PROCEDURES REFERENCED

- FMG 1.4 - Data Recording - Field Books/Digital Recording
- FMG 5.1 - Water Level Measurements
- FMG 6.15 - PFAS/PFOA Sampling
- FMG 8.0 - Field Instruments - Use/Calibration
- FMG 9.0 - Equipment Decontamination

PROCEDURAL GUIDELINES

The following describes four techniques for groundwater sampling: "Low Stress/Low Flow Methods", "Typical Sample Methods", "Passive Diffusion Bag Sample Methods", and "Direct-Push Methods".

"Low Stress/Low Flow Methods" will be employed to collect representative groundwater samples, and minimize the impact of particulates such as sediment/colloids. Parameters typically affected by particulates present in the sample include PCBs, SVOCs, and inorganic constituents (metals).

The "Typical Sample Methods" will be employed where groundwater samples are collected for the analysis of parameters less sensitive to the presence of particulates such as VOCs and general chemistry.

The "Passive Diffusion Bag (PDB) Sample Methods" are typically employed for the collection of VOCs.

The "Direct-Push Methods" are typically employed for pre-screening areas for chemical presence to aid in determining well placement, or the need for further study.

Note: If non-aqueous phase liquids (NAPL) (light or dense) are detected in a monitoring well, groundwater sample collection will not be conducted, and the Project Manager and Client must be contacted to determine a course of action.
If deemed necessary to sample groundwater from below a LNAPL layer, a suggested sampling procedure has been presented at the end of this Procedural Guidelines section. However, that analysis will always be suspect and results must always be qualified.

Preparatory Requirements

- Verify well identification and location using borehole log details and location layout figures. Note the condition of the well and inform the Project Manager of any required repair work.
- Prior to opening the well cap, measure the breathing space above the well casing with a PID to establish baseline levels. Repeat this measurement once the well cap is opened. If either of these measurements exceeds the air quality criteria in the Health and Safety Plan, field personnel should adjust their PPE accordingly.
- Prior to commencing the groundwater purging/sampling tasks, water level and total well depth measurements must be obtained to determine the volume of water in the well. Refer to FMG 5.1 - Water Level Measurements for details. In some settings, it may be necessary to allow time for the water level to equilibrate. This condition exists if a water tight seal exists at the well cap and the water level has fluctuated above the top of screen; creating a vacuum or pressurized area within the well casing. Three (3) water level checks will verify static water level conditions or changing conditions.
- Calculate the water volume in the well. Typically overburden well volumes consider only the quantity of water standing in the well screen and riser; bedrock well volumes are calculated on the quantity of water within the open corehole and within the overburden casing.

Well Purging and Stabilization Monitoring (Low Stress/Low Flow Method)

Note: The low stress/low flow method described below is the preferred procedure for most OXY Sites. Bladder pumps/submersible variable rate pumps or peristaltic pumps are typically employed.

- Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified by the project requirements. The pump or tubing should be placed in the well as early as possible before sampling is initiated (this is to minimize well disturbance). In some programs, it may be necessary to install the pumping equipment/tubing approximately 24 hours prior to purging. Peristaltic tubing placement should include a tubing "clamp" at the well head, to minimize vibration transfer into the water column. The pump or tubing intake must be at the mid-point of the well screen to prevent disturbance and resuspension of any sediment in the screen base. Bedrock well sampling may require pump/tubing placement at the depth of specific fracture zone areas or other areas identified within the project-specific Work Plan.
- Before starting the pump, measure the water level again with the pump in the well leaving the water level measuring device in the well when completed.
- Purge the well at 100 to a maximum of 500 milliliters per minute (mL/min). During purging, the water level should be monitored approximately every 5 minutes, or as appropriate. A steady flow rate should be maintained that results in drawdown of 0.3 feet or less. The rate of pumping should not exceed the natural flow rate conditions of the well being sampled. Note: Care should be taken to maintain pump suction and to avoid entrainment of air into the tubing. Equipment should be free from per- and polyfluoroalkyl substances (PFAS)/perfluorooctanoic acid (PFOA) if PFAS/PFOA may be present, is being sampled for or may be sampled for in the future. See FMG 6.15-PFAS/PFOA Sampling for special considerations.

- Record adjustments made to the pumping rates and water levels immediately after each adjustment.
- Calibrate field instrument and document calibration activity. Calibration shall be performed in accordance with manufacturer's recommendations and FMG 8.0 - Field Instruments - Use/Calibration.
- During the purging of the well, monitor and record the field indicator parameters (pH, temperature, conductivity, oxidation-reduction (redox) potential (ORP), dissolved oxygen (DO), and turbidity) approximately every 5 minutes. Stabilization is achieved when three (3) consecutive readings for each parameter are within the following limits:
  - pH ±0.1 pH units of the average value of the three readings;
  - temperature ±3 percent of the average value of the three readings;
  - conductivity ±0.005 milliSiemen per centimeter (mS/cm) of the average value of the three readings for conductivity <1 mS/cm and ±0.01 mS/cm of the average value of the three readings for conductivity >1 mS/cm;
  - ORP ±10 millivolts (mV) of the average value of the three readings;
  - DO ±10 percent of the average value of the three readings; and
  - turbidity ±10 percent of the average value of the three readings, or a final value of less than 5 nephelometric turbidity units (NTU).
- Should stabilization not be achieved for all field parameters, purging is continued until a maximum of 8 well screen volumes have been purged from the well. Since low-flow purging (LFP) likely will not draw groundwater from a significant distance above or below the pump intake, the screen volume is based upon a 5-foot (1.4 m) screen length. After purging 8 well screen volumes, purging is continued if the purge water remains visually turbid and appears to be clearing, or if stabilization parameters are varying slightly outside of the stabilization criteria listed above and appear to be approaching stabilization.
- If low-turbidity samples are critical to the project goals, purging will be extended until turbidity has been reduced to 5 NTU or less.
- The pump should not be removed from the well between purging and sampling.
- Once stabilization has been achieved, direct the discharge of the pump tubing to the appropriate sample containers as specified in the sampling order presented below.
  (see Sampling Techniques)

Well Purging and Stabilization Monitoring (Typical Method)

- The use of bailers for well purging is not recommended due to the surging of the groundwater within the well casing and the potential to increase suspended solids. Submersible bladder pumps are preferred but peristaltic pumps can be used for shallow small (>2.0-inch interior diameter) wells. The pump intake/tubing is typically placed at the mid-point of the screen within overburden wells. Bedrock well sampling may require pump/tubing placement in specific fracture zone areas or other areas identified within the project-specific Work Plan.

- Purge the well until three (3) consecutive well volume measurements of temperature and specific conductivity are approximately plus or minus 10 % and if the pH values are within 1 pH unit of the last three (3) value averages, and the groundwater turbidity values are less than the project-specific Work Plan requirements. If stabilization has not occurred after five (5) well volumes have been removed, continue purging and monitoring until eight (8) well volumes have been removed. Purging rates should not exceed the natural flow rate of groundwater into the well if using very low flow sampling. Elevated purging rates may result in excessive drawdown of the water column, introducing sediment/particulates into the sample and allow oxidation of sediments prior to sample collection.
- Groundwater turbidity may be evaluated by a visual examination or use of a nephelometer. Work Plan-specific goals may exist for turbidity values which may require extending the purging or require an alternate purging method.
- Purging and stabilization activities using a bailer should be performed at the top of the water column, within the riser piping/above the well screen. This will minimize the potential for sediment disturbance/suspension in the screen area and move water from the formation into the well screen/riser area in an effort to remove stagnant groundwater within the well. Bottom-loading bailers are generally employed. The lowering and removal actions are performed slowly to minimize well disturbance. Once stabilization has been attained, the sample aliquots are collected directly from the bailer.
- In the event the well goes dry (poor yielding formations), allow sufficient groundwater recharge to occur and perform sample collection.

Passive Diffusion Bag (PDB) Sampling Technique

Passive diffusion bag sampling techniques are used when sampling for VOCs (excluding certain ketones, ethers and alcohols). PDBs are simple to deploy, eliminate the collection and disposal of purged groundwater, and significantly reduce the cost of sampling. Verify the regulatory agency identifies PDB sampling as an accepted form of sampling for VOCs prior to utilizing the sampling technique.

Passive diffusion bags are made of low density polyethylene which acts as a semi-permeable membrane. The PDBs are either unfilled or prefilled by the manufacturer, are cylindrical in shape and come in a variety of sizes. Prefilled PDBs are filled with ASTM Type II certified, laboratory grade, analyte free, deionized water. Passive diffusion bag sampling methods are as follows:

- Hang the PDB sampler from the provided stainless-steel cable, connect the PDB to the top stainless-steel clip of the line then secure the bottom using a zip-tie.
- Lower the PDB into the monitoring well at the well screen interval. The hanging assembly is labeled and pre-sized for correct sampling depth. The stop cap will keep the bag at the desired depth.
- Wait a minimum of 14 days, or until equilibrium has been achieved between the water in the sampler and the surrounding groundwater prior to retrieving the PDB.
- Wind up the cable, release the PDB from the steel clip and cut the zip-tie.

- Cut a notch at the top of the PDB with decontaminated scissors and gently pour the water into the sample bottles.
- Dispose of PDB appropriately.

Direct-Push Sampling Technique

Generally, the direct-push sampling methods are employed for "pre-screening" groundwater quality (typically VOCs) in selected areas. This method is generally used to evaluate the need for permanent monitoring wells or determine the need for further study. The sampling technique is a direct-push protected-screen sampling technique as described in ASTM D6001 (Standard Guide for Direct Push Water Sampling for Geoenvironmental Investigations). The direct-push sampling technique is summarized as follows:

- Advance borehole to the target depth below the groundwater table.
- Remove the drill rod, assemble the direct–push sample tool and attach it to the drill rod.
- Lower the sample device to the bottom of the borehole using the drill rod.
- Advance the sample device approximately 3 feet into the bottom of the borehole by hydraulically pushing the drill rod.
- Withdraw the drill rods approximately 1 to 2 feet to retract the screen sleeve and to expose the sampler screen to the formation.
- Alternatively, a number of direct-push tools exist that do not require an advance borehole and can be driven directly to the target depth and retracted for sample collection.
- Allow at least 15 minutes from exposing the sampler screen to sample collection to allow silt in the sampler to settle. In tight formations, a longer wait time may be required to allow sufficient groundwater to enter the screen. In some clays, the sample device may not collect sufficient water volume to obtain a sample.
- Lower a small bailer into the sampler, discard initial bail (to acclimate bailer), and collect a water sample. A few bailer volumes may be required to obtain a sufficient volume of water sample. Alternatively, a "Waterra" check ball affixed to tubing maybe employed to collect a groundwater sample, or a peristaltic pump.
- Remove and clean the sampler device after completion of sample collection. Decontaminate sampler for next sample event.

This sampling technique is prone to sediment presence due to the lack of a well screen and sand pack and the limited purging performed before sample collection. A project variance will be required if non-VOC constituents are collected for analysis and results should be qualified on tables as to collection method.

Sampling Techniques

- If an alternate pump is utilized (i.e., Typical Method), the first pump discharge volumes (or bailer volumes) should be discarded to allow the equipment a period of acclimation to the groundwater.
- Samples are typically collected directly from the pump with the groundwater sample discharged into the appropriate sample container. Avoid handling the interior of the bottle or bottle cap and don new gloves for each well sampled to avoid cross-contamination of the sample.
- Order of sample collection:
  - VOCs;
  - SVOCs and PCBs;
  - Total organic carbon (TOC);
  - Total organic halogens (TOX);
  - Extractable organics;
  - Total metals;
  - Dissolved metals;
  - Phenols;
  - Cyanide;
  - Sulfate and chloride;
  - Nitrate and ammonia; and
  - Radionuclides.
- For low stress/low flow sampling, samples should be collected at a flow rate between 100 and 250 mL/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 feet.
- For VOC sample containers, the pumping rate should not exceed 100 mL/min. Samples should be transferred directly to the final container 40 mL glass vials completely full and topped with a Teflon cap (if not sampling for PFAS/PFOA). (NOTE: DO NOT OVERFILL AND DISPLACE SAMPLE PRESERVATIVE) Once capped the vial must be inverted and tapped to check for headspace/air presence (bubbles). If air is present the sample vial will be discarded, and re-collected until free of air.
- Field filtration will be performed if required by the project-specific Work Plan. Sediment presence can interfere or bias sample results; false positive findings have been observed when turbid samples are analyzed. Field filtration can eliminate this concern; generally applicable to inorganic/PCB analysis. In-line disposable filter cartridges are generally the easiest and quickest method for field filtration.
- Sample labels/sample identification. All samples must be labeled in accordance to GHD's Laboratory Program including:
  - A unique sample number;
  - Date and time;

- Parameters to be analyzed;
- Project Reference ID; and
- Sampler's initials.

- Labels should be secured to the bottle(s) and should be written in indelible inks or preprinted.
- Field laboratory analysis can be conducted in the field using kits (i.e. HACH Kits or similar). Field analysis can also include alkalinity, chloride, total hardness, iron, etc. The manufacturer's instructions must be followed to ensure the correct result is obtained. Data determined from the field analysis will be recorded on the appropriate field forms (see FMG 6.9 - Field Quality Control Samples appropriate forms).

Groundwater Sampling Techniques Below LNAPL Layers

Sampling and analysis of groundwater below a LNAPL layer is not performed at OXY Sites. The rationale for avoiding groundwater analysis below a LNAPL layer is as follows:

- The potential for sample "cross-contamination" with a trace amount of NAPL is very probable; analytical data will be biased "high" based upon this concern and the method and conditions should be noted on any results collected using this technique.
- Analytical data generated from this scenario does not represent "dissolved" constituent presence in groundwater. Dissolved constituents are "best" determined in downgradient locations.

In some instances, it may be required to perform groundwater sampling below a LNAPL layer, possibly at the request of a regulatory group. This should not be done without the prior approval of the Client. If absolutely necessary, this type of sampling may be accomplished in accordance with the following procedure:

- Determine the LNAPL depth and thickness using an interface probe or clear bottom loading bailer.
- Determine the sampling depth, selecting a sample point as far away as possible from the LNAPL interface.
- Using a "capped" outer tube or piping (i.e., 1-inch diameter polyethylene), insert the outer tube to the selected sample interval. The cap should be a slip-on cap affixed to the outer tube using a short "leash" (i.e., stainless steel wire or equivalent). This allows cap recovery once the sampling is complete.
- Insert the sample line (3/8-inch diameter tubing) into the outer tube and "push out" the end cap for sample line entry into the sampling interval.
- Perform purging and sampling using a peristaltic pump.
- Monitor the groundwater level and/or the NAPL level to ensure the LNAPL layer is not drawn to sampling depth. If LNAPL drawdown occurs evaluate the need to proceed further and consider terminating sampling activity.

- This sample should not be referred to on any analysis as a groundwater sample. It should always be referred to as a groundwater/NAPL mixture (GW/NAPL designation).

Sampling Techniques for Per- and Polyfluoroalkyl Substances (PFAS)/Perfluorooctanoic acid (PFOA) by LC/MS/MS

Sampling for PFAS/PFOA is becoming more common. When sampling for PFAS/PFOA, caution must be taken to avoid cross contamination and false positives. Prior to sampling PFAS/PFOA, contact the project laboratory to define a PFAS/PFOA target list. It is recommended to collect additional field/equipment blanks prior to and during sampling to check for residual PFAS/PFOA on sampling equipment due to the potential for cross-contamination issues and the need for very low reporting limits. PFAS/PFOA sampling methods are as follows:

- Using new nitrile gloves, sample for PFAS/PFOA first prior to collecting samples for any other parameter.
- Do not place the bottle cap on any other surface when collecting the sample.
- Avoid all contact with the inside of the sample bottle or its cap.
- After the sample has been collected and capped, place the sample bottle(s) in an individual sealed plastic bag (Ziploc) separate from all other sample parameter bottles.
- Make sure all equipment and sampling containers do not contain potential PFAS containing materials, such as Teflon. Samplers should ensure to the extent possible that PPE and any lotions/etc. do not contain PFAS/PFOA.

Due to the very low reporting levels of PFAS/PFOA, care must be taken during sample collection. The following table summarizes the do's and don'ts of sampling for PFAS/PFOA:

Do Not Use Items	**Do Use Items**
Field Equipment Items	
No Teflon containing materials including Teflon lined bottle caps and bailers	High-density polyethylene (HDPE) and Low-density polyethylene (LDPE)
	Acetate liners for soil samples
No Teflon tubing	Silicon tubing
No waterproof field books	Loose paper (non-waterproof)
No plastic clipboards, binders, or spiral hardcover notebooks	Aluminum field clipboards or with Masonite
No Post-It Notes	Ball-point pens
No chemical (blue) ice packs	Regular ice
Field Clothing and PPE Items	
No new clothing or water resistant, waterproof, or stain-treated clothing, clothing containing Gore-Tex	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of synthetic or natural fibers (preferable cotton)
No clothing laundered using fabric softener	No fabric softener

Do Not Use Items	Do Use Items
No boots containing Gore-Tex	Boots made with polyurethane and polyvinyl chloride (PVC)
No Tyvek	Cotton Clothing
No cosmetics, moisturizers, hand cream, or other related products on the morning of sampling.	Sunscreens – All Organic Natural Sunscreen, that are "free" or "natural" Check the label
	Insect repellents- various natural products, DEET, but check the label prior to use
Sample Container Items	
No LDPE or glass containers	HDPE or polypropylene
No Teflon -lined caps	Lined or unlined HDPE or polypropylene caps
Rain Gear Items	
No waterproof or resistant rain gear	Tent that is only touched or moved prior to & following sampling activities
Equipment Decontamination Items	
No Decon 90	Alconox and/or Liquinox
No water from an on-site well	Potable water from municipal drinking water supply
Food Items	
No food and drink, with exceptions noted on the right	Bottled water and hydration drinks (Gatorade and Powerade) to be brought and consumed only in the staging area

EQUIPMENT/MATERIALS

- pH meter, conductivity meter, nephlometer, ORP meter, DO meter, temperature gauge.
- Field filtration units (if required).
- Purging/sampling equipment:
  - Peristaltic pump (not suitable for VOCs[1]/SVOCs, or drawing water from depths greater than 25 feet[2]);
  - Suction pumps (not suitable for LFP, VOCs/SVOCs, or depths greater than 25 feet);
  - Submersible pumps (suitable for VOCs/SVOCs only at low flow rates);
  - Air lift pumps (not suitable for VOCs/SVOCs);
  - Bladder pumps (suitable for LFR and VOCs/SVOCs);
  - Inertia pumps (gaining acceptability for VOCs/SVOCs, generally not suited for OXY programs); and
  - Bailers.
- Water level probe.
- Sampling materials (containers, log book/forms, coolers, chain-of-custody).
- Project Work Plan.

- Health and Safety Plan.

Note[1]: Peristaltic pump use for VOC collection is acceptable on select EPA/RCRA sites; this technique has gained acceptance in select areas. Where it is permissible to collect VOCs using a peristaltic pump, collection must be performed at a low flow rate (Michigan allows VOC sampling with the peristaltic pump).
Acceptability of the collection of VOCs using the peristaltic pump should be evaluated before the sampling program commences, commonly performed during the project Work Plan development and approval process.
Note[2]: Exception is noted in locations that the suction line can be placed at the desired sample depth (i.e., 100 feet), and the natural recharge maintains a water level within 25 feet of the ground surface.

Field Notes

Field notes must document field activities and measurements collected during the sampling activities. FMG 1.4 - Data Recording - Field Books/Digital Recording describes the data/recording procedure for field activities. The log book/field file should document the following for each well sampled:

- Identification of well.
- PID readings before and after well opening (if required).
- Well depth.
- Static water level depth and measurement technique.
- Sounded well depth.
- Presence of immiscible layers and detection/collection method.
- Well yield – high or low.
- Purge volume, pumping rate, and final disposition.
- Time well purged.
- Measured field parameters and meter calibration records.
- Purge/sampling device used.
- Well sampling sequence.
- Sample appearance.
- Sample odors.
- Sample volume.
- Types of sample containers and sample identification.
- Preservative(s) used.
- Parameters requested for analysis.

- Field analysis data and method(s).
- Sample distribution and transporter.
- Analytical laboratory.
- Chain-of-custody number for shipment to laboratory.
- Field observations on sampling event.
- Name(s) of sampling personnel.
- Climatic conditions including air temperature.
- Problems encountered, and any deviations made from the established sampling protocol.

A standard log form for documentation and reporting groundwater purging and sampling events are presented on Form FMG 6.4-01 - Well Purging Field Information, Form FMG 6.4-02 - Sample Collection Data Sheet, and Form FMG 6.4-03 - Monitoring Well Record for Low-Flow Purging.

Groundwater/Decontamination Fluid Disposal

The project Work Plan will identify the required disposal procedures for groundwater and decontamination fluids. Groundwater disposal methods will vary on a case-by-case basis but may range from:

- Off-site treatment at private treatment/disposal facilities or public owned treatment facilities.
- On-site treatment at Facility-operated facilities.
- Direct discharge to the surrounding ground surface, allowing groundwater infiltration to the underlying subsurface regime (if State allows).
- Direct discharge to impervious pavement surfaces, allowing evaporation to occur.

Decontamination fluids should be segregated and collected separately from wash waters/groundwater containers. Often small volumes of solvents used during the day can be allowed to evaporate if left in an open pail. In the event evaporation is not possible or practical, off-site disposal arrangements must be made with the Facility Resources Manager and the Client.

REFERENCES

ALS, Passive Diffusion Bags (PDBs), http://www.alsglobal.com/us

ASTM D5474 - Guide for Selection of Data Elements for Groundwater Investigations.

ASTM D4696 - Guide for Pore-Liquid Sampling from the Vadose Zone.

ASTM D5979 - Guide for Conceptualization and Characterization of Groundwater Systems.

ASTM D5903 - Guide for Planning and Preparing for a Groundwater Sampling Event.

ASTM D4448 - Standard Guide for Sampling Groundwater Wells.

ASTM D6001 - Standard Guide for Direct Push Water Sampling for Geo-Environmental Investigations.

SGS Sampling, Shipping & Handling or Per and Polyfluorinated Alkyl Substances (PFAS) By LC/MS/MS Fact Sheet – 2017.

USEPA Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (EPA/540/S -95/504).

USEPA RCRA Groundwater Monitoring: Draft Technical Guidance (EPA/530-R-93-001).

WELL PURGING FIELD INFORMATION FORM

JOB# _ _ _ _ _ - _ _

SITE/PROJECT NAME: ____________________

WELL# _ _ _ _ _ _ _

WELL PURGING INFORMATION

PURGE DATE (MM DD YY)	SAMPLE DATE (MM DD YY)	WATER VOL. IN CASING (LITRES/GALLONS)	ACTUAL VOLUME PURGED (LITRES/GALLONS)

PURGING AND SAMPLING EQUIPMENT

PURGING EQUIPMENT.........DEDICATED Y N (CIRCLE ONE)

SAMPLING EQIPMENT.........DEDICATED Y N (CIRCLE ONE)

PURGING DEVICE ☐ A - SUBMERSIBLE PUMP D - GAS LIFT PUMP G - BAILER X- ____________ PURGING OTHER (SPECIFY)

B - PERISTALTIC PUMP E - PURGE PUMP H - WATERRA®

SAMPLING DEVICE ☐ C - BLADDER PUMP F - DIPPER BOTTLE X- ____________ SAMPLING OTHER (SPECIFY)

PURGING DEVICE ☐ A - TEFLON D - PVC X- ____________ PURGING OTHER (SPECIFY)

B - STAINLESS STEEL E - POLYETHYLENE

SAMPLING DEVICE ☐ C - POLYPROPYLENE X- ____________ SAMPLING OTHER (SPECIFY)

PURGING DEVICE ☐ A - TEFLON D - POLYPROPYLENE F - SILICONE X- ____________ PURGING OTHER (SPECIFY)

B - TYGON E - POLYETHYLENE G - COMBINATION TEFLON/POLYPROPYLENE

SAMPLING DEVICE ☐ C - ROPE X- ____________ (SPECIFY) X- ____________ SAMPLING OTHER (SPECIFY)

FILTERING DEVICES 0.45 ☐ A - IN-LINE DISPOSABLE B - PRESSURE C - VACUUM

FIELD MEASUREMENTS

WELL ELEVATION ________ (m/ft) GROUNDWATER ELEVATION ________ (m/ft)

DEPTH TO WATER ________ (m/ft) WELL DEPTH ________ (m/ft)

pH	TURBIDITY	CONDUCTIVITY	ORP	DO	SAMPLE TEMPERATURE
(std)	(ntu)	(µm/cm) AT 25°C	(mV)	(mg/L)	(°C)
(std)	(ntu)	(µm/cm) AT 25°C	(mV)	(mg/L)	(°C)
(std)	(ntu)	(µm/cm) AT 25°C	(mV)	(mg/L)	(°C)
(std)	(ntu)	(µm/cm) AT 25°C	(mV)	(mg/L)	(°C)
(std)	(ntu)	(µm/cm) AT 25°C	(mV)	(mg/L)	(°C)

FIELD COMMENTS

SAMPLE APPEARANCE: ________ ODOR: ________ COLOR: ________ TURBIDITY: ________

WEATHER CONDITIONS: WIND SPEED ________ DIRECTION ________ PRECIPITATION Y/N OUTLOOK ________

SPECIFIC COMMENTS ____________________

I CERTIFY THAT SAMPLING PROCEDURES WERE IN ACCORDANCE WITH APPLICABLE OXY PROTOCOLS

DATE PRINT SIGNATURE

FMG MODIFICATIONS MUST BE ACCOMPANIED BY A REVISION REQUEST FORM APPROVED BY THE PROJECT MANAGER

SAMPLE COLLECTION DATA SHEET - GROUNDWATER SAMPLING PROGRAM

PROJECT NAME _____ PROJECT NO. _____

SAMPLING CREW MEMBERS _____ SUPERVISOR _____

DATE OF SAMPLE COLLECTION _____

[Note: For 2" dia. well, 1 ft. = 0.14 gal (imp) or 0.16 gal (us)]

Sample I.D. Number	Well No.	Measuring Point Elev. (ft. AMSL)	Bottom Depth (ft. btoc)	Water Depth (ft. btoc)	Water Elevation (ft. AMSL)	Well Volume (gallons)	Bailer Volume No. Bails	Volume Purged (gallons)	Field pH	Field Temp.	Field Cond.	Time	Sample Description & Analysis

Additional Comments: _____

Copies to: _____

FMG MODIFICATIONS MUST BE ACCOMPANIED BY A REVISION REQUEST FORM APPROVED BY THE PROJECT MANAGER

MONITORING WELL RECORD FOR LOW-FLOW PURGING

Project Data:

Project Name: _____ Date: _____

Ref. No.: _____ Personnel: _____

Monitoring Well Data:

Well No.: _____ Screen Length (ft): _____

Measurement Point: _____ Depth to Pump Intake (ft)[(1)]: _____

Constructed Well Depth (ft): _____ Well Diameter, D (in): _____

Measured Well Depth (ft): _____ Well Screen Volume, V_S (mL)[(2)]: _____

Depth of Sediment (ft): _____ Initial Depth to Water (ft): _____

Time	Pumping Rate (mL/min)	Depth to Water (ft)	Drawdown from Initial Water Level[(3)] (ft)	pH	Temperature °C	Conductivity (mS/cm)	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Volume Purged, V_p (mL)	No. of Well Screen Volumes Purged[(4)]

Notes:

(1) The pump intake will be placed at the well screen mid-point or at a minimum of 2 ft above any sediment accumulated at the well bottom.

(2) The well screen volume will be based on a 5-foot screen length, $V_S=p*(D/2)^2*(5*12)*(2.54)^3$

(3) The drawdown from the initial water level should not exceed 0.3 ft.

(4) Purging will continue until stabilization is achieved or until 20 well screen volumes have been purged (unless purge water remains visually turbid and appears to be clearing, or unless stabilization parameters are varying slightly outside of the stablization criteria and appear to be stablizing), No. of Well Screen Volumes Purged= Vp/Vs.

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NON-AQUEOUS PHASE LIQUID (NAPL)

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OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

NON-AQUEOUS PHASE LIQUID (NAPL)

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure is for monitoring the presence of dense and light non-aqueous phase liquids (DNAPL and LNAPL), and collection of NAPL samples for laboratory analysis in monitoring, observation, and extraction wells.

It should be noted that groundwater sampling and analysis should not be performed in locations where NAPL has been identified.

PROCEDURES REFERENCED

- FMG 5.1 - Water Level Measurements
- FMG 9.0 - Equipment Decontamination

PROCEDURAL GUIDELINES

- Conduct well identification, inspection, and opening in accordance with FMG 5.1 - Water Level Measurements.
- NAPL level measurements are best conducted using a dual phase interface probe. The interface probe uses an optical liquid sensor, in conjunction with an electric circuit to detect the top of a phase-separated liquid and the interface between the phase layer and water (water level). The procedure for use of this probe is:
- For LNAPL:
  - Lower the probe tip into the center of the well until discontinuous beeping is heard (this indicates the top of the LNAPL has been detected). Grasp the calibrated tape at the reference point and note reading. Confirm the reading by slowly raising and lowering the probe to the level of the phase layer.

- Once the top of the phase layer is confirmed, slowly lower the probe until a continuous sound is heard. This indicates that the water level has been encountered. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
- Decontaminate the submerged end of the tape and probe prior to the next use in accordance with the Work Plan requirements.

- For DNAPL:
  - Lower the probe tip in the center of the well to the bottom of the well, a discontinuous beeping will be heard if DNAPL is present. Grasp the calibrated tape at the reference point and note reading.
  - Once the bottom of the well is confirmed, slowly raise the probe until a continuous sound is heard. This indicates that the water level has been encountered and represents the top of the DNAPL layer. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
  - Decontaminate the submerged end of the tape and probe prior to the next use and collect a decontamination blank following each cleaning.
- Alternative NAPL measurement methods exist in the event an interface probe is unavailable or not functioning properly. These methods tend to be less accurate than the interface probe but may be used to establish an estimated NAPL measurement.
  - **Clear Bailer** – A clear bottom-loading bailer may be used to estimate NAPL thickness if floating or denser than water. If NAPL presence is suspected, the bailer is carefully lowered to the location of suspected NAPL presence (top of water column/base of water column), and slowly removed and examined for NAPL. If present, the column of NAPL within the clear bailer can be measured to estimate the NAPL thickness within the groundwater column.
  - **Weighted Cord** – Primarily used for DNAPL measurements, a weighted "cotton" string or cord may be lowered to the base of the well and inspected upon retrieval. Typically, the lower DNAPL layer will "coat" the string indicating the approximate thickness of this layer.

<u>Well NAPL Sampling</u>

- Prior to sampling, the level of NAPL in the well should be measured as identified above.
- Various sampling devices can be employed to acquire fluid samples from the top and bottom of the well, including the following:
  - Bottom-loading bailer;
  - Double check value bailer (produces most reliable results);
  - Peristaltic pump for shallow wells (<25 feet in depth); or
  - Inertia pump for deeper wells (up to 300 feet in depth).
- Transfer NAPL to sample containers for shipment to laboratory. NAPL can be sampled to evaluate the physical properties of the fluid or to evaluate chemical composition.

- Decontaminate equipment prior to next use.

Note: Groundwater sampling shall not be performed in locations where NAPL is present.

EQUIPMENT/MATERIAL

- Interface probe.
- Bottom-loading bailer.
- Double check valve bailer.
- Peristaltic pump.
- Inertia pump.
- Work Plan.
- Health and Safety Plan.

REFERENCES

Cohen, Robert M., Mercer, James W. (GeoTrans, Inc.), Robert S. Kerr Environmental Research Laboratory "DNAPL Site Evaluation" Office Research and Development. U.S. Environmental Protection Agency

Cohen, R.M., Brayda, A.P., Shaw, S.T., and Spaulding, C.P.; Fall 1992 "Evaluation of Visual Methods to Detect NAPL in Soil and Water", Groundwater Monitoring Review, Volume 12 No. 4, pp. 132-141.

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SAMPLE HANDLING AND SHIPPING

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SAMPLE HANDLING AND SHIPPING

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

Sample management is the continuous care given to each sample from the point of collection to receipt at the analytical laboratory. Good sample management ensures that samples are properly recorded, properly labeled, not lost, broken, or exposed to conditions which may affect the sample's integrity and that the integrity of the sample can be defended even in court proceedings by the sampling team and the documentation.

All sample submissions must be accompanied with a chain-of-custody (COC) document to record sample collection and submission. When possible, sampling should be batched to prevent completing validation for a small set of samples.

The following sections provide the minimum standards for sample management.

PROCEDURAL GUIDELINES

Field Handling

Prior to entering the field area where sampling is to be conducted, especially at sites with defined exclusion zones, the sampler should ensure that all materials necessary to complete the sampling are on hand.

If samples must be maintained at a specified temperature after collection, proper coolers and ice/cool-packs must be brought out to the field. Consideration should be given to keeping reserve cooling media on hand if sampling events will be of long duration. Conversely, when sampling in extremely cold weather, proper protection of water samples, trip blanks, and field blanks must be considered.

Personnel performing groundwater sampling tasks must check the sample preparation and preservation requirements to ensure compliance with the Work Plan Quality Assurance Project Plan (QAPP). Typical sample preparation may involve pH adjustment (i.e., preservation), sample filtration and preservation, or simply cooling to 4°C. Sample preparation requirements vary from site to site and vary depending upon the analytical method for which the samples will be analyzed.

The sampling personnel must also confirm before the sample event, the amount of bottle filling required for the respective sample containers. Groundwater samples analyzed for volatile organic compounds (VOCs) must not have any headspace within the sample collection vial; whereas when collecting select analytes (i.e., metals) a headspace must be provided to allow addition of the required preservative.

Sample Labeling

Samples must be properly labeled as soon as practical after collection. Note that markers that generate VOCs (i.e. Sharpie® markers) should not be used to write on labels as they can create false positive VOC results in the sample.

Note that the data shown on the sample label is the minimum data required. The sample label data requirements are listed below for clarity.

i) Project name.
ii) Sample number.
iii) Sampler's initials.
iv) Date of sample collection.
v) Time of sample collection.
vi) Analysis required.
vii) Preservatives.

The Work Plan Quality Assurance/Quality Control (QA/QC) specification should be reviewed to determine any additional requirements.

Quite often the analytical laboratory supplying the containers will provide blank sample labels. If these are adequate and convenient they can be used.

Under certain field conditions it is impractical to complete and attach sample labels to the container at the point of sample collection. However, to ensure that samples are not confused, a clear notation should be made on the container with a permanent, non-VOC marker indicating the last three digits of the sample number. If the containers are too soiled or small for marking, the container can be put into a zip-lock bag which can then be labeled.

No one sample number format is adequate for every type of sampling activity. Prior to the start of every project or sub-sampling event within the project, Project Managers and field personnel

should devise a sample number format. Sample number formats should be as simple and short as possible. Simple number formats will reduce transcription errors by both Consultants and lab personnel. The sample number format should be comprehensive enough to allow for easy location of detailed sample data within the Site log books. Sample format must also be consistent with any future data management activities. OXY is migrating to digital recording to minimize transcription errors and reduce management costs.

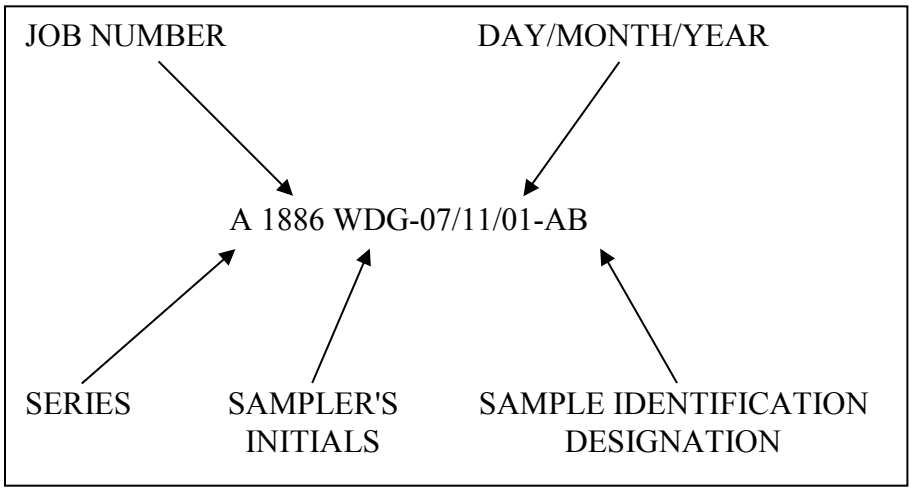
Sample Labels/Sample Identification

All samples must be labeled with:

- A unique sample number (never to be re-used, nor likely to be).
- Date and time.
- Parameters to be analyzed.
- Job number.
- Sampler's initials.

Labels should be secured to the sample container (bottle, Summa® canister, etc.) and should be written in indelible, non-VOC inks. It is also desirable to place wide clear tape over the label before packing in a cooler for label protection during transportation.

The unique sample identification number may follow the format recommended below, or a specific sample protocol for labeling may be specified in the project Work Plan.



This format has been selected to maximize the information content of the sample number. Minor modifications are certainly reasonable.

i) Series is a letter, which designates a group of samples. This might include sample round, or might designate sample type (e.g., sediment, soil, volatile analysis, Round 2 Lower Aquifer wells), or sample source. For example, "A" might mean samples of influent to

some treatment system, "B" might mean samples of effluent. Letters should be used, not numbers. Series is optional.

ii) Job number together with the series number will allow easier tracking of samples.

iii) Sampler's initials will allow identification of the sampler, and so allow all project personnel to contact the correct person for information regarding that sample and its collection. The use of three initials is requested. Special arrangements will need to be made if two individuals have the same initials.

iv) Sample date will allow monitoring of actual holding time of samples and should ensure that all sample numbers are unique, even if sample location designation is used in a system, as opposed to assigned at random.

v) Sample identification designation will identify the sample and can be any numerical or letter designation.

The decision of how to assign sample numbers should be made at the beginning of a job or phase and should be consistent throughout the job.

Packaging

When possible, sample container preparation and packing for shipment should be completed in a well organized and clean area, free of any potential cross-contaminants.

Sample containers should be prepared for shipment as follows:

i) Containers should be wiped clean of all debris/water using paper towels (paper towels must be disposed of with other contaminated materials).

ii) Clear, wide packing tape should be placed over the sample label for protection.

While there is no one "best" way to pack samples for shipment, the following packing guidelines should be followed.

i) Plan time to pack your samples (and make delivery to shipper if applicable). Proper packing and manifesting takes time. A day's worth of sampling can be easily wasted due to a few minutes of neglect when packing the samples.

ii) Always opt for more coolers and more padding rather than crowd samples. The cost associated with the packing and shipment of additional coolers is usually always small in comparison with the cost of having to re-sample due to breakage during shipment. Make sure though to minimize the number of COCs and batch samples where possible to reduce the laboratory cost and validation costs that are incurred with each set of samples.

iii) Do not bulk pack. Each sample must be individually padded.

iv) Large glass containers (1 L and up) require much more space between containers.

v) Ice is not a packing material due to the reduction in volume when it melts.

The following is a list of standard guidelines which must be followed when packing samples for shipment.

i) When using ice for a cooling media, always double bag the ice in zip-lock bags.

ii) Double-check to ensure trip and temperature blanks have been included for all shipments containing VOCs, or where otherwise specified in the QAPP.

iii) Enclose the COC form in a zip-lock bag and place copies in each cooler.

iv) Ensure custody seals (two, minimum) are placed on each cooler. Coolers with hinged lids should have both seals placed on the opening edge of the lid. Coolers with "free" lids should have seals placed on opposite diagonal corners of the lid. Place clear tape over custody seals.

v) Ensure that all "Hazardous Material" stickers/markings have been removed from coolers being used which previously contained such materials.

vi) Ensure all proper containers/shipping labels required for the sample shipment are used/adhered to the sample packaging

Note: Never store sterile sample containers in enclosures containing equipment which use any form of fuel or volatile petroleum-based product. An alternate means of secure storage must be planned for.

When conducting sampling in freezing conditions at sites without a heated storage area (free of potential cross contaminants), trip blanks and temperature blanks not being used in a QA/QC role should be isolated from coolers immediately after receipt. Trip and temperature blanks should be double-bagged and kept from freezing.

Chain-of-Custody

COC forms will be completed for all samples collected. The form documents the transfer of sample containers. OXY is in the process of migrating to digital COCs.

The COC record, completed at the time of sampling, will contain, but not be limited to, the sample number, date and time of sampling, and the name of the sampler. The COC document will be signed and dated by the sampler when transferring the samples.

Each sample cooler being shipped to the laboratory will contain a COC form. The COC form will consist of four copies which will be distributed as follows: The shipper will maintain a copy while the other three copies will be enclosed in a waterproof envelop within the cooler with the samples. The cooler will then be sealed properly for shipment. If one COC is used and there are multiple coolers, copies of the COC should be placed in all coolers. The number of coolers must be written on the COC. Make sure the laboratory knows when there are multiple coolers it is still one batch. The laboratory, upon receiving the samples, will complete the three remaining copies. The laboratory will maintain one copy for their records. One copy will be returned to the Field QA/QC Officer upon receipt of the samples by the laboratory. One copy will be returned with the data deliverables package.

COC records are legal documents and may be evidence in court. They must be completed and handled accordingly.

The following list provides guidance for the completion and handling of all COCs.

i) COCs used should be Consultant standard forms or those supplied by the analytical laboratory. Do not use any COC forms from other labs, even if the heading is blocked out.

ii) COCs must be completed in black ball-point ink only.

iii) COCs must be completed neatly using printed text.

iv) If a simple mistake is made, line out the error with a single line and initial and date next to it.

v) Each separate sample entry must be sequentially numbered.

vi) The use of "Ditto" or quotation marks to indicate repetitive information in columnar entries should be avoided. If numerous repetitive entries must be made in the same column, place a continuous vertical arrow between the first entry and the next different entry.

vii) When more than one COC form is used for a single shipment, each form must be consecutively numbered using the "Page ___ of ___" format. Try to batch as much as possible.

viii) If necessary, place additional instructions directly onto the COC. Do not enclose separate loose instructions.

ix) Include a contact name and phone number on the COC in case there is a problem with the shipment.

x) Before using an acronym on a COC, define clearly the full interpretation of your designation [i.e., Polychlorinated Biphenyls (PCBs)].

Shipment

In all but a few cases, the QA/QC plan for the field work will require shipment of samples by overnight carrier. When possible, samples may be held to ship a batch of samples together by overnight carrier. Samples must be kept at proper temperatures and received at the laboratory with adequate holding times remaining. Issues can be avoided by planning in advance and discussing with the laboratory when holding samples in the field.

Prior to the start of the field sampling, the carrier should be contacted to determine if pickup can be made at the field site location. If pickup at the field site can be made, the "no-later-than" time for having the shipment ready must be determined.

If no pickup is available at the site, the nearest pickup or drop-off location should be determined. Again, the "no-later-than" time for each location should be determined.

Sufficient time must be allowed not only for packaging but also for delivery of samples if this becomes necessary. Driving at high rates of speed in order to make the drop time is unacceptable.

Sample shipments must not be left at unsecured or questionable drop locations (i.e., if the cooler will not fit in a remote drop box do not leave the cooler unattended next to the drop box).

Some overnight carriers do not in fact provide "overnight" shipment to/from some locations. Do not assume; call the carrier in advance before the start of the field work. If overnight shipment is provided, make sure that the correct overnight delivery timeframe is selected. If the samples are collected and to be shipped on a Friday, ensure that the lab will have someone working that Saturday to accept the shipment. All transfers of sample control should be documented on the COC.

Copies of all shipment manifests must be maintained in the field file.

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FIELD INSTRUMENTS – USE/CALIBRATION

LIST OF FORMS
(Following Text)

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FIELD INSTRUMENTS – USE/CALIBRATION

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

A significant number of field activities involve usage of electronic instruments to monitor for environmental screening and health and safety purposes. It is imperative the instruments are used and maintained properly to optimize their performance and minimize the potential for inaccuracies in the data obtained, and to insure worker's health and safety is not compromised. The equipment should also be evaluated for potential per- and polyfluroalkyl substances (PFAS)/perfluorooctanoic acid (PFOA) presence if there is a potential for cross contaminating analytical samples.

This FMG provides guidance on the usage, maintenance and calibration of electronic field equipment, whether for equipment owned by the Consultant or Contractor, or equipment obtained from a rental agency.

PROCEDURES REFERENCED

- FMG 1.4 - Data Recording – Field Books/Digital Recording

PROCEDURAL GUIDELINES

- All monitoring equipment will be in proper working order and operated for the purpose for which it was intended, in accordance manufacturer's recommendations before bringing it to the field or using it in the field.
- Field personnel will be responsible for ensuring the equipment is maintained and calibrated in the field to extent practical or returned for office or manufacturer maintenance or calibration if warranted. Calibration is discussed in greater detail below.

- A copy of the Operating Instructions, Maintenance and Service Manual for the equipment being used during a task will be kept with the equipment on-site until the task has been completed and the equipment is no longer on-site.
- Instruments will be operated only by personnel trained in the proper usage and calibration. In the event certification of training is required, personnel will have documentation of such certification with them on site at all times.
- Personnel must be aware that certain instruments are rated for operation within a limited range of conditions such as temperature and humidity. Usage of such instruments in conditions outside these ranges will only proceed with proper approval by a project manager and/or health and safety supervisor as appropriate.
- Instruments that contain radioactive source material, such as x-ray fluorescence analyzers or moisture-density gauges require specific transportation, handling, and usage procedures that are generally associated with a license from the Nuclear Regulatory Commission (NRC) or an NRC-Agreement State. Under no circumstance will operation of such instruments be allowed on site unless by properly authorized and trained personnel, using the proper personal dosimetry badges or monitoring instruments.

Calibration

Calibration of an electronic instrument is critical to insure it is operating properly for its intended use. Such instruments are often sensitive to changes in temperature or humidity, or chemical vapors in the working atmosphere, and as a result their response and ability to monitor conditions and provide data can change significantly.

Calibration of instruments shall be performed and documented in accordance with the manufacturer's recommendations. This includes the following parameters:

- Frequency.
- Use of proper calibration gases or chemical standards.
- Requirements for factory calibration.

Instrument calibration shall be performed in accordance with the following manufacturer recommendations or the suggested "minimum" calibration frequency:

Instrumentation Classification/Group		***Instrumentation***	***Representative Manufacturer Recommended Calibration Frequency***	***Minimum Recommended Calibration Frequency***
Health and Safety	Air Monitoring (Real-Time):	PID, FID, compound-specific or multi-gas meter (typ.), etc.	No Recommendation, Daily or As Needed	Daily
	Air Sampling (non-Real-Time):	Flow meter, personal air sampling device, etc.	Per Manufacturer's Recommendations	Per Manufacturer's Recommendations

Instrumentation Classification/Group		*Instrumentation*	*Representative Manufacturer Recommended Calibration Frequency*	*Minimum Recommended Calibration Frequency*
	Air Monitoring for Confined Space Entry (Real-Time)	Four Gas Meter or Multi-Gas meter with O, LEL, CO, H2S sensors	Daily or before each entry As Needed	Daily
Other Monitoring	Water Sampling:	pH, Cond., Temp., ORP, DO, etc.	Per Manufacturer's Recommendations	Daily, or As Needed
	Physical Parameters:	Velocity/flow meter, pressure transducer, water level meter, oil-water interface probe, etc.	Per Manufacturer's Recommendations	Per Manufacturer's Recommendations
	Other:	Miscellaneous (Troxler nuclear density, etc.)	Per Manufacturer's Recommendations	Per Manufacturer's Recommendations

Notes:

1. Some instrumentation requires factory calibration only.
2. If a significant change in conditions occurs, or in dangerous atmosphere conditions, more frequent calibration should be performed.

Calibration Gas Safety

Several instruments such as photoionization detectors (PIDs), flame ionization detectors (FIDs), oxygen meters, explosimeters, combustible gas indicators, and many others require use of calibration gasses contained in compressed gas cylinders. Many of these gases are combustible or explosive. Care shall be taken to minimize the potential for injury from the use of such compressed gases. Transport, handling, and storage of cylinders, where necessary, shall be performed in accordance with applicable Department of Transportation (DOT) regulations and site requirements.

Calibration will only be performed in areas free of sources of spark, flame, or excessive heat. Smoking will not be allowed in the vicinity of calibration gas usage areas.

Documentation of Calibration

Instrument calibration activities will be documented. Form FMG 8.0-01 - Instrument Calibration Record shall be used to record applicable calibration and maintenance activities. In addition, protocol for documentation outlined in FMG 1.4 - Data Recording - Field Books/Digital Recording shall be followed.

Intrinsically Safe Requirements

Certain work locations may be such that dangerous, ignitable, or explosive conditions exist. In such cases, it may be necessary to utilize only equipment that is rated as "Intrinsically Safe".

Intrinsically safe instrumentation is designed with limited electrical and thermal energy levels to eliminate the potential for ignition of hazardous mixtures.

For site work requiring operation of monitoring instruments in Class I, Division I locations [as defined by the National Fire Protection Agency (NFPA)] only instrumentation rated as Intrinsically Safe will be used. Such equipment (including all accessories and ancillary equipment) must be rated to conform to Underwriters Laboratories (UL) Standard 913, for use in a Class I, Division 1, Groups A, B, C, and D locations. It is also recommended the equipment conform with CSA Standard 22.2, No. 157-92.

Upon completion of the field activities, equipment shall be returned to the possession of the Consultant, Contractor, or Rental Agency accompanied by a written summary of any problems encountered with its use or calibration.

Equipment shall be properly prepared for shipping, including insuring that residual gases (if applicable) are removed from the instrument, and accompanying containers of compressed gases or fluids are properly labeled and sealed.

Equipment Decontamination

Equipment that comes in contact with Site media (water level meters, water quality meters) must be cleaned **before** removal from the site to ensure that chemicals are not transferred to other sites. It is the responsibility of the person who requisitioned the equipment to ensure appropriate cleaning before returning the equipment. Equipment decontamination procedures are typically site specific for unique site compounds.

EQUIPMENT/MATERIALS

- Monitoring equipment specific to work plan tasks.
- Manufacturer's instructions, operation and maintenance information.
- Associated calibration gases, aqueous standards, etc.
- Appropriate shipping containers to facilitate transport without damage to equipment.

REFERENCES

Underwriters Laboratories, Inc. (https://www.ul.com/) Standard UL 913.

National Fire Protection Agency (https://www.nfpa.org/).

Canadian Standards Association (CSA) (https://www.csagroup.org/) Standard 22.2 No. 157.

Form FMG 8.0-01

INSTRUMENT CALIBRATION RECORD

PROJECT _____ **PROJECT MANAGER** _____

LOCATION _____ **FIELD REP.** _____

DATE _____

Instrument	Date Calibrated	By	Standard Used	Decontamination, Maintenance, or Repair Performed	Remarks

Other Remarks: _____

FMG MODIFICATIONS MUST BE ACCOMPANIED BY A REVISION REQUEST FORM APPROVED BY THE PROJECT MANAGER

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EQUIPMENT DECONTAMINATION

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 9.0
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

EQUIPMENT DECONTAMINATION

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

This procedure describes decontamination of field equipment potentially exposed to contaminants. Proper decontamination is required to reduce the risk of transfer of contaminants from areas of contamination to other areas and to minimize the potential for cross-contamination that would compromise sample quality. The degree of decontamination required will be dependent on the nature of the activity, equipment used, and on the amount of exposure to contaminants.

PROCEDURES REFERENCED

- FMG 2.0 - Subsurface Investigations
- FMG 5.0 - Aquifer Characterization
- FMG 6.0 - Sample Collection for Laboratory Analysis
- FMG 6.15 – PFAS/POFA Sampling
- FMG 8.0 - Field Instruments – Use/Calibration
- FMG 10.0 - Waste Characterization

PROCEDURAL GUIDELINES

Decontamination activities must be performed in a controlled area outside any exclusion zones established on the site. Care must be taken to minimize the potential for transfer of contaminated materials to the ground or onto other materials. Regardless of the size or nature of the equipment being decontaminated, the process will utilize a series of steps that involve removal of gross material (dirt, grease, oil, etc.), washing with a detergent, and multiple rinsing steps. In lieu of a series of washes and rinse steps, steam cleaning with low-volume, high-pressure equipment (i.e., steam cleaner) is acceptable.

Drill rigs, backhoes, and other exploration equipment must be decontaminated prior to initiating site activities, in between exploration locations to minimize cross-contamination potential, and prior to mobilizing off site after completion of site work. Heavy equipment is generally best deconned with a combination of steam-cleaning equipment and detergent scrubbing. Particular attention should be paid to parts in direct contact with contaminants, e.g., shovels, tires, augers, drilling decks, etc.

Control and containerization of all decontamination fluids is critical. A decontamination pad must be constructed that is appropriate for the size and type of equipment being decontaminated. At a minimum, the decontamination pad will have the following elements:

- An impermeable barrier capable of containing decontamination fluids.
- A low point where fluids will collect and can be pumped into appropriate containers.
- Durability to withstand equipment such as vehicle and foot traffic.
- Appropriate ancillary equipment such as racks to place decontaminated equipment to drain without further exposure to contaminated fluids.
- Labels to alert personnel as to the potential presence of contaminated materials.

Decontamination of Specific Sampling Equipment

Note there is a preference to use pre-packaged disposable equipment rather than create potential for cross contamination and the time spent on decontamination.

The following specific decontamination procedure is recommended:

- Brush loose soil off equipment.
- Wash equipment with laboratory grade detergent (i.e., Alconox or equivalent). Make sure it's appropriate for the types of contaminants [i.e., per- and polyfluoroalkyl substances (PFAS)/ perfluorooctanoic acid PFOA)] (see FMG 6.15 – PFAS/POFA Sampling for further details).
- Rinse with tap water (three rinses minimum).
- Rinse equipment with reagent grade methanol for VOC samples (this requirement may not be appropriate for sites where methanol is a contaminant of concern).
- Rinse equipment with nitric acid for metal samples (especially important for sites with potentially high metals concentrations.
- Rinse equipment with distilled water.
- Allow water to evaporate before reusing equipment

Decontamination of Monitoring Equipment

Because monitoring equipment is difficult to decontaminate, care should be exercised to *prevent* contamination. Sensitive monitoring instruments should be protected when they are at risk of exposure to contaminants. This may include enclosing them in plastic bags allowing an opening for the sample intake. Ventilation ports should not be covered.

If contamination does occur, decontamination of the equipment will be required; however, immersion in decontamination fluids is not possible. As such, care much be taken to wipe the instruments down with detergent-wetted wipes or sponges, and then with deionized water-wetted wipes or sponges.

Disposal of Wash Solutions and Contaminated Equipment

All contaminated wash water, rinsates, solids and materials used in the decontamination process that cannot be effectively decontaminated (such as polyethylene sheeting) will be containerized and disposed of in accordance with applicable regulations and OXY requirements. All containers will be labeled with an indelible marker as to contents and date of placement in the container, and any appropriate stickers required [such as polychlorinated bipheyls (PCBs)].

Sampling of containerized wastes will be performed immediately upon completion of the investigations to minimize storage time on site. Storage of decontamination wastes on site will not exceed 90 days under any circumstances.

Level C Decontamination Procedures

The general Level C decontamination procedures to be used when leaving the exclusion zone are as follows:

Step 1: Equipment drop.
Step 2: Outer boot cover, outer glove and suit wash with decontamination solution or detergent/potable water.
Step 3: Outer boot cover, outer glove and suit rinse with potable water.
Step 4: Tape removal around outer boots and gloves and deposit in PPE waste receptacle properly labeled for disposal.
Step 5: Boot cover removal.
Step 6: Outer glove removal.
Step 7: Suit removal. If disposable place in PPE waste receptacle.
Step 8: Respirator removal. Clean and disinfect for next use.
Step 9: Inner glove removal and disposal in PPE waste receptacle.
Step 10: Wash hands, face, and neck and shower as soon as possible at the end of the day/shift.

It should be noted that the steps above can vary slightly dependent on the task and what PPE is required (e.g., reusable or disposable). Decontamination of Level C PPE is generally accomplished

using detergents (surfactants) in water combined with a physical scrubbing action. This process will remove most forms of surface contamination including dusts, many inorganic chemicals, and some organic chemicals.

EQUIPMENT/MATERIALS

Decontamination equipment and solutions are generally selected based on ease of decontamination and disposability.

- Polyethylene sheeting.
- Metal racks to hold decontaminated equipment.
- Soft-bristle scrub brushes or long-handle brushes for removing gross contamination and scrubbing with wash solutions.
- Large galvanized wash tubs, stock tanks, or wading pools for wash and rinse solutions.
- Plastic buckets or garden sprayers for rinse solutions.
- Large plastic garbage cans or other similar containers lined with plastic bags can be used to store contaminated clothing.
- Contaminated liquids and solids should be segregated and containerized in DOT-approved plastic or metal drums, appropriate for off-site shipping/disposal if necessary.

REFERENCES

ASTM D5088 - Practice for Decontamination of Field Equipment Used at Non-Radioactive Waste Sites.

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WASTE CHARACTERIZATION

FORMER CITIES REFINERY	FIELD METHOD GUIDELINE NO.: FMG 10.0
EAST CHICAGO, INDIANA	EFFECTIVE DATE: AUGUST 17, 2018
OXY USA, INC	
REVISION NO.: 1	REVISION DATE:

WASTE CHARACTERIZATION

It is mandatory that all field activities are performed in a manner that is consistent with Occupational Safety and Health (OSHA) regulations and OXY's health and safety policy. Prior to completing any field activities, the project-specific Health and Safety Plan (HASP) must be finalized, reviewed, and understood. **In addition, all field activities must comply with federal, state, and local rules and regulations at all times. Any questions that arise should be discussed and resolved with the OXY Project Manager.**

INTRODUCTION

The following procedure describes the techniques for characterization of investigation derived waste (IDW) for disposal purposes.

It is important to review the health and safety and the waste disposal requirements for the plant with both the OXY plant Environmental Engineers and the plant Resource Manager (RM) from the OXY-contracted Resource Management Company, prior to any work. The IDW containment and management procedures, profile sampling requirements, and strategy should be reviewed to provide an estimate to the RM of the waste volumes.

It should be noted that the plant RM will be managing the IDW that is generated. Waste characterization sampling will be performed as directed by the plant RM. The waste characterization results and anticipated quantities will inform the plant RM of the volume of IDW produced. The RM will complete the waste profile and arrange for disposal.

PROCEDURAL GUIDELINES

IDW may consist of soil cuttings (augering, boring, well installation soils, test pit soils, etc.), rock core or rock flour (from coring, reaming operations), groundwater (from well development, purging, and sampling activities), decontamination fluids, personal protective equipment (spent gloves, tyveks) (PPE), and disposal equipment (DE).

This procedure applies when disposition of investigation soils and/or groundwater is required in accordance with the project Work Plan. Generally, this procedure is applicable to plants where the OXY Project Manager has assessed the areas of investigation and has developed a waste handling plan. In some areas and/or sections within a plant it is permitted to return soil cuttings/test pit soils and groundwater to the source area (RCRA guidance allows waste management

techniques within an area of concern without 'triggering' new points of waste generation) subject to OXY Project Manager approval. This is also allowed by some States. In other areas it may not be practical to return cutting/soils to their origin, and are better handled by this characterization/disposal procedure. This practice is consistent with United States Environmental Protection Agency (USEPA) procedure for IDW at RCRA facilities and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) sites (Reference 1, 2).

Typically investigative derived wastes are dealt with following "Best Management Practices"; and are not considered RCRA characteristic or listed waste until proven to be listed and/or identified characteristically hazardous waste. Evidence has to be definitive. Investigative soils and groundwater should not be considered a listed waste (in most circumstances) due to the lack of generator knowledge concerning chemical source, chemical origin, and timing of chemical introduction to the subsurface. Consequently, waste sampling and characterization is performed to determine if the wastes exhibit a characterization of hazardous waste. Once the waste characterization and a determination is made, best management practices apply consistent with RCRA.

The disposal of soil cuttings and/or purged groundwater must be reviewed on a case-by-case basis prior to initiation of field activities. Two scenarios typically exist:

i) Sufficient plant and/or site information exists and State regulations allow that investigative cuttings and/or purged groundwater to be placed back into the borehole or spread on the ground surface. No disposal required.

ii) Site conditions warrant that all materials handled will be contained and disposed of consistent with the RCRA and/or State requirements.

DISPOSAL PROCEDURES

The following outlines the waste characterization procedures to be employed when IDW disposal is required. The OXY Remediation Team is now working with the plant RM to manage IDW disposal. Waste characterization sampling will be performed as directed by the OXY Environmental Engineer(s). The waste characterization results and anticipated quantities will inform the plant RM of the volume of IDW produced. The RM will then set up a direct Purchase Order (PO) with the OXY Remediation Team to complete the waste profile and dispose of the waste (e.g. contact the OXY Remediation Team Project Manager).

Soil/Rock Cuttings

Soils removed from boring activities and well construction tasks (including, rock flour from bedrock coring) will be contained within an approved container, suitable for transportation and disposal.

- Once placed into the approved container, any free liquids (i.e., groundwater) will be poured off for disposal as waste fluids, or solidified within the approved container using a

solidification agent such as speedy-dri (or equivalent). No free liquid as determined by the "paint filter test" shall be present.

- Contained soils will be screened for the presence of volatile organic compounds (VOCs), using a photoionization detector (PID); this data will be logged for future reference.
- Once screened, full and closed, the container will be labeled in accordance with the plant labeling requirements and placed into the plant container storage area. At a minimum, the following information will be shown on each container label: date of filling/generation, plant name, source of soils (i.e., borehole or well), and plant contact and any additional regulatory labelling requirements. If necessary, the exterior of the container will be cleaned to remove any lose dirt/cuttings.
- Prior to container closure, representative samples from a percentage of the containers will be collected for waste characterization purposes and submitted to the project laboratory. The waste characterization sampling scheme will be dictated by the Work Plan, coordinated with the plant RM, and will establish the volume of soils required for analysis (depending on parameters required), the number of containers considered representative, the homogenization procedure, volatile analysis collection procedure (if required), and preparation handling requirements. Typically at a location where an undetermined site-specific parameter group exists, sampling and analysis may consist of the full RCRA Waste Characterization (ignitability, corrosivity, reactivity, toxicity), or a subset of the above based upon data collected, historical information, and generator knowledge. This will be determined under the plant RM's direction, with the approval of OXY.

Groundwater

Well construction development, purging, and sampling groundwater which requires disposal will be contained. Containment may be performed in 55-gallon drums, tanks suitable for temporary storage (i.e., Nalgene or plant provided tanks 500 to 1,000 gallons) or if large volumes of groundwater are anticipated, drilling "frac" tanks may be utilized (20,000 gallons ±), or tanker trailer (5,000 to 10,000 gallons ±). In all cases, the container/tank used for groundwater storage must be clean before use such that cross-contamination does not occur. Do not mix grout purge or mixing water with other well fluids, as the high pH from cement grout can create an unintended hazardous waste. Grout water should be drummed separately.

Decontamination Waters/Decontamination Fluids

- Decontamination waters and/or fluids will be segregated, contained, and disposed of accordingly.
- Decontamination waters may be disposed of with the contained groundwater once analytical results have been acquired. Depending on the extent of chemistry present it may be appropriate to discharge the decontamination waters to the Publicly Owned Treatment Works (POTW); or discharge to an on-site treatment system; or send off-site for treatment. Proper permitting may be required.

- Spent Solvent/Acid Rinses - Solvents and acids used during decontamination activities must be segregated and disposed separately from the groundwater/decontamination water. Often if only small amounts of solvents are involved these can be left to evaporate. If large volumes are involved then containerization, labeling, and storage is required.

PPE/DE

- Several disposal options exist for spent PPE/DE generated from investigation tasks. The options typically employed are:
  - i) Immediately disposed of within on-site dumpster/municipal trash, if properly decontaminated; or
  - ii) If known to be contaminated with RCRA hazardous waste, disposed of off-site at a RCRA Subtitle C facility; or alternatively PPE/DE decontaminated and disposed of on site within dumpster/municipal trash; or
  - iii) Contained and stored until the final remedy is implemented.

WASTE CHARACTERIZATION PROCEDURES

Waste characterization will be performed under the plant RM's direction, with the approval of OXY. The Work Plan, in coordination with the plant RM, will identify the appropriate sampling strategy and analytes required to determine the IDW characteristics and disposal requirements. USEPA SW-846 (Reference 5, Chapters 9 and 10) describes the rationale for sampling plan development and sampling procedures. Generally, random sampling and preparation of a composite sample of the media is employed for most investigative programs.

Sampling procedures for IDW are:

- Solid Wastes - Grab sampling using pre-cleaned sample spoons from bulk piles, lugger boxes, or as drums are being filled is commonly employed. In some instances, sufficient media mixing may be evident to permit drum sampling from a random number of drums by accessing only the top solids. In other instances where stratification is evident, a sample trier/hand auger or device to collect from the entire vertical profile is required. Typically, a composite sample(s) from representative areas of the container(s) is homogenized and submitted for analysis. If VOCs are being evaluated, compositing and homogenization is not permitted. Individual grab samples are typically required for VOCs.
- Waste Waters - Grab sampling techniques using pre-cleaned bailers or sampling pumps are typically employed. Waters in bulk are typically sampled once using a bailer or pump. The Work Plan will outline the appropriate sample frequency and analytes necessary to adequately

characterize the contained waters. Facility sewer discharge permit parameters will be evaluated when disposal to the POTW is being considered.

Note: If non-aqueous phase liquid (NAPL) is present special sampling and handling requirements will apply. Precautions to separate the NAPL from the wastewater will commonly be employed, due to the special material handling and waste disposal requirements when dealing with phase materials.

- Spent Solvent/Acid Rinses - The need for sampling must be determined in consultation with the waste management organization handling the materials. If known that only the solvent and/or acids are present, then direct disposal/treatment using media specific options maybe possible without sampling (i.e., incineration).
- PPE/DE - Typically not sampled and can be included with the disposal of the solid wastes or decontaminated and disposed in the plant refuse.

EQUIPMENT/MATERIALS

- Sample spoons, trier, auger.
- Sample mixing bowl.
- Sampling bailer, or pump.
- Sample glassware.

REFERENCES

USEPA RCRA - Guidance and Policies: Management of Remediation Waste Under RCRA (October 1998).

USEPA RCRA - Management of Contaminated Media (October 1998).

USEPA CERCLA Guidance (Options Relevant to RCRA Facilities): Guide to Management of Investigation-Derived Wastes (January 1992).

USEPA Office of Solid Waste - SW-846 Chapter 9 Sampling Plan, Chapter 10 Sampling Methods (September 1986).

about GHD

GHD is one of the world's leading professional services companies operating in the global markets of water, energy and resources, environment, property and buildings, and transportation. We provide engineering, environmental, and construction services to private and public sector clients.

John-eric Pardys
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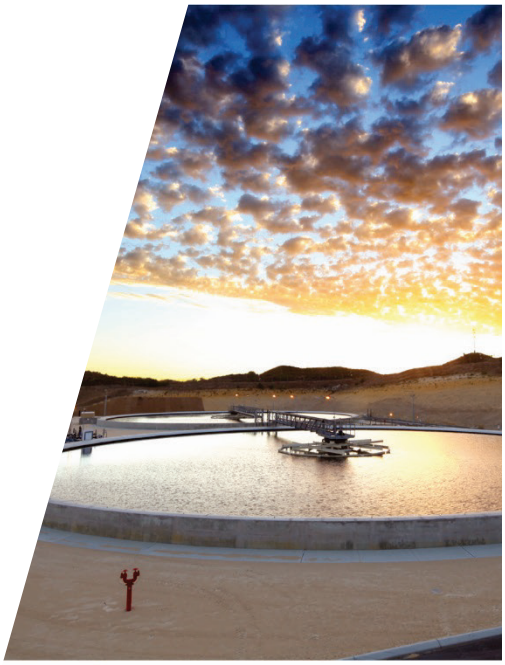
Appendix B
Quality Assurance Project Plan (QAPP)



Quality Assurance Project Plan

Former Cities Refinery,
East Chicago, Indiana

OXY USA, Inc.

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Appendix Index

QAPP Worksheet #1 & 2: Title and Approval Page
(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)4

1. Project Identifying Information
   a. Former Cities Refinery (INR 000 123 927)]
   b. East Chicago, State of Indiana
   c. GHD Project Number 11208111

2. Settling Work Parties' Project Coordinator

   /s/ Rick Passmore 07/31/2020 _____ Signature
   GSH Project Coordinator – Rick Passmore Date

3. Settling Work Parties' Supervising Contractor - GHD

   /s/ Michael Tomka 07/31/2020 _____ Signature
   Project Director – Michael Tomka Date

   /s/ John-Eric Pardys 07/31/2020 _____ Signature
   Project Manager – John-Eric Pardys Date

   /s/ Angela Bown 07/31/2020 _____ Signature
   Quality Assurance Manager – Angela Bown Date

   /s/ James Abston 07/31/2020 _____ Signature
   Project Chemist – James Abston Date

4. United States Environmental Protection Agency - Region 5

   /s/ Renee Wawczak 07/13/2020 _____ Signature
   Corrective Action Project Manager – Renee Wawczak Date

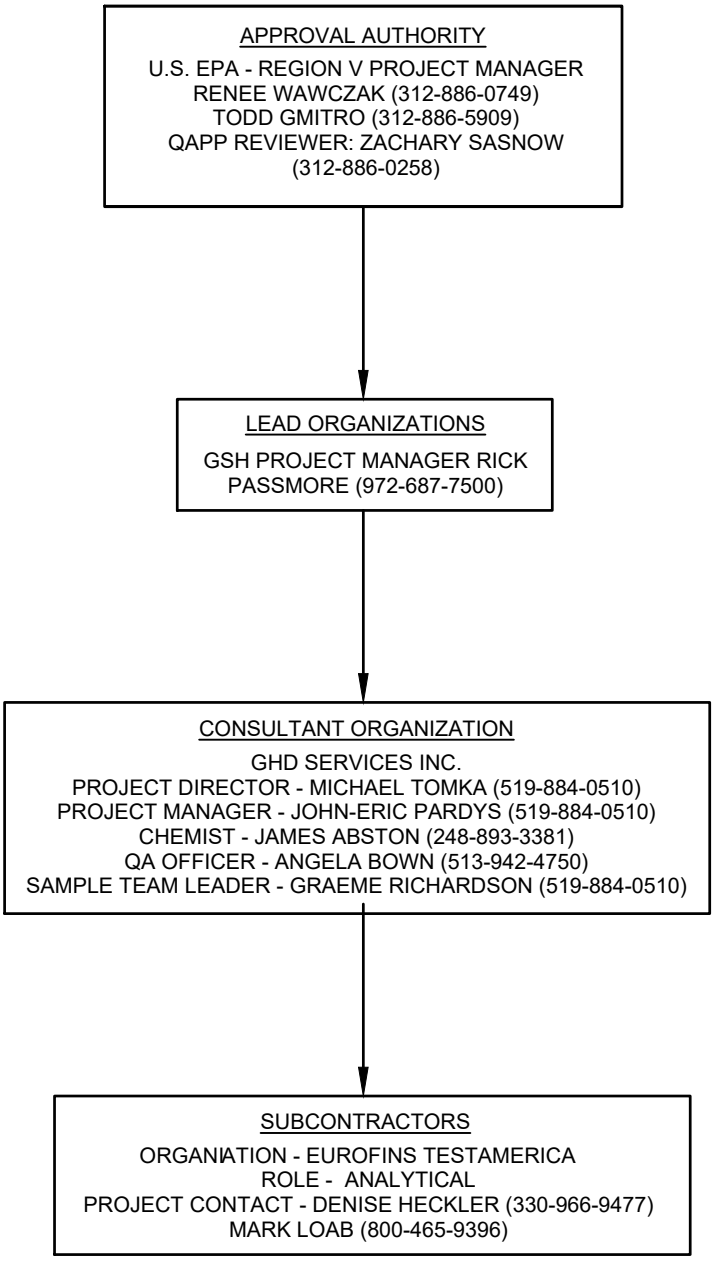
   /s/ Todd Gmitro 07/08/2020 _____ Signature
   Corrective Action Project Manager – Todd Gmitro Date

   /s/ Zachary Sasnow 07/09/2020 _____ Signature
   QAPP Reviewer – Zachary Sasnow Date

5. List plans and reports from previous investigations relevant to this project

   Investigation Results Report – Phase I, submitted October 31, 2019

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)


APPROVAL AUTHORITY
U.S. EPA - REGION V PROJECT MANAGER
RENEE WAWCZAK (312-886-0749)
TODD GMITRO (312-886-5909)
QAPP REVIEWER: ZACHARY SASNOW
(312-886-0258)
LEAD ORGANIZATIONS
GSH PROJECT MANAGER RICK
PASSMORE (972-687-7500)
CONSULTANT ORGANIZATION
GHD SERVICES INC.
PROJECT DIRECTOR - MICHAEL TOMKA (519-884-0510)
PROJECT MANAGER - JOHN-ERIC PARDYS (519-884-0510)
CHEMIST - JAMES ABSTON (248-893-3381)
QA OFFICER - ANGELA BOWN (513-942-4750)
SAMPLE TEAM LEADER - GRAEME RICHARDSON (519-884-0510)
SUBCONTRACTORS
ORGANIATION - EUROFINS TESTAMERICA
ROLE - ANALYTICAL
PROJECT CONTACT - DENISE HECKLER (330-966-9477)
MARK LOAB (800-465-9396)

QAPP Worksheet #4, 7, & 8: Personnel Qualifications and Sign-off Sheet
(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

Organization: U.S. EPA

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature*/Date
Renee Wawczak	EPA Project Manager			/s/ Renee Wawczak 07/13/2020
Todd Gmitro	EPA Corrective Action Project Manager	M.S. Geology (30 years)		/s/ Todd Gmitro 07/08/2020
Zachary Sasnow	QAPP Reviewer			/s/ Zachary Sasnow 07/09/2020

Organization: GHD Services Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature*/Date
Michael Tomka	Project Manager	B.A.Sc. in Civil Engineering/28 years	P.E.	/s/ Michael Tomka 07/31/2020
James Abston	Project Chemist/Data Validator	BS in Physics and Mathematics/28 years		/s/ James Abston 07/31/2020
Angela Bown	QA Manager	B.S. Environmental Management; A.S. Laboratory Technology; 30+ years of experience in environmental laboratory operations and data validation		/s/ Angela Bown 07/31/2020

Organization: Eurofins TestAmerica, Canton

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature*/Date
Denise Heckler	Laboratory Project Manager	BS Chemistry, Youngstown State University, 1988. 30 years environmental lab experience		/s/ Denise Heckler 07/31/2020
Mark Loeb	Quality Assurance Manager	BS Chemistry, University of Akron, 31 years environmental lab experience		/s/ Mark Loeb 07/31/2020

* Signatures indicate personnel have read and agree to implement this QAPP as written.

QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory agency interface	GSH	Rick Passmore	rick_passmore@oxy.com 713-215-7622	Analytical data and project information such as changes to the QAPP, schedule and field activities will be forwarded to the Agency per the SOW.
Regulatory agency interface – Alternate	GHD Services, Inc.	Michael Tomka	michael.tomka@ghd.com 519-884-0510	Analytical data and project information such as changes to the QAPP, schedule and field activities will be forwarded to the Agency per the SOW.
Regulatory agency interface	USEPA Region 5 Remedial Project Managers	Renee Wawczak Todd Gmitro	312-886-0749 312-886-5909	Provide feedback to GHD regarding analytical data and project information such as changes to the QAPP, schedule and field activities.
Field, data, and reporting progress reports; unexpected events; emergencies; non-conformances	GHD Services Inc.	Michael Tomka	michael.tomka@ghd.com 519-884-0510	Frequent updates on all routine aspects of the project, and immediate updates on non-routine aspects of the project will be provided by phone and/or email to
Field progress reports	GHD Services Inc.	Graeme Richardson	graeme.richardson@ghd.com 519-884-0510	Daily field progress reports will be phoned or emailed to the GHD PM.
Stop work due to safety issues	GHD Services Inc.	All Personnel	All Personnel	STOP WORK IMMEDIATELY-NOTIFY PM.
QAPP changes prior to field work	GHD Services Inc.	Angela Bown	Angela.Bown@ghd.com	Changes to the QAPP prior to field work will be made by Angela Bown and approved by PM as needed.
QAPP changes during project execution	GHD Services Inc.	Angela Bown	Angela.Bown@ghd.com	Changes to the QAPP during project execution will be made by Angela Bown and approved by PM as needed.
EPA QAPP Review	USEPA Region 5	Zachary Sasnow	sasnow.zachary@epa.gov 312-886-0258	Changes to the QAPP will be reviewed and approved by Zachary Sasnow.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Field corrective actions	GHD Services Inc.	Graeme Richardson	graeme.richardson@ghd.com 519-884-0510	Field corrective actions will be documented by FIELD TECH and communicated to the GHD PM immediately.
Sample receipt variances	TestAmerica, Inc.	Denise Heckler Mark Loeb	Denise.Heckler@testamericainc.com Mark.Loeb@testamericainc.com 800-456-9396	Sample receipt variances will be documented by Denise Heckler or Mark Loeb and communicated to James Abston within 48 hours.
Laboratory quality control variances	TestAmerica, Inc.	Denise Heckler Mark Loeb	Denise.Heckler@testamericainc.com Mark.Loeb@testamericainc.com 800-456-9396	Laboratory quality control variances will be documented by Denise Heckler or Mark Loeb and communicated to James Abston within 48 hours.
Analytical corrective actions	TestAmerica, Inc.	Denise Heckler Mark Loeb	Denise.Heckler@testamericainc.com Mark.Loeb@testamericainc.com 800-456-9396	Analytical corrective actions will be documented by Denise Heckler or Mark Loeb and communicated to Angela Bown within 48 hours.
Data verification issues, e.g., incomplete records	GHD Services Inc.	James Abston	James.Abston@ghd.com	Data verification issues will be documented by James Abston and will notify Denise Heckler or Mark Loeb of any incomplete lab records and request corrective actions.
Data validation issues, e.g., on-compliance with procedures	GHD Services Inc.	James Abston	James.Abston@ghd.com	Data validation issues will be documented by James Abston in the data validation report. PM will be notified of deficiencies as needed.
Data review corrective actions	GHD Services Inc.	James Abston	James.Abston@ghd.com	Corrective actions will be documented in the data validation report by James Abston. PM will be notified of corrective actions as needed.

QAPP Worksheet #9: Project Planning Session Summary
(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)

Date of planning session: September 24, 2019
Location: USPEA Region V, Chicago Office
Purpose: Phase I results

Participants:

Name	Organization	Title/Role	Email/Phone
Renee Wawczak	U.S. EPA	Project Manager	wawczak.renee@epa.gov
Mario Mangino			mangino.mario@epa.gov
Todd Gmitro		Project Manager	gmitro.todd@epa.gov
Mike Beedle		Project Director	beedle.michael@epa.gov
Scott Buckner	CITGO	Project Manager	Sbuckne@citgo.com
Peter Krivas		Facility Manger	Pkrivas@citgo.com
Rick Passmore	GSH	Project Manager	Rick_passmore@oxy.com
Michael Tomka	GHD	Project Manager	Michael.tomka@ghd.com
Matt Rousseau		LNAPL Expert	Matt.Rousseau@ghd.com

Notes/Comments:
- Chemical of Potential Concern (COPCs) were identified consistent with the draft CAF provided by EPA (TCL VOCs, TCL SVOCs, TAL Metals, and 1,4-dioxane)

Consensus decisions made:
- No objections to the list were identified.

Action Items:

Action	Responsible Party	Due Date

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

Conceptual Site Model (CSM)

Current and Future Site Land Use

Current Land Use: Industrial None
Projected Future Land Use: Industrial or Commercial

Current and Future Surrounding Property Land Use

Mixed residential, commercial, industrial and limited recreational

Sources and Extent of Known Contamination

Sources of contamination could include wastes from former petroleum refinery related operations. Previous investigations have identified the presence of select volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), and metals.

Sufficient data is not currently available to conclusively determine the extent of contamination.

The extent of impacts of VOCs, SVOCs, 1,4-dioxane, and metals will be confirmed in subsequent phases of the RFI.

Contamination Transport/Migration Pathways

Contaminant transport and migration pathways include the following:
- Hydrocarbon migration through the subsurface to groundwater
- Groundwater to surface water (Grand Calumet River)
- Hydrocarbon vapor intrusion potential

Exposure Receptors

Potential on-site exposure receptors include:
- Routine workers
- Maintenance or construction workers
- Trespassers

Potential off-site exposure receptors include:
- Routine workers
- Maintenance or construction workers
- Trespassers
- Residential

Potential off-site ecological receptors include:
- Natural area to the south
- Grand Calumet River

Exposure Point and Exposure Mediums include:
- Surface soil
- Subsurface soil
- Groundwater
- Indoor air

Exposure Routes

Potential exposure routes include:
- Soil dermal direct contact
- Soil or groundwater ingestion
- Soil vapor inhalation from contaminated soil or groundwater
- Inhalation of fugitive dust

It is noted that institutional or engineering controls will be employed to prevent exposure by any of these potential exposure routes, and that none of these pathways have been confirmed to exist as of this date, but will continue to be investigated as part of the RFI.

Discussion of Unknowns and Uncertainty

The delineation of Constituents of Potential Concern (COPCs) is currently unknown and ongoing. Historical data and knowledge are being used to design a biased sampling plan for the Former Refinery.
The current COPCs, as supported by historical investigations, for soil and groundwater are as follows:
- Target compound list (TCL) VOCs (Method 8260),
- TCL SVOCs (Method 8270),
- target analyte list (TAL) metals (Method 6010/7470) and
- 1,4-dioxane.

QAPP Worksheet #11: Project/Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

1. **State the Problem**

From approximately 1929 to 1972, Empire Refining Company, and then Cities, or subsidiaries of Empire or Cities operated a refining and bulk storage terminal complex consisting of approximately 322 total acres, of which the crude oil refinery operations were located on portions of the 93.5 acre Former Cities Refinery. The refining operation ceased on or about 1972. The bulk storage terminal continued to operate subsequent to closure of the refinery and is currently owned and operated by CITGO (since 1983).

The refinery operations formerly located on the Facility produced gasoline, diesel, tractor fuel, kerosene, fuel oil, range oil, petroleum coke, naphtha, and other related materials.

In accordance with Section 13 of the Order the objectives of the Resource Conservation and Recovery Act Facility Investigation Remedy Selection Track (RCRA First) program are to:

- Determine the nature and extent of releases of hazardous waste and hazardous constituents at or from the Facility
- Identify and evaluate interim corrective measures to control human exposures to contamination or to stabilize the migration of contaminated groundwater
- Demonstrate human exposures to contamination are under control and migration of contaminated groundwater is stabilized

2. **Identify the Goals of the Study**

The data to be collected as part of the (RCRA First) as specified in the Order and the Corrective Action Frameworks (CAFs) are necessary to determine the nature and extent of impacts to groundwater and soil such that informed decisions can be made regarding potential risks to human and ecological receptors. The data will be used to update the CSM and address data gaps. The data will be compared to screening levels and will receive site-specific evaluation to assess risk to receptors. The data will therefore ultimately help identify potential remedial alternatives to address or prevent exposure to contamination present at concentrations that pose an unacceptable risk to receptors.

3. **Identify Information Inputs**

Presents the rationale and types of data that are required to fill data gaps in the CSM. The data may be used for evaluation of risk (ecological and human health).

4. **Define the Boundaries of the Study**

The boundaries of the study area are shown in the CAFs. The list of constituents of potential concern (COPC) are as follows:

- Target Compound List (TCL) volatile organic compounds (VOCs) (Method 8260/8011)
- TCL semi-volatile organic compounds (SVOCs) (Method 8270)
- Target Analyte List (TAL) metals (Method 6010/7470)
- 1,4-dioxane.

5. **Develop the Analytic Approach**

Laboratory analytical methods are presented in Worksheet #28.

6. **Specify Performance or Acceptance Criteria**

The analytical results will be compared to the project action levels (PALs) detailed on Worksheet #15. To compare site data to the applicable PALs, the selected laboratory must be able to achieve Reporting Detection Limits (RDLs) that are low enough to measure constituent concentrations below the PALs to ensure laboratory sensitivity is sufficient. In cases where conventional test methods are not able to achieve detection limits that are lower than PALs, rules for evaluating the data are required that help the Project Team determine with reasonable satisfaction whether the constituent poses a potentially unacceptable risk. Analytical data reported by the laboratory use the following reporting conventions: all concentrations less than the MDL and RDL will be considered non-detects and will be reported with a “U” qualifier; between the MDL and RDL will be reported with a “J” qualifier; and at or above the RDL will be reported with no qualifier. In the event that a target analyte has a PAL between the MDL and RDL, the “J” flagged data will be accepted to achieve project goals. The inability to quantifiably compare individual analytes to PALs with confidence must be addressed in the risk evaluation uncertainty analysis in each risk assessment.

7. **Develop the Detailed Plan for Obtaining Data**

The basis for the sampling design is to fill data gaps by collecting additional data. Refer to Worksheet #17 for details regarding Sample Design and Rationale, and Worksheets #19, 20, 24-28, and 30 for analysis design requirements. GHD will collect the samples and submit to Eurofins for analyses. The laboratory will submit a final complete analytical report in electronic format. The analytical report submitted by the laboratory shall conform to all reporting and deliverable requirements. Files for the data shall be inventoried and maintained by GHD and shall consist of the following; work plan and supporting plans; project logbooks, field data records, sample identification documents; chain-of-custody records; laboratory data; correspondence; report notes; calculations etc.; references; copies of pertinent literature; photos; maps; drawings, etc. and final report.

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater/Soil/LNAPL
Analytical Group or Method: TCL VOC: SW-846 8260C
SOP: NC-MS-019, Rev. 6 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	BFB Tune	BFB Tune Criteria must be met per SW-846 Method 8260C
Analytical Accuracy/Bias (laboratory)	Initial Calibration (ICAL) Curve	%RSD and %D must be met per SW-846 Method 8260C; COD (R^2) $\geq$ 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8260C
Analytical Sensitivity (laboratory)	Continuing Calibration (CCAL) Standards	%D or % Drift must be met per SW-846 Method 8260C; Minimum Mean Response Factors must be met per SW-846 Method 8260C
Analytical Accuracy/Bias (laboratory)	Internal Standards	50-200% Recovery of the response of the previous continuing calibration standard
Analytical Accuracy/Bias (laboratory)	Surrogates	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples-Second Source	Must meet established Laboratory Limits*
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates - Second Source	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Method Blanks	No target analyte concentrations > RDL
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	Must meet established Laboratory acceptance criteria*

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/bias (contamination)	Field Blanks/Trip Blanks	No target analyte concentrations > RDL
Overall Precision	Field Duplicates	Waters: RPD ≤ 50% when VOCs are detected in both samples ≥ 5 times RDL Soils/LNAPL : RPD ≤ 100% when VOCs are detected in both samples ≥ 5 times RDL
Completeness	See Worksheet #34	See Worksheet #34

[i]

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Group or Method: DBCP & EDB/SW-846 8011
SOP: NC-GC-040, Rev. 2 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (laboratory)	Initial Calibration Curve (ICAL)	%RSD must be met per SW-846 Method 8011; COD (R^2) > 0.99 for linear or quadratic curves, if used
Analytical Accuracy/Bias (laboratory)	Continuing Calibration Verification (CCV) Standards	%D must be met per SW-846 Method 8011
Analytical Accuracy/Bias (laboratory)	Surrogates	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples - Second Source	Must meet established Laboratory Limits*
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates - Second Source	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Blanks (Method blanks & continuing calibration blanks)	No target analyte concentrations > RDL
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	Must meet established Laboratory acceptance criteria*
Overall accuracy/bias (contamination)	Field Blanks	No target analyte concentrations > RDL
Overall Precision	Field Duplicates	RPD ≤ 50% when analytes are detected in both samples ≥ 5 times RDL
Analytical Precision (laboratory)	Dual Column Results	RPD ≤ 40% between primary and confirmation columns
Completeness	See Worksheet #34	See Worksheet #34

[i]

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater/Soil/LNAPL
Analytical Group or Method: TCL SVOC/SW-846 8270D
SOP: NC-MS-018, Rev. 8 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	DFTPP Tune	DFTPP Tune Criteria must be met per SW-846 Method 8270D
Analytical Accuracy/Bias (laboratory)	Initial Calibration (ICAL) Curves	%RSD and %D must be met per SW-846 Method 8270D; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D
Analytical Sensitivity (laboratory)	Continuing Calibration (CCAL) Standards	%D or %Drift must be met per SW-846 Method 8270D; Minimum Mean Response Factors must be met per SW-846 Method 8270D
Analytical Accuracy/Bias (laboratory)	Internal Standards	50-200% of the response of the previous continuing calibration standard
Analytical Accuracy/Bias (laboratory)	Surrogates	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples-Second Source	Must meet established Laboratory Limits*
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates - Second Source	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Method Blanks	No target analyte concentrations > RDL
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	Must meet established Laboratory acceptance criteria*[i]
Overall accuracy/bias (contamination)	Field Blanks	No target analyte concentrations > RDL

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	Waters: RPD ≤ 50% when SVOCs are detected in both samples ≥ 5 times RDL Soils/LNAPL: RPD ≤ 100% when SVOCs are detected in both samples ≥ 5 times RDL
Completeness	See Worksheet #34	See Worksheet #34

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Group or Method: TCL SVOC/SW-846 8270D-SIM
SOP: ED-MSS-009, Rev. 7 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	DFTPP Tune	DFTPP Tune Criteria must be met per SW-846 Method 8270D-SIM
Analytical Accuracy/Bias (laboratory)	Initial Calibration (ICAL) Curve	%RSD and %D must be met per SW-846 Method 8270D-SIM; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D-SIM
Analytical Sensitivity (laboratory)	Continuing Calibration (CCAL) Standards	%D or %Drift must be met per SW-846 Method 8270D-SIM; Minimum Mean Response Factors must be met per SW-846 Method 8270D-SIM
Analytical Accuracy/Bias (laboratory)	Internal Standards	50-200% of the response of the previous continuing calibration standard
Analytical Accuracy/Bias (laboratory)	Surrogates	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples-Second Source	Must meet established Laboratory Limits*
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates - Second Source	Must meet established Laboratory Limits*
Analytical Accuracy/Bias (laboratory)	Laboratory Method Blanks	No target analyte concentrations > RDL
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	Must meet established Laboratory acceptance criteria*[i]
Overall accuracy/bias (contamination)	Field Blanks	No target analyte concentrations > RDL

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	RPD ≤ 50% when SVOCs are detected in both samples ≥ 5 times RDL
Completeness	See Worksheet #34	See Worksheet #34

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater/Soil/LNAPL
Analytical Group or Method: TAL Metals/SW-846 6010
SOP: NC-MT-012, Rev. 9 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (laboratory)	Initial Calibration Curve Standards	Initial Calibration Criteria must be met per SW-846 Method 6010 Minimum R $\geq$ 0.995
Analytical Accuracy/Bias (laboratory)	Initial Calibration Verification (ICV)-Second Source	Analyze at the beginning of each analytical run; 90-110% Recovery
Analytical Accuracy/Bias (laboratory)	Initial calibration blank (ICB)	Analyze immediately after ICV; No target analyte concentrations > RDL
Analytical Sensitivity (laboratory)	Low Level Continuing Calibration Verification (CCV) Standard	Analyze at the beginning of each analytical run; 80-120% Recovery
Analytical Accuracy/Bias (laboratory)	Interference Check Solution Analysis (ICSAB)	Analyze at the beginning of each analytical run; 80-120% Recovery
Analytical Accuracy/Bias (laboratory)	Interference Check Solution Analysis (ICSA)	Analyze at the beginning of each analytical run; results for the non-interfering elements with reporting limits $\leq$ 10 ug/L must fall within $\pm$ 2 times the RL from zero. ICSA results for the noninterfering elements with RLs > 10 ug/L must fall within $\pm$ RL from zero.
Analytical Sensitivity (laboratory)	Continuing Calibration Verification (CCV) Standard	Analyze every 10 samples; 90-110% Recovery
Analytical Accuracy/Bias (laboratory)	Continuing Calibration blank (CCB)	Analyze every 10 samples; No target analyte concentrations > RDL
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples-Second Source	80-120% Recovery
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates-Second Source	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Method Blank	No target analyte concentrations > RDL

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	75-125% recovery; RPD ≤ 20% for results > 5 times RDL
Analytical Accuracy/Bias (matrix interference)	Laboratory Duplicate	RPD ≤ 20% for results > 5 times RDL
Analytical Accuracy/Bias (matrix interference)	Serial Dilution	%D ≤ 10% (20% for 6010D) for analyte concentrations > 10 times RDL
Overall accuracy/bias (contamination)	Field Blanks	No target analyte concentrations > RDL
Overall Precision	Field Duplicates	Waters: RPD ≤ 50% when metals are detected in both samples ≥ 5 times RDL Soils/LNAPL: RPD ≤ 100% when metals are detected in both samples ≥ 5 times RDL
Completeness	See Worksheet #34	See Worksheet #34

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater/Soil/LNAPL
Analytical Group or Method: Mercury/SW-846 7470A/SW-846 7471
SOP: NC-MT-014, Rev. 9 (Appendix 1)
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (laboratory)	Initial Calibration Standards	Initial Calibration Criteria must be met per SW-846 Method 7470A Minimum R ≥ 0.995
Analytical Sensitivity (laboratory)	Initial Calibration Verification (ICV) Standard-Second Source	Analyze at the beginning of each analytical run; 90-110% Recovery
Analytical Accuracy/Bias (laboratory)	Initial calibration blank (ICB)	Analyze immediately after ICV; No target analyte concentrations > RDL
Analytical Sensitivity (laboratory)	Detection Limit Standard (CRA)	50-150% Recovery
Analytical Sensitivity (laboratory)	Continuing Calibration Verification (CCV) Standard	Analyze every 10 samples; 80-120% Recovery
Analytical Accuracy/Bias (laboratory)	Continuing Calibration Blank (CCB)	Analyze every 10 samples; No target analyte concentrations > RDL
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples-Second Source	80-120% Recovery
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates-Second Source	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Method Blank	No target analyte concentrations > RDL
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates	75-125% recovery; Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy/Bias (matrix interference)	Laboratory Duplicate	Waters: RPD ≤ 20% for results > 5 times RDL Soils/LNAPL: RPD ≤ 35% for results > 5 times RDL
Overall accuracy/bias (contamination)	Field Blanks	No target analyte concentrations > RDL
Overall Precision	Field Duplicates	Waters: RPD ≤ 50% when metals are detected in both samples ≥ 5 times RDL Soils/LNAPL: RPD ≤ 100% when metals are detected in both samples ≥ 5 times RDL
Completeness	See Worksheet #34	See Worksheet #34

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: LNAPL
Analytical Method: SW-846 Method 8260C
SOP: NC-MS-019, Rev. 6 (Appendix 1)
Concentration level (if applicable): Low

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
71-55-6	1,1,1-Trichloroethane	NA	NA	1000	1000	21.0
79-34-5	1,1,2,2-Tetrachloroethane	NA	NA	1000	1000	8.90
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	NA	NA	2000	2000	39.0
79-00-5	1,1,2-Trichloroethane	NA	NA	1000	1000	12.0
75-34-3	1,1-Dichloroethane	NA	NA	1000	1000	17.0
75-35-4	1,1-Dichloroethene	NA	NA	1000	1000	18.0
120-82-1	1,2,4-Trichlorobenzene	NA	NA	1000	1000	7.30
96-12-8	1,2-Dibromo-3-chloropropane	NA	NA	1000	1000	50.0
106-93-4	1,2-Dibromoethane	NA	NA	1000	1000	10.0
95-50-1	1,2-Dichlorobenzene	NA	NA	1000	1000	8.60
107-06-2	1,2-Dichloroethane	NA	NA	1000	1000	10.0
78-87-5	1,2-Dichloropropane	NA	NA	1000	1000	8.20
541-73-1	1,3-Dichlorobenzene	NA	NA	1000	1000	4.80

NOTES:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
106-46-7	1,4-Dichlorobenzene	NA	NA	1000	1000	8.00
78-93-3	2-Butanone	NA	NA	4000	4000	43.0
591-78-6	2-Hexanone	NA	NA	4000	4000	20.0
108-10-1	4-Methyl-2-pentanone	NA	NA	4000	4000	48.0
67-64-1	Acetone	NA	NA	4000	4000	170
71-43-2	Benzene	NA	NA	4000	4000	12.0
75-27-4	Bromodichloromethane	NA	NA	1000	1000	9.90
75-25-2	Bromoform	NA	NA	1000	1000	19.0
74-83-9	Bromomethane	NA	NA	2000	2000	29.0
75-15-0	Carbon disulfide	NA	NA	1000	1000	12.0
56-23-5	Carbon tetrachloride	NA	NA	1000	1000	6.40
108-90-7	Chlorobenzene	NA	NA	1000	1000	6.40
75-00-3	Chloroethane	NA	NA	2000	2000	61.0
67-66-3	Chloroform	NA	NA	1000	1000	8.80
74-87-3	Chloromethane	NA	NA	2000	2000	14.0
156-59-2	cis-1,2-Dichloroethene	NA	NA	1000	1000	6.90
10061-01-5	cis-1,3-Dichloropropene	NA	NA	1000	1000	7.90
110-82-7	Cyclohexane	NA	NA	2000	2000	40.0
124-48-1	Dibromochloromethane	NA	NA	1000	1000	12.0
75-71-8	Dichlorodifluoromethane	NA	NA	2000	2000	16.0
100-41-4	Ethylbenzene	NA	NA	1000	1000	5.40
98-82-8	Isopropylbenzene	NA	NA	1000	1000	6.50
79-20-9	Methyl Acetate	NA	NA	5000	5000	25.0
1634-04-4	Methyl tert-Butyl Ether	NA	NA	4000	4000	7.10
108-87-2	Methylcyclohexane	NA	NA	1000	1000	12.0
75-09-2	Methylene chloride	NA	NA	1000	1000	77.0
100-42-5	Styrene	NA	NA	1000	1000	5.60
127-18-4	Tetrachloroethene	NA	NA	1000	1000	12.0
108-88-3	Toluene	NA	NA	1000	1000	17.0

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
156-60-5	trans-1,2-Dichloroethene	NA	NA	1000	1000	9.20
10061-02-6	trans-1,3-Dichloropropene	NA	NA	1000	1000	20.0
79-01-6	Trichloroethene	NA	NA	1000	1000	9.70
75-69-4	Trichlorofluoromethane	NA	NA	2000	2000	16.0
75-01-4	Vinyl chloride	NA	NA	2000	2000	18.0
1330-20-7	Total Xylenes	NA	NA	2000	2000	6.20

Notes:

A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Method: SW-846 Method 8260C
SOP: NC-MS-019, Rev. 6 (Appendix 1)
Concentration level (if applicable): Low

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
71-55-6	1,1,1-Trichloroethane	640000	B	5.00	5.00	0.820
79-34-5	1,1,2,2-Tetrachloroethane	8400	B	5.00	5.00	1.43
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	910000	B	5.00	5.00	1.28
79-00-5	1,1,2-Trichloroethane	2100	B	5.00	5.00	1.13
75-34-3	1,1-Dichloroethane	50000	B	5.00	5.00	0.693
75-35-4	1,1-Dichloroethene	320000	B	5.00	5.00	0.903
120-82-1	1,2,4-Trichlorobenzene	81000	B	5.00	5.00	0.572
96-12-8	1,2-Dibromo-3-chloropropane	74	B	10.0	10.0	3.61
106-93-4	1,2-Dibromoethane	500	B	5.00	5.00	0.770
95-50-1	1,2-Dichlorobenzene	380000	B	5.00	5.00	1.11
107-06-2	1,2-Dichloroethane	6400	B	5.00	5.00	0.772
78-87-5	1,2-Dichloropropane	22000	B	5.00	5.00	0.851
541-73-1	1,3-Dichlorobenzene	NA	NA	5.00	5.00	0.816

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
106-46-7	1,4-Dichlorobenzene	36000	B	5.00	5.00	0.882
78-93-3	2-Butanone	28000000	B	20.0	20.0	3.56
591-78-6	2-Hexanone	280000	B	20.0	20.0	4.08
108-10-1	4-Methyl-2-pentanone	3400000	B	20.0	20.0	3.71
67-64-1	Acetone	85000000	B	25.0	25.0	21.0
71-43-2	Benzene	17000	B	5.00	5.00	0.698
75-27-4	Bromodichloromethane	4100	B	5.00	5.00	0.679
75-25-2	Bromoform	270000	B	5.00	5.00	2.40
74-83-9	Bromomethane	9500	B	5.00	5.00	0.988
75-15-0	Carbon disulfide	740000	B	5.00	5.00	1.16
56-23-5	Carbon tetrachloride	9100	B	5.00	5.00	3.25
108-90-7	Chlorobenzene	390000	B	5.00	5.00	0.916
75-00-3	Chloroethane	2100000	B	5.00	5.00	1.22
67-66-3	Chloroform	4500	B	5.00	5.00	0.788
74-87-3	Chloromethane	150000	B	5.00	5.00	1.04
156-59-2	cis-1,2-Dichloroethene	220000	B	5.00	5.00	0.651
10061-01-5	cis-1,3-Dichloropropene	25000	B	5.00	5.00	1.44
110-82-7	Cyclohexane	280000	B	10.0	10.0	1.38
124-48-1	Dibromochloromethane	120000	B	5.00	5.00	2.78
75-71-8	Dichlorodifluoromethane	120000	B	5.00	5.00	0.943
100-41-4	Ethylbenzene	81000	B	5.00	5.00	1.05
98-82-8	Isopropylbenzene	270000	B	5.00	5.00	0.832
79-20-9	Methyl Acetate	29000000	B	25.0	25.0	3.40
1634-04-4	Methyl tert-Butyl Ether	660000	B	5.00	5.00	0.820
108-87-2	Methylcyclohexane	NA	NA	10.0	10.0	1.23
75-09-2	Methylene chloride	490000	B	25.0	25.0	12.0
100-42-5	Styrene	870000	B	5.00	5.00	1.16
127-18-4	Tetrachloroethene	110000	B	5.00	5.00	0.730
108-88-3	Toluene	820000	B	5.00	5.00	0.773

CAS Number	Analyte	Project Action Limit (PAL) (µg/kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
156-60-5	trans-1,2-Dichloroethene	1900000	B	5.00	5.00	0.465
10061-02-6	trans-1,3-Dichloropropene	25000	B	5.00	5.00	1.03
79-01-6	Trichloroethene	5700	B	5.00	5.00	0.633
75-69-4	Trichlorofluoromethane	1200000	B	5.00	5.00	1.08
75-01-4	Vinyl chloride	830	B	5.00	5.00	0.837
1330-20-7	Total Xylenes	260000	B	10.0	10.0	1.59

Notes:

A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 8260C
SOP: NC-MS-019, Rev. 6 (Appendix 1)
Concentration level (if applicable): Low

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
71-55-6	1,1,1-Trichloroethane	200	A	1.00	1.00	0.240
79-34-5	1,1,2,2-Tetrachloroethane	0.76	C	1.00	1.00	0.130
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	10000	C	1.00	1.00	0.410
79-00-5	1,1,2-Trichloroethane	5	A	1.00	1.00	0.0900
75-34-3	1,1-Dichloroethane	28	C	1.00	1.00	0.170
75-35-4	1,1-Dichloroethene	7	A	1.00	1.00	0.190
120-82-1	1,2,4-Trichlorobenzene	70	A	1.00	1.00	0.260
95-50-1	1,2-Dichlorobenzene	600	A	1.00	1.00	0.150
107-06-2	1,2-Dichloroethane	5	A	1.00	1.00	0.210
78-87-5	1,2-Dichloropropane	5	A	1.00	1.00	0.150
541-73-1	1,3-Dichlorobenzene	NA	NA	1.00	1.00	0.150
106-46-7	1,4-Dichlorobenzene	75	A	1.00	1.00	0.160
78-93-3	2-Butanone	5600	C	10.0	10.0	1.16

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
591-78-6	2-Hexanone	38	C	10.0	10.0	0.540
108-10-1	4-Methyl-2-pentanone	6300	C	10.0	10.0	0.420
67-64-1	Acetone	14000	C	10.0	10.0	5.41
71-43-2	Benzene	5	A	1.00	1.00	0.130
75-27-4	Bromodichloromethane	80	A	1.00	1.00	0.170
75-25-2	Bromoform	80	A	1.00	1.00	0.760
74-83-9	Bromomethane	7.5	C	1.00	1.00	0.420
75-15-0	Carbon disulfide	810	C	1.00	1.00	0.280
56-23-5	Carbon tetrachloride	5	A	1.00	1.00	0.260
108-90-7	Chlorobenzene	100	A	1.00	1.00	0.140
75-00-3	Chloroethane	21000	C	1.00	1.00	0.830
67-66-3	Chloroform	80	A	1.00	1.00	0.130
74-87-3	Chloromethane	190	C	1.00	1.00	0.200
156-59-2	cis-1,2-Dichloroethene	70	A	1.00	1.00	0.160
10061-01-5	cis-1,3-Dichloropropene	4.7	C	1.00	1.00	0.610
110-82-7	Cyclohexane	70	C	1.00	1.00	0.240
124-48-1	Dibromochloromethane	80	A	1.00	1.00	0.390
75-71-8	Dichlorodifluoromethane	200	C	1.00	1.00	0.350
100-41-4	Ethylbenzene	700	A	1.00	1.00	0.110
98-82-8	Isopropylbenzene	450	C	1.00	1.00	0.0900
79-20-9	Methyl Acetate	20000	C	10.0	10.0	1.72
1634-04-4	Methyl tert-Butyl Ether	140	C	1.00	1.00	0.0700
108-87-2	Methylcyclohexane	NA	NA	1.00	1.00	0.330
75-09-2	Methylene chloride	5	A	1.00	1.00	2.62
100-42-5	Styrene	100	A	1.00	1.00	0.100
127-18-4	Tetrachloroethene	5	A	1.00	1.00	0.150
108-88-3	Toluene	1000	A	1.00	1.00	0.140
156-60-5	trans-1,2-Dichloroethene	100	A	1.00	1.00	0.190

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
10061-02-6	trans-1,3-Dichloropropene	4.7	C	1.00	1.00	0.670
79-01-6	Trichloroethene	5	A	1.00	1.00	0.100
75-69-4	Trichlorofluoromethane	5200	C	1.00	1.00	0.450
75-01-4	Vinyl chloride	2	A	1.00	1.00	0.200
1330-20-7	Total Xylenes	10000	A	2.00	2.00	0.150

Notes:

A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018

B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6

C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6

[1] Reporting Detection Limit

[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 8011
SOP: NC-GC-040, Rev. 2 (Appendix 1)
Concentration level (if applicable): Low

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
96-12-8	1,2-Dibromo-3-chloropropane	0.2	A	0.02	0.02	0.00300
106-93-4	1,2-Dibromoethane	0.05	A	0.02	0.02	0.00600

[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: LNAPL
Analytical Method: SW-846 Method 8270D
SOP: NC-MS-018, Rev. 8 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
92-52-4	1,1'-Biphenyl	NA	NA	20000	20000	432
123-91-1	1,4-Dioxane	NA	NA	20000	20000	780
108-60-1	bis(2-Chloroisopropyl)ether	NA	NA	20000	20000	312
95-95-4	2,4,5-Trichlorophenol	NA	NA	20000	20000	294
88-06-2	2,4,6-Trichlorophenol	NA	NA	20000	20000	408
120-83-2	2,4-Dichlorophenol	NA	NA	20000	20000	318
105-67-9	2,4-Dimethylphenol	NA	NA	20000	20000	408
51-28-5	2,4-Dinitrophenol	NA	NA	96000	96000	2460
121-14-2	2,4-Dinitrotoluene	NA	NA	20000	20000	342
606-20-2	2,6-Dinitrotoluene	NA	NA	20000	20000	348
91-58-7	2-Chloronaphthalene	NA	NA	20000	20000	378
95-57-8	2-Chlorophenol	NA	NA	20000	20000	216
91-57-6	2-Methylnaphthalene	NA	NA	20000	20000	59.4

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
95-48-7	2-Methylphenol	NA	NA	20000	20000	402
88-74-4	2-Nitroaniline	NA	NA	96000	96000	306
88-75-5	2-Nitrophenol	NA	NA	20000	20000	204
91-94-1	3,3'-Dichlorobenzidine	NA	NA	96000	96000	294
99-09-2	3-Nitroaniline	NA	NA	96000	96000	192
534-52-1	4,6-Dinitro-2-methylphenol	NA	NA	96000	96000	2820
101-55-3	4-Bromophenyl-phenylether	NA	NA	20000	20000	282
59-50-7	4-Chloro-3-methylphenol	NA	NA	20000	20000	3060
106-47-8	4-Chloroaniline	NA	NA	20000	20000	312
7005-72-3	4-Chlorophenyl phenyl ether	NA	NA	20000	20000	216
108-39-4; 106-44-5	3 & 4-Methylphenol	NA	NA	40000	40000	1200
100-01-6	4-Nitroaniline	NA	NA	96000	96000	216
100-02-7	4-Nitrophenol	NA	NA	96000	96000	4860
83-32-9	Acenaphthene	NA	NA	20000	20000	55.2
208-96-8	Acenaphthylene	NA	NA	20000	20000	84.0
98-86-2	Acetophenone	NA	NA	20000	20000	330
120-12-7	Anthracene	NA	NA	20000	20000	138
1912-24-9	Atrazine	NA	NA	20000	20000	660
100-52-7	Benzaldehyde	NA	NA	20000	20000	414
56-55-3	Benzo[a]anthracene	NA	NA	20000	20000	84.0
50-32-8	Benzo[a]pyrene	NA	NA	20000	20000	138
205-99-2	Benzo[b]fluoranthene	NA	NA	20000	20000	138
191-24-2	Benzo[ghi]perylene	NA	NA	20000	20000	108
207-08-9	Benzo[k]fluoranthene	NA	NA	20000	20000	138
111-91-1	Bis(2-chloroethoxy)methane	NA	NA	20000	20000	960
111-44-4	Bis(2-chloroethyl)ether	NA	NA	20000	20000	246

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
117-81-7	Bis(2-ethylhexyl) phthalate	NA	NA	20000	20000	960
85-68-7	Butyl benzyl phthalate	NA	NA	20000	20000	222
105-60-2	Caprolactam	NA	NA	20000	20000	468
86-74-8	Carbazole	NA	NA	20000	20000	558
218-01-9	Chrysene	NA	NA	20000	20000	59.4
53-70-3	Dibenz[a,h]anthracene	NA	NA	20000	20000	90.0
132-64-9	Dibenzofuran	NA	NA	20000	20000	49.8
84-66-2	Diethyl phthalate	NA	NA	20000	20000	372
131-11-3	Dimethyl phthalate	NA	NA	20000	20000	384
84-74-2	Di-n-butyl phthalate	NA	NA	20000	20000	300
117-84-0	Di-n-octyl phthalate	NA	NA	20000	20000	660
206-44-0	Fluoranthene	NA	NA	20000	20000	55.8
86-73-7	Fluorene	NA	NA	20000	20000	78.0
118-74-1	Hexachlorobenzene	NA	NA	20000	20000	84.0
87-68-3	Hexachlorobutadiene	NA	NA	20000	20000	150
77-47-4	Hexachlorocyclopentadiene	NA	NA	96000	96000	174
67-72-1	Hexachloroethane	NA	NA	20000	20000	294
193-39-5	Indeno(1,2,3 cd)pyrene	NA	NA	20000	20000	120
78-59-1	Isophorone	NA	NA	20000	20000	210
91-20-3	Naphthalene	NA	NA	20000	20000	456
98-95-3	Nitrobenzene	NA	NA	20000	20000	246
621-64-7	n-Nitroso-di-n-propylamine	NA	NA	20000	20000	53.4
86-30-6	n-Nitrosodiphenylamine	NA	NA	20000	20000	384
87-86-5	Pentachlorophenol	NA	NA	20000	20000	2700

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
85-01-8	Phenanthrene	NA	NA	20000	20000	66.0
108-95-2	Phenol	NA	NA	20000	20000	342
129-00-0	Pyrene	NA	NA	20000	20000	60.0

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Method: SW-846 Method 8270D
SOP: NC-MS-018, Rev. 8 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
92-52-4	1,1'-Biphenyl	66000	B	50.0	50.0	17.0
123-91-1	1,4-Dioxane	74000	B	150	150	15.0
108-60-1	bis(2-Chloroisopropyl)ether	1000000	B	100	100	10.0
95-95-4	2,4,5-Trichlorophenol	8800000	B	150	150	69.0
88-06-2	2,4,6-Trichlorophenol	88000	B	150	150	64.0
120-83-2	2,4-Dichlorophenol	270000	B	150	150	44.0
105-67-9	2,4-Dimethylphenol	1800000	B	150	150	40.0
51-28-5	2,4-Dinitrophenol	180000	B	330	330	142
121-14-2	2,4-Dinitrotoluene	24000	B	200	200	62.0
606-20-2	2,6-Dinitrotoluene	5000	B	200	200	56.0
91-58-7	2-Chloronaphthalene	6700000	B	50.0	50.0	14.0
95-57-8	2-Chlorophenol	550000	B	50.0	50.0	10.0
91-57-6	2-Methylnaphthalene	3430000	B	15.0	15.0	1.96
95-48-7	2-Methylphenol	4500000	B	200	200	31.0

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
88-74-4	2-Nitroaniline	880000	B	200	200	40.0
88-75-5	2-Nitrophenol	NA	NA	50.0	50.0	13.0
91-94-1	3,3'-Dichlorobenzidine	17000	B	100	100	43.0
99-09-2	3-Nitroaniline	NA	NA	200	200	49.0
534-52-1	4,6-Dinitro-2-methylphenol	7100	B	330	330	80.0
101-55-3	4-Bromophenyl-phenylether	NA	NA	50.0	50.0	14.0
59-50-7	4-Chloro-3-methylphenol	8800000	B	150	150	45.0
106-47-8	4-Chloroaniline	38000	B	150	150	30.0
7005-72-3	4-Chlorophenyl phenyl ether	NA	NA	50.0	50.0	14.0
108-39-4; 106-44-5	3 & 4-Methylphenol	4500000	B	400	400	29.0
100-01-6	4-Nitroaniline	350000	B	200	200	60.0
100-02-7	4-Nitrophenol	NA	NA	330	330	94.0
83-32-9	Acenaphthene	5000000	B	15.0	15.0	2.86
208-96-8	Acenaphthylene	NA	NA	15.0	15.0	4.01
98-86-2	Acetophenone	2500000	B	100	100	11.0
120-12-7	Anthracene	25000000	B	15.0	15.0	2.41
1912-24-9	Atrazine	34000	B	200	200	36.0
100-52-7	Benzaldehyde	1200000	B	100	100	23.0
56-55-3	Benzo[a]anthracene	15000	B	15.0	15.0	3.41
50-32-8	Benzo[a]pyrene	1500	B	15.0	15.0	9.34
205-99-2	Benzo[b]fluoranthene	15000	B	15.0	15.0	6.50
191-24-2	Benzo[ghi]perylene	NA	NA	15.0	15.0	7.10
207-08-9	Benzo[k]fluoranthene	150000	B	15.0	15.0	6.93
111-91-1	Bis(2-chloroethoxy)methane	270000	B	100	100	12.0

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
111-44-4	Bis(2-chloroethyl)ether	3200	B	100	100	12.0
117-81-7	Bis(2-ethylhexyl) phthalate	550000	B	70.0	70.0	51.0
85-68-7	Butyl benzyl phthalate	4100000	B	70.0	70.0	22.0
105-60-2	Caprolactam	43000000	B	330	330	75.0
86-74-8	Carbazole	NA	NA	50.0	50.0	19.0
218-01-9	Chrysene	1500000	B	15.0	15.0	1.49
53-70-3	Dibenz[a,h]anthracene	1500	B	15.0	15.0	6.92
132-64-9	Dibenzofuran	100000	B	50.0	50.0	13.0
84-66-2	Diethyl phthalate	71000000	B	70.0	70.0	31.0
131-11-3	Dimethyl phthalate	NA	NA	70.0	70.0	14.0
84-74-2	Di-n-butyl phthalate	NA	NA	70.0	70.0	22.0
117-84-0	Di-n-octyl phthalate	NA	NA	70.0	70.0	28.0
206-44-0	Fluoranthene	3400000	B	15.0	15.0	4.45
86-73-7	Fluorene	3400000	B	15.0	15.0	2.74
118-74-1	Hexachlorobenzene	2900	B	15.0	15.0	2.85
87-68-3	Hexachlorobutadiene	17000	B	50.0	50.0	12.0
77-47-4	Hexachlorocyclopentadiene	2500	B	330	330	62.0
67-72-1	Hexachloroethane	25000	B	50.0	50.0	9.00
193-39-5	Indeno(1,2,3 cd)pyrene	15000	B	15.0	15.0	7.36
78-59-1	Isophorone	8000000	B	50.0	50.0	12.0
91-20-3	Naphthalene	53000	B	15.0	15.0	2.41
98-95-3	Nitrobenzene	71000	B	100	100	13.0
621-64-7	n-Nitroso-di-n-propylamine	1100	B	50.0	50.0	11.0
86-30-6	n-Nitrosodiphenylamine	1500000	B	50.0	50.0	12.0
87-86-5	Pentachlorophenol	14000	B	150	150	58.0

CAS Number	Analyte	Project Action Limit (PAL) (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory-RDL[1] (µg/Kg)	Laboratory-MDL[2] (µg/Kg)
85-01-8	Phenanthrene	NA	NA	15.0	15.0	2.23
108-95-2	Phenol	27000000	B	50.0	50.0	8.00
129-00-0	Pyrene	2500000	B	15.0	15.0	2.14

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 8270D
SOP: NC-MS-018, Rev. 8 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
92-52-4	1,1'-Biphenyl	0.83	C	1.00	1.00	0.492
108-60-1	bis(2-Chloroisopropyl)ether	710	C	1.00	1.00	0.551
95-95-4	2,4,5-Trichlorophenol	1200	C	5.00	5.00	1.99
88-06-2	2,4,6-Trichlorophenol	12	C	5.00	5.00	1.80
120-83-2	2,4-Dichlorophenol	46	C	2.00	2.00	0.262
105-67-9	2,4-Dimethylphenol	360	C	2.00	2.00	0.518
51-28-5	2,4-Dinitrophenol	39	C	10.0	10.0	6.21
121-14-2	2,4-Dinitrotoluene	2.4	C	5.00	5.00	2.07
606-20-2	2,6-Dinitrotoluene	0.49	C	5.00	5.00	2.13
91-58-7	2-Chloronaphthalene	750	C	1.00	1.00	0.483
95-57-8	2-Chlorophenol	91	C	1.00	1.00	0.273
91-57-6	2-Methylnaphthalene	36	C	0.200	0.200	0.111
95-48-7	2-Methylphenol	930	C	1.00	1.00	0.209
88-74-4	2-Nitroaniline	190	C	2.00	2.00	0.510

Notes:
A - MCLs
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
88-75-5	2-Nitrophenol	NA	NA	2.00	2.00	0.564
91-94-1	3,3'-Dichlorobenzidine	1.3	C	5.00	5.00	1.15
99-09-2	3-Nitroaniline	NA	NA	2.00	2.00	0.566
534-52-1	4,6-Dinitro-2-methylphenol	1.5	C	5.00	5.00	2.82
101-55-3	4-Bromophenyl-phenylether	NA	NA	2.00	2.00	0.499
59-50-7	4-Chloro-3-methylphenol	1400	C	2.00	2.00	0.296
106-47-8	4-Chloroaniline	3.7	C	2.00	2.00	0.316
7005-72-3	4-Chlorophenyl phenyl ether	NA	NA	2.00	2.00	0.551
108-39-4; 106-44-5	3 & 4-Methylphenol	930	C	2.00	2.00	
100-01-6	4-Nitroaniline	38	C	2.00	2.00	0.917
100-02-7	4-Nitrophenol	NA	NA	10.0	10.0	2.17
83-32-9	Acenaphthene	530	C	0.200	0.200	0.172
208-96-8	Acenaphthylene	NA	NA	0.200	0.200	0.125
98-86-2	Acetophenone	1900	C	1.00	1.00	0.366
120-12-7	Anthracene	1800	C	0.200	0.200	0.135
1912-24-9	Atrazine	3	C	2.00	2.00	0.952
100-52-7	Benzaldehyde	190	C	2.00	2.00	0.759
56-55-3	Benzo[a]anthracene	0.3	C	0.200	0.200	0.171
50-32-8	Benzo[a]pyrene	0.2	A	0.200	0.200	0.173
205-99-2	Benzo[b]fluoranthene	2.5	C	0.200	0.200	0.154
191-24-2	Benzo[ghi]perylene	NA	NA	0.200	0.200	0.178
207-08-9	Benzo[k]fluoranthene	25	C	0.200	0.200	0.140
111-91-1	Bis(2-chloroethoxy)methane	59	C	1.00	1.00	0.455

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
111-44-4	Bis(2-chloroethyl)ether	0.14	C	1.00	1.00	0.402
117-81-7	Bis(2-ethylhexyl) phthalate	6	A	5.00	5.00	2.22
85-68-7	Butyl benzyl phthalate	160	C	2.00	2.00	0.666
105-60-2	Caprolactam	9900	C	5.00	5.00	0.934
86-74-8	Carbazole	NA	NA	1.00	1.00	0.490
218-01-9	Chrysene	250	C	0.200	0.200	0.186
53-70-3	Dibenz[a,h]anthracene	0.25	C	0.200	0.200	0.151
132-64-9	Dibenzofuran	7.9	C	1.00	1.00	0.561
84-66-2	Diethyl phthalate	15000	C	5.00	5.00	3.82
131-11-3	Dimethyl phthalate	NA	NA	2.00	2.00	0.515
84-74-2	Di-n-butyl phthalate	NA	NA	5.00	5.00	1.80
117-84-0	Di-n-octyl phthalate	NA	NA	2.00	2.00	0.821
206-44-0	Fluoranthene	800	C	0.200	0.200	0.160
86-73-7	Fluorene	290	C	0.200	0.200	0.169
118-74-1	Hexachlorobenzene	1	A	0.200	0.200	0.161
87-68-3	Hexachlorobutadiene	1.4	C	1.00	1.00	0.543
77-47-4	Hexachlorocyclopentadiene	50	A	10.0	10.0	1.76
67-72-1	Hexachloroethane	3.3	C	1.00	1.00	0.395
193-39-5	Indeno(1,2,3 cd)pyrene	2.5	C	0.200	0.200	0.135
78-59-1	Isophorone	780	C	1.00	1.00	0.324
91-20-3	Naphthalene	1.7	C	0.200	0.200	0.109
98-95-3	Nitrobenzene	1.4	C	1.00	1.00	0.514
621-64-7	n-Nitroso-di-n-propylamine	0.11	C	1.00	1.00	0.253
86-30-6	n-Nitrosodiphenylamine	120	C	1.00	1.00	0.440
87-86-5	Pentachlorophenol	1	A	10.0	10.0	3.10

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
85-01-8	Phenanthrene	NA	NA	0.200	0.200	0.167
108-95-2	Phenol	5800	C	1.00	1.00	0.128
129-00-0	Pyrene	120	C	0.200	0.200	0.175

Notes:
A - MCLs
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 8270D-SIM
SOP: ED-MSS-009, Rev. 7 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
123-91-1	1,4-Dioxane (by SIM)	4.6	C	0.400	0.400	0.174

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: LNAPL
Analytical Method: SW-846 Method 6010
SOP: NC-MT-012, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7429-90-5	Aluminum	NA	NA	20.0	20.0	5.33
7440-36-0	Antimony	NA	NA	2.00	2.00	0.359
7440-38-2	Arsenic	NA	NA	1.50	1.50	0.316
7440-39-3	Barium	NA	NA	20.0	20.0	0.362
7440-41-7	Beryllium	NA	NA	0.500	0.500	0.0540
7440-43-9	Cadmium	NA	NA	0.500	0.500	0.0480
7440-70-2	Calcium	NA	NA	500	500	36.5
7440-47-3	Chromium	NA	NA	1.00	1.00	0.151
7440-48-4	Cobalt	NA	NA	1.00	1.00	0.200
7440-50-8	Copper	NA	NA	2.50	2.50	0.236
7439-89-6	Iron	NA	NA	20.0	20.0	6.94
7439-92-1	Lead	NA	NA	1.00	1.00	0.282
7439-95-4	Magnesium	NA	NA	500	500	46.1
7439-96-5	Manganese	NA	NA	1.50	1.50	0.309
7440-02-0	Nickel	NA	NA	4.00	4.00	0.233
7440-09-7	Potassium	NA	NA	500	500	36.1

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7782-49-2	Selenium	NA	NA	2.00	2.00	0.469
7440-22-4	Silver	NA	NA	1.00	1.00	0.0810
7440-23-5	Sodium	NA	NA	500	500	62.8
7440-28-0	Thallium	NA	NA	2.00	2.00	0.399
7440-62-2	Vanadium	NA	NA	5.00	5.00	0.822
7440-66-6	Zinc	NA	NA	5.00	5.00	1.37

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Method: SW-846 Method 6010
SOP: NC-MT-012, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7429-90-5	Aluminum	100000	B	20.0	20.0	5.33
7440-36-0	Antimony	43	B	2.00	2.00	0.359
7440-38-2	Arsenic	9.5	B	1.50	1.50	0.316
7440-39-3	Barium	21000	B	20.0	20.0	0.362
7440-41-7	Beryllium	220	B	0.500	0.500	0.0540
7440-43-9	Cadmium	99	B	0.500	0.500	0.0480
7440-70-2	Calcium	NA	NA	500	500	36.5
7440-47-3	Chromium	NA	NA	1.00	1.00	0.151
7440-48-4	Cobalt	32	B	1.00	1.00	0.200
7440-50-8	Copper	4300	B	2.50	2.50	0.236
7439-89-6	Iron	77000	B	20.0	20.0	6.94
7439-92-1	Lead	400	B	1.00	1.00	0.282
7439-95-4	Magnesium	NA	NA	500	500	46.1
7439-96-5	Manganese	2500	B	1.50	1.50	0.309
7440-02-0	Nickel	2100	B	4.00	4.00	0.233
7440-09-7	Potassium	NA	NA	500	500	36.1
7782-49-2	Selenium	550	B	2.00	2.00	0.469

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7440-22-4	Silver	550	B	1.00	1.00	0.0810
7440-23-5	Sodium	NA	NA	500	500	62.8
7440-28-0	Thallium	1.1	B	2.00	2.00	0.399
7440-62-2	Vanadium	550	B	5.00	5.00	0.822
7440-66-6	Zinc	32000	B	5.00	5.00	1.37

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 6010
SOP: NC-MT-012, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
7429-90-5	Aluminum	20000	C	200	200	47.3
7440-36-0	Antimony	6	A	20.0	20.0	7.46
7440-38-2	Arsenic	10	A	15.0	15.0	4.05
7440-39-3	Barium	2000	A	200	200	1.33
7440-41-7	Beryllium	4	A	5.00	5.00	0.601
7440-43-9	Cadmium	5	A	5.00	5.00	0.203
7440-70-2	Calcium	NA	NA	5000	5000	307
7440-47-3	Chromium	100	A	10.0	10.0	0.625
7440-48-4	Cobalt	6	C	10.0	10.0	0.752
7440-50-8	Copper	1300	A	25.0	25.0	3.55
7439-89-6	Iron	14000	C	200	200	26.0
7439-92-1	Lead	15	A	10.0	10.0	2.77
7439-95-4	Magnesium	NA	NA	5000	5000	259
7439-96-5	Manganese	430	C	15.0	15.0	2.12
7440-02-0	Nickel	390	C	40.0	40.0	2.20
7440-09-7	Potassium	NA	NA	5000	5000	557
7782-49-2	Selenium	50	A	20.0	20.0	5.96

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
7440-22-4	Silver	94	C	10.0	10.0	0.623
7440-23-5	Sodium	NA	NA	5000	5000	560
7440-28-0	Thallium	2	A	20.0	20.0	2.68
7440-62-2	Vanadium	86	C	50.0	50.0	5.56
7440-66-6	Zinc	6000	C	50.0	50.0	9.67

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample-Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: LNAPL
Analytical Method: SW-846 Method 7471
SOP: NC-MT-014, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7439-97-6	Mercury	NA	NA	0.100	0.100	0.0180

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
NA – Not Applicable
[1] Reporting Detection Limit
[2] Non Sample –Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Method: SW-846 Method 7471
SOP: NC-MT-014, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (mg/Kg)	PAL Reference	Project Quantitation Limit Goal (mg/Kg)	Laboratory-RDL[1] (mg/Kg)	Laboratory-MDL[2] (mg/Kg)
7439-97-6	Mercury	3.1	B	0.100	0.100	0.0180

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample –Specific Method Detection Limit

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Method: SW-846 Method 7470A
SOP: NC-MT-014, Rev. 9 (Appendix 1)
Concentration level (if applicable):

CAS Number	Analyte	Project Action Limit (PAL) (µg/L)	PAL Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-RDL[1] (µg/L)	Laboratory-MDL[2] (µg/L)
7439-97-6	Mercury	2	A	0.200	0.200	0.130

Notes:
A – Maximum Contaminant Level, EPA 822-F-18-001, March 2018
B - 2019 IDEM Screening & Closure Levels, Soil Exposure, Direct Contact, Residential; Table A-6
C - 2019 IDEM Screening & Closure Levels, Ground Water, Tap, Residential; Table A-6
[1] Reporting Detection Limit
[2] Non Sample –Specific Method Detection Limit

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

Scope and objectives of the investigation include characterization of the nature and extent of COPCs to characterize the horizontal and vertical delineation of COPC-impacted soil and groundwater. The RFI will be completed in a phased approach.

OXY and CITGO may coordinate efforts to increase efficiency and avoid duplication for elements of the corrective action where applicable to both parties. OXY and CITGO are proceeding under separate AOCs for the Refinery Site and CITGO Terminal respectively.

Perimeter Investigation

In May 2019 and August 2019, OXY and CITGO jointly conducted a perimeter soil and groundwater screening investigation as set forth in the Site Perimeter Screening Investigation Work Plan dated April 5, 2019 (approved by EPA). The investigation utilized real-time field screening technologies designed to qualitatively detect polycyclic aromatic hydrocarbons (PAHs) and volatile organic compounds (VOCs), using laser-induced fluorescence (LIF) and membrane interface probe (MIP).

In September 2019, OXY and CITGO (and other appropriate persons) met with EPA to present the Phase I results.

RFI Investigation

The results of the Perimeter investigation, a review of the historical operations, and physical setting of the Facility were used in the development of the scope, expectations, and objectives for the RFI Investigation. The scope was discussed and agreed upon during the September 2019 meeting between OXY and EPA.

Respondents submitted the RFI Work Plan in February, 2020 for EPA review and approval. The RFI Work Plan including sampling locations, vertical extent of sampling, density of sampling, and screening data shall be reviewed and approved by EPA consistent with the approved CAF systematic planning process.

Initial soil and groundwater analysis will include the following chemicals of potential concern (COPCs); target compound list (TCL) volatile organic compounds (VOCs) (Method 8260), TCL semi-volatile organic compounds (SVOCs) (Method 8270), target analyte list (TAL) metals (Method 6010/7470) and 1,4-dioxane. The COPCs may be reduced during subsequent phases of investigation. The investigation will also determine the nature and extent of contamination in soil or groundwater or where potentially preferential pathways for historical contamination are identified.

The RFI investigation is intended to be iterative and adaptive based on conditions encountered in the field. Pending the results of the initial investigation, additional phases may be identified during or after the quarterly monitoring program. Additional phases may include a subsequent Off-site groundwater investigation. Subsequent sampling locations, vertical extent of sampling, and density of screening data shall be reviewed and approved by EPA consistent with the approved CAF systematic planning process.

An interim RFI data report summarizing the results of the RFI Work Plan and an updated Conceptual Site Model (CSM), including proposed additional activities, if any, will be prepared for submittal to the U.S. EPA following two rounds of

quarterly groundwater sampling. Subsequent quarterly groundwater results will be included in either the quarterly progress reports or in a subsequent interim RFI data report(s) (if applicable).

Once it has been determined that sufficient data has been obtained to describe the nature and extent of any releases of hazardous waste and hazardous constituents at or from the Facility that may pose an unacceptable risk to human health and the environment, a final RFI Report will be developed and submitted to the U.S. EPA by no later than October 1, 2021 unless a revised date is agreed to by both U.S. EPA and OXY. The RFI report will describe the nature and extent of any releases of hazardous waste and hazardous constituents at or from the Facility that do or do not pose an unacceptable risk to human health and the environment, and provide the basis for those conclusions, including an evaluation of the risks. The investigation shall include a consensus driven balance between qualitative and quantitative high-resolution investigation techniques.

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Sample ID	Matrix[1]	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
Proposed Monitoring Locations are presented on Figure 3.1 See Worksheet #27 for Sample ID protocol	Groundwater	Water table	Characterize groundwater quality	TCL VOCs, TCL SVOCs, TAL Metals	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.4	If LNAPL is present, no groundwater sample will be collected from the well reporting LNAPL but from a downgradient location with no LNAPL
Proposed Monitoring Locations are presented on Figure 3.1 See Worksheet #27 for Sample ID protocol	Soil	0-2 and immediately above the water table	Characterize shallow soil quality	TCL VOCs, TCL SVOCs, TAL Metals	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.1	If the water table is less than 4-ft below ground surface only one sample will be collected.
IDW – water See Worksheet #27 for Sample ID protocol	Wastewater	NA	Characterize for disposal	TCL VOCs, TCL SVOCs, TAL Metals, PCBs, RCRA Characteristics	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 10.0	
IDW – soil See Worksheet #27 for Sample ID protocol	Soil	NA	Characterize for disposal	TCLP VOCs, TCLP SVOCs, TCLP Metals, PCBs, RCRA Characteristics	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 10.0	

[1]Key: SS = surface soil, S = soil, SD = sediment, GW = groundwater, SW = surface water

Sample ID	Matrix[1]	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
LNAPL See Worksheet #27 for Sample ID protocol	NAPL	NA	Characterize NAPL	TCL VOCs, TCL SVOCs, TAL Metals	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.5	Only if sufficient LNAPL can be recovered.

Note: Additional phases of investigation or sampling events may be required, as necessary. Depending on the results of the initial sampling event(s), additional investigation locations may be added between sampling events, where warranted. The need for further data collection will be based on the investigation results, and will be completed in consultation with the U.S. EPA. Separate work plans will be prepared for additional phases of investigation, as necessary, which will include investigation locations, vertical extent, density, methodology, COPCs, screening data, and corresponding rationale. Separate work plans will be reviewed and approved by U.S EPA consistent with the approved CAF systematic planning process.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times
(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)

Laboratory: Eurofins TestAmerica, Canton
4101 Shuffel Street NW
North Canton, Ohio 44720
Denise Heckler (denise.heckler@testamericainc.com)
Back-up Laboratory: Various Eurofins TestAmerica Network Laboratories
Sample Delivery Method: FedEx

Analyte/ Analyte Group	Matrix	Method/ SOP	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Volatile Organic Compounds (VOCs)	Water	SW-846 8260C NC-MS-019, Rev. 6 (Appendix 1)	3, 40-ml VOA vials w/ PTFE-faced silicone septum	pH < 2 with HCl; 4 ± 2°C	NA	14 days	15 BD
DBCP & EDB	Water	SW-846 8011; NC-GC-040, Rev. 2 (Appendix 1)	3, 40-ml VOA vials w/ PTFE-faced silicone septum	pH < 2 with HCl; 4 ± 2°C	NA	14 days	15 BD
Semivolatile Organic Compounds (SVOCs)	Water	SW-846 8270D; NC-MS-018, Rev. 8 (Appendix 1)	1 L amber glass w/PTFE lined lid	4 ± 2°C	7 days	40 days	15 BD
Semivolatile Organic Compounds (SVOCs)-SIM	Water	SW-846 8270D-SIM; ED-MSS-009, Rev. 7 (Appendix 1)	250 mL amber glass w/PTFE lined lid	4 ± 2°C	7 days	40 days	15 BD
TAL Metals	Water	SW-846 6010; NC-MT-012, Rev. 9 (Appendix 1)	500 ml HDPE	pH < 2 with HNO_3; 4 ± 2°C	NA	180 Days	15 BD

Analyte/ Analyte Group	Matrix	Method/ SOP	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Mercury	Water	SW-846 7470A; NC-MT-014, Rev. 9 (Appendix 1)	500 ml HDPE	pH < 2 with HNO_3; 4 ± 2°C	NA	28 days	15 BD
Volatile Organic Compounds (VOCs)	Soil	SW-846 8260C NC-MS-019, Rev. 6 (Appendix 1)	EnCore or TerraCores	4 ± 2°C	48 hours	14 days	15 BD
Semivolatile Organic Compounds (SVOCs)	Soil	SW-846 8270D; NC-MS-018, Rev. 8 (Appendix 1)	8 ounce glass jar w/PTFE lined lid	4 ± 2°C	14 days	40 days	15 BD
TAL Metals	Soil	SW-846 6010; NC-MT-012, Rev. 9 (Appendix 1)	8 ounce glass jar w/PTFE lined lid	4 ± 2°C	NA	180 days	15 BD
Mercury	Soil	SW-846 7471; NC-MT-014, Rev. 9 (Appendix 1)	8 ounce glass jar w/PTFE lined lid	4 ± 2°C	NA	28 days	15 BD
TCL VOCs	LNAPL	SW-846 8260C NC-MS-019, Rev. 6 (Appendix 1)	2 ounce glass jar w/ PTFE lined lid	4 ± 2°C	NA	14 days	15 BD
TCL SVOCs	LNAPL	SW-846 8270D; NC-MS-018, Rev. 8 (Appendix 1)	4 ounce glass jar w/ PTFE lined lid	4 ± 2°C	14 days	40 days	15 BD
TAL Metals	LNAPL	SW-846 6010; NC-MT-012, Rev. 9 (Appendix 1)	4 ounce glass jar w/ PTFE lined lid	4 ± 2°C	NA	180 days	15 BD
Mercury	LNAPL	SW-846 7471; NC-MT-014, Rev. 9 (Appendix 1)	4 ounce glass jar w/ PTFE lined lid	4 ± 2°C	NA	28 days	15 BD

QAPP Worksheet #20: Field QC Summary
(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Task/Event	Matrix	Analyte/Analytical Group	Field Samples	Field Blanks (1/10)	Field Duplicates (1/10)	Matrix Spike/Matrix Spike Duplicates (1/20)	Trip Blanks (1/cooler for VOCs)	Total # analyses
Monitoring Well installation and Quarterly Groundwater Sampling Events (4)	Groundwater	TCL VOCs	12	2	2	1	1	18
		DBCP & EDB	12	2	2	1	1	18
		TCL SVOCs	12	2	2	1	NA	17
		1,4-Dioxane-SIM	12	2	2	1	NA	17
		TAL Metals	12	2	2	1	NA	17
		Mercury	12	2	2	1	NA	17
Monitoring Well installation	Soil	TCL VOCs	24	3	3	2	NA	32
		TCL SVOCs	24	3	3	2	NA	32
		TAL Metals	24	3	3	2	NA	32
		Mercury	24	3	3	2	NA	32
Monitoring Well installation and Quarterly Groundwater Sampling Events (4)	LNAPL	TCL VOCs	TBD	TBD	TBD	TBD	TBD	TBD
		TCL SVOCs	TBD	TBD	TBD	TBD	TBD	TBD
		TAL Metals	TBD	TBD	TBD	TBD	TBD	TBD
		Mercury	TBD	TBD	TBD	TBD	TBD	TBD

Note: Additional phases of investigation or sampling events may be required, as necessary. Depending on the results of the initial sampling event(s), additional investigation locations may be added between sampling events, where warranted. Additional sample phases or events will be conducted with the same QA/QC procedures and frequency as identified on Worksheet #20. The need for further data collection will be based on the investigation results, and will be completed in consultation with the U.S. EPA. Separate work plans will be prepared for additional phases of investigation, as necessary, which will include investigation locations, vertical extent, density, methodology, COPCs, screening data, and

corresponding rationale. Separate work plans will be reviewed and approved by U.S EPA consistent with the approved CAF systematic planning process.

QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

SOP # or reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified for Project? Y/N	Comments
GHD Field Method Guidelines	Revision 1 –August 17, 2018	GHD Services, Inc.	Section 6.1: Surficial Soil Sample Collection for Laboratory Analysis	N	
GHD Field Method Guidelines	Revision 1 –August 17, 2018	GHD Services, Inc.	Section 6.4: Groundwater Sample Collection for Laboratory Analysis	N	
GHD Field Method Guidelines	Revision 1 –August 17, 2018	GHD Services, Inc.	Section 9.0: Equipment Decontamination	N	
GHD Field Method Guidelines	Revision 1 –August 17, 2018	GHD Services, Inc.	Section 10.0: Waste Characterization	N	
GHD Field Method Guidelines	Revision 1 –August 17, 2018	GHD Services, Inc.	Section 6.5 Non-Aqueous Phase Liquid (NAPL) Sample Collection for Laboratory Analysis	N	

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
YSI 3560 Water Quality Meter (pH, temperature, conductivity, ORP)	Calibration	GHD Field Method Guidelines Rev. 1 – August 17, 2018– Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
YSI 3560 Water Quality Meter (pH, temperature, conductivity, ORP)	Maintenance	Operators Manual	Equipment Coordinator	Annually	See Operators Manual	See Operators Manual
YSI 3560 Water Quality Meter (pH, temperature, conductivity, ORP)	Testing	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
YSI 3560 Water Quality Meter (pH, temperature, conductivity, ORP)	Inspection	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline 8.0

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
YSI 52 (dissolved oxygen)	Calibration	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline 0
YSI 52 (dissolved oxygen)	Maintenance	Operators Manual	Equipment Coordinator	Annually	See Operators Manual	See Operators Manual
YSI 52 (dissolved oxygen)	Testing	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
YSI 52 (dissolved oxygen)	Inspection	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
DRT-15C (turbidity)	Calibration	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
DRT-15C (turbidity)	Maintenance	Operators Manual	Equipment Coordinator	Annually	See Operators Manual	See Operators Manual
DRT-15C (turbidity)	Testing	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
DRT-15C (turbidity)	Inspection	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
PID Meter	Calibration	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
PID Meter	Maintenance	Operators Manual	Equipment Coordinator	Annually	See Operators Manual	See Operators Manual

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
PID Meter	Testing	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
PID Meter	Inspection	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline
Dual Phase Probe	Maintenance	Operators Manual	Equipment Coordinator	Annually	See Operators Manual	See Operators Manual
Dual Phase Probe	Testing	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0	Field Crew Chief	Daily	See Field Method Guideline	See Field Method Guideline

QAPP Worksheet #23: Analytical SOPs
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

Eurofins TestAmerica, Canton SOP #	Title and Date	Definitive or Screening Data	Matrix/Analytical Group	Equipment Type	‡Modified for Project? Y/N
NC-MS-019, Rev. 6 (Appendix 1)	*Determination of Volatile Organics by GC/MS Based on Methods 8260C, 8260B, and 8260A, 7/23/18*	Definitive	Water & Soil/VOCs	GC/MS	N
NC-GC-040, Rev. 2 (Appendix 1)	*Ethylene Dibromide (EDB), and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography, 1/7/19*	Definitive	Water/VOCs	GC	N
NC-MS-018, Rev. 8 (Appendix 1)	*GC/MS ANALYSIS BASED ON METHODS 8270C, 8270D, and 8270E, 7/2/19*	Definitive	Water & Soil/SVOCs	GC/MS	N
ED-MSS-009, Rev. 7 (Appendix 1)	*Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270D, 6/8/18*	Definitive	Water/SVOCs	GC/MS	N
NC-MT-012, Rev. 9 (Appendix 1)	*INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR ELEMENT ANALYSES, 7/1/19*	Definitve	Water & Soil/Metals	ICP/AES	N
NC-MT-014, Rev. 9 (Appendix 1)	*PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS AND SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, 8/5/19*	Definitive	Water & Soil/Mercury	CVAA	N

Eurofins TestAmerica, Canton SOP #	Title and Date	Definitive or Screening Data	Matrix/Analytical Group	Equipment Type	‡Modified for Project? Y/N
NC-IP-011, Rev. 8 (Appendix 1)	*ACID DIGESTION FOR AQUEOUS SAMPLES, 10/22/19*	NA	Water/Metals	Hot Plate/Hot Block	N
NC-OP-025, Rev. 9 (Appendix 1)	*CLEANUP PROCEDURES FOR ORGANIC EXTRACTABLE SAMPLES, 1/25/19*	NA	All/Organics	Various	N
NC-OP-037, Rev. 7 (Appendix 1)	*CONTINUOUS LIQUID/LIQUID EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3520C AND 600 SERIES, 10/12/18*	NA	Water/Organics	Various	N
NC-OP-038, Rev. 8 (Appendix 1)	*SEPARATORY FUNNEL EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3510C AND 600 SERIES, 10/16/19*	NA	Water/Organics	Various	N
NC-09-040, Rev. 6(Appendix 1)	*SOXHLET (TRADITIONAL) EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3540C, 5/25/18*	NA	Soil/Organics	Various	N
ED-ORP-002, Rev. 11 (Appendix 1)	*Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C, 3/26/18*	NA	Water/Organics	Various	N
NC-QAM-001, Rev 6 (Appendix 1)	*Quality Assurance Manual, 4/2/19*	NA	NA	NA	N

Eurofins TestAmerica, Canton SOP #	Title and Date	Definitive or Screening Data	Matrix/Analytical Group	Equipment Type	‡Modified for Project? Y/N
NC-OP-043, Rev. 3 (Appendix 1)	*Waste Dilution – SW846 Method 3580A, 5/14/18*	NA	Waste/Organics	Various	N
NC-IP-010, Rev. 8 (Appendix 1)	*Acid Digestion for Solid Samples – SW846 Method 3050B, 10/16/18*	NA	Solid/Metals	Various	N

‡ A brief summary of project-specific SOP modifications must be provided on this worksheet or referenced.

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
GC/MS-VOCs	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	NC-MS-019, Rev. 6 (Appendix 1)
GC-VOCs	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	NC-GC-040, Rev. 2 (Appendix 1)
GC/MS-SVOCs	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	NC-MS-018, Rev. 8 (Appendix 1)
GC/MS-SVOCs-SIM	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	ED-MSS-009, Rev. 7 (Appendix 1)
ICP-AES	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	NC-MT-012, Rev. 9 (Appendix 1)
CVAA	Per SOP	Per SOP	Per SOP	Per SOP	Per SOP	Denise Heckler/Lab PM	NC-MT-014, Rev. 9 (Appendix 1)

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

The project team should determine whether it is necessary to complete all fields in this table. For example, if the selected laboratory is operating under a quality system that conforms to ISO 17025:2005, then the activities documented in this table will be documented in the laboratory's quality manual (however named). In this case, it may be acceptable to simply reference the quality manual (including revision number and date.) If the project has specific requirements that are different from those contained in the laboratory's quality manual, however, this table should be completed for those items.

Instrument / Equipment	Maintenance/Testing/Inspection Activity	Reference
GC/MS-VOCs	Per NC-QAM-001, Rev. 6, 4/2/19	Eurofins Environment Testing TestAmerica-North Canton, Ohio – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19
GC/MS-SVOCs	Per NC-QAM-001, Rev. 6, 4/2/19	Eurofins Environment Testing TestAmerica-North Canton, Ohio – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19
GC-VOCs	Per NC-QAM-001, Rev. 6, 4/2/19	Eurofins Environment Testing TestAmerica-North Canton, Ohio – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19
ICP-AES	Per NC-QAM-001, Rev. 6, 4/2/19	Eurofins Environment Testing TestAmerica-North Canton, Ohio – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19
CVAA	Per NC-QAM-001, Rev. 6, 4/2/19	Eurofins Environment Testing TestAmerica-North Canton, Ohio – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal
(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)

Sampling Organization: GHD Services Inc.
Laboratory: Eurofins TestAmerica, Canton
Method of sample delivery (shipper/carrier): FedEx
Number of days from reporting until sample disposal: 180

Activity	Organization and title or position of person responsible for the activity	SOP reference
Inspection of sampling supplies and equipment	GHD/Field Technician	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 8.0
Sample labeling	GHD/Field Technician	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.10
Chain-of-custody form completion	GHD/Field Technician	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.10
Packaging	GHD/Field Technician	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.10
Shipping coordination	GHD/Field Technician	GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.10
Sample receipt, inspection, & log-in	Eurofins TestAmerica, Canton/Sample Custodian	Eurofins TestAmerica, Canton – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19 (Appendix 1)
Sample custody and storage	Eurofins TestAmerica, Canton/Sample Custodian	Eurofins TestAmerica, Canton – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19 (Appendix 1)
Sample disposal	Eurofins TestAmerica, Canton/Sample Custodian	Eurofins TestAmerica, Canton – Quality Assurance Manual, NC-QAM-001, Rev. 6, 4/2/19 (Appendix 1)

Sample Identification

No one sample identification (ID) format is adequate for every type of sampling activity. Prior to the start of every project or sub-sampling event within the project, Project
Managers (PM) and field personnel should devise a sample ID format. Sample IDs **must be unique** and formats should be as simple and consistent as possible. Simple sample
IDs will reduce transcription errors by both GHD and lab personnel. The sample ID format should be comprehensive enough to allow for easy location of detailed sample data
within the GHD log books. This information and other related information should be entered in the field logbook or sample key which will facilitate entry into GHD's project database.

The unique sample ID may follow one of the two formats recommended below, or a specific sample protocol for labeling may be specified in the project Work Plan or QAPP.

MC-ZZZ-LOC-MMDDYY-XX-NNN

Where:

1. MC (Matrix Code) = WG-groundwater, SO-soil
2. ZZZ = abbreviated name of the Site
3. LOC = Well number or sample location identification
4. MMDDYY = Date in month/day/year
5. XX = Sampler's first and last initials
6. NNN = Sequential number for an event or project starting with 001

This format has been selected to maximize the information content of the sample ID. Minor modifications are certainly reasonable.

1. Series letters designate a group of samples. This will typically identify sample matrix (e.g., sediment, soil, groundwater, surface water, air, etc.), or sample source. For example, "WG" means samples of groundwater, "SO" means samples of soil. Letters should be used, not numbers. The sample ID matrix code is not necessarily the same as the matrix code identified in the field sample key or GHD database, it is a simple two letter code used to define matrix as established for the project.

2. Abbreviated name of the Site together with the series number will allow easier tracking of samples.

3. Sample date will allow monitoring of actual holding time of samples and should ensure that all sample numbers are unique, even if sample location designation is used in a system, as opposed to assigned at random.

4. Sampler's initials will allow identification of the sampler and so allow all project personnel to contact the correct person for information regarding that sample and its collection. The use of three initials is preferred. Special arrangements will need to be made if two individuals have the same initials.

5. The sequential number designation will identify the sample, and can be any numerical or letter designation. With multiple sampling crews collecting samples it is advantages to assign blocks of sequential numbers to each thereby avoiding repetition.

Field QC samples should be identified in the following manner:

1. Field blank samples – Field blank samples including equipment blanks, equipment rinsate blanks, media or bottle blanks, and ambient blanks shall be identified in the same format as investigative samples with no indication that they are QC samples. The field logbook shall identify them as QC samples.

2. Field duplicate samples – Field duplicate samples including field replicates, splits and full duplicates shall also be identified in the same format as investigative samples. The field logbook should identify them as QC samples.

3. Trip blank samples – Trip blank samples are used during the collection and transportation of samples for VOC analysis (including light range TPH). Trip blanks are primarily for water samples but may include soils if field preservation is performed. Trip blanks shall be identified in the same format as investigative samples. The field logbook shall identify them as QC samples.

4. Matrix spikes and matrix spike duplicates – These are technically laboratory QC samples but typically require additional volume (triple volume). They shall not be assigned separate sample IDs from the investigative sample as they are just additional volume.

The decision of how to assign sample numbers shall be made at the beginning of a job or phase, and shall be consistent throughout the job. Effort shall be made to eliminate use of the same sequential number on a project.

It is imperative that sample IDs be unique, use of sample locations (well numbers or boreholes numbers) that may repeated may cause problems when data is entered into the database (e.g., data may be overwritten). Samples collected on different days may have the same sequential number, but will have a unique date to identify the different samples. Samples collected on the same day shall include a sample depth or other appropriate difference in the 'LOC' field along with a unique sequential number to avoid potential database issues.

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater/Soil/LNAPL
Analytical Group: VOCs
Analytical Method/SOP: SW-846 8260C/SOP# NC-MS-019, Rev. 6 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
BFB Tune	One every 12 hours	BFB Tune Criteria must be met per SW-846 Method 8260C	Re-tune and reanalyze BFB.	Analyst / Laboratory Quality Assurance Officer	BFB Tune Criteria must be met per SW-846 Method 8260C
Initial Calibration (ICAL) Curve	As needed	%RSD and %D must be met per SW-846 Method 8260C; COD (R^2) $\geq$ 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8260C	Re-calibrate	Analyst / Laboratory Quality Assurance Officer	%RSD and %D must be met per SW-846 Method 8260C; COD (R^2) $\geq$ 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8260C
Continuing Calibration Check	One every 12 hours	%D and Minimum Mean Response Factors must be met per SW-846 Method 8260C	Re-calibrate and reanalyze.	Analyst / Laboratory Quality Assurance Officer	%D and Minimum Mean Response Factors must be met per SW-846 Method 8260C
Internal Standards (IS)	Every sample must be spiked with appropriate IS compounds	50-200% Recovery	Reanalyze samples with outlying recoveries. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	50-200% Recovery

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Surrogates	Every sample must be spiked with appropriate surrogate compounds	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Laboratory Control Sample/LCS Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	Reanalyze LCS/LCSD and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*[i]	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater
Analytical Group: GC-VOCs
Analytical Method/SOP: SW-846 8011/SOP# NC-GC-040, Rev. 2 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Initial Calibration	As Needed	%RSD must be met per SW-846 Method 8011; COD (R^2) > 0.99 for linear or quadratic curves, if used	Re-calibrate	Analyst / Laboratory Quality Assurance Officer	%RSD must be met per SW-846 Method 8011; COD (R^2) > 0.99 for linear or quadratic curves, if used
Initial/Continuing Calibration Verification (ICV/CCV)	Beginning of sequence (ICV), end of sequence and one per every 10 samples analyzed.	%D must be met per SW-846 Method 8011	Re-calibrate and reanalyze. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	%D must be met per SW-846 Method 8011
Surrogates	Every sample must be spiked with appropriate surrogate compounds	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Laboratory Control Sample/LCS Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	Re-prep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Re-prep and reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Initial/Continuing Calibration Blank (ICB/CCB)	Beginning of sequence (ICB), end of sequence and one per every 10 samples analyzed.	No target analyte concentrations > RDL	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*[i]

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater/Soil/LNAPL
Analytical Group: SVOC
Analytical Method/SOP: SW-846 8270D/SOP# NC-MS-018, Rev. 8 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
DFTPP Tune	One every 12 hours	DFTPP Tune Criteria must be met per SW-846 Method 8270D	Re-tune and reanalyze DFTPP.	Analyst / Laboratory Quality Assurance Officer	DFTPP Tune Criteria must be met per SW-846 Method 8270D
Initial Calibration (ICAL) Curve	As needed	%RSD and %D must be met per SW-846 Method 8270D; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D	Re-calibrate	Analyst / Laboratory Quality Assurance Officer	%RSD and %D must be met per SW-846 Method 8270D; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D
Continuing Calibration Check	One every 12 hours	%D and Minimum Mean Response Factors must be met per SW-846 Method 8270D	Re-calibrate and reanalyze.	Analyst / Laboratory Quality Assurance Officer	%D and Minimum Mean Response Factors must be met per SW-846 Method 8270D

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Internal Standards (IS)	Every sample must be spiked with appropriate IS compounds	50-200% Recovery	Reanalyze samples with outlying recoveries. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	50-200% Recovery
Surrogates	Every sample must be spiked with appropriate surrogate compounds	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Laboratory Control Sample/LCS Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	Re-prep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Re-prep and reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater
Analytical Group: SVOCs-SIM
Analytical Method/SOP: SW-846 8270D-SIM/SOP# ED-MSS-009, Rev. 7 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
DFTPP Tune	One every 12 hours	DFTPP Tune Criteria must be met per SW-846 Method 8270D	Re-tune and reanalyze DFTPP.	Analyst / Laboratory Quality Assurance Officer	DFTPP Tune Criteria must be met per SW-846 Method 8270D
Initial Calibration (ICAL) Curve	As needed	%RSD and %D must be met per SW-846 Method 8270D-SIM; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D-SIM	Re-calibrate	Analyst / Laboratory Quality Assurance Officer	%RSD and %D must be met per SW-846 Method 8270D-SIM; COD (R^2) > 0.99 for linear or quadratic curves, if used. Minimum Mean Response Factors must be met per SW-846 Method 8270D-SIM
Continuing Calibration Check	One every 12 hours	%D and Minimum Mean Response Factors must be met per SW-846 Method 8270D	Re-calibrate and reanalyze.	Analyst / Laboratory Quality Assurance Officer	%D and Minimum Mean Response Factors must be met per SW-846 Method 8270D

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Internal Standards (IS)	Every sample must be spiked with appropriate IS compounds	50-200% Recovery	Reanalyze samples with outlying recoveries. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	50-200% Recovery
Surrogates	Every sample must be spiked with appropriate surrogate compounds	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Laboratory Control Sample/LCS Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	Re-prep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Re-prep and reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	Must meet Laboratory Limits*	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Must meet Laboratory Limits*

[i]

[i] * Established Laboratory acceptance limits and criteria are included in Appendix 2

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater/Soil/LNAPL
Analytical Group: TAL Metals
Analytical Method/SOP: SW-846 6010/TA-NC SOP# NC-MT-012, Rev. 9 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Initial Calibration	Daily	Initial Calibration Criteria must be met per SW-846 Method 6010 Minimum R $\geq$ 0.995	Perform routine maintenance. Re-calibrate	Analyst / Laboratory Quality Assurance Officer	Initial Calibration Criteria must be met per SW-846 Method 6010 Minimum R $\geq$ 0.995
Initial/Continuing Calibration Verification (ICV/CCV)	Beginning of sequence (ICV), end of sequence and one per every 10 samples analyzed.	90-110% Recovery	Re-calibrate and reanalyze all samples processed with the outlying ICV/CCV.	Analyst / Laboratory Quality Assurance Officer	90-110% Recovery
Low Level Continuing Calibration Verification (CCV) Standard	Analyze at the beginning of each analytical run	80-120% Recovery	Re-analyze once. Re-calibrate and reanalyze all samples processed with the outlying ICV/CCV.	Analyst / Laboratory Quality Assurance Officer	80-120% Recovery
Interference Check Solution Analysis (ICSAB)	Beginning and end of sequence or every 12 hours	Analyze at the beginning of each analytical run; 80-120% Recovery	Re-calibrate and reanalyze all samples processed with the outlying ICV/CCV.	Analyst / Laboratory Quality Assurance Officer	Analyze at the beginning of each analytical run; 80-120% Recovery

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Interference Check Solution Analysis (ICSA)	Beginning and end of sequence or every 12 hours	Analyze at the beginning of each analytical run; results for the non-interfering elements with reporting limits < 10 ug/L must fall within + 2 times the RL from zero. ICSA results for the noninterfering elements with RLs > 10 ug/L must fall within + RL from zero.	Re-calibrate and reanalyze all samples processed with the outlying ICV/CCV.	Analyst / Laboratory Quality Assurance Officer	Analyze at the beginning of each analytical run; results for the non-interfering elements with reporting limits < 10 ug/L must fall within + 2 times the RL from zero. ICSA results for the noninterfering elements with RLs > 10 ug/L must fall within + RL from zero.
Laboratory Control Sample	One per preparatory batch of up to 20 samples.	80-120% Recovery	Re-prep and reanalyze LCS and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	80-120% Recovery
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Re-prep and reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Initial/Continuing Calibration Blank (ICB/CCB)	Beginning of sequence (ICB), end of sequence and one per every 10 samples analyzed.	No target analyte concentrations > RDL	Re-calibrate and reanalyze all samples processed with the contaminated ICB/CCB.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	75-125% recovery Waters: RPD ≤ 20% Soils: RPD ≤ 35%	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	75-125% recovery Waters: RPD ≤ 20% Soils: RPD ≤ 35%
Laboratory Duplicate	One per preparatory batch of up to 20 samples.	Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL
Serial Dilution	One per preparatory batch of up to 20 samples.	%D ≤ 20% for analyte concentrations > 10 times RDL	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	%D ≤ 20% for analyte concentrations > 10 times RDL

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater/Soil/LNAPL
Analytical Group: Mercury
Analytical Method/SOP: SW-846 7470A/SW-846 7471/SOP# NC-MT-014, Rev. 9 (Appendix 1)

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Initial Calibration	Daily	Initial Calibration Criteria must be met per SW-846 Method 7470A or 7471 Minimum R > 0.995	Perform routine maintenance. Re-calibrate.	Analyst / Laboratory Quality Assurance Officer	Initial Calibration Criteria must be met per SW-846 Method 7470A or 7471 Minimum R > 0.995
Initial Calibration Verification (ICV)	Beginning of sequence	90-110% Recovery	Re-calibrate and reanalyze all samples processed with the outlying ICV.	Analyst / Laboratory Quality Assurance Officer	90-110% Recovery
Detection Limit Standard (CRA)	Beginning of sequence	50-150% Recovery	Re-analyze. Re calibrate and reanalyze all samples processed with the outlying CRA.	Analyst / Laboratory Quality Assurance Officer	50-150% Recovery
Continuing Calibration Verification (CCV)	One per every 10 samples analyzed.	80-120% Recovery	Re-calibrate and reanalyze all samples processed with the outlying CCV.	Analyst / Laboratory Quality Assurance Officer	80-120% Recovery

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Laboratory Control Sample/LCS Duplicate	One per preparatory batch of up to 20 samples.	80-120% Recovery RPD ≤ 20%	Re-prep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	80-120% Recovery RPD ≤ 20%
Method Blank	1 per preparatory batch of up to 20 samples.	No target analyte concentrations > RDL	Re-prep and reanalyze the method blank and all samples processed with the contaminated blank. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Initial/Continuing Calibration Blank (ICB/CCB)	Beginning of sequence (ICB), end of sequence and one per every 10 samples analyzed.	No target analyte concentrations > RDL	Re-calibrate and reanalyze all samples processed with the contaminated ICB/CCB.	Analyst / Laboratory Quality Assurance Officer	No target analyte concentrations > RDL
Matrix Spike/Matrix Spike Duplicate	One per preparatory batch of up to 20 samples.	75-125% recovery Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	75-125% recovery Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
Laboratory Duplicate	One per preparatory batch of up to 20 samples.	Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL	None. Qualify data as needed.	Analyst / Laboratory Quality Assurance Officer	Waters: RPD ≤ 20% for results > 5 times RDL Soils: RPD ≤ 35% for results > 5 times RDL

QAPP Worksheet #29: Project Documents and Records
(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Sample Collection and Field Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Field logbook or data collection sheets	Field Technician - TBD	John-Eric Pardys	Project File
Laboratory Supplied Chain-of-Custody Forms	Field Technician - TBD	John-Eric Pardys	Project File
Air Bills	Field Technician - TBD	John-Eric Pardys	Project File
Contractor Daily QC Reports	Field Technician - TBD	John-Eric Pardys	Project File
Deviations	Field Technician - TBD	John-Eric Pardys	Project File
Corrective Action Reports	Field Technician - TBD	John-Eric Pardys	Project File
Correspondence	Field Technician - TBD	John-Eric Pardys	Project File
Photos, maps, & drawings	Field Technician - TBD	John-Eric Pardys	Project File

Project Assessments			
Record	**Generation**	**Verification**	**Storage location/archival**
Field audit checklists	Field Technician - TBD	John-Eric Pardys	Project File
Data verification checklists	James Abston	John-Eric Pardys	Project File
Data validation report	James Abston	John-Eric Pardys	Project File
Data usability assessment report	James Abston	John-Eric Pardys	Project File

Project Assessments			
Record	**Generation**	**Verification**	**Storage location/archival**
Corrective Action Forms	James Abston	John-Eric Pardys	Project File
Correspondence	James Abston	John-Eric Pardys	Project File
Laboratory QA Plan	James Abston	John-Eric Pardys	Project File

Laboratory Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Sample Receipt, Custody & Tracking Records	Sample Custodian	Denise Heckler	Laboratory Files
Standard Traceability Logs	Analyst	Denise Heckler	Laboratory Files
Equipment Calibration Logs	Analyst	Denise Heckler	Laboratory Files
Sample Prep Logs	Analyst	Denise Heckler	Laboratory Files
Instrument Run Logs	Analyst	Denise Heckler	Laboratory Files
Equipment Maintenance, Testing, and Inspection Logs	Analyst	Denise Heckler	Laboratory Files
Deviation Reports	Analyst	Denise Heckler	Laboratory Files
Corrective Action Forms	Analyst	Denise Heckler	Laboratory Files
Instrument Calibration & Method Performance Summaries	Analyst	Denise Heckler	Laboratory Files
Reported Sample Results	Analyst	Denise Heckler	Laboratory Files
Reported Results for Standards, QC Check, and QC Samples	Analyst	Denise Heckler	Laboratory Files

Laboratory Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Raw Data for Field Samples, QC Checks, and QC samples	Analyst	Denise Heckler	Laboratory Files
Laboratory Case Narrative	Analyst	Denise Heckler	Laboratory Files
Lab Qualifier Definitions	Analyst	Denise Heckler	Laboratory Files
MDL Study Results	Analyst	Denise Heckler	Laboratory Files
Data Package Completeness Checklists	Analyst	Denise Heckler	Laboratory Files
Extraction/Clean-Up Records	Analyst	Denise Heckler	Laboratory Files
Sample Disposal Records	Sample Custodian	Denise Heckler	Laboratory Files
Correspondence	Laboratory Project Manager	Denise Heckler	Laboratory Files

Parameter	**Level 2 Data Package [1]**	**Level 4 Data Package [1]**	**Equis 4-file EDD**
Groundwater & Soil			
TCL VOCs	X	X	X
SW-846 8011 (Groundwater only)	X	X	X
TCL SVOCs	X	X	X
SVOCs-SIM (Groundwater only)	X	X	X
TAL Inorganics (including Hg)	X	X	X

[1] See Table below for Required Items for Level 2 and Level 4 Data Packages.

Documents are to be retained for 5 years after EPA issues the Acknowledgement of Termination pursuant to the Administrative Order on Consent. Respondent shall preserve and retain all non-identical copies of Records (including records in electronic form).

Required Item	Level 2 Data Package	Level 4 Data Package
General Report Deliverables		
Sample ID Check (COC versus Lab Deliverables)	x	x
Sample Dates/Times and Sample Receipt Date/Time	x	x
Sample Condition Upon Receipt	x	x
Laboratory Methods/Procedures	x	x
Parameter List	x	x
Laboratory Reporting & Detection Limits establishment & verification	x	x
Case Narrative/Definitions/Corrective Action Reports	x	x
Sample Specific and Batch QC Results	x	x
Sample Preservation and Holding Times	x	x
Method Blank Results	x	x
Field Blank Results (Trip and Rinsate Blanks)	x	x
System Monitoring Compounds (Surrogates) Recoveries	x	x
MS/MSD Recoveries & RPDs-Organics	x	x
MS/MSD, MS/MD Recoveries & RPDs-Inorganics	x	x
Laboratory Control Sample (LCS) Recoveries	x	x
Serial Dilution Results	x	x
Post Digestion Spike Recoveries	x	x
Field Duplicates Results	x	x
Expanded Data Elements		
Instrument Performance Check Forms		x
Initial Calibration Summary		x
Continuing Calibration Forms		x
Initial Calibration Verification Forms		x
Continuing Calibration Verification Forms		x
Internal Standards Summary Form		x
Instrument Blanks Forms		x
ICP Interference Check Samples Forms		x

Required Item	Level 2 Data Package	Level 4 Data Package
Compound Identification-library search		x
Chromatography raw data		x
Compound/Analyte Quantitation (raw data)		x
QC Sample raw data		x
Preparation logs raw data		x
Other records (call logs, copies of logbook pages, etc…)		x

QAPP Worksheet #31, 32, & 33: Assessments and Corrective Action
(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Readiness Review	Project Manager	One assessment one week prior to mobilization.	TBD	Readiness Review Memorandum and Checklist	24 hours following assessment
Field Sampling TSA	Project Chemist	One each on first day of soil and groundwater sampling events.	TBD	TSA Memorandum and Checklist	24 hours following assessment
Management Review	Project Manager and QA Officer	Interim Management Review following site mobilization. Final management review upon completion of field work.	TBD	QA Management Report	48 hours following Management Review
Field Audit	USEPA Region 5 Remedial Project Manager	At the discretion of the USEPA Region 5 Remedial Project Manager	TBD	TBD	TBD

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Readiness Review	Task Manager	Readiness Review Corrective Action Response	24 hours from receipt of Readiness Review Memorandum	As directed by PM	Project Manager and QA Officer
Field Sampling TSA	Field Task Manager	Field Sampling Corrective Action Response	24 hours from receipt of Memorandum	Field Task Leader	Project Manager and QA Officer
Laboratory TSA	On-site Analytical Manager	On-site Analytical Corrective Action Response	48 hours from receipt of Memorandum and before further analyses can be conducted.	On-site Analytical Manager	Project Chemist
Management Reviews	Task Manager	QA Management Response	48 hours from receipt of QA Management Report	As assigned in QA Management Response	Project Manager and QA Officer

QAPP Worksheet #34: Data Verification and Validation Inputs
(UFP-QAPP Manual Section 5.2.1 and Table 9)
(EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (completeness)	Validation (conformance to specifications)
	Planning Documents/Records		
1	Approved QAPP	**X**	
2	Contract	**X**	
4	Field SOPs	**X**	
5	Laboratory SOPs	**X**	
	Field Records		
6	Field logbooks	**X**	**X**
7	Equipment calibration records	**X**	**X**
8	Chain-of-Custody Forms	**X**	**X**
9	Sampling diagrams/surveys	**X**	**X**
10	Drilling logs	**X**	**X**
11	Geophysics reports	**X**	**X**
12	Relevant Correspondence	**X**	**X**
13	Change orders/deviations	**X**	**X**
14	Field audit reports	**X**	**X**
15	Field corrective action reports	**X**	**X**
	Analytical Data Package		
16	Cover sheet (laboratory identifying information)	**X**	**X**
17	Case narrative	**X**	**X**
18	Internal laboratory chain-of-custody	**X**	**X**
19	Sample receipt records	**X**	**X**
20	Sample chronology (i.e., dates and times of receipt, preparation, & analysis)	**X**	**X**
21	Communication records	**X**	**X**
22	RDL/MDL establishment and verification	**X**	**X**
23	Standards Traceability	**X**	**X**
24	Instrument calibration records	**X**	**X**
25	Definition of laboratory qualifiers	**X**	**X**
26	Results reporting forms	**X**	**X**
27	QC sample results	**X**	**X**
28	Corrective action reports	**X**	**X**
29	Raw data	**X**	**X**
30	Electronic data deliverable	**X**	**X**

QAPP Worksheet #35: Data Verification Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbook	QAPP, GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 1.4	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed and results are documented.	Daily – Field Leader At conclusion of field activities - Project Manager
Chain-of-custody forms	QAPP, GHD Field Method Guidelines Rev. 1 – August 17, 2018 – Section 6.10	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily - Field Leader At conclusion of field activities - Project Chemist
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the CoCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Before release – Laboratory QA Manager Upon receipt - Project Chemist
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QA Officer

QAPP Worksheet #36
Data Validation Procedures

Data Validator: GHD Services Inc.

Analytical Group/Method:	**TCL VOCs – SW-846 8260**	**TCL SVOCs – SW-846 8270 and SW-846 8270-SIM**
Data deliverable requirements:	Level 4 Data Package; Equis 4-file EDD	Level 4 Data Package; Equis 4-file EDD
Analytical specifications:	WS 28-01	WS 28-02
Measurement performance criteria:	WS 12-01	WS 12-02
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	10%	10%
Percent of results to be recalculated:	10%	10%
Validation procedure:	"Analytical Data Quality Assessment and Validation Standard Operating Procedure", GHD, Draft Document; Professional Judgment; and applicable guidance from "National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016	"Analytical Data Quality Assessment and Validation Standard Operating Procedure", GHD, Draft Document; Professional Judgment; and applicable guidance from "National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016
Validation code (see table below):	Validation Code and Label Identifier Table	Validation Code and Label Identifier Table

"Analytical Data Quality Assessment and Validation Standard Operating Procedure" is included in Appendix 3.

Analytical Group/Method:	**DBCP & EDB – SW-846 8011**
Data deliverable requirements:	Level 4 Data Package; Equis 4-file EDD
Analytical specifications:	WS 28-03
Measurement performance criteria:	WS 12-03
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	10%
Percent of results to be recalculated:	10%
Validation procedure:	“Analytical Data Quality Assessment and Validation Standard Operating Procedure”, GHD, Draft Document; Professional Judgment; and applicable guidance from "National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016

Analytical Group/Method:	**TAL Metals – SW-846 6010**	**Mercury – SW-846 7470 and SW-846 7471**
Data deliverable requirements:	Level 4 Data Package; Equis 4-file EDD	Level 4 Data Package; Equis 4-file EDD
Analytical specifications:	WS 28-08	WS 28-09
Measurement performance criteria:	WS 12-08	WS 12-09
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	10%	10%
Percent of results to be recalculated:	10%	10%
Validation procedure:	“Analytical Data Quality Assessment and Validation Standard Operating Procedure”, GHD, Draft Document; Professional Judgment; and applicable guidance from "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016	“Analytical Data Quality Assessment and Validation Standard Operating Procedure”, GHD, Draft Document; Professional Judgment; and applicable guidance from "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
Validation code (see table below):	Validation Code and Label Identifier Table	Validation Code and Label Identifier Table

Validation Code and Label Identifier Table		
Validation Code	**Validation Label**	**Description/Reference**
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
J+	The result is an estimated quantity, but the result may be biased high.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
J-	The result is an estimated quantity, but the result may be biased low.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016 "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA 540-R-2016-001, September 2016
NJ	The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.	"National Functional Guidelines for Superfund Organic Methods Data Review", USEPA 540-R-2016-002, September 2016

QAPP Worksheet #37: Data Usability Assessment
(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

Identify personnel (organization and position/title) responsible for participating in the data usability assessment:

John-Eric Pardys– GHD-Project Manager
James Abston – GHD-Project Chemist/Data Validator
Angela Bown – GHD-QA Manager
TBD – GHD-Risk Assessor
TBD – GHD-Geologist/Hydrogeologist
TBD – GHD-Field Task Leader
TBD – GHD-Statistician

Describe how the usability assessment will be documented:

Data will be validated using the analytical methods and SOPs referenced on Worksheet #23 and Worksheet #36. In order to assess data quality, these documents specify the consideration of statistical values such as percent recovery, relative percent difference, percent relative standard deviation, and percent difference and the use of equations such as those used to calculate response and calibration factors, quantitation limits and analyte concentrations. A data validation report will be prepared for the sampling event. The report will summarize any identified trends, correlations, or anomalies so that the data user can make informed decisions on the use of the data.

Completeness is defined as the ratio of the number of valid measurements to the total number of measurements necessary to achieve a specified level of confidence in decision making. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient. The QA objective for completeness is to collect and analyze all environmental samples in a manner such that valid data are obtained from a minimum of 95 percent of the samples.

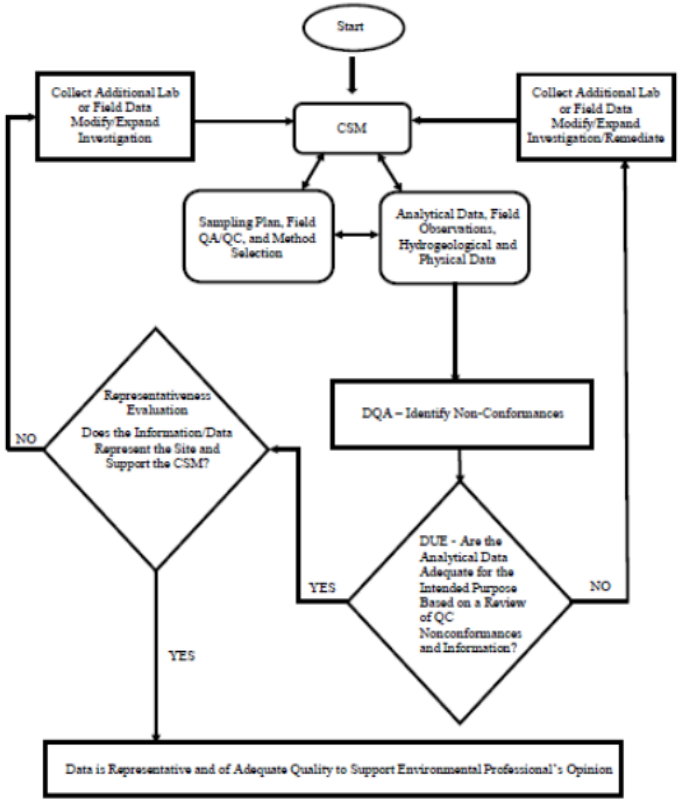
When possible, the percent completeness for each set of samples will be calculated as follows:

$$\% \text{ Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100$$

Summarize the data usability assessment process including statistics, equations, and computer algorithms that will be used to analyze the data:

Step 1	**Review the project's objectives and sampling design** Review the key outputs defined during systematic planning to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.
Step 2	**Review the data verification and data validation outputs** Review available QA reports, including the data verification and data validation reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.
Step 3	**Verify the assumptions of the selected statistical method** Verify whether underlying assumptions for selected statistical methods are valid. Common assumptions include the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, etc. Depending on the robustness of the statistical method, minor deviations from assumptions usually are not critical to statistical analysis and data interpretation. If serious deviations from assumptions are discovered, then another statistical method may need to be selected.
Step 4	**Implement the statistical method** Implement the specified statistical procedures for analyzing the data and review underlying assumptions. For decision projects that involve hypothesis testing consider the consequences for selecting the incorrect alternative; for estimation projects (e.g., establishing a boundary for surface soil contamination), consider the tolerance for uncertainty in measurements.
Step 5	**Document data usability and draw conclusions** Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss data quality indicators. Assess the performance of the sampling design and Identify limitations on data use. Update the conceptual site model and document conclusions. Prepare the data usability summary report which can be in the form of text and/or a table.

Figure 1: DQA and DUE Flow Chart



DQA – **Data Quality Assessment** is an assessment of the laboratory quality control data, the laboratory report, and the laboratory narrative by the investigator to identify and summarize QC non-conformances.

DUE – **Data Usability Evaluation** is an evaluation by the investigator to determine if the analytical data (that may include non-conformances) are of sufficient quality for the intended purpose. The DUE uses the results of the DQA and evaluates the quality of the analytical data in relation to the project-specific DQOs and the intended use of the data.

Appendices

Appendix 1
Laboratory SOPs

Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8260C, 8260B, AND 8260A

[Method: EPA Methods 8260A, 8260B, and 8260C]

Approvals (Signature/Date):

Technology Specialist	06/18/18 Date	Health & Safety Coordinator	06/18/18 Date
Quality Assurance Manager	06/28/18 Date	Technical Director	07/23/18 Date

This SOP was previously identified as SOP No. NC-MS-019, Rev 5, dated 3/11/16

Facility Distribution No. _____ Distributed To:_____

The controlled copies of this SOP are the PDF copy of the SOP that is posted to the laboratory's SOP Directory and, where applicable, the copy of the SOP that is printed and placed in the General Chemistry SOP binder. Printed or electronic copies of this SOP distributed outside of the facility are considered uncontrolled.

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges, and other solid matrices.

1.2. This SOP is applicable to Methods 8260B and 8260C. It may also be used for analysis following Method 8260A.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap sample delivery, gas chromatograph/mass spectrometric (GC/MS) separation and detection procedure. Reporting limits are available in the LIMS. Reporting limits will be proportionately higher for samples that require a dilution.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike (LCS) samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved (in the field or in the laboratory) by extracting the volatile analytes into methanol. Soil samples may also be preserved with sodium bisulfate or by freezing for storage, then thawing and purging directly.

2.3. In the purge and trap process, an inert gas is bubbled through the sample at ambient temperature or at 40^{o}C (40^{o}C required for low-level soils) and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and back-flushed with inert gas to desorb the components onto a GC column. The GC column is gradually heated to separate the components; when they elute they are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response ratio for a characteristic ion produced by that compound to the response of the

applicable internal standard (IS) in the sample, compared against the same ratio for the target compound and IS in the calibration standards.

3. DEFINITIONS

3.1. Refer to the TestAmerica Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks (MBs) as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. The use of ultra-high purity (UHP) gases pre-purged, purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Refer to SOP NC-QA-020 for additional information on holding blanks.

4.3. Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample depending upon the nature of the sample matrix and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially on an autosampler. Whenever an unusually concentrated sample is analyzed, it must be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample must be diluted.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. Cut-resistant gloves MUST be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.

5.3. Primary Materials Used

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made. MS VOA samples may be prepared outside of the hood, unless it is known that concentrations are high.

5.6. The preparation of hazardous standards and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit. MS VOA standards may be prepared outside of the hood due to low concentrations of analytes.

5.7. All work must be stopped in the event of a known or potential compromise to the health

and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the laboratory Group Leader.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the TestAmerica Corporate Environmental Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by annual refresher training.

5.9. Specific Safety Concerns or Requirements

5.9.1. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.9.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.9.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.9.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger

6.2. Syringe: 5, 25, or 50 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.01 g

6.4. Glassware

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon® liners.

6.4.2. Volumetric flasks: 10 mL, 100 mL, and 500 mL class A with ground-glass stoppers.

6.5. Spatula: Wood splints, small and large.

6.6. Disposable pipettes: Pasteur, 5 ¾ in.

6.7. Pipetters: Drummond, 30 uL to 100 uL, and 10 uL to 25 uL

6.8. Waste Dilution Pipette: 1 mL capacity

6.9. Methanol Dispensers: 5 mL, 10 mL, and 50 mL

6.10. pH paper: Wide range, pH 0-14.

6.11. Chlorine Test Paper: 0 ppm to 10 ppm

6.12. Gases

6.12.1. Helium: UHP, gr. 5, 99.999%.

6.12.2. Nitrogen: UHP from cylinders or gas generators may be used as an alternative to helium for purge gas.

6.13. Purge and Trap Device: The purge and trap device consists of the sparger, trap, and desorber.

6.13.1. Sparger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low-level soils are purged directly from a VOA vial.

6.13.2. Trap: A variety of traps may be used, depending on the target analytes required. Vocarb 3000 trap, OI 10, or an equivalent may be used if the Quality Control criteria are met. (Refer also to instrument operating manuals located within the laboratory).

6.13.3. Desorber: The desorber must be capable of rapidly heating the trap to at least 180°C.

6.13.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low-level soil analysis.

6.14. GC/MS System – HP (Agilent) GC/MSD 5973/5975 or equivalent

6.14.1. Autosampler – Archon or OI

6.14.2. The GC system must be capable of temperature programming.

6.14.3. GC Columns: Capillary Column: 20m x 0.18 ID DB-624 or equivalent, with 1 µm film thickness.

6.14.4. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron (eV) energy in the electron impact (EI) mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected into the gas chromatograph column inlet.

6.14.5. GC/MS Interface: Direct introduction to the mass spectrometer is used in the Canton MS VOA laboratory, but any interface that achieves all acceptance criteria may be used.

6.14.6. Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The NIST/EPA mass spectral library must be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.14.7. Chemstation software is used in Canton MS VOA laboratory for data acquisition.

6.14.8. CHROM software is used in Canton MS VOA laboratory for data processing.

7. REAGENTS AND STANDARDS

7.1. Reagents and standards information is recorded and available in the laboratory LIMS system. Preparation dates and expiration dates, concentrations, and stock standard identifications are all stored in the LIMS system.

7.2. Reagents

7.2.1. Methanol: Purge and Trap grade, high purity

7.2.2. Reagent Water: High purity water that meets the requirements for an MB when analyzed (see Section 9.4). Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight.

7.2.3. Hydrochloric Acid: Reagent grade or equivalent

7.2.4. Sodium bisulfate: Reagent grade or equivalent

7.3. Standards

7.3.1. Calibration Standard

7.3.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon®-sealed screw-cap bottles with minimal headspace at -10° to -20°C. Note that standard/spiking concentrations or vendors are subject to change.

7.3.1.2. Working standards: A working solution containing the compounds of interest is prepared from the stock solution(s) in methanol on a weekly basis. These standards are stored in the freezer or as recommended by the manufacturer.

7.3.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.3.1.4. If stock or secondary dilution standards are purchased in sealed ampoules, they may be used up to the manufacturer's expiration date.

7.3.1.5. Additional information can be found in SOP NC-QA-017.

7.3.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 3 for internal standard components.

7.3.3. Surrogate Standards: Refer to Table 4 for surrogate standard components and spiking levels.

7.3.4. Laboratory Control Sample Spiking Solutions: Refer to Table 5 for LCS components. Spiking levels are available via the LIMS. One LCS is included in every batch.

7.3.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 5. A MS/MSD is included in every batch.

7.3.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromofluoro-benzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.3.7. All standard preparation information is detailed in the LIMS standards and reagents module.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. Water samples are normally preserved at pH $\leq$ 2 with hydrochloric acid. Unpreserved water samples must be analyzed within seven days of sampling.

8.3. Solid samples are field preserved with sodium bisulfate solution or by freezing upon receipt at the laboratory for low-level analysis, or with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Analysis must be completed 14 days from sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. Following shipment back to the lab, the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to collect three use two additional 5g EnCore™ samples, one for methanol and two for low-level soils.

8.5. Sample collection for medium level analysis using EnCore™ samplers

8.5.1. When the samples are received at the lab, extrude each (nominal) 5g sample into a tared VOA vial containing 5 mL methanol. Obtain the weight of the soil added to the vial and record in LIMS prep batch.

8.5.2. Add the correct amount of surrogate spiking mixture. 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.5.3. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 50 µg/mL solution for a nominal 5 g sample). Reduce the volume of methanol added to ensure the final volume is 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.5.4. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.5.5. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6. Sample collection for medium-level analysis using field methanol preservation

8.6.1. At client request, the methanol addition and weighing may be performed in the field.

8.6.2. When the samples are returned to the lab, obtain the weight of the soil added to the vial and record in LIMS prep batch.

8.6.3. Add the correct amount of surrogate spiking mixture. (Add 5 μL of 2500 ug/mL solution for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.6.4. Add the correct amount of matrix spiking solution to the MS and MSD samples. (Add 100 μL of 50 μg/mL solution for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.6.5. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 μL of spike to 5 mL methanol. Refer to Section 17.2 for Michigan project criteria.

8.6.6. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7. Low-level procedure

8.7.1. If low detection limits are required (typically < 50 μg/kg), low-level soil preservation must be used. However, it is also necessary to take a sample for the medium-level (field methanol preserved or using the EnCore™ sampler) procedure in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method (Varian Archon, O.I. 4552, or equivalent).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample location to allow for reruns if necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials for sodium bisulfate preservation by adding a magnetic stir bar, approximately 1g of sodium bisulfate, and 5 mL of reagent water. Prepare vials for preservation by freezing by adding a stir bar and 5 mL reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g, and record the weight in the LIMS prep batch.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45 degree angle in the freezer.

8.8. Unpreserved Soils

8.8.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on Method 5030A and Method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.9. Methanol Preserved - Field Preservation for ISM (Incremental Sampling Method) Samples

8.9.1. Prior to sample collection for ISM samples, the client must contact the laboratory PM and notify them of the number of increments that will be sampled. The laboratory supplies the sample bottle containing the appropriate amount of Methanol needed to preserve the sample. The ratio of Methanol to sample is normally 1:1. Approximately 5 g of sample are collected per increment.

Note: Each bottle supplied by the lab has a “Contains Methanol” label affixed. After the appropriate volume of Methanol has been added, the bottle with the Methanol gets weighed. The tare weight must be recorded on the label.

8.9.2. The sampler places approximately 5 g per increment into the bottles supplied by the lab. The samples must be stored at 4 ± 2°C.

8.9.3. The ISM preserved samples have a 14 day holding time from sampling completion to analysis.

8.9.4. Matrix spike compounds and surrogate compounds are spiked into sample aliquots and batch QC prior to analysis.

8.10. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C ± 2°C with no headspace.

8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However, they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. Holding blanks are used in all refrigerators used to store samples for volatile compound analyses. Holding blanks are held in storage for a minimum of seven days. These holding blanks are analyzed weekly. The holding blanks are replaced every two weeks.

Note: An unpreserved sample must be analyzed when 2-Chloroethyl vinyl ether (2-CLEVE) is a compound of interest, as it cannot be reliably recovered from a preserved sample. If an unpreserved sample is received with 2-CLEVE requested as a target analyte, the sample must be analyzed within 7 days of collection. If 2-CLEVE is reported from an acid-preserved sample, or from an unpreserved sample past the 7-day holding time, the PM must be notified. The analyst must generate the appropriate NCM to explain that 2-CLEVE cannot be reliably recovered from acidified samples.

9. QUALITY CONTROL

9.1. Batch

9.1.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

9.1.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the TestAmerica QC program document (QA-003) for further details of the batch definition.

9.2. Control Limits

9.2.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.2.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

9.2.3. Control limits may also be project or site specific.

9.3. Surrogates

9.3.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 4. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze if either of the above checks reveal a problem.
- Re-prepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

9.3.2. It is only necessary to re-prepare/re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.3.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-analysis or flagging of the data is required.

9.3.4. For concrete matrix, Dibromofluoromethane may have poor recovery in samples and matrix spikes. If the surrogate does not meet criteria, no further action is required due to matrix effect.

9.3.5. Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks (MBs)

9.4.1. For each batch of samples, analyze a MB. The MB is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the MB consists of reagent water. For medium-level samples, the MB consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the MB is carried through the entire analytical procedure. The MB must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at ten times the blank level.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, or 2-butanone; chloroform is a common laboratory contaminant for SPLP), the data may be reported with qualifiers, if the concentration of the analyte is less than five times the reporting limit. Such action may only be taken after consultation with and agreement from the client.

- Re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.4.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated sample results are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the TestAmerica QC Program document, Policy QA-003, for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

9.5.1. For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (see Table 5), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits in the sample, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and re-analysis of the batch. The exceptions are as follows: (a) insufficient sample for re-preparation (b) expired holding times, (c) the failing compound(s) is a poor performing compound, or (d) the LCS is biased high and the samples are non-detect for those analytes.

- If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation and explanation.

9.5.2. Refer to the TestAmerica QC Program document (Policy QA-003) for further details of the corrective action.

9.5.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements must be negotiated with the client. n-Hexane must be spiked and reported for the LCS for Ohio VAP samples.

9.5.4. If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. For each QC batch, analyze an MS and MSD. Spiking compounds are given in Table 5. Compare the percent recovery and relative percent difference (RPD) to that from the laboratory-specific, historically-generated limits.

9.6.2. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2.1. If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-analysis of the batch.

9.6.2.2. The matrix spike/duplicate must be analyzed at the same dilution as the un-spiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. See Canton SOP NC-QA-029.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system with a continuing calibration verification (CCV) standard.

10.1.2. Recommended Instrument settings

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	To give at least 5 scans/peak, but not to exceed 2 seconds/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications

Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 0.4 – 0.6 mL/minute

10.2. Gas chromatograph suggested temperature program

10.2.1. BFB Analysis

Initial Temperature:	100°C
Initial Hold Time:	0.1 minute
Temperature Program:	20°C/minute
Final Temperature:	200°C

10.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	2minutes
Temperature Program:	15°C/minute
Final Temperature:	200°C
Final Hold Time:	3 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 6 for a maximum of a 50 ng injection or purge of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each 12-hour time period. The 12-hour time period begins at the moment of the BFB injection.

10.4. Initial Calibration (IC)

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. See the LIMS for details on calibration levels. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained by using a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40 µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

NOTE: For Method 8260C. Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. However, with improvements in instrumentation and more reliance on the autosampler, an

option is available depending on the project-specific data quality requirements for allowing the autosampler (or using a manual technique) to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option the surrogate standards in the initial calibration can be averaged to develop a response factor and an effective one point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout and the recovery of this known concentration can easily be attained without demonstrating if the response is linear. Under a second calibration option, the surrogates can be calibrated in the same manner as the target analytes, however, the laboratory should have the latitude to employ either option given the instrument system limitations and the ability to meet the project's data quality objectives.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 3. Target compounds must reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Table 10 for a list of characteristic ions. See Equation 1, Section 12, for calculation of response factor.

10.4.4. For Method 8260B, the % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 9 for the CCCs. These criteria must be met before sample analysis begins.

10.4.4.1. Calibration Check Compound (CCC) (Method 8260B only)

10.4.4.1.1. CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

10.4.4.2. System Performance Check Compounds (SPCC) (Method 8260B only)

10.4.4.2.1. SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 7 for the SPCC compounds for Method 8260B and required minimum response factors. Refer to Table 8 for the recommended minimum average relative response factor criteria for initial and continuing calibration verification for Method 8260C.

10.4.6. Weighting of Data Points

10.4.6.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.

10.4.7. For any analyte with % RSD >15%, linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response. The analyst must consider instrument maintenance to improve the linearity of response. Nonlinear calibration models cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but in a narrower calibration range. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of five points are maintained (six points for quadratic) and the quantitation range is adjusted accordingly. Otherwise, the coefficient of determination r^2 must be $\geq$ 0.99. For Method 8260C, % RSD is ± 20% for each target analyte.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial five-point calibration for low-level soils must be heated to 40ºC for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five-point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low-level soils analysis requires the use of a closed vial autosampler. Each standard for analysis of sodium bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1g sodium bisulfate. The standards are heated to 40ºC for purging. All low-level soil samples, standards, and blanks must also be heated to 40ºC for purging.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. Detection of the non-standard analytes at the low concentration is required. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) re-analyzed for quantitation. However, if the analyte is not detected, the non-detect is reported and no further action is necessary.

10.5. Initial Calibration Verification (ICV)

10.5.1. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For Method 8260B, the recovery for CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to six compounds > 50%.

10.5.1.1. For Method 8260C, the acceptance criteria is 70-130% for each target analyte, with the exception of the analytes listed in section 17.4 that have acceptance criteria of 50- 150%:

10.6. Continuing Calibration

10.6.1. The calibration must be verified every 12 hours.

10.6.2. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.6.3. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in Equation 4, Section 12.3.4.

10.6.4. For Method 8260B, the % difference or % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The exception to this is if the CCC fails criteria on the high side and the samples are ND, the results may be reported with proper narration. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 7. In addition, the percent drift of all analytes must be ≤ 50% with allowance for up to six target analytes to have percent drift > 50%.

10.6.4.1. For Method 8260C, all compounds of interest must be verified at 20%, with the exception of the compounds listed in section 17.4 that must be verified with percent difference or percent drift of 50%.

10.6.5. If the CCCs and/or the SPCCs do not meet the criteria in Section 10.5.3 and Table 7, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis

begins. Extensive corrective action such as a different type of column will require a new initial calibration. For Method 8260C, any sample non-detects for an analyte that fails the SOP criteria low, must have a low level CCV (CCV at the RL) in the batch as a sensitivity demonstration. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detect samples to be reported without flagging.

10.6.6. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration equation) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

10.7. Secondary Calibration Testing

10.7.1. In order to be in compliance with NELAC and SW-846 the method 8260C non-average curve types need to be evaluated both by COD and by a secondary test. The secondary test used for method 8260C is the %RSE test. RSE (Relative Standard Error) is calculated for the curve. If the curve fails the RSE limit, the calibration curve may not provide accurate quantitation. RSE uses the ICVRSE limit type.

10.7.2. The RSE acceptance limit criterion (expressed as %) for the calibration model is the same as the RSD limit for individual RF or Avg. of Individual in the determinative method. The RSE limit for method 8260C is set at =20% for good performing compounds and =30% for poor performing compounds.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation must be completely documented using a Nonconformance Memo and approved by a Supervisor or Group Leader.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described. See SOP NC-QA-029.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Where available, the project manager's notes and historical information available in the LIMS system must be consulted for details on sample matrix, special instructions, and/or dilutions.

11.2.3. Aqueous samples must be checked for proper preservation. Sample vials used for screening purposes are checked prior to being placed on the instrument. Any sample with a pH greater than 2 is considered unpreserved and must be analyzed within 7 days of sampling. Sample vials used for the analysis are checked for proper preservation after analysis has been completed. Both the screening vial pH and the analysis vial pH will be noted.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank. See Section 9.4 for method blank preparation.

11.3.2.1. It is not necessary to re-analyze batch QC with re-analyses of samples. However, any reruns must be part of a valid batch.

11.3.3. Dilutions must be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample, then serial dilutions must be made in volumetric flasks. Dilutions may also be prepared in a 40 mL vial. An appropriate amount of water is added to the vial. The sample is added using an appropriate syringe.

11.3.3.1. Estimate a dilution that will give analyte concentrations in the upper half of the calibration range.

11.4. Methanol Extract Soils

11.4.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe. Refer to Section 17.2 for Michigan project requirements.

Note: All soil samples prepared as a methanol extraction will be corrected for moisture content using the factor in the equation 12.3.6.

11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1. Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1g.

11.5.2. Quickly add 4 mL of methanol, then add 5μL of a 2500 μg/mL surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for two minutes to mix thoroughly. For an MS/MSD or LCS, 4.9 mL of methanol, 5μL of a 2500 μg/mL surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.5.3. Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 μL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 5μL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1μL will be added to the water in the syringe.

11.6. Aqueous and low-level soil sample analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1. Units which sample from the VOA vial must be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.6.2. If the autosampler uses automatic IS/SS injection no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

11.6.3. Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1g in 5 mL).

11.6.4. Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.6.5. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7. Aqueous Samples Not Directly Sampled from VOA Vials

11.7.1. All samples and standard solutions must be at ambient temperature before analysis.

11.7.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.7.3. Add 50 ng of each internal and surrogate standard. The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 10 µg/L solution for a 5 mL sample). Inject the sample into the purging chamber. Alternately, the internal and surrogate standards can be added automatically by the autosampler.

11.7.3.1. For TCLP samples, use 1.0 mL of TCLP leachate with 4 mL reagent water. (Note: TCLP reporting limits will be five times higher than the corresponding aqueous limits.)

11.7.4. Purge the sample for 11 minutes (trap must be below 35ºC).

11.7.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.7.6. Desorb and bake time and temperature are optimized for the type of trap in use (see vendor's instructions). The same conditions must be used for all samples and standards.

11.8. Analyzing aqueous samples in the soil mode

11.8.1. Measure out 10 mL of aqueous sample using a gas tight syringe

11.8.2. Expel into a clean 40 mL VOC vial

11.8.3. Load into autosampler

11.8.4. Autosampler adds 5 mL of reagent water and 1 µl each of surrogate and internal standard

11.8.5. Analyze as described in Section 11.6.

11.9. *Low-Level Solids Analysis using discrete autosamplers, Methods 8260A and 5030A*

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of

comparability with previous data. It is no longer part of SW-846.

11.9.1. This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.9.2. Weigh out 5g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method. Add 5 mL of organic free water to the VOA vial. Add surrogate/internal standard (and matrix spike solutions if required.). Analyze as described in Section 11.6.

11.9.3. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle, and store in a clean Teflon®-capped vial at 4°C until analysis. Analyze as described in Section 11.4.

11.9.4. The above steps must be performed rapidly and without interruption to avoid loss of volatile organics.

11.10. Medium-Level Soil/Sediment and Waste Samples

11.10.1. Sediments/soils and waste that are insoluble in methanol

11.10.1.1. Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.10.1.2. Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample, add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

Note: Sections 11.10.1.1 and 11.10.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.11. Initial review and corrective actions

11.11.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.11.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required. Re-analysis must be at the same dilution, if matrix interference is not observed.

11.11.2.1. The internal standard response in samples is compared to the associated continuing calibration standard. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected.

11.12. Dilutions

11.12.1. If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.12.2. Guidance for Dilutions Due to Matrix

11.12.2.1. If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample must be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

11.12.3. Reporting Dilutions

11.12.3.1. The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Target compound mass spectra for

standard reference must be obtained on the user's GC/MS by analysis of the calibration standards.

12.1.2. Two criteria must be satisfied to verify target compound identification: (1) elution of sample component at the same GC retention time as the same component in the standard, and (2) correspondence of the sample component and the standard component characteristic ions. See Table 10 for a list of the characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.2.1. The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.2.2. The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.3. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst must report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions must agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks.

(Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches must the analyst assign a tentative identification.

12.3. Calculations

12.3.1. Response Factor (RF)

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Amount of the internal standard, ng

C_x = Amount of the compound being measured, ng

12.3.2. Standard Deviation (SD)

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

Where:

X_i = Value of X at i through N

N = Number of points

X = Average value of X_I

12.3.3. Percent Relative Standard Deviation (%RSD)

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = Mean of RF values in the curve

12.3.4. Percent Drift Between the Initial Calibration and the Continuing Calibration

Equation 4

$$\%\ \text{Drift} = \frac{C_{expecte} - C_{found}}{C_{expecte}} \times 100$$

Where:

$C_{expecte}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target Compound and Surrogate Concentrations

12.3.5.1. Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is ≤ 15% (< 20% for method 8260C).

12.3.5.2. Calculation of Concentration Using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{\overline{RF}}$$

12.3.5.3. Calculation of Concentration using Linear Fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.4. Calculation of Concentration Using Quadratic Fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

Where:

x is defined in Equations 8, 9, and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.5. Calculation of ***x*** for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

X = ug/L

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.6. Calculation of ***x*** for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, D_f, A_{is}, same as for water

V_t = Volume of total extract, mL

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%moisture}{100}$$

12.3.5.7. Calculation of ***x*** for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, A_{is}, same as for water

D = as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to *x* as defined in Equations 5 through 10.

12.3.6. Calculation for solids with methanol correction applied

12.3.6.1. SW-846 8000C, Section 11.10.5 states the following: Solid samples with a significant moisture content (>10%), designated for volatile organic analysis, that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. This total volume is then expressed as Vt in the sample concentration calculations provided. Therefore, in order to report results for volatiles analysis of samples containing significant moisture content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as Vt. The total solvent/water volume is calculated as follows:

Equation 11

uL solvent/water V_t = *[mL of solvent + ((%moisture/100) x g of sample)] x 1000 uL/mL*

V_t solvent/water is then used in the formula in section 12.3.5.6.

12.3.7. MS/MSD Recovery

Equation 12

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike Sample result
SR = Sample Result
SA = Spiked amount

12.3.8. Relative % Difference (RPD) calculation for the MS/MSD:

Equation 13

$$\textbf{RPD} = \frac{|\textbf{MSR - MSDR}|}{\frac{1}{2}\textbf{(MSR + MSDR)}} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result
MSDR = Matrix spike duplicate result

12.3.9. Relative Standard Error (%RSE) for Secondary Calibration Testing

Equation 14

$$\%RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 / (n-p)}$$

X_i = True value of the calibration level i

X_i' = Measured concentration of calibration level i

P = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)

N = Number of calibration points

12.4. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is defined in QA SOPs NC-QA-021 and CA-Q-S-006. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client.

13.2. Initial Demonstration

13.2.1. Each analyst must have initial demonstration of competence (IDOC) data on file for each method he or she performs. A continuing DOC (CDOC) must be performed annually as evidence of on-going competence to perform these analyses.

13.2.2. An MDL study must be performed and documented for each analysis method and each matrix.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Specific training requirements are outlined in the Quality Assurance Manual.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.2. All waste will be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.3. The following waste streams are produced when this method is carried out.

15.3.1. **Acidic material from the auto-sampler**: Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 5.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a flammable liquid in a solvent waste container identified as "Flammable Liquid Waste".

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal.

16. REFERENCES

16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994

16.1.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030C, Rev 3, May 2003

16.1.4. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996

16.1.5. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030A, Rev 1, July 1992

16.1.6. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996

16.1.7. SW846, Test Methods for Evaluating Solid Waste Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, Draft Revision 1, July 2002

16.1.8. SW846, Test Methods for Evaluation Solid Waste, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C, Revision 3, August 2006.

16.1.9. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.10. TestAmerica Corporate Environmental Health and Safety Manual, **CW-E-M-001** and TestAmerica **Canton Facility Addendum And Contingency Plan**, current version

16.1.11. Corporate Quality Management Plan (CQMP), current version

16.1.12. Revision History

Historical File:		Revision 2: 02/17/12		
(formerly CORP-MS-0002NC)		Revision 3: 06/29/12		
Revision 2.0: 12/15/97		Revision 4: 05/21/14		
Revision 2.1: 03/06/00		Revision 5: 03/11/16		
Revision 2.2: 11/28/00				
Revision 2.3: 05/23/01				
Revision 2.4: 09/27/04				
Revision 2.5: 04/03/07				
Revision 0: 06/30/08 (NC-MS-019)				
Revision 1: 01/07/09				

**4/17/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. Standards and Reagents, NC-QA-017

16.2.6. Laboratory Holding Blanks, NC-QA-020

16.2.7. Selection of Calibration Points, CA-T-P-002

16.2.8. Calibration Curves (General), CA-Q-S-005

16.2.9. Acceptable Manual Integration Practices, CA-Q-S-002

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW846 in order to improve the reliability of qualitative identification.

17.2. The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.2.1. Modify Sections 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 10g sample).

17.2.2. Modify Sections 8.5.5 and 8.6.9 (add 200 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.3. Modify Sections 8.5.6 and 8.6.10 (add 200 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.4. Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration range for water of 0.5 ug/L to 100 ug/L. Some analytes are prepared at higher concentrations.

17.3. Analysis of aqueous samples in the soil mode: A 10 mL aliquot of the sample is poured into a 40 mL vial, 5 mL of reagent water is added, and the sample is loaded in the autosampler.

17.4. The minimum response factors recommended by Method 8260C were modified to define the performance of analytes on the columns and conditions used.

The initial calibration verification criteria and continuing calibration acceptance criteria were modified to define acceptance limits for the following poor performing compounds, which include a) compounds that do not purge well, b) compounds that have erratic response, and c) compounds that are common lab contaminants whose contribution to reference standard peak areas can adversely affect the calibration causing the potential for high or low bias to calculated results and recoveries.

ICV/CCV Poor Performer list

Acetone
4-Methyl-2-pentanone
1,2-Dibromo-3-chloropropane
1,4-Dioxane
2-Butanone
Hexachlorobutadiene
Methylene Chloride
Naphthalene
1,2,4-Trichlorobenzene
Methyl acetate
2-Hexanone
cis-1,3-Dichloropropene

Table 1: Compound List with CAS Numbers

Compound	CAS Number
Dichlorodifluoromethane	75-71-8
Chloromethane	74-87-3
Bromomethane	74-83-9
Vinyl chloride	75-01-4
Chloroethane	75-00-3
Trichlorofluoromethane	75-69-4
Acrolein	107-02-8
Acetone	67-64-1
Trichlorotrifluoroethane	76-13-1
Iodomethane	74-88-4
Carbon disulfide	75-15-0
Methylene chloride	75-09-2
tert-Butyl alcohol	75-65-0
1,1-Dichloroethene	75-35-4
1,1-Dichloroethane	75-34-3
Trans-1,2-Dichloroethene	156-60-5
Acrylonitrile	107-13-1
Methyl *tert*-butyl ether (MTBE)	1634-04-4
Hexane	110-54-3
cis-1,2-Dichloroethene	156-59-2
1,2-Dichloroethene (Total)	540-59-0
Tetrahydrofuran	109-99-9
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
Dibromomethane	74-95-3
2-Butanone	78-93-3
1,4-Dioxane	123-91-1
1,1,1-Trichloroethane	71-55-6
Carbon tetrachloride	56-23-5
Bromodichloromethane	75-27-4
1,2-Dichloropropane	78-87-5
cis-1,3-Dichloropropene	10061-01-5
Trichloroethene	79-01-6
1,2-Dibromoethane	106-93-4
1,2,3-Trichloropropane	96-18-4
1,1,2-Trichloroethane	79-00-5
Benzene	71-43-2
Ethylmethacrylate	97-63-2

Compound	CAS Number
Trans-1,3-Dichloropropene	10061-02-6
Bromoform	75-25-2
4-Methyl-2-pentanone	108-10-1
2-Hexanone	591-78-6
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,1,2,2-Tetrachloroethane	79-34-5
2-Chloroethyl vinyl ether	110-75-8
Vinyl acetate	108-05-4
Chlorobenzene	108-90-7
Ethylbenzene	100-41-4
Styrene	100-42-5
t-1,4-Dichloro-2-butene	110-57-6
m and p Xylenes	
o-xylene	95-47-6
Total xylenes	1330-20-7
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
2,2-Dichloropropane	590-20-7
Bromochloromethane	74-97-5
1,1-Dichloropropene	563-58-6
Bromodichloromethane	75-27-4
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
Isopropylbenzene	98-82-8
Bromobenzene	108-86-1
n-Propylbenzene	103-65-1
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
1,3,5-Trimethylbenzene	108-67-8
Tert-Butylbenzene	98-06-6
1,2,4-Trimethylbenzene	95-63-6
Sec-butylbenzene	135-98-8
4-Isopropyltoluene	99-87-6
n-Butylbenzene	104-51-8
1,2,4-Trichlorobenzene	120-82-1
Napthalene	91-20-3
Hexachlorobutadiene	87-68-3
1,2,3-Trichlorobenzene	87-61-6

Compound	CAS Number
Cyclohexane	110-82-7
Methyl Acetate	79-20-9
Methyl cyclohexane	108-87-2
Epichlorohydrin	106-89-8
Propylene Oxide	75-56-9
2-Ethyltoluene	611-14-3
3-Ethyltoluene	620-14-4
1-Chlorohexane	544-10-5
Ethylene Oxide	75-21-8
1,1,1,2-Tetrachloroethane	630-20-6
1,2-Dibromo-3-chloropropane	96-12-8
3-Chloro-1-propene	107-05-1
Dichlorofluoromethane	75-43-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
n-Heptane	142-82-5
1,3-Dichloropropene, Total	542-75-6
Butadiene	106-99-0
Ethyl acrylate	140-88-5
n-Butyl acrylate	123-86-4
Trihalomethanes, Total	

Table 2: Appendix IX Compounds with CAS Numbers

Compound	CAS Number
Acetonitrile	75-05-8
Isopropyl ether	108-20-3
Chloroprene	126-99-8
n-Butanol	71-36-3
Propionitrile	107-12-0
Methacrylonitrile	126-98-7
Methyl methacrylate	80-62-6
Ethyl Acetate	141-78-6
2-Nitropropane	79-46-9
Cyclohexanone	108-94-1

Compound	CAS Number
Isopropylbenzene	98-82-8
1,2,3-Trimethylbenzene	526-73-8
1,3,5-Trichlorobenzene	108-70-3
Benzyl chloride	100-44-7
Pentachloroethane	76-01-7
Tert-Amyl methyl ether	994-05-8
Tert-Butyl ethyl ether	637-92-3
2-Methylnaphthalene (Michigan only)	91-57-6

Table 3 - Internal Standards

Compound	Standard Concentration µg/mL (may vary per matrix)	Quantitation ion
Fluorobenzene	50 – 250	96
Chlorobenzene-d5	50 – 250	117
1,4-Dichlorobenzene-d4	50 – 250	152

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 4 - Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL (may vary per matrix)
1,2-Dichloroethane-d_4	50 – 250
Dibromofluoromethane (not required for Method 8260C)	50 – 250
Toluene-d_8	50 - 250
4-Bromofluorobenzene	50 – 250

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

2) Recovery limits for surrogates are g enerated from historical data and are maintained by the QA Dept.

3) There is no corrective action for Dibromofluoromethane for Method 8260C.

Table 5: Matrix Spike / LCS Compounds

Compound
1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloro-1,2,2-trifluoroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloroethene (total)
1,2-Dichloropropane
1,3,5-Trimethylbenzene 1,3-Dichlorobenzene
1,3-Dichloropropane 1,4-Dichlorobenzene
1,4-Dioxane
2,2-Dichloropropane
2-Butanone
2-Chloroethyl Vinyl Ether
2-Chlorotoluene
2-Methyl-2-propanol
2-Hexanone
3-Chloro-1-propene
4-Chlorotoluene
4-Methyl-2-pentanone
Acetone
Acetonitrile
Acrolein
Acrylonitrile
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chloroethane
Chloroform
Chloromethane

Compound
cis-1,2-Dichloroethene
cis-1,3-Dichloropropene
Cyclohexane
Dibromochloromethane
Dibromomethane
Dichlorodifluoromethane
Dichlorofluoromethane
Ethylbenzene
Ethyl ether
Ethyl methacrylate
Hexachlorobutadiene
Iodomethane
Isobutyl alcohol
Isopropylbenzene
Methyl acetate
Methyl tert-butyl ether (MTBE)
Methylcyclohexane
Methylene chloride
Naphthalene
n-Butylbenzene
n-Heptane
n-Hexane (Ohio VAP only)
n-Propylbenzene
p-Isopropyltoluene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethene
Toluene
trans-1,2-Dichloroethene
Tetrahydrofuran
trans-1,3-Dichloropropene
trans-1,4-Dichloro-2-butene
Trichloroethene
Trichlorofluoromethane
Vinyl Acetate
Vinyl chloride
Xylenes (total)

Note: *This list is subject to change without notice*

Table 6 - BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 7 - SPCC Compounds and Minimum Response Factors for Method 8260B

Compound	Methods 8260B and 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 8 - Method 8260C. Recommended Average Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification		
Volatile Compound	**Minimum Response Factor**	**Typical Response Factor**
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.050	0.255
Chloroethane	0.050	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.050	0.302
Acetone	0.010	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.05	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.010	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.150	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.150	0.424
cis-1,3-Dichloropropene	0.150	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.050	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.150	0.606
2-Hexanone	0.050	0.536
Dibromochloromethane	0.100	0.652
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408

Table 10 - Method 8260C: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification (cont'd)		
Volatile Compound	**Minimum Response Factor**	**Typical Response Factor**
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

Table 9 - CCC Compounds for Method 8260B

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	30	20
1,1-Dichloroethene	30	20
Chloroform	30	20
1,2-Dichloropropane	30	20
Toluene	30	20
Ethylbenzene	30	20
n-Hexane (Ohio VAP only)	30	20

Table 10 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98

Table 10 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	

Table 10 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98
Epichlorohydrin	57	49	
Propylene oxide	58	43	
2-Ethyltoluene	105	120	
3-Ethyltoluene	105	120	
1-Chlorohexane	91	55	
Ethylene oxide	44	43	

** The primary ion must be used for quantitation unless interferences are present, in which case a secondary ion may be used.*

*** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.*

TABLE 11:
Internal Standards with Corresponding Analytes and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Fluorobenzene**	**Fluorobenzene Cont'd**
trans-1,4-Dichloro-2-butene	Dichlorodifluoromethane	Methyl methacrylate
1,1,2,2-Tetrachloroethane	Chloromethane	2-Chloroethyl vinyl ether
Bromobenzene	Vinyl chloride	Methylcyclohexane
n-Propylbenzene	Bromomethane	cis-1,3-Dichloropropene
2-Chlorotoluene	Chloroethane	4-Methyl-2-pentanone
4-Chlorotoluene	Trichlorofluoromethane	Butadiene
1,3,5-Trimethylbenzene	Acrolein	Dichlorofluoromethane
Pentachloroethane	Acetone	2-Methyl-2-propanol
Tert-butylbenzene	Ethyl ether	Hexane
1,2,4-Trimethylbenzene	1,1-Dichloroethene	Isobutyl alcohol

sec-Butlybenzene	Iodomethane	n-Heptane
1,3-Dichlorobenzene	Acrylonitrile	Acetonitrile
1,4-Dichlorobenzene	Methylene chloride	Ethyl acetate
4-Isopropyltoluene	1,1,2-Trichloro-1,2,2-trifluoroethane	Propionitrile
1,2-Dichlorobenzene	Methyl acetate	n-Butanol
n-Butylbenzene	3-Chloro-1-propene	Ethyl acrylate
1,2-Dibromo-3-chloropropane	Carbon disulfide	Dibromofluoromethane (Surr)
1,2,4-Trichlorobenzene	trans-1,2-Dichloroethene	1,2-Dichloroethane-d_4 (Surr)
Naphthalene	Methyl tert-butyl ether	
Hexachlorobutadiene	1,1-Dichloroethane	
1,2,3-Trichlorobenzene	Vinyl acetate	
1,2,3-Trichloropropane	2-Chloro-1,3-butadiene	**Chlorobenzene-d_5**
Cyclohexanone	2-Butanone	Chlorobenzene
1,2,3-Trimethylbenzene	Isopropyl ether	Ethylbenzene
Benzyl chloride	Methacrylonitrile	Styrene
1,3,5-Trichlorobenzene	cis-1,2-Dichloroethene	1,1,2,2-Tetrachloroethane
2-Methylnaphthalene	Chlorobromomethane	Tetrachloroethene
	Chloroform	1,3-Dichloropropane
	2,2-Dichloropropane	Ethylene dibromide
	tert-Butly ethyl ether	Xylenes
	Tetrahydrofuran	Ethyl methacrylate
	1,1,1-Trichloroethane	2-Hexanone
	1,1-Dichloropropene	Chlorodibromomethane
	Cyclohexane	1,1,2-Trichloroethane
	Carbon tetrachloride	1-Chlorohexane
	1,2-Dichloroethane	Bromoform
	Benzene	Isopropylbenzene
	tert-Amyl methyl ether	trans-1,3-Dichloropropene
	Dibromomethane	1,1,2-Trichloroethane
	1,2-Dichloropropane	Toluene
	Trichloroethene	n-Butyl acetate
	2-Nitropropane	Toluene-d_8 (Surr)
	Dichlorobromomethane	4-Bromofluorobenzene (Surr)
	1,4-Dioxane	

**The closest eluting internal standard is used.*

Table 12: Poor Performers and Common Lab Contaminants

Compound	Poor Performer	Common Contaminant
1,1,2-Trichloro-1,2,2-trifluoromethane	X	
1,2,4-Trichlorobenzene	X	
1,2-Dibromo-3-chloropropane	X	
1,3-Dichloropropene, Total	X	
1,4-Dioxane	X	
2,2-Dichloropropane	X	
2-Butanone	X	
2-Chloroethyl vinyl ether	X	
2-Hexanone	X	
2-Methyl-2-propanol	X	
2-Nitropropane	X	
3-Chloro-1-propene	X	
4-Methyl-2-pentanone	X	
Acetone	X	X

Compound	Poor Performer	Common Contaminant
Acrolein	X	
Acrylonitrile	X	
Bromoform	X	
Bromomethane	X	
Butadiene	X	
Chlorobromomethane	X	
Chloroethane	X	
Chloromethane	X	
cis-1,3-Dichloropropene	X	
Cyclohexanone	X	
Dibromomethane	X	
Dichlorodifluoromethane	X	
Dichlorofluoromethane	X	
Ethyl methacrylate	X	
Ethylene dibromide	X	
Hexachlorobutadiene	X	
Hexane	X	
Isobutyl alcohol	X	
Methacrylonitrile	X	
Methyl acetate	X	
Methylene chloride	X	X
Naphthalene	X	
n-Heptane	X	
Pentachloroethane	X	
Tetrahydrofuran	X	
trans-1,4-Dichloro-2-butene	X	
Trichlorofluoromethane	X	

Title: Ethylene Dibromide (EDB), and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography

Method: SW846 8011

Approvals (Signature/Date):

Technology Specialist	12/28/18 Date	Health & Safety Coordinator	01/07/19 Date
Quality Assurance Manager	12/28/18 Date	Technical Director	12/28/18 Date

This SOP was formerly known as NC-GC-040 Rev. 1, dated 6/24/13

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used when EPA Method 8011 is applied to the analysis of Ethylene Dibromide (EDB) and 1,2-Dibromo-3-chloropropane (DBCP) in aqueous matrices by GC/ECD.

1.2. Analytes and reporting limits are listed in Table 1

2. SUMMARY OF METHOD

2.1. EDB and DBCP extracts of standards and samples are prepared by microextraction using hexane. The extract is injected into a capillary gas chromatograph (GC) column and separated for analysis by an electron capture detector (ECD). After the initial preparation step, the sample is introduced into the GC and the concentration of each target analyte is measured by the detector response within a defined retention time (RT) window, relative to the response of the reference standards. Sample results above the reporting limit must be confirmed using a dissimilar column.

3. DEFINITIONS

3.1. Refer to the Test America Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. All glassware is cleaned per SOP NC-QA-014.

4.2. Interferences in the GC analysis can arise from many compounds which are amenable to gas chromatography and give a measurable response on the electron capture detector (ECD). Phthalate esters, which are common contaminants found in plastics, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica Canton Corporate Environmental Health and Safety Manual for a complete description of personal protection equipment.

5.2. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and

discarded; other gloves must be cleaned immediately. Nitrile gloves provide adequate protection against the solvents used in this method.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a de-fatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Note: Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. All ^{63}Ni sources (ECD) must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.5. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

5.6. Exposure to chemicals must be limited as much as reasonably achievable. All samples with stickers that read "Caution/Use Hood!" must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. Opened containers of neat standards must be handled in a fume hood.

5.8. Sample extracts and standards, which are in a flammable solvent, must be stored in an explosion-proof refrigerator.

5.9. When using hydrogen gas as a carrier, all precautions listed in the CSM must be observed.

5.10. Standard preparation and dilution must be performed inside an operating fume hood.

5.11. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.12. There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.13. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph (GC) equipped with Electron Capture Detectors (ECDs)

6.2. Software: ChemStation or equivalent

6.3. Analytical columns and run conditions (See Table 2)

6.4. Microsyringes and syringes: various sizes, for standards preparation, sample injection, and extract dilution

6.5. Autosampler vials, inserts, and caps

6.6. Class A volumetric flasks: various sizes

6.7. Transfer pipettes: disposable

6.8. Balance: capable of weight determination to the nearest 0.1 g

6.9. Shaker table

6.10. Bottle top dispensers

6.11. Pipettes

6.12. Disposable pipette tips

6.13. VOA vials

6.14. Carrier gas: Hydrogen

6.15. Makeup gas: Nitrogen

7. REAGENTS AND STANDARDS

7.1. Reagents:

7.1.1. Hexane Extraction Solvent: UV grade, J.T. Baker Ultra-Resi Analyzed or equivalent.

7.1.2. Methyl Alcohol - ACS Reagent Grade: Demonstrated to be free of analytes.

7.1.3. Sodium Chloride, NaCl - ACS Reagent Grade: For pretreatment before use, pulverized a batch of NaCl and place in a muffle furnace. Increase the temperature to approximately 400°C for 30 minutes. Place in a bottle and cap.

7.1.4. Sodium Thiosulfate

7.1.5. Hydrochloric Acid HCL: Reagent Grade

7.1.6. Reagent Water: Concentrations of any analyte of interest must be below the method detection limit.

7.1.7. Ethylene Dibromide (EDB): CAS # 106-93-4, Restek or equivalent.

7.1.8. 1,2-Dibromo-3-chloropropane (DBCP): CAS # 96-12-8, Restek or equivalent.

7.1.9. 1,1,1,2-Tetrachloroethane (TCE): CAS # 630-20-6, Restek or equivalent.

7.2. Standards:

7.2.1. Stock Standards: Stock standards are purchased as certified solutions or prepared from pure solutions.

7.2.2. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.

7.2.3. Stock standard solutions must be replaced after one year. The expiration date for all working standards is 6 months.

7.2.4. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then the vendor's expiration date is used. Refer to SOP NC-QA-017, Standards and Reagents, for additional information. The standard preparation information is detailed in the LIMS standards and reagents module.

7.3. Calibration Standards

7.3.1. EDB and DBCP are purchased at 2000 ug/mL in Methanol. The stock standard is then made by diluting 250 uL of the 2000 ug/mL standard to a 10 mL final volume with methanol. The final concentration of this standard is 50 ug/mL.

7.3.2. Initial calibration verification standards are similar to calibration standards, but are from a different source. EDB and DBCP standards are purchased with each component at 2000 ug/mL in methanol. A stock standard is made by taking 250 uL of the 2000 ug/mL standard to a 10 ml final volume in methanol. Concentration is 50 ug/mL. A working standard is then made by taking 10 uL of stock standard to a 10 mL final volume with Methanol. Working standard concentration is 0.05 ug/mL.

7.3.3. Surrogate Standards: 1,1,1,2-Tetrachloroethane. The surrogate is purchased at 2000 ug/mL. A stock standard is made by taking 250 uL of the 2000 ug/mL standard to a 10.0 ml final volume in methanol. Concentration is 50 ug/mL. A working standard is then made by taking 10uL of stock standard to a 10 mL final volume with Methanol. Working standard concentration is 0.05 ug/mL.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. All samples are shipped and stored at < 4 °C. Vials should not have headspace. If they do, the project manager should be contacted and a nonconformance should accompany the sample.

8.2. All samples must be preserved with either HCL or $Na_2S_2O_3$.

8.3. Samples must be extracted and analyzed within 14 days from the date of collection. If samples are not preserved then samples must be extracted and analyzed within 7 days of collection. Analysts will be notified of unpreserved samples immediately upon receipt.

8.4. Samples are stored in 40 mL VOA vial with Teflon-lined septa.

9. QUALITY CONTROL

9.1. Batch definition

9.1.1. The batch is a set of up to 20 samples of the same matrix processed at the same time using the same procedures and reagents. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). Laboratory generated QC samples (Method Blank, LCS and MS/MSD do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with an MS and an un-spiked sample duplicate (DU).

9.2. Method Blank (MB)

9.2.1. For each batch of samples, analyze an MB. The MB consists of reagent water

9.2.2. The MB must not contain any analyte of interest at, or above, the reporting limit or at, or above, 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

Note: Some programs require that the MB be clean to ½ the RL. Method notes should inform the analyst if the samples are part of a special program. Analysts are responsible for checking the program requirements.

9.2.3. Corrective action

9.2.3.1. Corrective action may include re-analysis of the MB. If the re-analysis fails to meet criteria, re-extraction and re-analysis of samples associated with an unacceptable MB is required when concentrations greater than the RL are detected in the samples.

9.2.3.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers.

9.3. Laboratory Control Samples (LCS)

9.3.1. For each batch of samples, analyze an LCS. The LCS contains the analytes of interest and the surrogate required for the analysis. The LCS standard contains the same analytes as the matrix spike. If any LCS analyte is outside the laboratory established historical control limits, corrective action must occur.

9.3.2. Corrective Action

9.3.2.1. Corrective action may include re-analysis of the LCS. If the re-analysis fails to meet criteria, re-extraction and re-analysis of the batch may be needed. If the LCS is biased high, samples that have no analytes detectable above the RL may be reported with proper narration.

9.3.3. LCS compound lists and surrogates are included in the reagent module of the LIMS.

9.4. Matrix Spikes/Spike Duplicates (MS/MSD)

9.4.1. For each QC batch, analyze an MS/MSD. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory-specific historically generated limits.

9.4.2. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur unless samples for this compound are ND. The initial corrective action must be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, adverse matrix effect is indicated, the laboratory operation is in control and analysis may proceed.

9.4.3. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory process is out of control and corrective action must be taken.

9.4.4. The MS/MSD must be analyzed at the same dilution as the un-spiked sample.

9.5. Surrogates

9.5.1. Surrogates are added during the preparation procedure. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits.

9.5.2. Method Blank

9.5.2.1. Surrogates are added to the MB and the MB is carried through the entire analytical procedure. The MB must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the MB and affected samples will normally be required. If surrogate recoveries are high, and the samples are non-detect, the data may be reported with proper narration.

9.5.3. LCS

9.5.3.1. The LCS must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the LCS has served the purpose of demonstrating effectiveness of the extraction process. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. None of the surrogate recoveries can fall below 10% in the LCS. If surrogate recoveries are high, and the samples have no detections above the RL, the data may be reported with proper narration.

9.5.4. Instrument QC

9.5.4.1. Surrogates in the calibration standards must meet the same criteria as the ICAL. See section 10 for ICAL criteria.

9.5.4.2. Surrogates in the CCVs must have a %D of <40%.

9.5.5. Samples

9.5.5.1. If the surrogate is outside limits, the following corrective actions must take place (except for dilutions greater than 5X):

9.5.5.1.1. Check all calculations for error.

9.5.5.1.2. Ensure instrument performance is acceptable.

9.5.5.1.3. Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.

9.5.5.2. It is only necessary to re-prepare / re-analyze a sample once to demonstrate poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.5.5.3. If the surrogates are out of control for the sample and MS/MSD, then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If the sample is out of control and the MS/MSD is in control, then re-preparation or flagging of the data is required. Re-preparation includes the parent sample and the MS/MSD.

9.5.6. Refer to TestAmerica Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.6. Control Limits

9.6.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

10. CALIBRATION AND STANDARDIZATION

10.1. For this method, a calibration standard is spiked into water and extracted in the same manner as samples. The calibration levels are made from this extract.

10.2. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low-level standard must be at, or below, the reporting limit. The other standards define the working range of the detector.

10.3. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns or

replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance, unless CCV criteria cannot be met.

10.4. With the exception of section 10.5 below, it is not acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve and the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

10.5. A level may be removed from the calibration if the reason can be clearly documented (for example, a broken vial or no purge run). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be re-analyzed and the re-analysis used for the calibration. All initial calibration points in a single calibration curve must be analyzed without any changes to instrument conditions, and all points in a single calibration curve must be analyzed within 24 hours.

10.6. External standard calibration

10.6.1. Quantitation by the external standard method assumes a proportional relationship between the analyte or surrogate response and the concentration that is the same in all of the calibration standards and the samples. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected is used to prepare a calibration curve.

10.7. Calibration Curve Fits

10.7.1. The calculations for all calibration curve fits are found in section 12.

10.7.2. Weighted linear regression, average calibration factor, non-weighted linear regression, or quadratic curves may be used to fit the data. - Average calibration factor is the preferred calibration model used for 8011.

10.8. Average calibration factor (CF)

10.8.1. The average CF (external calibration) may be used if the average percent relative standard deviation (% RSD) of all the CFs taken together is $\leq$ 10%.

10.9. Linear Regression / Weighted Linear Regression

10.9.1. Linear / weighted linear regressions must have a minimum of 5 calibration levels with the lowest being at or below the reporting limit. The correlation coefficient (r) must be >0.990.

10.10. Quadratic Curve

10.10.1. A quadratic calibration curve must only be used if the analyst has reason to believe that a linear or average model does not fit the normal concentration-to-response behavior of the detector. A quadratic curve fit may be used only if the compounds have historically exhibited a non-linear response and cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but within a narrower calibration range.

10.10.2. A quadratic calibration curve must have a minimum of 6 calibration levels with the lowest being at or below the reporting limit. The coefficient of determination (r^2) must be > 0.990.

10.11. Evaluation of Calibration Curves

10.11.1. The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.11.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level.

Note: When average calibration factors are used, %RSE is equivalent to %RSD.

10.12. The following requirements must be met for any calibration to be used.

10.12.1. Response must increase with increasing concentration.

10.12.2. If a curve is used, the calculated intercept of the curve at zero response must be less than ± the reporting limit for the analyte.

10.12.3. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 10%.

10.12.4. Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination (COD) may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point.

Note: The surrogates must be judged against these same criteria.

10.13. Weighting of Data Points

10.13.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and must be used when an average calibration factor is not appropriate, as long as the data system has this capability.

10.14. Initial Calibration- The calibration is performed by extracting one 0.5ug/L standard which is then diluted to prepare a 7 point curve.

10.14.1. Add 35 mL of water to one 40-mL vial.

10.14.2. Add 6 g of NaCl to the water.

10.14.3. Cap and gently shake the contents of the 40-mL vial until the sodium chloride dissolves.

10.14.4. Add 350uL of the calibration standard to the vial.

10.14.5. Add 2.0 ml of Hexane to the vial.

10.14.6. Cap and place on the shaker table for 1 minute approximately.

10.14.7. After water and hexane phases have separated, transfer the hexane layer to an autosampler vial.

10.14.8. Individual calibration standards are then prepared from the extract as indicated below.

Calibration Level	1	2	3	4	5	6	7
Volume of calibration standard (0.05ug/L) brought to 1 mL final volume with hexane (uL)	40	80	160	200	250	400	500
Final Concentration (ug/L)	0.02	0.04	0.08	0.1	0.125	0.2	0.25

10.15. Initial Calibration Verification (ICV)

10.15.1. The ICV is analyzed immediately after an initial calibration. The acceptance criterion is ± 40%.

10.15.2. If the percent drift of the analytes is greater than ±40%, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the percent drift still varies by more than ±40%, a new calibration curve may be required.

10.16. Continuing Calibration Verification (CCV)

10.16.1. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.16.2. A mid-level standard is used for the calibration verification.

10.16.3. The calibration must be verified by the analysis of a mid-point calibration standard (CCV) at the beginning of the analytical sequence, after every 20 injections (max 12 hours), and at the end of the sequence.

Note: Various programs require a CCV every 10 injections. Analysts are responsible for checking the program requirements.

10.16.4. The CCV acceptance criterion is ± 40%.

10.16.5. Corrective Actions for Continuing Calibration Failures

10.16.5.1. If a CCV fails to meet criteria, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the re-analysis still fails to meet criteria, a new calibration curve may be required.

10.16.5.2. Samples must be bracketed with passing CCVs. All samples analyzed immediately prior to, and immediately following the failing CCV must be re-analyzed.

10.16.5.3. Sample results that are below the reporting limit may be reported when a CCV is biased high. Such action must be addressed in the case narrative.

10.17. Quality Reference Sample (QRS)

10.17.1. The QRS must be analyzed at least weekly when method is analyzed. This contains all compounds at 0.1 ug/L. 80 uL of the initial calibration verification

standard and 70 uL of the surrogate standard is added to 35 mL of water and extracted.

10.17.2. Control limits for both analytes and the surrogate are 60 – 140%. If the percent recovery for either analyte or surrogate falls outside of the control limits, reanalyze the QRS sample once.

10.17.3. If the recovery of either analyte is still not within control limits, reanalyze all samples associated with the failed analyte. If the surrogate recovery is still not within control limits, check the surrogate recovery in the LCS. If the surrogate recovery of the LCS is within control limits, report the data and narrate the non-conformance. If the surrogate recovery in the LCS is also not within control limits, re-preparation and reanalysis of the batch is required.

10.18. Retention Time Windows

10.18.1. Retention time (RT) windows must be determined for all analytes.

10.18.2. Initial determination of Retention time windows

10.18.2.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration or the first CCV in the daily analytical sequence, whichever is more recent.

10.18.2.2. Evaluate the deviation from expected retention time for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours. Calculate the standard deviation of these retention times.

10.18.2.3. If three days of analytical data are not available, use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

10.18.2.4. Multiply the standard deviation by 3. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at 0.01 min.

10.18.2.5. An alternative method to determine the retention time window is to multiply the maximum deviation of all points by 1.5. The minimum retention time window is 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.

10.18.2.6. If the retention time windows for analytes of interest overlap, the analyte must be confirmed on a dissimilar column.

10.18.3. Ongoing evaluation of retention time windows

10.18.3.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day and verified every 12 hours. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

10.18.3.2. Matrix spike analytes may fall outside the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

10.18.3.3. If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

10.18.3.4. Retention time windows should be reliably narrower than +/- 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

11. PROCEDURE

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.2. Sample Preparation:

11.2.1. Remove samples and standards from storage and allow them to reach room temperature.

11.2.2. For samples and field reagent blanks, contained in 40 mL vials, remove the container cap. Discard approximately 7.4 mL of sample volume using a mechanical pipette. Replace the container cap and weigh the container with contents to the nearest 0.1g and record this weight in the Worksheet Tab (See Attachment 1) in a TALS prep batch for subsequent volume determination.

11.2.3. For calibration standards, method blanks, and LCS measure a 35 mL volume using a Dispensette bottle dispenser (or equivalent) and transfer it to a 40 mL sample container.

11.2.4. Remove the container cap and add 6g NaCl to the sample.

11.2.5. Recap the sample container and dissolve the NaCl by shaking by hand for about 20 seconds.

11.2.6. Add 70 uL of surrogate to all samples for a final concentration of 0.10 ug/L.

11.2.7. For the MS/MSD and LCS, 70 uL of standard is added for a concentration of 0.10 ug/L.

11.2.8. Remove the cap and add 2.0 mL of hexane. Recap and place on the shaker table for approximately 1 minute. Allow the water and hexane phases to separate. (If stored at this stage, keep the container upside down.)

11.2.9. Remove the cap and carefully transfer approximately 0.5 mL of the hexane layer into an auto injector vial using a disposable glass pipette.

11.3. Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second auto injector vial. Reserve this second vial at 4°C for a reanalysis if necessary.

11.4. Transfer the first sample vial to an auto injector for analysis.

11.5. Determination of Sample Volume

11.5.1. For samples and field blanks, remove the cap from the sample container.

11.5.2. Discard the remaining sample/hexane mixture. Shake off the remaining few drops using short, brisk wrist movements.

11.5.3. Reweigh the empty container with original cap and record this weight in the worksheet tab of the TALS prep batch. This net weight (in g) is equivalent to the volume of water (in mL) extracted.

11.6. Suggested gas chromatographic conditions are given in Table 2.

11.7. Sample Analysis

11.7.1. The sample extract must be injected using the same injection volume used for the calibration standards.

11.7.2. If highly contaminated samples are expected, it is acceptable to analyze solvent blanks or primers at any point in the run. Solvent blanks may not be routinely analyzed prior to QC samples.

11.8. Qualitative Identification

11.8.1. Tentative identification occurs when a peak is found within the RT window of an analyte, at a concentration above the RL, or above the MDL if J flags (J=estimated value) are requested.

11.8.2. Identification is confirmed if a peak is also present in the RT window for that analyte on the confirmatory column at a concentration greater than the reporting limit (MDL, if J flag required).

11.9. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are automatically documented by the analyst and data system. Chromatograms before and after manual integration are required. Additional information on manual integration can be found in SOP CA-Q-S-002.

11.10. Reporting of Data

11.10.1. The preferred reporting approach is the primary column approach, where the analyst evaluates the opening CCV and the data and designates a primary column to report data from. All data is reported from the primary column in an analytical run, as long as the RPD between columns is less than 40%.

11.10.2. When the RPD between the primary and confirmation column exceeds 40%, it is an indication of likely matrix interference. Every situation is different and the analyst must rely on their experience in choosing which data to report. A common issue is addressed below:

11.10.2.1. When there is interference with an analyte of interest, the higher result is likely biased due to matrix; therefore, the lower result is reported.

11.11. Dilutions

11.11.1. Samples may be screened to determine the appropriate dilution for the initial run.

11.11.2. If concentrations of any analyteexceed the working range as defined by the calibration standards, then the sample must be diluted with hexane and re-analyzed.

11.11.3. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range when allowed by sample matrix.

11.11.4. It may be necessary to dilute samples due to matrix. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

11.11.5. If the sample is run at a dilution and only minor matrix interferences are observed, then the sample should be re-analyzed at a more concentrated dilution in an attempt to target the upper half of the calibration range.

11.11.6. The most concentrated dilution with no target compounds above the calibration range should be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgment and client site history will be factors in the reporting of multiple dilutions.

11.12. Interferences

11.12.1. If peak detection is prevented by interferences, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

11.13. Analytical Documentation

11.13.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.13.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

Note: When making new standards, it is required that all information entered into TALS should be reviewed by another analyst.

11.13.3. Record sample and associated QC information in the LIMs. Level I and Level II technical reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calibration Calculations

Calibration Factor (CF) for external calibration and Response Factor $CF = \frac{A_s}{C_s}$

Where A_s = Peak area (or height) in standard

C_s = Concentration of standard injected

12.1.1. Evaluating the linearity of the initial calibration using an Average Calibration fit:

$$Mean\ CF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where n = the number of calibration standards

$$Standard\ Deviation\ (SD) = \sqrt{\frac{\sum_{i=1}^{n}(CF_i - \overline{CF})^2}{n-1}}$$

Where n = the number of calibration standards

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$

Note: If the RSD of the calibration or response factors is less than or equal to 10%, the average calibration or response factor may be used to determine sample concentrations.

12.1.2. Percent Difference (%D)

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where CF_c = The calibration factor from the CCV or ICV

$\overline{CF}$ = The average calibration factors from the initial calibration

12.2. Linear regressions

y = ax + b

Where y = Instrument response (area or height)

a = Slope of the line

x = Concentration of the calibration standard

b = The intercept

12.2.1. Correlation Coefficient

$$r = \frac{\sum d_x d_y}{\sqrt{\sum d_x^2 \sum d_y^2}}$$

Where:

d_x = deviation of x from the mean

d_y = deviation of y from the mean

12.2.2. Relative Standard Error (%RSE)

$$\%RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{n}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(n-p)}}$$

Where: n = Number of points in the curve

p = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)

C_i = True concentration for level i

PC_i = Predicted concentration for level i

12.2.3. Percent Drift for CCV and ICV

$$\%Drift = 100\% \times \frac{C_{actual} - C_{found}}{C_{actual}}$$

Where: C_{actaul} = Known concentration in standard

C_{found} = Measured concentration in CCV or ICV

12.2.4. Curve weighting may be beneficial for linear regressions. Curve weighting improves the linearity at the low end of the calibration curve. Weighted curve calculations are similar to the unweighted calculations above, but use 1/x or $1/x^2$ (where x = concentration) to determine the regression instead of the concentration.

12.3. Quadratic calibration fit (only to be used if applicable)

$y = ax^2 + bx + c$

Where: y = Response

x = Concentration
a = Curvature
b = Slope
c = Intercept

12.3.1. Coefficient of Determination (COD)

$$COD = r^2 = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - y_i)^2}{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2}$$

Where: y_{obs} = Observed response (area) for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration

y_i = Calculated response at each concentration from the initial calibration

n = Total number of calibration points

p = Number of adjustable parameters in the polynomial equation (2 for second order polynomial)

12.4. Concentration

12.4.1. Aqueous

$$Concentration, \frac{ug}{L} = \frac{C_{ex}V_t}{V_o}$$

Where C_{ex} = On-column concentration as determined in sections 12.1-12.3. (ug/L)

V_t = Final extract volume (ml)

V_o = Initial sample volume (mL)

13. METHOD PERFORMANCE

13.1. Demonstration of Capability

13.1.1. Performance limits for the four replicate initial demonstration of capability are recovery of 60 -140%. The spiking level should be equivalent to a mid level calibration.

13.2. Method Detection Limit

13.2.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP CA-Q-S-006.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.2. Solvent waste generated by the extraction.

15.2.3. Acidic and/or alkaline waste generated in the laboratory.

15.2.4. All contaminated disposable glassware utilized in the laboratory. This waste is placed in a trash container and disposed in the trash dumpster located near the shipping/receiving dock.

15.2.5. **Discarded samples:** These samples are collected in the solid debris drum.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8011

16.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.4. Corporate Quality Management Plan (CQMP), current version

16.5. Reporting Results for Methods that Require Dual Column, CA-Q-P-004

16.6. Policy for Determining RT Windows for GC/ECD Tests, CA-T-P-005

16.7. Revision History

Historical File:				
Revision 0: 05/23/12				
Revision 1: 01/14/16				

**4/17/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.8. Associated SOPs and Policies, current version

16.8.1. QA Policy, QA-003

16.8.2. Glassware Washing, NC-QA-014

16.8.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.8.4. Detection and Quantitation Limits, CA-Q-S-006

16.8.5. Standards and Reagents, NC-QA-017

16.8.6. Acceptable Manual Integration Practices, CA-Q-S-002

16.8.7. Calibration Curves (General), CA-Q-S-005

16.8.8. Selection of Calibration Points, CA-T-P-002

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. In Section 5.11 (Check Standard) of Method 8011, it states that the aqueous calibration standards should be prepared every 8 hours. The laboratory

prepares the aqueous check standard with the calibration curve prior to analysis of each analytical batch, or group of samples analyzed under the same calibration curve. The analytical batch may contain one or more preparation batches.

17.1.2. The QRS is prepared and analyzed at 1.0 ug/L instead of 0.25 ug/L.

17.2. Tables

Table 1
Standard Analyte List, CAS Numbers, and Reporting Limits

Compound	CAS #	Reporting Limit
Ethylene Dibromide	106-93-4	0.02 ug/L
1,2-Dibromo-3-Chloropropane	96-12-8	0.02 ug/L

TABLE 2
Suggested Instrumental Conditions

Parameter	Recommended Conditions
Injection port temp	200°C
Detector temp	320°C
Temperature program	35°C for 2min, 10°C/min to 110°C, 25°C/min to300,
Column 1	CLPesticide I, 30m, 0.32mm ID, 0.5µm
Column 2	CLPesticide II, 30m, 0.32 mm ID, 0.25µm
Injection	4 µL, -
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Glass, Restek or equivalent

Title: GC/MS ANALYSIS BASED ON METHODS 8270C, 8270D, AND 8270E

[Method: SW846 8270C, 8270D, and 8270E]

Approvals (Signature/Date):

Technology Specialist — Date: 06/24/19	Health & Safety Coordinator — Date: 06/21/19
Quality Assurance Manager — Date: 06/26/19	Technical Director — Date: 07/01/19

This SOP was previously identified as SOP NC-MS-018, Rev 7, dated 3/30/18

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1. SCOPE AND APPLICATION

1.1. This method is based upon SW846 8270C, 8270D, and 8270E, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 3a and 3b for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be analyzed by this method. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before quantitative sample results may be reported.

1.2. The following compounds may require special treatment when being determined by this method:

1.2.1. Benzidine exhibits poor chromatography and can be subject to oxidative losses during solvent concentration. Neutral extraction should be performed if this compound is expected.

1.2.2. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.2.3. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.

1.2.4. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.2.5. Hexachlorophene is not amenable to analysis by this method.

1.2.6. 3-Methylphenol cannot be separated from 4-methylphenol under the conditions specified in this method.

1.3. Refer to the LIMS for specific reporting limits. Reporting limits will be proportionately higher for sample extracts that require dilution.

2. SUMMARY OF METHOD

2.1. Aqueous samples are extracted with methylene chloride using a separatory funnel and/or a continuous extractor. Solid samples are extracted with methylene chloride / acetone using sonication, or soxhlet extractor. The extract is dried, concentrated to a final volume of 2 mL for waters and soils, and analyzed by GC/MS. Extraction procedures are detailed in SOPs NC-OP-037, NC-OP-038, NC-OP-039, NC-OP-040, NC-OP-042, and NC-OP-043.

2.2. The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) equipped with a narrow-bore fused silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected directly to the GC.

2.3. Identification of target analytes is accomplished by comparing their electron impact mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard (IS), using a calibration curve appropriate to the intended application.

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks (MBs) as described in the Quality Control section below. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the instrumental analysis, preparation, and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample source to sample source, depending upon the nature of the site.

4.4. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of solvent to check for cross contamination.

4.5. Phthalate contamination is commonly observed in this analysis and its occurrence must be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY PRECAUTIONS

5.1. Employees must abide by the policies and procedures in the Corporate

Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.6. Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read “Caution/Use Hood!” must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.10. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica Canton associate. The situation must be reported immediately to a Laboratory Supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph/Mass Spectrometer (GC/MS) system: An analytical system complete with a temperature-programmable GC, suitable for split/splitless injection, and all required accessories, including syringes, analytical columns, and gases. The capillary column must be directly coupled to the MS source.

6.2. Column: 30m x 0.25mm ID, 0.5µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

NOTE: A suitable alternative column may be substituted as long as its performance is documented to meet the requirements of the method.

6.3. Mass Spectrometer (MS): Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The MS must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 2 when the GC/MS tuning standard is injected through the GC.

6.4. GC/MS Interface: Any direct GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5. Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also allow integration of the abundances in any EICP between specified times or scan-number limits. (This is used to quantify TICs: The most recent version of the EPA/NIH Mass Spectral Library is recommended for TIC library searches.)

6.6. Syringes: 5 µL and 10 uL Hamilton Laboratory grade syringes or equivalent.

6.7. Carrier gas: Ultra high purity helium.

6.8. Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. A minimum five-point calibration curve is prepared. The standard preparation information and calibration levels are detailed in the LIMS. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point must be at or below the reporting limit. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit (RL) and the high standard defines the upper limit or end of the range of the calibration. For Ohio VAP work, the low standard must be at, or below, the RL.

7.2. An IS solution is prepared by diluting a purchased standard. The standard preparation information is detailed in the standards and reagents module in LIMS. Compounds in the IS Mix are acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}.

7.3. Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. Preparation information is detailed in the standards and reagents module in LIMS for the Organic Prep group. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 6.

7.4. GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. The standard preparation information is detailed in the standards and reagents module in LIMS. Pentachlorophenol, benzidine, and DDT, must also be included in the Tuning Standard to assess chromatographic performance. All components are at 25 ug/mL.

7.5. The standards listed in Sections 7.1 to 7.4 must be refrigerated at $\leq 6^{o}C$ when not in use. Standards may be stored at -10^{o}C to -20^{o}C if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. Additional information on standards preparation, tracking, and storage can be found in SOP NC-QA-017

8. SAMPLE PRESERVATION AND STORAGE

8.1. Sample extracts are stored at $4 \pm 2^{o}C$. Samples and extracts must be stored in suitable glass containers with Teflon®-lined caps. (Extracts will be stored for 30 days after invoicing.)

8.2. Water samples are extracted within seven days of sampling, and the extracts are analyzed within 40 days of extraction. Solids, sludges, and organic liquids are extracted within 14 days of sampling and the extracts are analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). Batches are defined at the sample preparation stage. Batches must be kept together through the whole extraction process, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Eurofins TestAmerica Canton QC Program document (QA-003) for further details of the batch definition.

9.2. Method Blank (MB)

9.2.1. A MB is prepared and analyzed with each batch of samples. The MB consists of reagent water for aqueous samples and sodium sulfate for soil samples. Surrogates are added and the MB is carried through the entire extraction and analysis procedure. The MB must not contain any analyte of interest at or above the reporting limit (except common lab contaminants, see below). Any MB contamination above the RL must be less than 1/10 of the measured concentration of any sample in the associated preparation batch. For Wisconsin the MB must be clean down to ½ the RL.

9.2.2. If the analyte is a common laboratory contaminant the data may be reported with qualifiers if the concentration of the analyte in the MB is less than five times the RL. Such action must be taken in consultation with the client.

9.2.3. Re-analysis of any samples with reportable concentrations of analytes found in the MB is required unless other actions are agreed upon with the client.

9.2.4. If there is no target analyte greater than the RL in the samples associated with an unacceptable MB the data may be reported with qualifiers. Such action should be taken in consultation with the client. NOTE: For Ohio VAP work, there can be no target analyte greater than the RL in the MB. All samples associated with an unacceptable MB must be re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction (b) expired holding times, or (c) the analytes detected in the MB are non-detect in the associated samples.

9.2.5. The MB must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the MB has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the MB and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.2.6. If re-analysis of the batch is not possible due to limited sample volume or other constraints, the MB is reported, all associated samples are flagged with a "B", and appropriate comments must be made in a narrative to provide further documentation.

9.2.7. Refer to the Eurofins TestAmerica Canton QC Program document (QA-003) for further details of the corrective actions.

9.3. Laboratory Control Sample (LCS)

9.3.1. A LCS is prepared and analyzed with every batch of samples. All control analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 4 and/or 5 unless otherwise specified by a client or agency.

9.3.2. If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 10% or greater if the compound is on the client's list. Corrective action may include re-extraction and re-analysis of the batch. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.3.3. If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not re-analyzing might be that the MS and MSD are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

9.3.4. If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.3.5. The LCS must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted. The exceptions are as follows: (a) insufficient sample for re-extraction (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

9.3.6. Ongoing monitoring of the LCS over time provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. A MS/MSD is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (see Tables

4 and/or 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically-generated limits.

9.4.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. For client specific samples, corrective action may include re-preparation and re-analysis of the batch.

9.4.3. The MS/MSD must be analyzed at the same dilution as the un-spiked sample, even if the MS compounds will be diluted out.

9.5. Surrogates

9.5.1. Every sample, MB, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery [%R]) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 6.

9.5.2. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

9.5.2.1. Check all calculations for error.

9.5.2.2. Ensure that instrument performance is acceptable.

9.5.2.3. Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.

9.5.2.4. It is only necessary to re-prepare / re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (MB, LCS, MS/D), up to one surrogate per fraction may be outside of acceptance criteria, as long as the recovery is greater than 10%. **Note:** For Ohio VAP all surrogates must be within acceptance criteria. The exceptions for Ohio VAP are as follows: (a) insufficient sample for re-extraction, or (b) the surrogates are biased high and the samples are non-detect.

9.5.3. If the sample with surrogate recoveries outside the recovery limits was a sample used for a MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require re-analysis as this phenomenon would indicate a possible matrix problem.

9.5.4. If the sample is re-analyzed and the surrogate recoveries in the re-analysis are acceptable, then the problem was within the analyst's control and only the re-analyzed data must be reported (unless the re-analysis was outside holding times, in which case, reporting both sets of results may be appropriate).

9.5.5. If the re-analysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.6. Internal Standards

9.6.1. Every sample, MB, and QC sample (including calibration standards, ICV and CCV) is spiked with internal standards.

9.6.2. When compared to the daily CCV, internal standards must be within ± 0.5 minutes and peak area recoveries must be 50% to 200%.

9.6.3. Samples with failing internal standards must be re-analyzed "undiluted" unless matrix interference is apparent. If matrix interference is apparent, dilute the sample with methylene chloride using a syringe for re-analysis. When there is obvious interference causing the IS failure that corrective action will not remedy, data must be flagged with a qualifier indicating matrix interference. If the QC has failing internal standards, the batch must be re-prepped and re-analyzed.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. Deviations are not applicable for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is tuned for decafluorotriphenylphosphine (DFTPP), calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 1.

10.2. All standards and extracts are allowed to warm to room temperature before injecting.

10.3. Instrument Tuning

10.3.1. At the beginning of every 12-hour shift, the GC/MS system must be checked to see if acceptable performance criteria (Table 2) are achieved for DFTPP.

10.3.2. Inject the GC/MS tuning standard (Section 7.4). Obtain background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the MS and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.3. The GC/MS tuning standard must also be used to evaluate the inertness of the chromatographic system. The tailing factor for benzidine must be less than 3.0. The tailing factor for pentachlorophenol must be less than 5. For Method 8270D and 8270E, benzidine and pentachlorochlorophenol should not exceed a tailing factor of 2. DDT must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

NOTE: Breakdown and trailing factor are not applicable for LVI PAHs.

10.4. Initial Calibration

10.4.1. Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2. Compounds should be assigned to the IS with the closest retention time. Refer to Table 7 for internal standard corresponding analytes.

10.4.3. Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Add the appropriate amount of the IS mixture to result in 2 ng on column. (For example, 5 uL of 80 ppm IS mix is added to 100 uL of extract. This results in 2 ng per each 0.5 ul injection). The concentration ranges of all analytes can be easily accessed in the LIMS. For Ohio VAP work, the low standard must be at or below the reporting limit

10.4.4. For LVI analysis, 2 uL of 8 ppm IS mix is added to 100 uL of extract. The calibration standards are diluted by a factor of 10, however 10x more is injected (5 uL injected rather than the normal 0.5 uL), keeping the on-column amount the same as the non-LVI analytes (2 ng).

10.4.5. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Table 3 lists the analytes and characteristic ions analyzed in the laboratory. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12. For Method 8270C, verify that the SPCC and CCC criteria in Sections 10.4.6 and 10.4.8 are met. **No sample analysis maybe performed unless these criteria are met.** See section 10.4.7 for 8270D and 8270E ICAL criteria.

10.4.6. System Performance Check Compounds (SPCCs) (Method 8270C). The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.7. Initial Calibration Criteria for Method 8270D and 8270E

10.4.7.1. The RSD should be less than 20% for each analyte. For analytes that fail, use linear or quadratic curve with 0.99 correlation coefficient.

NOTE: If compliance with Method 8270C is required, the RSD limit is 15%.

10.4.7.2. No more than 10% of compounds can fail the 20%/0.99 correlation coefficient requirement.

10.4.7.3. If more than 10% of analytes fail both 20% RSD and 0.99 correlation coefficient, then recalibration is necessary.

10.4.7.4. Any individual analyte that fails both 20% RSD and 0.99 correlation coefficient criteria must have any positive result flagged as estimated and must be noted in the narrative.

10.4.7.5. For any analyte non-detect associated with a calibration that fails the 20% RSD/0.99 correlation coefficient/minimum response factor criteria, there must be a demonstration of adequate sensitivity at the quantitation limit. Successful analysis of a LLCCV is considered adequate demonstration for this purpose (see section 10.4.7.7).

10.4.7.6. Minimum response factor should be met, especially for the low level standard to verify the sensitivity.

10.4.7.7. Any individual analyte that fails the minimum response factor set in the SOP must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is

analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported with appropriate flagging. In general, Table 4 in the method should be used as guidance in setting minimum response factors in the SOP; but the RFs may be modified if appropriate (for example, if especially low-level analysis is performed).

10.4.7.8. For Method 8270D and 8270E, the minimum response factors are listed in Table 8 at the end of this SOP.

10.4.8. Calibration Check Compounds (CCCs) (Method 8270C). The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could also affect the CCCs.

10.4.8.1. If none of the CCCs are required analytes, project-specific calibration specifications must be agreed upon with the client.

10.4.8.2. CCC Compounds

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.9. Continuing Calibration Criteria for Method 8270D and 8270E

10.4.9.1. At least 80% of analytes must have a %D less than or equal to 20%.

10.4.9.2. Minimum response factors must be evaluated.

10.4.9.3. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst must evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using

quantitation from a curve, then the appropriate curve with no forced intercept must be used for quantitation.

10.4.9.4. If an analyte in the initial calibration is >15%, then calibration on a curve must be used. Quadratic curve fits must be used if the compound has historically exhibited a nonlinear response. The analyst must consider instrument maintenance to improve the linearity of response where appropriate. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve, it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of five points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear or quadratic curve must be used. The coefficient of determination (r^2) must be $\geq$ 0.990. If the coefficient of determination is < 0.990, then any hits for these compounds must be flagged as estimated. If a curve is not linear for any compound that is found in a sample, the result must be flagged as estimated. Linear is defined as <15% RSD or a coefficient of determination of 0.990.

Note: For Method 8270C, D, and E, analytes using the linear calibration fit should have the read back concentration of the low level standard evaluated. The read back concentration should be within 50% of the true value. Any sample detects for analytes that fail the read back criterion and are using a linear calibration must be flagged as estimated, and be described in the narrative.

Note: Some of the later-eluting PAH compounds exhibit greater variability at the low end of the calibration curve. Analysts' judgment is critical in assessing the validity of the curve at the low end, if the 50% criterion is exceeded. Any potential effects on sample results will be narrated in the analytical report.

Note: Several components do not respond well by this method (poor linearity). These compounds are indene, benzoic acid, benzaldehyde, caprolactam, 1,3,5-Trinitrobenzene, dinoseb, p-phenylenediamine, benzidine, alpha alpha-dimethyl phenethylamine, acrylamide, 4-Nitroquinoline-1-oxide, famphur, benzenethiol, kepone, and 2,4-Toluenediamine. If these compounds are requested by a client and hits are found, results will be flagged as estimated. Sensitivity as demonstrated by the low standard is sufficient to substantiate a non-detect.

10.4.9.5. Quantitation is performed using the calibration curve or average response factor from the initial curve.

10.5. Initial Calibration Verification (ICV)

10.5.1. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For 8270C, the CCC compounds must have ≤ 20% difference (%D) from the ICAL. Non-CCC compounds must have ≤ 50%D with an allowance of up to six compounds >50%.

10.5.2. If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples maybe analyzed. (Samples may be analyzed immediately after the ICAL and ICV) Otherwise, proceed to continuing calibration.

10.6. For Methods 8270D and 8270E, the suggested acceptance criteria limit is <30%D for all analytes. Continuing Calibration

10.6.1. At the start of each 12-hour period, analyze a GC/MS tuning standard. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 2.

10.6.2. Following a successful DFTPP analysis, the continuing calibration standard(s) (CCs) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the CC.

10.6.3. For Method 8270C, the following criteria must be met for the CC to be acceptable:

10.6.3.1. The SPCC compounds must have an average response factor of ≥ 0.05.

10.6.3.2. The percent difference or drift (both %D) of the CCC compounds from the initial calibration must be ≤ 20% (see Section 12 for calculations). In addition, the %D of all analytes must be ≤ 50%, with allowance for up to four compounds to be greater than 50%.

10.6.3.3. The IS area response must be within 50-200% of the response in the mid-level of the ICAL.

10.6.3.4. The IS retention times must be within 30 seconds of the retention times in the mid-level standard of the ICAL.

Note: Ohio VAP requires that samples with failing internal standards must be re-analyzed "undiluted" unless matrix interference is apparent. If matrix interference is apparent, dilute the sample with methylene chloride using a syringe for re-analysis. When there is obvious interference causing the IS failure that corrective action will not remedy, data must be flagged with a qualifier indicating matrix interference. If the QC has failing internal standards, the batch must be re-prepped and re-analyzed.

10.6.3.5. If none of the CCCs are required analytes, project specific calibration specifications must be agreed upon with the client.

10.6.3.6. For Method 8270D and 8270E, if any sample is non-detect for an analyte that fails the SOP criteria low, it must have a low level CCV at the RL) in the batch as a demonstration of sensitivity for the compound that failed criteria. The criterion for a passing LLCCV is detection only and a passing LLCCV allows non-detect samples to be reported with appropriate flagging.

10.6.4. Once the above criteria have been met, sample analysis will begin. IC average RFs (or the calibration curve) will be used for sample quantitation, not the CCRFs. Analysis will proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Samples are prepared following SOP NC-OP-037, NC-OP-038, NC-OP-039, NC-OP-040, NC-OP-041, or NC-OP-043.

11.2. Sample Analysis Procedure

11.2.1. Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2. Analyze all samples using the same instrument conditions as the preceding CC standard.

11.2.3. Add IS to the extract to result in 2 ng injected on column. Mix thoroughly before injection into the instrument. For LVI samples, the addition should result in 2 ng injected on column.

11.2.4. Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Chromatograms before and after manual integration, as well as the reason for performing the manual

integration are required. Additional information on manual integration can be found in SOP CA-Q-S-002.

11.2.7. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.2.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, or TICs) must be performed if required by the client. They are evaluated using the criteria in Section 12.3.

11.3. Dilutions

11.3.1. If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. The diluent used is methylene chloride. An appropriate dilution must be in the upper half of the calibration range. Samples should be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or has hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample should be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range if matrix allows.

11.3.2. Guidance for Dilutions Due to Matrix

11.3.2.1. If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids must be analyzed at a higher dilution to avoid destroying the column.

11.3.3. Reporting Dilutions

11.3.3.1. The most concentrated dilution with target compounds within the calibration range will be reported. Other dilutions will only be reported at client request.

11.3.4. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 $\pm 2^{o}$C protected from light in screw cap vials equipped with unpierced Teflon®-lined septa.

11.4. Retention Time Criteria for Samples

11.4.1. If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.4.2. If the retention time of any IS in any sample varies by more than 0.1 minute from the preceding CC standard, the data must be carefully evaluated to ensure no analytes have shifted outside their retention time windows.

11.5. Procedural Variations

11.5.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo (NCM). The NCM must be filed in the project file.

11.6. Troubleshooting Guide

11.6.1. Daily Instrument Maintenance

11.6.1.1. In addition to the checks listed in the instrument maintenance schedule in the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version, the following daily maintenance may be performed as needed.

11.6.1.2. Clip column as necessary.

11.6.1.3. Install new or cleaned injection port liner as necessary.

11.6.1.4. Install new septum as necessary.

11.6.1.5. Perform auto-tune.

11.6.2. Major Maintenance

11.6.2.1. A new ICAL may be necessary following major maintenance. Major maintenance includes changing the column, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards (target compounds) or from the NBS library (TICs). When a good user-generated spectrum for a compound cannot be produced, the NBS library must be used. Two criteria must be satisfied to

verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1. The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.1.2. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.1.3. The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.1.4. The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.2. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst must report that identification and proceed with quantitation.

12.2. Mass chromatogram searches

12.2.1. Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte, a mass chromatogram (EICP) search is made.

12.2.1.1. Hexachlorophene

12.2.1.1.1. Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d_{12} to at least 4 minutes after chrysene-d_{12}. If peaks for both ions coincide, then the analyst evaluates the spectrum for the presence of hexachlorophene. Quantitation is not possible without calibration. This is a present/not present determination only, no quantitative information can be provided.

12.3. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of

analyses being conducted or by client request. Computer-generated library search routines must not use normalization that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the experienced analyst assign a tentative identification. Guidelines for making tentative identification are:

12.3.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) must be present in the sample spectrum.

12.3.2. The relative intensities of the major ions must agree within ± 20 %. (Example: For an ion with an abundance of 50 % in the standard spectrum, the corresponding sample ion abundance must be between 30 % and 70 %.)

12.3.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.3.4. Ions present in the sample spectrum, but not in the reference spectrum, must be reviewed for possible background contamination or presence of co-eluting compounds.

12.3.5. Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.6. Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

Note: For water samples, the TIC searches begin with compounds eluting after the first surrogate (2-Fluorophenol). For solid samples, the TIC searches begin with compounds eluting after the Aldol Condensation Product. Any compounds eluting before these analytes are considered volatile analytes are reported in the volatile analysis. A possible exception to this general rule would be if an early eluting compound were the reason for a sample dilution.

12.3.7. If a client requests 10 TICs, the laboratory supplies a minimum of 10. Choosing the largest non-target peaks present in the sample chromatogram. For a request of 20 TICS, the laboratory would supply a minimum of 20, assuming that number of compounds was available.

12.4. Anyone evaluating data must be trained to handle isomers with identical mass spectra and close elution times. These include target compounds:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene

Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

12.4.1. Extra precautions concerning these compounds include closely scrutinizing retention time vs. the calibration standard and also checking that all isomers have distinct retention times.

12.4.2. A second category of problem compounds would be the poor responders or compounds that chromatograph poorly (or exhibit erratic response). Included in this category are:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

12.4.3. Manually checking the integrations is appropriate for these compounds.

12.5. Calculations

12.5.1. Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$RF =$ Mean of RFs from intial caibration for a compound

$SD =$ Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

$RFi =$ RF for each of the calibration levels

$N =$ Number of RF values

12.5.2. Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

$C_{actual} =$ Known concentration in standard

$C_{found} =$ Measured concentration using selected quantitation method

12.5.3. Concentration in the extract

12.5.3.1. The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.4. Average Response Factor

12.5.4.1. If the average of all the %RSDs of the response factors in the initial calibration is $\leq$ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.5. Linear fit

$$X_s = \frac{\left(\frac{A_s \times C_{is}}{A_{is}}\right) - b}{a} \times C_{is}$$

Where: X_s = Concentration in extract, µg/mL
A_s = Response for analyte
A_{is} = Concentration of internal standard
C_{is} = Intercept

12.5.6. Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}^2\right)$$

Where: C = Curvature

12.5.7. The concentration in the sample is then calculated.

12.5.7.1. Aqueous Calculation

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where: V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half the base/neutral extract and half the acid extract are combined, V_t=2,000)
V_o = Volume of water extracted (mL)

12.5.7.2. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex}V_t}{W_sD}$$

Where: W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry weight basis or one for a wet weight basis

12.5.8. MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where: S_{SR} = Spike sample result
S_R = Sample result
S_A = Concentration equivalent of spike added

12.5.9. Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where: RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.5.10. Relative response factor calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_x = Concentration of the compound being measured (µg/L)

C_{is} = Concentration of the specific internal standard (µg/L)

12.6. Calculation of TICs: The calculation of TICs) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

Results for TICs are not quantitative and are always reported as estimated "J."

12.7. Percent DDT breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The total ion current areas are used for this calculation

12.8. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1. Method Detection Limit (MDL)

13.1.1. Each laboratory must generate a valid MDL for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in Policy CA-Q-S-006 and SOP NC-QA-021.

13.2. Lower Limit of Quantitation Verification – The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

13.2.1. The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all preparation and clean-up methods which samples would be.

13.2.2. The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand.

Other clean matrices may be used in addition, for project specific requirements.

13.2.3. The LLOQ shall be verified annually on each instrument used for client sample analysis.

13.2.4. Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery. NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

13.2.5. If the LLOQ cannot be verified, it will be necessary to raise the RL to a concentration level that can be carried through the preparation and cleanup steps to meet recovery limits.

13.3. Initial Demonstration of Capability (IDOC)

13.3.1. Each analyst must make an IDOC for each individual analyte. Demonstrations of capability (DOCs) for both soil and water matrices is required. This requires analysis of four LCSs containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1.1. Four aliquots of the LCS are analyzed using the same procedures used to analyze samples, including sample preparation.

13.3.1.2. Calculate the average recovery and standard deviation of the recoveries for each analyte of interest.

13.3.1.3. If any analyte does not meet the LCS acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4. Training Qualification

13.4.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.4.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Eurofins TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.3. Waste Streams Produced by the Method

15.3.1. Vials containing sample extracts: These vials are placed in the vial waste located in the GC/MS laboratory.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), 8270D, Rev. 4, 2007

16.1.3. SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), 8270D, Rev. 5, 2014

16.1.4. SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), 8270E, Rev. 6, 2018

16.1.5. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

16.1.6. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.7. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.8. Corporate Quality Management Plan (CQMP), current version

16.1.9. Revision History

Historical File:		Revision 2.1: 01/25/99		Revision 0: 05/28/08 (NC-MS-018)
(formerly CORP-MS-0001NC)		Revision 2.2: 03/27/00		Revision 1: 12/16/08
		Revision 2.3: 02/15/01		Revision 2: 10/26/10
		Revision 2.4: 05/29/01		Revision 3: 04/25/13
		Revision 2.5: 04/25/02		Revision 4: 07/24/14
		Revision 2.6: 08/15/02		Revision 5: 03/01/16
		Revision 2.7: 11/12/02		Revision 6: 10/31/17
		Revision 2.8: 01/23/03		Revision 7: 03/30/18
		Revision 2.9: 06/18/03		
		Revision 2.10: 02/24/04		
		Revision 2.11: 02/03/06		
		Revision 2.12: 03/01/07		

16.2. Associated SOPs and policies, current version

16.2.1. Continuous Liquid/Liquid Extraction of Organic Compounds from Waters Based on Method SW-846 3520C and 600 Series, NC-OP-037

16.2.2. Separatory Funnel Extraction of Organic Compounds from Waters Based on Method SW-846 3510C and 600 Series, NC-OP-038

16.2.3. Sonication Extraction of Organic Compounds from Soils Based on Method SW-846 3550C, NC-OP-039

16.2.4. Soxhlet (Traditional) Extraction of Organic Compounds from Soils Based on Method SW-846 3540C, NC-OP-040

16.2.5. Microextraction of Organic Compounds from Waters Based on Method 3511, NC-OP-042

16.2.6. Waste Dilution, NC-OP-043

16.2.7. QA Policy, QA-003

16.2.8. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.9. Detection and Quantitation Limits, CA-Q-S-006

16.2.10. Standard and Reagents, NC-QA-017

16.2.11. Acceptable Manual Integration Practices, CA-Q-S-002

16.2.12. Calibration Curves (General), CA-Q-S-005

16.2.13. Section of Calibration Points, CA-T-P-002

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions from compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3. Method 8270E only requires the DFTPP tune standard to be analyzed once prior to an ICAL. There is no requirement for a daily DFTPP tune prior to sample and QC analysis. The laboratory will continue with a daily DFTPP tune following the tune criteria listed in Table 2and the tune evaluation will be the tighter criteria of methods 8270C or 8270D.

17.2. Tables and Appendices

TABLE 1: Suggested Instrument Conditions	
Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	60°C for 1 minutes, 50°C for 1 minute for LVI
Column Temperature Program	60 - 320°C at 35°C/min for 3 min 50 - 320°C at 35°C/min for 3 min for LVI
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's Specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 µl, or 5.0 ul for LVI
Carrier Gas	Helium at 30 cm/sec

TABLE 2: DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 – 80% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	25 - 75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 – 9% of mass 198
275	10 – 30% of mass 198
365	> 0.75% of mass 198
441	Present, but less than mass 443
442	40 - 110% of mass 198
443	15 - 24% of mass 442

TABLE 3a: Analytes (List 1) in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d_5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d_4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
Indene	115	116	89
3&4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d_5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d_8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
2,6-Dichlorophenol	162	164	63
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
1-Methylnaphthalene	142	141	115
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
Acetophenone	105		
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d_{10} (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154

2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
1,2,4,5-Tetrachlorobenzene	216		
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d_{10} (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
1,3-Dinitrobenzene	168		
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
2,3,4,6-Tetrachlorophenol	232		
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d_{14} (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d_{12} (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d_{12} (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 3b: Analytes (List 2) in Approximate Retention Time Order and Characteristic Ions			
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Acrylamide	71	44	55
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
1-Chloronaphthalene	162	127	164
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2,3,5,6-Tetrachlorophenol	232	230	131
2-Naphthylamine	143	115	
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
Ethyl parathion	97	109	291
4-Nitroquinoline-1-oxide	190	128	160
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,h)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 4: Method 8270C LCS Control Compounds	
LCS Compounds	**Spiking Level, Concentration Added = 20 ug/L**
1,1'-Biphenyl	20
1,2,4,5-Tetrachlorobenzene	20
1,2,4-Trichlorobenzene	20
1,2-Dichlorobenzene	20
1,3-Dichlorobenzene	20
1,3-Dinitrobenzene	20
1,4-Dichlorobenzene	20
1,4-Dioxane	20
1-Methylnaphthalene	20
2,2'-oxybis[1-chloropropane]	20
2,3,4,6-Tetrachlorophenol	20
2,4,5-Trichlorophenol	20
2,4,6-Trichlorophenol	20
2,4-Dichlorophenol	20
2,4-Dimethylphenol	20
2,4-Dinitrophenol	40
2,4-Dinitrotoluene	20
2,6-Dichlorophenol	20
2,6-Dinitrotoluene	20
2-Chloronaphthalene	20
2-Chlorophenol	20
2-Methylnaphthalene	20
2-Methylphenol	20
2-Nitroaniline	20
2-Nitrophenol	20
3&4-Methylphenol	20
3,3'-Dichlorobenzidine	40
3-Nitroaniline	20
4,6-Dinitro-2-methylphenol	40
4-Bromophenyl phenyl ether	20
4-Chloro-3-methylphenol	20
4-Chloroaniline	20
4-Chlorophenyl phenyl ether	20
4-Nitroaniline	20
4-Nitrophenol	40
Acenaphthene	20
Acenaphthylene	20
Acetophenone	20
Aniline	20
Anthracene	20
Atrazine	40
Azobenzene	20
Benzaldehyde	40
Benzidine	40
Benzoic acid	40
Benzo[a]anthracene	20
Benzo[a]pyrene	20
Benzo[b]fluoranthene	20
Benzo[g,h,i]perylene	20
Benzo[k]fluoranthene	20

Table 4: Method 8270C LCS Control Compounds	
LCS Compounds	**Spiking Level, Concentration Added = 20 ug/L**
Benzyl alcohol	20
Bis(2-chloroethoxy)methane	20
Bis(2-chloroethyl)ether	20
Bis(2-ethylhexyl) phthalate	20
Butyl benzyl phthalate	20
Caprolactam	40
Carbazole	20
Chrysene	20
Dibenz(a,h)anthracene	20
Dibenzofuran	20
Diethyl phthalate	20
Dimethyl phthalate	20
Di-n-butyl phthalate	20
Di-n-octyl phthalate	20
Fluoranthene	20
Fluorene	20
Hexachlorobenzene	20
Hexachlorobutadiene	20
Hexachlorocyclopentadiene	20
Hexachloroethane	20
Hexadecane	20
Indene	40
Indeno[1,2,3-cd]pyrene	20
Isophorone	20
Naphthalene	20
n-Decane	20
Nitrobenzene	20
N-Nitrosodimethylamine	20
N-Nitrosodi-n-propylamine	20
N-nitrosodiphenylamine	40
n-Octadecane	20
Pentachlorophenol	40
Phenanthrene	20
Phenol	20
Pyrene	20
Pyridine	40

Spike concentrations are subject to change without notice.

TABLE 5: TCLP LCS Compounds	
LCS Compounds	**Spiking Level, mg/L in extract**
1,4-Dichlorobenzene	0.08
2,4-Dinitrotoluene	0.08
Hexachlorobenzene	0.08
Hexachlorobutadiene	0.08
Hexachloroethane	0.08
2-Methylphenol	0.08
3-Methylphenol	0.08
4-Methylphenol	0.08
Nitrobenzene	0.08
Pentachlorophenol	0.08
Pyridine	0.08
2,4,5-Trichlorophenol	0.08
2,4,6-Trichlorophenol	0.08

Spike concentrations are subject to change without notice.

TABLE 6: Method 8270C Surrogate Compounds	
Surrogate Compounds	**Spiking Level, Conc. Added = 20 ug/L / 30 ug/L**
Nitrobenzene-d_5	20
2-Fluorobiphenyl	20
Terphenyl-d_{14}	20
Phenol-d_5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30

Spike concentrations are subject to change without notice.

Table 7: Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
1,4-Dioxane	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	N-Nitrosopiperidine	Isosafrole
Pyridine	Isophorone	1,2,4,5-Tetrachlorobenzene
2-Picoline	2-Nitrophenol	2,4,5-Trichlorophenol
N-Nitrosomethylethylamine	2,4-Dimethyphenol	2,4,6-Trichlorophenol
Acrylamide	Benzoic Acid	1,1'-Biphenyl
Methyl methanesulfonate	o,o',o''-Triethylphosphorothioate	2-Chloronaphthalene
N-Nitrosodiethylamine	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Ethyl methanesulfonate	alpha,alpha-Dimethyl phenethylamine	2-Nitroaniline
Benzaldehyde	2,4-Dichlorophenol	1,4-Naphthoquinone
Phenol	1,2,4-Trichlorobenzene	1,4-Dinitrobenzene
Aniline	Naphthalene	Dimethyl phthalate
Bis(2-chloroethyl)ether	4-Chloroaniline	1,3-Dinitrobenzene
Pentachloroethane	2,6-Dichlorophenol	2,6-Dinitrotoluene
2-Chlorophenol	Hexachloropropene	Acenaphthylene
n-Decane	Hexachlorobutadiene	3-Nitroaniline
1,3-Dichlorobenzene	Quinoline	2,4-Dinitrophenol
1,4-Dichlorobenzene	N-Nitrosodi-n-butylamine	Acenaphthene
Benzyl alcohol	Caprolactam	4-Nitrophenol
1,2-Dichlorobenzene	p-Phenylene diamine	2,4-Dinitrotoluene
2-Methylphenol	4-Chloro-3-methylphenol	Pentachlorobenzene
2,2'-oxybis[1-chloropropane]	Satrole, Total	Dibenzofuran
Indene	1-Methylnaphthalene	1-Naphthylamine
N-Nitrosopyrrolidine	2-Methylnaphthalene	2,3,5,6-tetrachlorophenol
3 & 4 Methylphenol	Nitrobenzene-d5	2,3,4,6-Tetrachlorophenol
N-Nitrosodi-n-propylamine		2-Naphthylamine
N-Nitrosomorpholine		Diethyl phthalate
Acetophenone		Hexadecane
2-Toluidine		Thionazin
Hexachloroethane		4-Chlorophenyl phenyl ether
2-Fluorophenol		N-Nitro-o-toluidine
Phenol-d5		4-Nitroaniline
		Fluorene
		2-Fluorobiphenyl (Surr)
		Hexachlorocyclopentadiene
		2,4,6-Tribromophenol

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Benzidine	Di-n-octyl phthalate
Diphenylamine	Pyrene	7,12-Dimethylbenz(a)anthracene
N-Nitrosodiphenylamine	Aramite, Total	Benzo[b]fluoranthene
Azobenzene	p-Dimethylamino azobenzene	Benzo[k]fluoranthene
Sulfotepp	Chlorobenzilate	Benzo[a]pyrene
1,3,5-Trinitrobenzene	Famphur	3-Methylcholanthrene
Phenacetin	Butyl benzyl phthalate	Dibenz[a,h]acridine
Diallate	3,3'-Dimethylbenzidine	Indeno[1,2,3-cd]pyrene
Phorate	2-Acetylaminofluorene	Dibenz(a,h)anthracene

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Bromophenyl phenyl ether	4,4'-Methylene bis(2-chloroaniline)	Benzo[g,h,i]perylene
Dimethoate	3,3'-Dichlorobenzidine	
Hexachlorobenzene	Bis(2-ethylhexyl) phthalate	
Atrazine	Benzo[a]anthracene	
4-Aminobiphenyl	Chrysene	
Pronamide	6-Methylchrysene	
Pentachlorophenol	Terphenyl-d14	
n-Octadecane		
Pentachloronitrobenzene		
Disulfoton		
Dinoseb		
Phenanthrene		
Anthracene		
Carbazole		
Methyl parathion		
Di-n-butyl phthalate		
Ethyl Parathion		
4-Nitroquinoline-1-oxide		
Methapyrilene		
Isodrin		
Fluoranthene		
4,6-Dinitro-2-methylphenol		

Table 8: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Actophenone	0.010
3&4-Methylphenol	0.600
N-Nitros-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroanaline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200

Table 8: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification	
Semivolatile Compounds	**Minimum Response Factor (RF)**
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroanaline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Dithyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phananthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,I)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometery (GC/MS), SW846 Method 8270D

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270D is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. TestAmerica Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270D.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5

Table 1

Compound	CAS No.	Compound	CAS No.
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270D Method Limit Groups in TALS (TestAmerica LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in TestAmerica Edison's Quality Assurance Manual (TestAmerica Edison Document No. ED-QA-LQM).

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following TestAmerica Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);

- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270)*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);
- ED-ORP-006 (*Extraction of Semivolatile Compounds in Soil Using Medium Level Extraction Techniques, SW846 Method 3550B)*.

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270D.

2.5 Standard procedure involves preparation of aqueous samples using a Reduced Volume Extraction (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270D are presented in this SOP.

2.6 This method is also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.4 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process, detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that

compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive. Benzidine, for example, is easily oxidized during extraction. Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene. Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 5890/6890/7890 (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP 7673/7683 autosampler (or

equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623 Zebron ZB-Semivolatiles, Catalog # 7HG-G027-11.

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to TestAmerica's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Toluene: J.T.Baker Resi-Analyzed, for Organic Residue Analysis (P/N 9460-03 or equivalent).

7.1.4. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.5. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from SPEX CertiPrep, Chem Service, Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep and Soils – Long Analyte List
- Full Volume Aqueous Prep and Soils – Short Analyte List
- Full Volume Aqueous Prep and Soils – Aromatic Amines
- Reduced Volume Aqueous Prep and Soils – Long Analyte List
- Reduced Volume Aqueous Prep and Soils – Short Analyte List
- Reduced Volume Aqueous Prep and Soils – Aromatic Amines

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
SPEX Super Mix (contains compounds listed in table below)	2000 *	SPEX	SVO-TANJ-16
8270 List 1/ Std #1 Megamix	Varied	Restek	567672
8270 List 1/ Std #7 N-Diphenylamine	2000	Restek	567676
8270 List 1/ Std #8	2000	Restek	568724
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#2 Amines	2000	Restek	567673
Custom Aromatic Amine Mix (see Table 5 below)	2000	Supelco	21892423
Custom Aromatic Amine Surrogate Standard (see Table 17A)	2000	Restek	569641
Bisphenol-A	1000	SPEX	S-509-MC

*SPEX Super Mix and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
SPEX Super Mix SPEX Catalog No. SVO-TANJ-16	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

Table 5 Supelco Custom Aromatic Amine Mix Catalog No. 2168334	
Analyte	**Concentration (PPM)**
Aniline	2000
o-Toluidine	2000
2-Ethylaniline	2000
2,4-Dimethylaniline	2000
3,4-Dimethylaniline	2000
2,3-Dimethylaniline	2000
2,4,5-Trimethylaniline	2000
4-Chloro-o-Toluidine	2000
4-Chloroaniline	2000
2-Naphthylamine	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 6 Full Volume Aqueous Prep and Soils – Long Analyte List Working Standards Preparation								
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**5 PPM**	**1 PPM**	**0.5 PPM**
8270 List 1/ Std #1 Megamix	1200ul	800ul	500ul	200ul	100ul	50ul	10ul	5ul
8270 List 1/ Std #7	600ul	400ul	250ul	100ul	50ul	25ul	-	-
8270 List 1/ Std #8	600ul	400ul	250ul	100ul	50ul	25ul	-	-
SPEX Super Mix	600ul	400ul	250ul	100ul	50ul	25ul	100ul*	50ul*
1,2,3,4-TCDD	-	-	100ul	-	-	-	-	-
8270 Surrogate Standard	240ul	160ul	100ul	40ul	20ul	10ul	100ul*	50ul*
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul	200ul	200ul
Bisphenol-A	600ul	400ul	250ul	100ul	50ul	25ul		
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Spex Super Mix and 8270 Surrogate Standard.

Table 7 Full Volume Aqueous Prep and Soils – Short Analyte List Working Standards Preparation						
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**5 PPM**
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul
8270 List 1/ Std#8	600ul	400ul	250ul	100ul	50ul	25ul
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**

Table 8 Full Volume Aqueous Prep and Soils - Aromatic Amines Working Standards Preparation						
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**0.5 PPM**
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul
Custom Aromatic Amine Mix	600ul	400ul	250ul	100ul	50ul	2.5ul
Custom Aromatic Amine Surrogate Std	600ul	400ul	250ul	100ul	50ul	2.5ul
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**

Table 9 Reduced Volume Extraction/LVI – Long Analyte List Working Standards Preparation								
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.2 PPM**	**0.1 PPM**
120 ppm Long Cal Std (see Table 6)	1.0 mL							
80 ppm Long Cal Std (see Table 6)		1.0 mL						
50 ppm Long Cal Std (see Table 6)			1.0 mL					
20 ppm Long Cal Std (see Table 6)				1.0 mL				
10 ppm Long Cal Std (see Table 6)					1.0 mL			
5.0 ppm Long Cal Std (see Table 6)						1.0 mL		
1.0 ppm Long Cal Std (see Table 6)							1.0 mL	
0.5 ppm Long Cal Std (see Table 6)								1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**	**5**	**5**

Table 10 Reduced Volume Extraction/LVI – Short Analyte List Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**
120 ppm Short Cal Std (see Table 7)	1.0 ml					
80 ppm Short Cal Std (see Table 7)		1.0 ml				
50 ppm Short Cal Std (see Table 7)			1.0 ml			
20 ppm Short Cal Std (see Table 7)				1.0 ml		

Table 10 Reduced Volume Extraction/LVI – Short Analyte List Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**
10 ppm Short Cal Std (see Table 7)					1.0 ml	
5.0 ppm Short Cal Std (see Table 7)						1.0 ml
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

Table 11 Reduced Volume Extraction/LVI -Aromatic Amine Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**0.1 PPM**
120 ppm Aromatic Amines Cal Std (see Table 8)	1.0 ml					
80 ppm Aromatic Amines Cal Std (see Table 8)		1.0 ml				
50 ppm Aromatic Amines Cal Std (see Table 8)			1.0 ml			
20 ppm Aromatic Amines Cal Std (see Table 8)				1.0 ml		
10 ppm Aromatic Amines Cal Std (see Table 8)					1.0 ml	
0.5 ppm Aromatic Amines Cal Std (see Table 8)						1.0 ml
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.1.2. **Initial Calibration Verification (full scan**): Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 12 8270/625 ICV -Long List Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #7 (2nd Lot)	125ul
8270 List 1/ Std #8 (2nd Lot)	125ul
SPEX Super Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
Bisphenol-A (2nd Lot)	125ul
Final Volume (ml)	**10**

Table 13 8270/625 ICV - Short List Working Standards Preparation	
Solution Name	**25 PPM**
8270 Internal Standard (2nd Lot)	200ul
8270 List 1/ Std#2 Amines (2nd Lot)	125ul
Final Volume (ml)	**10**

Table 14 Aromatic Amines ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 Internal Standard	200ul
Supelco Aromatic Amines 2nd Lot (Cat. No. 21467482)	125ul
Final Volume (ml)	**10**

Table 15 8270/625 ICV LVI - Long List Working Standards Preparation	
Solution Name	**5 PPM**
25PPM 8270/625 ICV (Long List) (see Table 12)	1.0 mL
Final Volume (ml)	**5**

Table 16 8270/625 ICV LVI -Short List Working Standards Preparation	
Solution Name	**5 PPM**
25PPM 8270/625 ICV (Short List) (see Table 13)	1.0 mL
Final Volume (ml)	**5**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 17 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.3.1 Surrogate Standards (Aromatic Amine Analysis): A 2000 ppm Surrogate Standard is purchased from Restek (Cat. # 569641) for use in spiking blanks, samples and associated QC prior to extraction and analysis of samples for Aromatic Amines (reference the applicable prep SOPs for spiking instructions).

Table 17a Aromatic Amine Surrogate Standards Solutions Restek Catalog Nos. 569641	
Surrogate Standard Compounds	**Concentration (PPM)**
Aniline-d5	5000
o-Toluidine-d9	5000
4-Chloroaniline-d4	5000

7.2.1.4. Internal Standards (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 18 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000

Table 18 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
Perylene-d12	2000

7.2.2. Calibration Standards (SIM analysis): The Edison lab currently analyzes only a select list of compounds by 8270D SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Accustandard and Spex. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 19- Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	100ppm	Accustandard	App-9-176
n-Nitrosodimethylamine	100ppm	Accustandard	APP-9-149
Hexachlorobenzene	100ppm*	Accustandard	APP-9-112
PAH Mix	100ppm	Accustandard	M-610
Bis(2-chloroethyl)ether	100ppm*	Accustandard	App-9-027
4,6-Dinitro-2-methylphenol	100ppm	Accustandard	P-3845
1,4-Dioxane	1000ppm**	Accustandard	APP-9-096

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 20 Full Volume Aqueous Prep – SIM Working Standards Preparation						
	0.025 PPM	**0.05 PPM**	**0.1 PPM**	**0.5 PPM**	**1.0 PPM**	**5.0 PPM**
Pentachlorophenol	10uL	25uL	50uL	50uL	100uL	250uL
n-Nitrosodimethylamine	10uL	25uL	50uL	50uL	100uL	250uL
PAH mix	2.5uL	5uL	100uL	25uL	50uL	100uL

Table 20 Full Volume Aqueous Prep – SIM Working Standards Preparation						
	0.025 PPM	**0.05 PPM**	**0.1 PPM**	**0.5 PPM**	**1.0 PPM**	**5.0 PPM**
Hexachlorobenzene	10uL	25uL	100uL	500uL	1000uL	2500uL
Bis(2-chloroethyl)ether	10uL	25uL	100uL	500uL	1000uL	250uL*
4,6-dinitro-2-methylphenol	50ul	100ul	200ul	200ul	250ul	500ul
1,4-Dioxane	20ul	50ul	100ul	100ul	200ul	500ul
ISTD	200uL	200uL	200uL	100uL	100uL	100uL
Final Volume (ml)	**10**	**10**	**10**	**5**	**5**	**5**

*For Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 21 Reduced Volume Extraction/LVI – SIM Working Standards Preparation						
	0.005 PPM	**0.01 PPM**	**0.02 PPM**	**0.10 PPM**	**0.20 PPM**	**1.0 PPM**
0.025 PPM Std (see Table 20)	1.0 mL					
0.05 PPM Std (see Table 20)		1.0 mL				
0.1 PPM Std (see Table 20)			1.0 mL			
0.5 PPM Std (see Table 20)				1.0 mL		
1.0 PPM Std (see Table 20)					1.0 mL	
5.0 PPM Std (see Table 20)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 6 using the stock standards detailed in Section 7.2.1.4 (above)

Table 22 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270D isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 23 -
Stock 1,4-Dioxane Isotope Dilution SIM Standards

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 24 -
Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane-d8	2000ppm	Restek	A0120108

Table 25 -
Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)

Standard Name	Concentration	Vendor	Catalog #
1,4-Dichlorobenzene-d4	2000ppm	Restek	A0121898

Table 26 -
Stock 1,4-Dioxane Isotope Dilution SIM Separate Source ICV

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane	1000ppm	Absolute	70373

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 27 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)							
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 4**	**Lev 6**	**ICV***
1,4-Dioxane	10	2	0.8	0.4	0.1	0.04	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 26.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Supelco Catalog # 47548-U) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in a standards logbook or in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to TestAmerica Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 6 months
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D

(1) : Reduced volume extaction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. .

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270D Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270D Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270D Method Limit Group for QC limits). **Note:** Three (3) surrogates are used when analyzing for Aromatic Amines (see Section 7.2.1.3.1).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198

DFTPP Key Ions and Abundance Criteria	
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;
9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section

7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 31) for the compound
Ais = Area characteristic ion (see Table 31) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 28 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 28:
Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200

Table 28: Minimum Response Factors	
Compound	**Minimum Response Factor**
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 28. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) for alternate fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.1 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be $\geq$ 0.99

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or or 0.99 correlation coefficient criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±30% of the standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. Initial Calibration Verification (ICV):.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.4. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is

required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check, and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 28. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of

the compounds in Table 28 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.6. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several minutes flush out the Sylon-Ct with toluene and finally with

methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300^{O}C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	42.0	50	43. 0	50	68.0	50
	74.0	50	128.0	50	129.0	50
	136.0	50	150.0	50	152.0	50
	93.0	50	66.0	50		
	58.0	50				
	88.0	50				

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	151.0	50	152.0	50	153.0	50
	154.0	50	162.0	50	164.0	50
	165.0	50	166.0	50		

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	94.0	50	101.0	50	142.0	50
	178.0	50	179.0	50	188.0	50
	202.0	50	264.0	50	266.0	50
	284.0	50				

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	120.0	50	228.0	50	229.0	50
	240.0	50				

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	138.0	50	139.0	50	252.0	50
	253.0	50	260.0	50	264.0	50
	267.0	50	276.0	50	278.0	50

Table 29: Target Compound - Primary and Monitoring Ions

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140^{o}C for 0.5 minutes
Column Temperature Program: 140^{o} to 320^{o}C at 22^{o}C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Screening: All samples extracts must be screened by GC/FID using the identical chromatographic conditions described in section 9.2. Screening is used to determine the dilution factor of the sample (if any) prior to GC/MS analysis (for additional details see TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision).

10.2.1.1. Aqueous samples: Prior to extract screening, the extract is diluted to 2ml and split into two 1-ml aliquots:

- One 1-ml aliquot is internal standardized with 20ul of the 2000 ng/ul internal standard solution for full scan analysis and is analyzed by GC/FID for screening.
- The other aliquot is archived for SIM analysis which is internal standardized with 20ul of 50ppm SIM Internal Standard

10.2.1.2. Soil samples: Final volume is 1ml and extracts are internal standardized with 20ul of the 2000 ng/ul internal standard solution and analyzed by GC/FID for screening.

10.2.1.3. After screening analysis, the chromatogram is evaluated for high concentrations of organics. Determine dilutions by comparing the peak heights of compounds in the sample with the internal standard. The ratio of naturally present compounds to internal standards must be <5:1.

10.2.1.4. Dilutions are made based on the screening analysis and prior to GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

$$DF = Ph / 5 \times Is$$

Where:

DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 30** to determine dilution and internal standard amount.

Table 30
Dilution Factor Calculations

DF Value	Volume of Sample (ul)	Volume of Methylene Chloride (ul)	Volume of ISTD (ul)
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10

*Prepare this dilution by serially diluting the >10, <20 dilution

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP) followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8270D are listed in Table 31.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be

made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration (µg/KG)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 31)
Ais = Area characteristic ion of associated internal std (See Table 31)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places

pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.2. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. TestAmerica Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.6. TestAmerica Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.7. TestAmerica Edison SOP No. ED-ORP-006, SW846 *Method 3550B- Extraction of Semi-Volatile Organic Compounds in Soil Using Medium--level Extraction Technique,* current revision.

15.8. TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. TestAmerica Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625,* current revision.

15.15. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. TestAmerica Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 7, date 06/08/2018
    - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
    - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
    - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
    - Added Section 9.2.4.4.5: CCV Poor Performers
    - Corrected number in section 9.2.4.5
    - Added Attachment 1 – Poor Performing Analytes
- Revision 6, date 01/12/2018:
    - Section 7.2.5 included to specify reagent and standard storage conditions.
    - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
    - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.
- Revision 5, dated 09/29/2017:
    - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
    - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
    - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
    - Section 7.2.1: added a list of full scan calibration list options.
    - Table 3: Renamed 'Full Scan Stock Standards'.
    - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
    - Section 9.2.3: updated CCV concentrations
    - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
    - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
    - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
    - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
    - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol SIM ICV standard.
    - Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
    - Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
    - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
    - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
    - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
    - Section 2.5: added text detailing the RVE/LVI options.
    - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
    - Section 6: updated to include newer GC, MS and autosampler models currently in use.
    - Section 6.1.3: added Zebron ZB column as an option.
    - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
    - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
    - Throughout document: removed references to Target and replaced with Chrom.
    - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
    - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
    - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
    - Section 8.0: Added Sample container and minimum sample size (250 ml) for

Reduced volume extraction.
- Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
- Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
- Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
- Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
- Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
- Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
- Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
- Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
- Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
- Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.
- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 **(Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR ELEMENT ANALYSES

[Methods: SW846 Methods 6010B, 6010C, 6010D, and EPA Method 200.7]

Approvals (Signature/Date):

Technology Specialist	05/30/19 Date	Health & Safety Coordinator	06/10/19 Date
Quality Assurance Coordinator	06/24/19 Date	Technical Director	07/01/19 Date

This SOP was previously identified as SOP NC-MT-012, Rev 8, dated 5/4/18

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of elements including metals in solution by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) using SW-846 Methods 6010B, 6010C, 6010D, and EPA Method 200.7. Table 1 of Appendix A lists the elements appropriate for analysis by Methods, 6010B, 6010C, 6010D, and 200.7. Additional elements may be analyzed under these methods provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity, and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Methods 6010B, 6010C, and 6010D are applicable to the determination of dissolved, suspended, total recoverable, and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP and other leachates/extracts. All matrices require digestion prior to analysis. Silver concentrations greater than 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples may be subject to error. Precipitation may occur in samples where silver concentrations exceed these levels, leading to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis. Silver concentrations must be below 0.1 mg/L in aqueous samples for the reasons discussed above.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multiple elements in solution using simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively-coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by a charge injection device (CID). The amplified photocurrents from the charge injection device (CID) are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background determination must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences must also be recognized and appropriate actions taken.

2.2. Refer to NC-IP-010, Acid Digestion of Soils by SW846 Method 3050B, and NC-IP-011, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, for details on sample preparation methods.

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version for additional definitions. Refer to Appendix B for a cross reference of method definitions.

4. INTERFERENCES

4.1. Physical, chemical, and spectral interference effects may contribute to inaccuracies in the determinations of elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of elements. Background correction is not required in cases where a background correction would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of another analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump or mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect. Chemical interferences are characterized by

molecular compound formation, ionization effects, and solute vaporization effects.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4-ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Note: Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE**: **AVOID looking directly at the plasma**.

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers must be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated pipettes

6.7. Class A volumetric flasks

6.8. Autosampler tubes

7. REAGENTS AND STANDARDS

7.1. Standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the solutions may be used for up to one year from receipt. They must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended, provided that the acceptance criteria described in laboratory-specific SOPs are met. Additional information can be found in SOP NC-QA-017. Standard or spiking concentrations, as well as vendors, are subject to change.

7.2. Working calibration, calibration verification solutions, and internal standard solutions must be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables 2 and 3 (Appendix A) for details regarding the working standard concentrations for

interference correction and spiking solutions. Refer to the reagents module in LIMS for details on standard or reagent preparation.

7.3. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.4. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to digestion.

8.3. Soil samples do not require preservation, but must be stored at 4°C ± 2° until the time of preparation.

8.4. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. Prior to analysis of any analyte using Methods 200.7, 6010B, 6010C, or 6010D the following requirements must be met.

9.1.2. Instrument Detection Limits (IDLs)

9.1.2.1. IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation; however, it should be understood that the lower limit of quantitation needs to be verified using the criteria in Section 9.9.

9.1.2.2. The IDL for each wavelength used for detection for each analyte must be determined for each instrument. The IDL must be determined annually. Whenever the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be re-determined. The IDL will be determined by multiplying the standard deviation obtained from the analysis of 7 consecutive measurements (, 6010B and 6010C) of a blank solution. 6010D and 200.7 require 10 consecutive replicates. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse at minimum.).

9.1.3. Linear Range Verification (LR):

9.1.3.1. The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the maximum concentration at which sample results can be reported. The standards used to verify the linear range must be analyzed during a routine analytical run, and must read within 10% of the expected value. Method 200.7 requires that any reading above 90% of the established LR must be diluted and re-analyzed. For Method 6010D, the upper linear range must be verified daily for samples exceeding the calibration range and must be within 10% of the true value. Samples cannot be reported above the calibration unless a standard is verified at or exceeding the concentration of the sample.

9.1.3.2. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from three different concentration standards across the estimated range. One standard must be near the expected upper limit of the estimated range. The concentration measured at the LDR must be within 10% of the true value.. If an adjustment is performed that may affect a LDR, then a new LDR must be determined. The LDR data must be documented and kept on file.

9.1.4. Lower Limit of Quantitation Check Standard (LLOQ/LLQC) – The LLOQ is the lowest point of quantitation for the laboratory. The LLOQ is a digested standard at or below the reporting limit. The LLOQ verification is analyzed on a quarterly basis for 6010D and on an as needed basis for 6010C to validate quantitation capability at low analyte concentration levels. In most cases the recovery limits for the LLOQ should be ± 35% of the true value and ± 30% for 6010C. The % RSD between successive LLOQ's should be ≤ 20%. If these limits cannot be met, the reporting limit must be reevaluated.

9.1.5. Background Correction Points:

9.1.5.1. To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side, adjacent to the wavelength, and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference, an algorithm is employed by the software for correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Purchased reference standards for individual elements are prepared and used for these determinations. Background correction points must be set prior to determining Inter-element Correction factors.

9.1.6. Inter-element Corrections (IECs):

9.1.6.1. ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If an adjustment is performed that may affect the IECs at any other time during the six month period, the IECs must be re-determined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination must be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. . An IEC must be established to compensate for any inter-element interference which results in a false analyte signal greater than $\pm$ the RL. For elements with a reporting limit of 10 µg/L or less, the signal must be $\pm$ two times the RL. To determine IECs, run a single element standard at the LDR. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element." These correction factors are updated in the software and automatically applied.

9.1.7. Rinse Time Determination:

9.1.7.1. Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 9.1.3) must be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes, it

may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Batch Definition

9.2.1. A batch is a group of no greater than 20 samples excluding QC samples (Laboratory Control Sample (LCS), Method Blank (MB), Matrix Spike (MS), and Matrix Spike Duplicate (MSD)) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.3. Method Blank (MB)

9.3.1. One MB must be processed with each preparation batch of up to 20 samples. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB must not contain any analyte of interest at, or above, the reporting limit (exception: common laboratory contaminants: see below) or at, or above, 10 % of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 10x higher than the MB contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit with the following exceptions: (a) insufficient sample for re-digestion (b) expired holding times, or (c) the elements detected in the MB are < RL for the associated samples. The 10x the concentration rule does not apply for OVAP.

Note: In cases where the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the MB is less than two times the RL. .

9.3.1.1. Re-preparation and re-analysis of all samples associated with an unacceptable MB is required when reportable concentrations are determined in the samples (see exception noted above).

9.3.1.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers.

9.3.1.3. If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

9.4. Laboratory Control Sample (LCS)

9.4.1. One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table 2 (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.4.1.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied for Method 6010B, 6010C, 6010D. For Method 200.7, control limits of 85-115% must be applied.

9.4.1.2. In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be addressed in the report narrative.

9.4.1.3. Corrective action will be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. The laboratory may re-analyze an aliquot of the LCS to verify the outlier; however, if the LCS exhibits the same anomaly upon re-analysis, the sample batch must be re-digested and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-digestion (b) expired holding times, or (c) the LCS is biased high and the sample results are < RL for those analytes.

9.5. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and

analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of un-spiked sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table 2 (Appendix A).

9.6.1.1. If the analyte recovery or RPD falls outside the acceptance range, the analyst shall determine if the MS/MSD is spiked properly and/or the matrix of the sample is the result of the MS/MSD or RPD failure. If it has been determined that the MS/MSD was not spiked properly and/or the failure is not a result of matrix then the sample along with the MS/MSD will be re-digested and analyzed.

Note: If client program requirements specify to confirm matrix interferences, re-preparation and re-analysis of the MS/MSD may be necessary.

9.6.1.2. If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported with a “4” flag.

9.6.1.3. For Method 6010C and 6010D samples – If the MS/MSD recoveries are unacceptable, the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and must be recovered to within 80% to 120% of the known value (75-125% for 6010D). If this spike fails, then the dilution test (serial dilution) must be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed and results are reported with narration.

9.7. Dilution test (Serial Dilution)

9.7.1. A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5X dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample after correction for dilution must agree within 10% (20% for Method 6010D) of the original sample determination when the original sample concentration is greater than 50 times the MDL for 6010B and 6010C, and 25 times the reporting limit for 6010D. If the results are not within acceptance criteria,

the possibility of chemical or physical interference exists and the data are flagged.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMS.

9.9. Method Detection Limits (MDLs) and MDL Checks

9.9.1. MDLs and MDL Checks are established by the laboratory as described in SOP CA-Q-S-006.

9.9.2. MDLs are easily accessible via the LIMS.

9.10. Nonconformance and Corrective Action

9.10.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. Deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Initial Calibration

10.2.1. Optimize and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank after each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. . Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument standardization date and time must be included in the raw data

10.3. Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.3.1. Calibration accuracy is verified by analyzing an ICV, prepared from a second source standard, immediately after the initial calibration. For analyses conducted under Method 200.7, the ICV must fall within ±5% of the true value for that solution and have an RSD of <3% from the replicate (minimum of three) exposures. For Methods 6010B, 6010C, and 6010D

the ICV must fall within ±10% of the true value for that solution. The % RSD must be <5% from the replicate (minimum of three) exposures for Method 6010B.

10.3.2. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB is prepared with reagent water and must contain the same concentrations of the acids used to prepare the standards. The ICB result must fall within ± the RL from zero for each element.

10.3.3. If either the ICV or ICB fail to meet criteria, the analytical sequence must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified (see Section 11.6 for the required run sequence). Exceptions: If the concentration in the ICB is > RL, sample results < RL can be reported, with an NCM. If the ICV result is outside of criteria high, sample results < RL can be reported, with an NCM.

10.4. Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)

10.4.1. Calibration accuracy is monitored throughout the analytical run through the analysis of a reference standard after every 10 samples and at the end of the analytical sequence. The CCV is prepared from stock standards from the primary supplier (or separate source from that used to prepare the ICV) at the approximate mid-range concentration The CCV for all methods must fall within ±10% of the true value for that solution. For Methods 6010B and 200.7, the RSD from replicate (minimum of three) exposures must be <5%. For Method 6010C and 6010D, there is no criterion for RSD from replicate exposures.

10.4.2. A CCB is analyzed immediately following each CCV (see Sections 11.6 for required run sequence). The CCB is the same solution that is used for the ICB and must contain the same concentrations of the acids used to prepare the standards. The CCB result must fall within ± RL from zero. If the CCB concentration is less than 10-times that of the associated samples, results may be reported if narrated and/or flagged. This is not acceptable for Ohio VAP samples.

10.4.3. If a mid-run CCV or CCB fails, all of the affected samples must be re-analyzed with valid CCV/CCB pairs (refer to Section 11.6 for an illustration of the appropriate rerun sequence). Exceptions: If the concentration in the CCB is > RL, sample results < RL can be reported, with an NCM. If the CCV result is outside of criteria high, sample results < RL can be reported, with an NCM.

10.5. Interference Check Solution Analysis (ICSA/ICSAB)

10.5.1. The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents.

Refer to Table 3 (Appendix A) for the details of ICSA and ICSAB composition. Custom multi-element ICS solutions must be used. All analytes must be spiked into the ICSAB solution; therefore, if a non-routine analyte is required, then it must be manually spiked into the ICSAB using a certified ultra-high purity single-element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number, then the non-routine elements can be controlled with the ICSA. Aluminum, iron, calcium, and magnesium must always be included in the ICSA/ICSAB pair in every sequence.

10.5.2. The ICSA and ICSAB solutions must be run at the beginning of the run (see Section 11.6 for required run sequence).

10.5.3. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analytical sequence must be terminated, the problem corrected, the instrument re-calibrated, and the samples rerun.

10.5.4. ICSA results for the non-interfering elements with reporting limits $\leq$ 10 ug/L must fall within $\pm$ 2 times the RL from zero. ICSA results for the non-interfering elements with RLs > 10 μg/L must fall within $\pm$ RL from zero. If the ICSA results for the non-interfering elements do not meet the above criteria the field sample data must be evaluated as follows.

10.5.4.1. If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted (vendor contact required if contamination is suspected).

10.5.4.2. If the affected element was not a requested analyte, then the sample data can be accepted.

10.5.4.3. If the analyst determines that interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than $\pm$ 2 x the RL from zero, then the field sample data can be accepted.

10.5.4.4. If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than $\pm$ 2 x the RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than ten times the analyte response in the ICSA.

10.5.4.5. If the data does not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary, and the affected samples re-analyzed, or the sample results must be manually corrected through application of the new IEC to the raw results.

10.6. Low Level ICV/Low Level CCV [ICVL/CCVL]

10.6.1. The ICVL/CCVL for Method 6010C must be within the 70 – 130% recovery range and analyzed at the beginning and end of the analytical sequence. .The ICVL criteria for Method 6010D is within 80-120% and analyzed at the beginning of the analytical sequence. In addition, a CCVL can be analyzed on a more frequent basis. If any analyte is outside the range indicated, the ICVL/CCVL may be re-analyzed once. If the results fall within the required values upon re-analysis, no further corrective action needs to be taken. If still outside the acceptable range, then samples containing the affected analytes at similar concentrations cannot be reported and the samples must be re-analyzed.

Note: The only exception is if the ICVL/CCVL recoveries are biased high and the associated sample is below the RL for the parameter(s) of interest. This must be addressed in the project narrative.

10.6.2. RL Verification Standard (ICVL) Method 6010B: An independent standard is analyzed after the ICB to monitor the lab's ability to produce reliable results at or near the RL-level concentrations. There is no set acceptance criteria established for this standard, but generally results should be within 50% of the expected value. Individual program requirements may vary.

10.7. Laboratory support equipment must be calibrated per SOP NC-QA-004

11. PROCEDURE

11.1. A minimum of three exposures readings for each standard, field sample, and QC sample is required. The average of the exposures is reported.

11.2. Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution.

11.3. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV), blanks (ICB, CCB), interference checks (ICSA, ICSAB), and field samples (linear range) to improve the data review process.

11.4. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5. The following procedural guidelines must be followed when using an internal standard:

11.5.1. Typically used internal standards are Yttrium and Indium. (Note: Any element can be used that is not typically found in environmental samples)

11.5.2. The internal standard (IS) must be added to every sample and standard at the same concentration. The IS is added to each analytical sample automatically through use of a third pump channel and mixing T. Internal standards must be added to blanks, samples, and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.5.3. The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.5.4. The internal standard raw intensity counts must be printed on the raw data.

11.5.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.5.5.1. If the internal standard counts fall within ±50% of the counts observed in the calibration blank then the data is acceptable.

11.5.5.2. If the internal standard counts in the field samples are outside 50%-150% than the expected level, a dilution is needed due to matrix interference.

11.6. The following analytical sequence should be used for Methods 6010B, 6010C, 6010D and 200.7:

Instrument Calibration
ICV
ICB
ICVL (6010C/6010D)
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete the run
CCVL (6010C)

11.7. Refer to Quality Control Section 9.0 for Methods 6010B, 6010C, 6010D, and 200.7

quality control criteria.

11.8. Guidelines are provided in Appendix C on procedures to minimize contamination.

11.9. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted. Acid strength is maintained in the dilution of samples by diluting the sample with the dilution blank solution.

11.10. Nonconformance documentation must be filed in the project file.

11.11. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.12. Analytical Documentation

11.12.1. Record all analytical information in the LIMS, including any corrective actions or modifications to the method.

11.12.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.12.3. Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

11.12.4. Record all sample results and associated QC into the LIMS. Level I and Level II reviews are performed in the LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates is calculated according to the following equation:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg\,/\,L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples is calculated as follows:

$$mg\,/\,Kg, dry\,weight = \frac{C \times V \times D}{W}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading $\times$ 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1. Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in Eurofins TestAmerica Canton SOP NC-WC-024.

12.10.2. Reporting Limits

12.10.2.1. The Eurofins TestAmerica Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2.2. The Eurofins TestAmerica Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.2.3. Calculations: Cr^{+3} = total Cr – Cr^{+6}

12.11. Hardness by Calculation (SM2340B)

12.11.1. Total hardness can be determined by a calculation of the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L.

12.11.2. Total Hardness mg equivalent $CaCO_3$/L = [2.497 X (Ca concentration in mg/L)] + [4.118 x (Mg concentration in mg/L)]

12.11.3. The reporting limit is 33 mg/L.

12.12. Additional equations and calculations are listed in the following SOP: Calibration Curves (General), CA-Q-P-003.

13. METHOD PERFORMANCE

13.1. Each instrument must have initial demonstration of performance data on file for each analyte of interest. ..

13.2. Refer to Table 1 in Appendix A for the list of analytes that may be analyzed using this SOP.

13.3. Training Qualification

13.3.1. The Group/Team Leader or the Supervisor has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the corporate environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. Acid waste consisting of sample and rinse solution: Any sample waste generated must be collected and disposed of in the acid waste drum located in the Metals Lab.

15.2.2. Standards must be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Final Update IV, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 3, February 2007

16.1.4. Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, SW-846, Final Update V, Method 6010D, Revision 4, October 2012

16.1.5. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7

16.1.6. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002

16.1.7. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.8. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9. Corporate Quality Management Plan (CQMP), current version

16.1.10. Revision History

Historical File:		Revision 1: 01/07/09		
(As CORP-MT-0001NC)		Revision 2: 02/22/11		
Revision 2.0: 10/27/97		Revision 3-A: 04/17/12		
Revision 2.1: 04/19/99		Revision 4: 09/13/13		
Revision 3.1: 10/04/00		Revision 5: 4/30/15		
Revision 3.2: 01/19/01		Revision 6: 10/13/16		
Revision 3.3: 12/05/01		Revision 7: 10/27/17		
Revision 3.4: 01/08/04		Revision 8: 05/04/18		
(As NC-MT-012)				

Revision 0: 01/08/04				

**4/9/19: changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. Eurofins TestAmerica Canton QC Program, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3. Detection and Quantitation Limits, CA-Q-S-006

16.2.4. Hexavalent Chromium (Colorimetric), NC-WC-024

16.2.5. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.6. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011

16.2.7. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from Methods 6010B and 200.7

17.1.1.1. Eurofins TestAmerica Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in-house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. In determining IECs because of lack of definition clarification for “concentration range around the calibration blank,” The laboratory uses the following criteria: +/- RL or +/- 2x RL if RL is 10ug/L or less.

17.1.1.3. Whenever a new or unusual matrix is encountered, a series of tests must be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because Eurofins TestAmerica Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, Eurofins TestAmerica Canton may select to perform a dilution test on one

sample in each prep batch. . At Eurofins TestAmerica Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. Eurofins TestAmerica Canton requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a specific client-identified sample.

17.1.2. Modifications from Method 200.7

17.1.2.1. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix. The former matrix provides improved performance, relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied to samples. The laboratory runs these methods concurrently; therefore, the solution used is used for all analyses.

17.1.2.2. Section 7.12 of Method 200.7 indicates that the quality control sample ICV must be prepared at a concentration greater than or equal to 1 ppm. This SOP specifies ICV concentrations which are appropriate to the mid range of the calibration. The intent of the ICV, verification of the primary calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.3. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 Section 10.4 states that results must fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The laboratory uses the following criteria: +/- RL or +/- 2x RL if RL is 10ug/L or less.

17.1.3. Modifications from Method 6010B

17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants may be allowed up to two times the reporting limit. This is not acceptable for Ohio VAP projects. For Ohio VAP projects, the MB must be clean down to the RL, unless the sample result is below the RL.

17.1.3.2. Method 6010B Section 8.6.1.3 states that the results of the calibration blank must be within three times the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean. Eurofins TestAmerica Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. .

APPENDIX A - TABLES

TABLE 1: Methods 200.7, 6010B, 6010C and 6010D Target Analyte List

Element	Symbol	CAS #
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-24-6
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6

TABLE 2: Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	Concentration (ug/L)
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	50000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Lithium	1000
Magnesium	50000
Manganese	500
Molybdenum	1000
Nickel	500
Potassium	50000
Selenium	2000
Silicon	1000
Silver	50
Sodium	50000
Strontium	1000
Thallium	2000
Vanadium	500
Zinc	500
Boron	1000
Tin	2000
Titanium	1000

Note: *Concentration are subject to change.*

TABLE 3: Interference Check Sample Concentrations

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Lithium		500
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silicon		10000
Silver	-	1000
Sodium	-	10000
Strontium		1500
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

APPENDIX B - CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW 6010B, 6010C, AND 6010D EUROFINS TESTAMERICA CANTON SOP

EPA 200.7	SW 6010B / 6010C / 6010D	Eurofins TestAmerica Canton SOP
Calibration blank	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory fortified blank (LFB)	Laboratory control sample	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX C - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept clean.

Powdered Gloves must not be used in the Metals Lab since the powder contains silica and zinc as well as other metallic analytes. Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS AND SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

[Method: MCAWW Method 245.1, SW846 Method 7470A, SW846 7471A, and 7471B]

Approvals (Signature/Date):

Technology Specialist	08/05/19 Date	Health & Safety Coordinator	07/30/19 Date
Quality Assurance Manager	07/26/19 Date	Technical Director	07/30/19 Date

This SOP was previously identified as SOP No. NC-MT-014, Rev 8, dated 3/18/19

Facility Distribution No. _____ Distributed To:_____
The controlled copies of this SOP are the PDF copy of the SOP that is posted to the laboratory's SOP Directory and, where applicable, the SOP that is printed and placed in the General Chemistry SOP binder. Printed or electronic copies of this SOP distributed outside of the facility are considered uncontrolled.

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW846 Methods 7470A, 7471A, and 7471B, and MCAWW Method 245.1.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants and potassium permanganate has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity, and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation, and volume of sample used.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed; however, Method 7471A is usually the method of choice. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in surface and saline waters, and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. Methods 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes, biological material, and sludge-type materials. All matrices require sample preparation prior to analysis.

1.6. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric (aqueous samples), or hydrochloric and nitric acids (soil samples). Organic mercury compounds are oxidized with potassium permanganate (aqueous and soil samples) and potassium persulfate (aqueous samples), and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control (QC) section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Chemical and physical interferences may be encountered when analyzing samples using this method.

4.3. Potassium permanganate, which is used to break down organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide (as sodium sulfide) do not interfere with the recovery of inorganic mercury from reagent water.

4.4. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.5. Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (maximum 25 mL); because during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride

4.6. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.7. Samples containing high concentrations of oxidizable organic materials, as evidenced by high Chemical Oxygen Demand (COD) levels, may not be completely

oxidized by this procedure. When this occurs, the recovery of mercury will be low. Reducing the volume of original sample used can eliminate this problem.

4.8. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Refer to Appendix B for Contamination Control Guidelines.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Mercury (10PPM in Reagent)	Oxidizer Corrosive Poison	0.1 g/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns

			and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydroxyl-amine Hydro-chloride	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The Ultra Violet (UV) light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory, and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature-controlled hot block or equivalent

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption cell with quartz end windows perpendicular to the longitudinal axis: Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury-specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL)

6.2.3. Peristaltic pump which can deliver 1 L/min

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min

6.2.5. Recorder or printer

6.2.6. Drying device to prevent condensation in cell

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. Plastic bottles – capable of holding 100 mL

6.4. Nitrogen or argon gas supply, welding grade or equivalent

6.5. Calibrated automatic pipettes

6.6. Class A volumetric flasks

6.7. Top-loading balance, capable of reading up to two decimal places

6.8. Thermometer (capable of accurate readings at 95 °C)

6.9. Disposable cups or tubes

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore Deionized Water (DI) system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm calibration and ICV) mercury standards are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017. Refer to the reagent module in the Laboratory Information Management System (LIMS) for details on standard preparation.

7.3. Working mercury standard (0.1 ppm): Take 2 mL of the 10 ppm stock standard (Section 7.2) and dilute to 200 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (300 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. Refer to the reagent module in LIMS for details on standard preparation.

7.4. Working ICV standard (0.1 ppm): Take 1 mL of the 10 ppm ICV stock standard (Section 7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. Refer to the reagent module in LIMS for details on standard preparation.

7.5. The calibration standards must be prepared fresh daily from the working standard (Section 7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles for solid samples or by transferring 0, 0.1, 0.25, 0.5, 2.5, and 5.0 mL aliquots of the working mercury standard into sample preparation bottles for aqueous samples and proceeding as specified in Section 11. (A calibration curve is valid for 24 hours from the completion of preparation.) The laboratory control sample (LCS) solution is prepared by transferring 5.0 mL (solids) or 2.5 mL (waters) of working standard into sample preparation bottles and proceeding as specified in Section 11. Refer to the reagent module in LIMS for details on standard preparation.

Note: Alternate approaches to standard preparation may be taken, and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I (Appendix A) are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table 1 (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.10. Sulfuric acid (H_2SO_4), concentrated, traces metal grade or better

7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.12. Stannous chloride solution: Add 50g $\pm$ 0.5g of stannous chloride and 25 mL of concentrated HCl, and bring to a final volume of 500 mL with DI water.

Note: Stannous sulfate may be used in place of stannous chloride. Prepare the stannous sulfate solution according to the recommendations provided by the instrument manufacturer.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 240g $\pm$ 0.5g of sodium chloride and 240g $\pm$ 0.5g of hydroxylamine hydrochloride to every 2000 mL of reagent water.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 100g of potassium permanganate for every 2000 mL of reagent water.

7.15. Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate for every 2000 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of sample collection to the time of sample analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples and biological material do not require preservation, but must be collected in wide-mouth glass jars with PFTE-lined lids and stored at 4° C ± 2° C (and/or freezing for tissues) until the time of analysis.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.2.1. Four aliquots of the laboratory check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3. A Preparation Batch is a group of up to 20 samples, excluding QC Samples (Laboratory Control Sample (LCS), Method Blank (MB), Matrix Spike (MS), Matrix Spike Duplicate (MSD)), that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain an MB, an LCS and an MS/MSD. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes. In some cases, at client request, it may be appropriate to process a MS and sample duplicate (DU) in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.4. Method Blank (MB)

9.4.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB must not contain any analyte of interest at, or above, the reporting or at, or above, 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the MB contamination level).

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be re-digested and re-analyzed.

9.4.2. Re-digestion and re-analysis of all samples associated with an unacceptable MB is required when reportable concentrations are determined in the

samples (see exception noted above).

9.4.3. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.4.4. If the above criteria are not met and re-analysis is not possible due to limited sample quantity, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5. Laboratory Control Sample (LCS)

9.5.1. One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits, the system is out of control and corrective action must occur. See Section 12 for the LCS calculation.

9.5.2. For Method 245.1, the LCS must be 85% - 115%. For Methods 7470A, 7471A, and 7471B, the laboratory control sample recovery must be 80%-120%.

9.5.3. Corrective action must be re-digestion and re-analysis of the batch unless the LCS recovery is greater than the upper control limit and the sample results are less than the RL. In that situation the results may be reported with proper narration.

9.6. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.7.1. One MS/MSD pair must be processed for each preparation batch. An MS is a field sample to which known concentrations of target analytes have been added. An MSD is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and MS. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates (DU) in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for

MS/MSD analysis. Spiking levels are provided in Table 1 (Appendix I). See Section 12 for the MS/MSD and Relative Percent Difference (RPD) calculation.

Note: for Method 245.1, an MS/MSD pair is required for every 10 samples.

9.7.2. If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. A control limit of, 70 – 130% for Method 245.1, and 20% RPD must be applied to the MS/MSD. A control limit of 80-120% for Methods 7470A, 7471A, and 7471B and 20% RPD must be applied to the MS/MSD.

9.7.3. If the analyte recovery or RPD falls outside the acceptance range, the analyst shall determine if the MS/MSD is spiked properly and/or the matrix of the sample is the result of the MS/MSD or RPD failure. If it has been determined that the MS/MSD was not spiked properly and/or the failure is not a result of matrix then the sample along with the MS/MSD will be re-digested and analyzed.

Note: If client program requirements specify to confirm matrix interferences, re-preparation and re-analysis of the MS/MSD may be necessary.

9.7.4. If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data are reported with a “4” as a flag. In the event an MS/MSD analysis is not possible, notation in the project narrative is required.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018

9.8.2. Control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMS

9.9. Method Detection Limits (MDLs) and MDL Checks

9.9.1. MDLs and MDL Checks are established by the laboratory as described in SOP CA-Q-S-006.

9.9.2. MDLs are easily accessible via the LIMS

9.10. Nonconformance and Corrective Action

9.10.1. Any deviations from QC procedures must be documented as a nonconformance. Procedural deviations are not allowed for Ohio VAP Projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.

10.2. Due to the differences in preparation protocols, separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a calibration blank. One standard must be at, or below, the reporting limit. Analyze standards in ascending order beginning with the calibration blank. Refer to Section 7 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥ 0.995, or the instrument must be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be re-digested.

10.7. Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.7.1. Calibration accuracy is verified by analyzing a second source standard ICV. The ICV result must fall within 5% (for method 245.1) or 10% (for methods 7470A, 7471A, and 7471B) of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within $\pm$ the reporting limit (RL) from zero. See Section 12 for the ICV calculation. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, and the instrument recalibrated (see Section 11.6.4. for required run sequence). The calibration curve standards are reanalyzed to determine if the failure was instrument related. If the cause of the ICV or ICB failure was not directly instrument-related, the corrective action must include re-digestion of the ICV,

ICB, CRA, CCV, and CCB with the calibration curve. For Ohio VAP, the sample batch must be re-digested.

Note: If the ICV and/or ICB fail criteria on the high side, samples with results below the reporting limit may be reported with proper narration.

10.8. Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)

10.8.1. Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard from the calibration curve.

10.8.2. The CCV result for Methods 7470A, 7471A, and 7471B must fall within 20% of the true value for that solution. For Method 245.1, the criterion is ± 10%. See Section 12 for the CCV calculation.

10.8.3. A CCB is analyzed immediately following each CCV (see Section 11.6.4 for required run sequence). The CCB result must fall within ± RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit, then the results can be reported. Sample results may be reported when bracketed by valid CCV/CCB pairs. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be re-digested.

Note: If the CCV and/or CCB fail criteria on the high side, sample results that are below the reporting limit may be reported with proper narration.

10.9. Detection Limit Standard (CRA) - A CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. It is recommended that the recovery be ± 50% of the true value. If the CRA recovery is outside of the recommended criterion, correct any problem and re-analyze. Re-calibration may be required. The CRA is only required when requested.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP projects.

11.3. Standard and Sample Preparation- Solids

11.3.1. All calibration and calibration verification standards (ICV, ICB, CCV, and CCB) are processed through the digestion procedure as well as the field samples.

11.3.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (Section 7.3) into a series of sample digestion bottles. The ICB/CCB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. For the ICV, transfer a 5 mL aliquot of the working ICV standard to the digestion bottle. The ICV standard must be from a source other than that used for the calibration standards. For the CCV, transfer a 5.0 mL aliquot of the working standard into a sample digestion bottle. Add reagent water to each standard bottle for a total volume of 10 mL.

Note: Alternate volumes and concentrations of standard may be prepared as long as the accuracy and final standard concentrations support laboratory or project reporting limits.

11.3.3. To each LCS standard, add 0.6 g of Teflon chips or other suitable solid matrix, 5 mL of reagent water and 5 mL of the working mercury standard (0.1 ppm) (see Section 7).

11.3.4. To the MB bottle, add 0.6 g of Teflon chips or other suitable solid matrix and 10 mL of reagent water.

11.3.5. For each sample, transfer 0.6 g ± 0.1 g of a well-mixed sample into a clean sample digestion bottle and add 10 mL of reagent water.

11.3.6. Add 5 ml of Aqua Regia to all containers.

11.3.7. Heat for two minutes in a hot block at 90 - 95 ° C.

11.3.8. Add 40 mL of distilled water.

11.3.9. Add 15 mL of potassium permanganate solution. Cover containers with digestion bottle lids.

11.3.10. Heat for 30 minutes in the hot block at 90 - 95 °C.

11.3.11. Record the time on and off of the hotblock in the batch info.

11.3.12. Check the temperature when heating is finished and note in batch information whether temperature requirement was met.

11.3.13. Cool

11.3.14. Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.3.15. Bring each standard, quality control sample, and sample up to a final volume of 100 mL with reagent water.

11.3.16. Samples are ready for analysis (section 11.6).

11.4. Sample preparation for incremental sampling method (ISM) solids

11.4.1. ISM samples are prepped and sub-sampled per SOP requirements for ISM. The Metals laboratory will receive a single sample aliquot from the Pre-Prep department containing approximately 3.0g of each sample inside a 500mL plastic container. The acceptable range for HG sample mass is approximately 2.5-3.5g.

11.4.1.1. 3.0g of Teflon boiling chips or other suitable solid matrix are weighed into a 500mL bottle for both the MB and LCS. Preparatory method for QC samples will be the same as for the client samples.

11.4.1.2. The laboratory control sample (LCS) is spiked with 25mL of the HG Calibration solution. Matrix spike (MS) and matrix spike duplicate (MSD) are spiked with 5mL of the HG Calibration solution.

11.4.1.3. Add 50mL of deionized water to each sample container except the LCS. Add 25mL of the reagent water into the LCS container for an even volume.

11.4.1.4. To each sample container, including MB and LCS, add 25mL of Aqua Regia.

11.4.1.5. Place each sample into individual sample slots inside the water bath. Heat samples for two minutes at 90-95 degrees Celsius.

11.4.1.6. Add 200mL of deionized water into each sample container.

11.4.1.7. Add 75mL of potassium permanganate solution.

11.4.1.8. Cover 500mL containers with its bottle lids.

11.4.1.9. Digest for 30 minutes inside the water bath at 90-95 degrees Celsius.

11.4.1.10. Add 30 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.4.1.11. Bring each standard, quality control sample, and sample up to a final volume of 500 mL with reagent water.

11.4.1.12. Samples are ready for analysis (section 11.6).

11.5. Standard and Sample Preparation Waters

11.5.1. All calibration and calibration verification standards (ICV, ICB, CCV, and CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.1, 0.25, 0.5, 2.5 and 5.0 mL aliquots of the working standard (Section 7.3) into a series of sample digestion bottles containing 50 mL of reagent water. For the ICV, transfer a 2.5 mL aliquot of the working standard to the digestion bottle containing 50 mL of reagent water. The ICV standard must be from a source other than that used for the calibration standards. For the CCV, transfer a 2.5 mL aliquot of the working standard into a sample digestion bottle containing 50 mL of reagent water.

11.5.2. The Method Blank (MB) consists of 50 mL of reagent water. The LCS consists of 2.5 mL of the HG working standard, and 50 mL of reagent water.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations support laboratory or project reporting limits.

11.5.3. Transfer 50 mL of well-mixed sample or standard to a clean sample digestion bottle. Continue preparation as described under Section 11.6.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.5.4. Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL of concentrated HNO_3.

11.5.5. Add 7.5 mL of potassium permanganate solution. For samples high in organic materials or chlorides, 7.5 mL may be insufficient to fully break down all organic mercury compounds. If the purple color does not persist for at least 15 minutes after the addition of potassium permanganate, the sample must be discarded and re-prepped at a dilution.

11.5.6. Add 4 mL of potassium persulfate solution, cover, and heat for two hours in a hot block at 90 - 95 °C.

11.5.7. Cool samples.

11.5.8. Add 3 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.5.9. Bring each standard, quality control sample and sample up to a final volume of 75mL with reagent water.

Note: The final volume for water samples is 50 mL prior to the addition of digestion reagents. After all prep reagents are added and the samples are digested, reagent water is added to bring each sample to a consistent 75 mL volume. The calibration curve and samples are prepared in the exact same manner. To avoid result and limit calculation errors, the 50 mL volume is recorded as the final sample volume in the preparation log in LIMS.

11.5.10. Samples are ready for analysis (section 11.6).

11.6. Sample Analysis

11.6.1. Automated determination: Refer to the instrument manual for details on instrument setup.

11.6.2. Create a calibration curve by plotting response of calibration standards vs. concentrations of mercury. Determine the mercury concentration in the samples from the linear fit of the calibration curve. The calibration acceptance criteria are listed in Section 10.6. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.6.3. All measurements must fall within the defined calibration range to be valid. Dilute and re-analyze all samples for analytes that exceed the highest calibration standard.

Note: For Method 245.1, the measurement must fall within 90% of the highest standard. Sample measurements that exceed 90% of the highest standard must be diluted and reanalyzed.

11.6.4. The following analytical sequence is consistent with Methods 7470A, 245.1, 7471A and 7471B.

Instrument Calibration
ICV

ICB
CRA if required
CCV
CCB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete the run
CCV
CCB

11.6.5. Refer to Quality Control Section 9.0 for quality control criteria.

Note: Samples include the MB, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.6.6. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.6.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

11.7. Analytical Documentation

11.7.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.7.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.7.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.7.4. Record all sample results and associated QC in LIMS. Level I and Level II reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike (MS) recoveries are calculated according to the following equation:

$$\%\ R\ = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
Matrix Spike (MS) = determined spiked sample concentration
Matrix Spike Duplicate (MSD) = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:
C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor must be omitted from the above equation.

12.6. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7. The Laboratory Control Sample (LCS) percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are generated by this method.

15.2.1.1. Acid Waste: This waste disposed of in the designated container labeled "Acid Waste".

15.2.1.2. Acid waste/aqueous waste generated by the analysis: Samples are disposed of in the acid waste drum located in the Metals lab. The contents of the drum are neutralized and released to the POTW.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury)

16.1.2. "Methods for the Chemical Analysis of Water and Wastes", Rev. 3.0 (1994)

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A

(Mercury)

16.1.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Revision 2, February 2007, Method 7471B (Mercury)

16.1.5. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.6. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.7. Corporate Quality Management Plan (CQMP), current version

16.1.8. Revision History

Historical File: (formerly Corp-MT-0007NC, NC-MT-011, and NC-MT-013)				
Revision 1.1: 04/17/97		Revision 2: 03/20/13		
Revision 2.2: 02/06/01		Revision 3: 06/05/13		
Revision 2.3: 05/15/01		Revision 4: 06/06/15		
Revision 2.4: 10/28/02		Revision 5: 06/15/15		
Revision 2.5: 11/24/04		Revision 6: 06/23/16		
Revision 0: 12/12/07 (011)		Revision 7: 02/13/18		
Revision 1: 01/07/09 (011)		Revision 8: 03/18/19		
Revision 0: 01/07/09 (013)				
Revision 0: 09/27/10 (014)				
Revision 1-A: 04/17/12				

**4/9/19: changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. Standards and Reagents, NC-QA-017

16.2.6. Calibration Curves (General), CA-Q-S-005

16.2.7. Selection Of Calibration Points, CA-T-P-002

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from Reference Method

17.1.1. Modifications from Method 7471A

17.1.1.1. Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2. Modifications from both Methods 7470A and 245.1

17.1.2.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, “Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology.”

17.1.3. Modifications from Method 7470A

17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit if the samples associated with the method blank are equal to or above the reporting limit.

17.1.4. Modifications from Method 245.1

17.1.4.1. Method 245.1 states that standards are not heated. Eurofins TestAmerica Canton prepares heated standards for this method.

17.1.4.2. Stannous Chloride is prepared in hydrochloric acid, instead of sulfuric acid, per instrument manufacturer recommendations.

17.1.4.3. Section 9.3.4 of the method states that the CCB must be less than the MDL. The laboratory uses the criteria that the CCB result must fall within ± RL from zero.

17.2. Tables and Appendices

APPENDIX A - TABLES

TABLE 1. MERCURY REPORTING LIMITS, CALIBRATION STANDARD, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Standard Aqueous RL (mg/L)	0.0002
TCLP RL (mg/L)	0.002
Std 0 (mg/L)	0
Std 1/CRA (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L)	0.010
ICV (mg/L)	0.005
CCV/Laboratory Control Sample (LCS) (mg/L)	0.005
LCS (mg/L)	0.005
Matrix Spike (MS) (mg/L)	0.001
TCLP Matrix Spike (MS)	0.005

APPENDIX B - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by Deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas should be kept clean.

Powdered Gloves should not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: ACID DIGESTION FOR AQUEOUS SAMPLES

[Methods: SW846 3005A, 3010A, and MCAWW 200 Series]

Approvals (Signature/Date):

Technology Specialist	08/30/18 Date	Health & Safety Coordinator	09/05/18 Date
Quality Assurance Manager	10/19/18 Date	Technical Director	10/05/18 Date

This SOP was previously identified as SOP No. NC-IP-011, Rev 7, dated 8/4/17

Facility Distribution No. _____ Distributed To:_____

The controlled copies of this SOP are the PDF copy of the SOP that is posted to the laboratory's SOP Directory and, where applicable, the SOP that is printed and placed in the General Chemistry SOP binder. Printed or electronic copies of this SOP distributed outside of the facility are considered uncontrolled.

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, and 3010A.

1.2. The applicability of each of these preparation protocols to specific elements is detailed in Tables 1 and 2(Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.

1.4. SW846 Method 3005A / MCAWW Method 200.8 are used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP and ICPMS.

1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total, total recoverable and dissolved metals determination by ICP.

1.6. SW846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP and ICPMS

2.1.1 A representative aliquot of sample is heated with nitric and hydrochloric acids and reduced to a low volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 / - Preparation for Total Metals Analysis by ICP

2.2.1 A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with 1:1 hydrochloric acid, filtered (if necessary), and diluted to volume.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron from the glassware will migrate into the sample solution during and following sample processing. For critical low-level determinations of boron, it is recommended quartz or plastic lab ware be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids.

4.5. Visual interferences or anomalies (such as dilution due to oily matrix) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates, or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.6. Exposure to hazardous chemicals must be maintained **as low as reasonably**

achievable. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid spills must be neutralized immediately, flushed with water, and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire-polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

EQUIPMENT AND SUPPLIES
Hot plate/digestion block: capable of maintaining a temperature of 90-95º C
Calibrated thermometer: range 0-110º C
Beakers, assorted sizes: Griffen, calibrated digestion beakers, or equivalent
Vapor recovery Device (Watch glasses, ribbed, or other device)
Whatman #41 filters, or equivalent
Funnels
Graduated cylinder: Various
Analytical balance capable of weighing to ± 0.01 g
Repeaters or suitable reagent dispensers
Pipettes and disposable tips: various volumes
Volumetric flasks: Class A
pH indicator strips: range 0-6
Plastic digestate storage bottles: Corning snap seals, or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the elements of interest as demonstrated through the analysis of method blanks (MB) as defined in the determinative SOPs.

7.2. Reagents

Reagents
Nitric Acid (HNO_3): Concentrated, trace metal grade or better
Hydrochloric Acid (HCl): Concentrated, trace metal grade or better

Reagents
1:1 Hydrochloric Acid: dilute concentrated HCl with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

7.3. Standards

7.3.1. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3.2. Working ICP laboratory control sample (LCS)/matrix spike (MS) spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards. Final concentrations are available in the LIMS. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS/MS solution must be made fresh every six months. The expiration date of the working ICP LCS/MS solution is the shorter of six months, the expiration date of the corresponding stock standard or when verification from an independent source indicates a problem.

7.3.3. The ICPMS LCS/MS solution is provided directly by the vendor. No further standard preparation is necessary.

7.3.4. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary. Final digestate spike concentrations are available in the LIMS.

7.3.5. The LCS/MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional element(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.3.6. Aqueous LCS and MS samples are prepared as described in Sections 9.4 and 9.3.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and must be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion. For metals samples that require preservation, the Sample Receiving Department must note the time of acid addition.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

8.4. For dissolved metals analysis, the samples must be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike / matrix spike duplicate (MS/MSD) If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. See Policy QA-003 for further definition of the batch. Laboratory generated QC (MB, LCS, MS/MSD) are not included in the sample count.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated element concentrations or false positive data.

9.2.2. Aqueous and TCLP MBs are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.

9.2.3. TCLP Leachate Blanks (LBs) are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must contain all elements of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

9.3.2. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution. The LCS is then processed through the appropriate procedure as described in Section 11.

Note: TCLP LCS is prepared by spiking 50 mL of leachate fluid with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS solution and taking it through the appropriate procedure as described in Section 11.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target elements have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the matrix spike) prepared and analyzed along with the sample and MS. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates (DU) in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.

Note: For Method 200.7, an MS/MSD pair is required for every 10 samples. 2 MS/MSD pairs must be analyzed if the batch contains more than 10 samples.

9.4.2. The aqueous MS sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution. The MS sample is then processed as described in Section 11.

9.4.3. The ICP TCLP MS/MSD sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution. For ICPMS

analysis, the MS/MSD is spiked the same as a water sample. The MS/MSD sample is then processed as described in Section 11.

Note: The TCLP matrix spike standard must be added prior to preservation of the leachate.

Note: If elements outside of the RCRA list are requested, l mL of additional spiking solution(s) is added.

9.6 Additional information on QC samples can be found in QA Policy QA-003.

9.7 Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMS.

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOP CA-Q-S-006.

9.8.2 MDLs are easily accessible via the LIMS.

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. Procedural deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1. The hot plate/hot block temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hot block temperature log

10.2. Support equipment used for this procedure will be checked for calibration as per SOP NC-QA-015, current version. .

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate

variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP projects.

11.2. All digestion procedures must be carried out in a properly functioning hood.

11.3. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.4. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the lab supervisor or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.

11.5. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab.

11.6. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.7. The following procedure must be followed for all aqueous sample preparations.

11.7.1. Mix sample by shaking the container.

11.7.2. Measure 50 mL of the sample into a calibrated digestion tube.

Note: For samples with particulate matter, the aliquot may be taken through a repeated series of shake and pour steps.

11.7.3. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions.

11.7.4. Measure 50 mL of reagent water into a calibrated digestion tube for the MB.

11.7.5. Measure 50 mL of reagent water into a calibrated digestion tube for the LCS and add the appropriate spiking solutions.

11.8. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP / ICPMS

11.8.1. To the sample container, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.8.2. Cover with ribbed watch glass.

11.8.3. Heat at 90-95°C until volume is reduced to less than or equal to 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of element and the sample must be re-prepared.

11.8.4. Cool the beaker in a fume hood.

11.8.5. If insoluble materials are present, filter the sample through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™

Note: If any samples in a preparation batch are filtered, the MB and LCS associated with that batch must also be filtered.

11.8.6. Rinse container, vapor recovery device, and filter paper with reagent water to ensure complete sample transfer.

11.8.7. Adjust the final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.9. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

11.9.1. To the sample container, add 3.0 mL of concentrated HNO_3.

11.9.2. Cover with ribbed watch glass.

Place container on hot block 90-95°C, and reflux until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing) and the volume is less than or equal to 20 mL. **NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY.** Doing so will result in the loss of element and the sample must be re-prepared.

11.9.3. Add 5 mL of 1:1 HCl.

11.9.4. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.9.5. Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™.

Note: If any samples in the QC batch are filtered, the MB and LCS associated with that batch must also be filtered.

11.9.6. Rinse container, vapor recovery device, and filter paper with reagent water to ensure complete sample transfer.

11.9.7. Adjust final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered, or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.10. Analytical Documentation

11.10.1. Record all analytical information in the LIMS, including any corrective actions or modifications to the method.

11.10.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each analyst must make an initial demonstration of capability and yearly continuing demonstrations of capability for each individual element. This requires analysis of four QC Check samples.

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Acidic waste containing nitric acid generated by the extraction: This waste is disposed of in the designated container labeled "Acid Waste".

15.2.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

16.1.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983

16.1.3 TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 2: 05/17/11		
(as CORP-IP-0003NC)		Revision 3-A: 06/28/12		
Revision 1.2: 03/20/00		Revision 4: 06/28/14		
Revision 1.3: 09/25/01		Revision 5: 08/06/14		
Revision 1.4: 02/19/03		Revision 6: 02/26/16		
Revision 1.5: 12/07/04		Revision 7: 08/04/17		
Revision 1.6: 02/07/07				
(as NC-IP-011)				
Revision 0: 01/07/09				
Revision 1: 01/28/10				

**4/17/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1 TestAmerica QC Program, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Detection and Quantitation Limits, CA-Q-S-006

16.2.5 Standards and Reagents, NC-QA-017

16.2.6 Subsampling, NC-IP-001

16.2.7 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW846 Methods 6010B, 6010C, and 200.7, NC-MT-012

16.2.8 Inductively Coupled Plasma – Mass Spectrometry, NC-MT-002

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference methods

17.1.1 Modifications applicable to SW-846 reference methods

17.1.1.1. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by Table 3-1 and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples. EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated "As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.1.2. Modifications Specific to Method 3010A

17.1.1.2.1. Section 11.8.3 of this SOP requires the sample be reduced to a volume –less than or equal to 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL, but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical elements through volatilization.

17.1.1.3. Modifications Specific to MCAWW Methods

17.1.1.3.1. It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846

methods and therefore were combined under the scope of this SOP. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

17.2. Appendices

APPENDIX A - TABLES

TABLE 1: Approved Preparation Method Elements - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element.

Note: *Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.*

TABLE 2: Approved Preparation Method Elements – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: *Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.*

TABLE 3: TCLP Reporting Limits and Regulatory Limits for ICP

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)
Arsenic	500	5000
Barium	10000	100000
Cadmium	100	1000
Chromium	500	5000
Lead	500	5000
Selenium	250	1000
Silver	500	5000

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept clean.

Powdered Latex Gloves must not be used in the metals laboratory since the powder contains zinc, as well as other metallic elements. Only unpowdered latex or nitrile gloves must be used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic elements.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: CLEANUP PROCEDURES FOR ORGANIC EXTRACTABLE SAMPLES

[Method: SW846 Method 3660B, 3665A, 3630C, 3610B and 3620C]

Approvals (Signature/Date):

Technology Specialist	01/24/19 Date	Health & Safety Coordinator	01/14/19 Date
Quality Assurance Manager	01/16/19 Date	Technical Director	01/15/19 Date

This SOP was previously identified as SOP NC-OP-025, Rev 8, dated 3/20/18

1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used when performing clean-ups using Sulfuric Acid (3665A), Copper and Tetrabutylammonium Sulfite (TBA) (3660B), Silica Gel (3630C), Potassium permanganate (3665A), Florisil (3620C), and Alumina (3610B) for semivolatile organic compound extracts-.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Sulfuric acid cleanup is used to remove certain pesticide compound interferences from organic extracts. Sulfuric acid cleanup may be used for pesticides dependent upon which pesticides are target analytes. The extract to undergo cleanup is mixed with sulfuric acid. The mixture is vortexed and the extract is separated and removed from the acid.

2.2. Copper and Tetrabutylammonium sulfite (TBA) cleanups are used to remove chromatographic interference caused by sulfur. The extract to undergo cleanup is mixed with either copper granules or TBA and Isopropyl alcohol. The mixture is vortexed and the extract is removed from the sulfur cleanup reagent.

2.3. Silica gel cleanup is used to remove chromatographic interferences caused by compounds of different chemical polarity. The sample extract is eluted through a Resprep ® Silica SPE cartridge (or equivalent), the eluate is further concentrated and the extract is then ready for analysis.

2.4. Potassium Permanganate ($KMNO_4$) cleanup is used to remove interferences from organic extracts. The extract that is to undergo cleanup is mixed with 5% permanganate ($KMNO_4$) solution. The mixture is vortexed, and the extract is separated and removed from the permanganate.

2.5. Florisil® is used for the cleanup of pesticide residues and other chlorinated hydrocarbons to remove steroids, ketones, esters, glycerides, alkaloids, and some carbohydrates from the sample extract. The sample extract is eluted through a Resprep ® Florisil ® SPE cartridge (or equivalent). The eluate is further concentrated and the extract is then ready for analysis.

2.6. Alumina cleanup is used to remove chromatographic interference caused by compounds of different chemical polarity. The sample extract is eluted through a Supelclean ™ LC-Alumina-B SPE cartridge (or equivalent), the eluate is further concentrated and the extract is then ready for analysis.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. The copper technique requires that the copper be very reactive, as evidenced by a bright shiny appearance (see Sec. 7.1.14 for the preparation of this reagent).

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Nitrile gloves should be used when performing this cleanup procedure. Latex and vinyl gloves provide no significant protection against organic solvents.

5.3. The following is a list of the materials that MAY be used in this method, which have a serious or significant hazard rating. **Note:** This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. The actual materials used during the cleanup will vary, depending on which compounds are to be filtered out of the sample extract. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Isopropyl alcohol	Flammable	400 ppm TWA	Inhalation of vapors irritates the respiratory tract. Exposure to high concentrations has a narcotic effect, producing symptoms of dizziness, drowsiness, headache, staggering, unconsciousness and possibly death.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/ m^3 –TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Florisil®	Irritant	TLV 10mg/ m^3 PEL 5mg/ m^3	May cause irritation if inhaled or adsorbed through the skin.
Potassium Permanganate	Poison Irritant	OSHA PEL 5 mg/m*f* (air)	Irritation to skin, eyes, lungs, mucous membranes, and GI tract. Metal poisoning hazard. Dermatitis.
Tetrabutylammo-nium hydrogen sulfate	Poison, Irritant, Hygroscopic agent	None Listed	Irritation to skin and eyes. Harmful if swallowed (do not induce vomiting). May be harmful if absorbed through skin.
Activated Copper	Toxin	0.1 mg/m^3 TWA (fumes) 1 mg/m^3 TWA (Dusts)	Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Avoid prolonged or repeated exposure. Do not ingest. May be harmful if swallowed.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 The addition of sulfuric acid to the sample extract has the potential to create an exothermic reaction. Both can cause severe burns.

5.5 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read “Caution/Use Hood!” **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 The preparation of hazardous standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the Laboratory Supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Bottles, amber: 250 mL, 1 L

6.2. Autopipettor or volumetric pipettes: 1 and 5 mL capacity

6.3. Disposable pipettes: Pasteur-type

6.4. Culture Tubes: 5 or 10 mL

6.5. Vortex Mixer

6.6. Beakers: Various

6.7. Top loading balance: Capable of accurately weighing $\pm$ 0.01 g

6.8. Centrifuge

6.9. Mechanical shaker

6.10. Manifold system to hold SPE cartridges

6.11. Glass bottles

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Hexane: Pesticide grade or equivalent

7.1.2. Methylene Chloride: Ultra-resi analyzed or equivalent

7.1.3. Sodium sulfite, anhydrous: reagent grade

7.1.4. Sulfuric acid (H_2SO_4), concentrated: reagent grade

7.1.5. 5% Potassium permanganate ($KMNO_4$) solution: reagent grade, purchased

7.1.6. Florisil®, activated: pesticide residue grade cartridges such as Resprep® Florisil® SPE cartridges or equivalent

7.1.7. Silica Gel: chromatography grade only cartridges such as Resprep® Silica SPE cartridges or equivalent

7.1.8. Supelclean™ LC-Alumina-B SPE cartridges or equivalent

7.1.9. Acetone: reagent grade

7.1.10. Isopropyl alcohol - Pesticide grade or equivalent

7.1.11. Organic free reagent water

7.1.12. Tetrabutylammonium hydrogen sulfate: reagent grade

7.1.13. Tetrabutylammonium (TBA)-Sulfite Reagent

7.1.13.1. Dissolve 3.39 g of Tetrabutylammonium hydrogen sulfate per 100 mL reagent water and mix well. Extract the100 mL aliquot of deionized water and Tetrabutylammonium hydrogen sulfate solution three times with 20 mL of hexane. Dispose of hexane layer. **Slowly** add 25 g anhydrous sodium sulfite per 100 mLs and shake until dissolved. Store this solution in an amber bottle with PTFE- lined screw cap at room temperature for at least one month

7.1.14. Granulated Activated Copper, 30 mesh Restek part number 26136 or equivalent

7.1.14.1. Clean copper granules by rinsing with hexane. Place copper granules in a glass jar and cover with hexane. Place jar on the mechanical shaker and allow to mix for approximately 5 minutes. Remove the excess hexane and dry under a stream of nitrogen.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. See the individual extraction or analytical procedure.

9. QUALITY CONTROL

9.1. The method blank and laboratory control sample associated with any samples subjected to any of the types of cleanups covered in this SOP must also be subjected to the same cleanup procedure(s).

9.2. Refer to the analytical SOPs NC-GC-007, NC-GC-045, NC-GC-042, NC-GC-043, NC-GC-046, and NC-GC-013 for QC requirements

9.3. Nonconformance and Corrective Action

9.3.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action. Procedural deviations are not allowed for Ohio VAP samples.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP samples.

11.3. Allow all extracts to reach room temperature if they were in cold storage.

11.4. All PCB extracts will initially go through the sulfuric acid stripping and screening process, followed by further cleanups based on screen results.

11.5. Sample Preparation

11.5.1. Sulfuric acid

11.5.1.1. Add approximately 5 mL of H_2SO_4 to 2 mL of sample extract into a culture tube.

11.5.1.2. Vortex for about one minute

11.5.1.3. Centrifuge

11.5.1.4. Remove the extract (top layer) from the acid, and place in a clean culture tube. Examine the extract layer for color and visible emulsions. Repeat as necessary.

11.5.2. Tetrabutylammonium sulfite (TBA)

11.5.2.1. Into a culture tube, add approximately 1-2 mLs of sample extract, 1 mL of TBA solution, and 2 mL of isopropyl alcohol. Shake

vigorously or vortex for one minute, after which a precipitate will form.

11.5.2.2. Add 5 mL of reagent water and shake or vortex for one minute. Allow the phases to separate or centrifuge.

11.5.2.3. Remove the sample extract (top layer) from the TBA.

11.5.2.4. Repeat as necessary.

11.5.3. Copper clean up

11.5.3.1. Transfer approximately 1-2 mLs of sample extract onto a culture tube.

11.5.3.2. Add approximately 2 g of cleaned copper granules to the centrifuge tube. (The copper will fill the tube to approximately the 0.5 mL mark). Vigorously mix the extract and the copper for at least 1 min on the mechanical shaker or vortex. Allow the phases to separate.

11.5.3.3. Separate the extract from the copper by drawing off the extract with a disposable pipette and transfer to a clean vial.

Note: This separation is necessary to prevent further degradation of the pesticides.

11.5.4. Silica Gel for PCB

11.5.4.1. A 1 gram silica gel cartridge is utilized: Restek catalog No. 24038 or equivalent.

11.5.4.2. Condition the cartridge with 4 mL of hexane. Slowly open the cartridge valve to allow a few drops per cartridge to pass through the sorbent bed and manifold to remove all of the air bubbles. Close the valve and allow the solvent to soak the entire sorbent bed for 5 minutes.

11.5.4.3. Slowly open the cartridge valve to allow the hexane to pass through the cartridge. Close the cartridge valve when there is still approximately 1mm of solvent above the sorbent bed. Do not allow cartridge to become dry.

Note: If the cartridge goes dry repeat the conditioning step.

11.5.4.4. Transfer 2 mL of extract to the cartridge. Allow the extract to soak the sorbent bed for 1 minute. Open the cartridge valve to allow the

extract to pass through the cartridge bed at approximately 2 mL per minute.

11.5.4.5. When the extract has mostly passed through the cartridge, but before the cartridge becomes dry, rinse the extract sample vial with 0.5 mL of hexane, and add this to the cartridge to complete the quantitative transfer.

11.5.4.6. Close the cartridge valve when there is still approximately 1mm of hexane above the sorbent bed, ensuring that the cartridge never goes dry.

11.5.4.7. Place a new culture tube into the sample rack corresponding to the cartridge position.

11.5.4.8. Add 5 mL of hexane to the cartridge. Allow the solvent to soak the sorbent bed for 1 minute. Slowly open the cartridge valve and collect the eluate into the culture tube. Allow the cartridge to completely drain into the culture tube. Close the cartridge valve. Remove the sample tube and concentrate down to 2 mL using an N-Evap. This eluate will contain the PCBs as well as some pesticides.

11.5.5. Silica Gel for DRO

11.5.5.1. A 1 gram silica gel cartridge is utilized: Restek catalog No. 24038 or equivalent.

11.5.5.2. Condition the cartridge with 4 mL of methylene chloride. Slowly open the cartridge valve to allow a few drops per cartridge to pass through the sorbent bed and manifold to remove all of the air bubbles. Close the valve and allow the solvent to soak the entire sorbent bed for 5 minutes.

11.5.5.3. Slowly open the cartridge valve to allow the methylene chloride to pass through the cartridge. Close the cartridge valve when there is still approximately 1mm of solvent above the sorbent bed. Do not allow cartridge to become dry.

Note: If the cartridge goes dry repeat the conditioning step.

11.5.5.4. Transfer 2 mL of extract to the cartridge. Allow the extract to soak the sorbent bed for 1 minute. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 mL per minute.

11.5.5.5. Close the cartridge valve when there is still approximately 1mm of extract above the sorbent bed, ensuring that the cartridge never goes dry.

11.5.5.6. Place a new culture tube into the sample rack corresponding to the cartridge position.

11.5.5.7. Add 5 mL of methylene chloride to the cartridge. Allow the solvent to soak the sorbent bed for 1 minute. Slowly open the cartridge valve and collect the eluate into the culture tube

11.5.5.8. When the extract has mostly passed through the cartridge, but before the cartridge becomes dry, rinse the extract sample vial with 0.5 mL of methylene chloride, and add this to the cartridge to complete the quantitative transfer.

11.5.5.9. Allow the cartridge to completely drain into the culture tube. Close the cartridge valve. Remove the sample tube and concentrate down to 2 mL using an N-Evap. This eluate will contain the Diesel Range Organics.

11.5.6. Potassium Permanganate (KMNO4)

11.5.6.1. Add approximately 5 mLs of 5% $KMNO_4$ solution to approximately 2 mLs of extract.

11.5.6.2. Vortex for about one minute

11.5.6.3. Centrifuge

11.5.6.4. Remove the extract (top layer) from the $KMNO_4$ solution and place in a clean culture tube. Repeat the cleanup up to three times if necessary.

11.5.7. Florisil

11.5.7.1. A 1 gram Florisil cartridge is utilized: Restek catalog No. 24031 or equivalent

11.5.7.2. Condition the cartridge with 4 mL of hexane. Slowly open the cartridge valve to allow a few drops per cartridge to pass through the sorbent bed and manifold to remove all of the air bubbles. Close the valve and allow the solvent to soak the entire sorbent bed for 5 minutes.

11.5.7.3. Slowly open the cartridge valve to allow the hexane to pass through the cartridge. Close the cartridge valve when there is still approximately 1mm of solvent above the sorbent bed. If the cartridge goes dry, repeat the conditioning step.

11.5.7.4. Transfer 1 mL of sample to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at a drop-wise rate until the hexane level reaches the top of the top frit. Stop the flow.

11.5.7.5. Fill the cartridge with a 90:10 hexane/acetone solution.

11.5.7.6. Place a culture tube into the sample rack corresponding to the cartridge position and restore the drop-wise flow to collect the effluent from the cartridge until 10 ml has been collected. Additional 90:10 hexane/acetone solution will need to be added to collect 10 ml. Stop the flow once 10 mL has been collected.

11.5.7.7. Remove the sample and concentrate down to the final volume of slightly less than 1 ml. Use hexane to bring back up to 1 ml.

Note: If employed in pesticide extracts, a pesticides performance check solution must be recovered at 80 to 110%, the recovery of trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected

11.5.7.8. See manufacturer specifications about storage of the cartridges since they are critical for optimum performance.

11.5.8. Alumina

11.5.8.1. A 1 gram Alumina cartridge is utilized: Supelco Part No. 57084 or equivalent

11.5.8.2. Condition the cartridge with 4 mL of hexane. Slowly open the cartridge valve to allow a few drops per cartridge to pass through the sorbent bed and manifold to remove all of the air bubbles. Close the valve and allow the solvent to soak the entire sorbent bed for about 5 minutes.

11.5.8.3. Slowly open the cartridge valve to allow the hexane to pass through the cartridge. Close the cartridge valve when there is still approximately 1mm of solvent above the sorbent bed. Do not allow cartridges to become dry. If the cartridge goes dry, repeat the conditioning step.

11.5.8.4. Transfer 2 mL of the sample extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 ml/minute; do not exceed 5 mL/minute.

11.5.8.5. When the extract has mostly passed through the cartridge, but before the cartridge becomes dry, rinse the sample vial with 0.5 mL of hexane and add this to the cartridge to complete the quantitative transfer. Close the cartridge valve when there is approximately 1mm of solvent above the sorbent bed, ensuring that the cartridge never goes dry.

11.5.8.6. Place a new culture tube into the sample rack corresponding to the cartridge position.

11.5.8.7. Add one tube volume of hexane to the cartridge. Allow the solvent to soak the sorbent bed for about 1 minute or less. Slowly open the cartridge valve and collect the eluate into the test tube. Close the cartridge valve. Repeat this step one more time. Remove the sample and concentrate down to the final volume of 2 mL using an N-Evap.

11.6. Sample Analysis

11.6.1. Refer to SOPs NC-GC-013, NC-GC-046, NC-GC-045, NC-GC-042, NC-GC-043 and NC-GC-007 for sample analysis.

11.7. Analytical Documentation

11.7.1. All cleanups must be documented.

11.7.2. Record all analytical information in the LIMS, including any corrective actions or modifications to the method.

11.7.3. Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Training Qualifications

13.1.1. The Group/Team Leader has the responsibility to ensure that this procedure is

performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

15.3. Waste Streams Produced by the Method

15.3.1. Glassware from extract cleanup / lab trash: Glassware, such as culture tubes, disposable pipettes, etc., is placed in the laboratory trash containers.

15.3.2. Copper granules are collected for recycling.

15.3.3. Sulfuric acid waste from the cleanup: Sulfuric is collected in a five-gallon container labeled "Sulfuric Acid Waste" located in the fume hood in the GC Prep Room.

15.3.4. Flammable solvent waste collected during silica gel prep and elution. This is collected in a 2.5-gallon waste container labeled "Flammable Waste" located in the GC Prep Room.

15.3.5. Waste hexane: This is collected in a 2.5 gallon waste container labeled "Flammable Waste" located in the GC Prep Room.

15.3.6. Flammable solvent waste collected during TBA sulfite preparation and Potassium permanganate waste: This is collected in a 2.5-gallon waste container labeled "Flammable Waste" located in the GC Prep Room.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition?, Florisil® Cartridge Cleanup, Method 3620C

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Silica Gel Cleanup, Method 3630C, Revision 3, December 1996

16.1.3. SW-846, Test methods for Evaluating Solid Waste, Third Edition, Sulfuric Acid/Permanganate Cleanup, Method 3665A, Revision 1, December 1996

16.1.4. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Sulfur Cleanup, Method 3660A

16.1.5. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Sulfur Cleanup, Method 3660B, Revision 2, December 1996

16.1.6. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Alumina Cleanup Method 3610B, Revision 2, December 1996

16.1.7. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.8. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9. Corporate Quality Management Plan (CQMP), current version

16.1.10. Revision History

Historical File:				
Revision 0: 08/17/06		Revision 4: 03/04/11		Revision 8: 03/20/18
Revision 1: 04/30/08		Revision 5; 08/05/13		
Revision 2: 01/07/09		Revision 6: 09/30/14		
Revision 3: 02/02/10		Revision 7: 12/14/15		

**4/17/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. Gas Chromatographic Analysis Based on Methods 8082 and 8082A, SOP NC-GC-045

16.2.2. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and

8081B, SOP NC-GC-042

16.2.3. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, SOP NC-GC-043

16.2.4. Analysis of Pesticides and PCBs by Method 608, SOP NC-GC-007

16.2.5. Analysis of Pesticides and PCBs by Method 608.3, SOP NC-GC-046

16.2.6. Total Petroleum Hydrocarbons by Wisconsin DNR Modified DRO Method, NC-GC-013

16.2.7. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Troubleshooting Guide

17.1.1. Sulfuric Acid

17.1.1.1. If the acid and extract will not separate, try diluting the extract before acid cleanup.

Title: CONTINUOUS LIQUID / LIQUID EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3520C AND 600 SERIES

[Methods: SW846 3520C and 600 Series]

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Technical Director	Date

This SOP was previously identified as SOP No. NC-OP-037, Rev 6, dated 11/7/17

Facility Distribution No. _____ **Distributed To:_____**

The controlled copies of this SOP are the PDF copy of the SOP that is posted to the laboratory's SOP Directory and, where applicable, the SOP that is printed and placed in the General Chemistry SOP binder. Printed or electronic copies of this SOP distributed outside of the facility are considered uncontrolled.

1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, and TCLP leachate, matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS) using Continuous Liquid/Liquid Extraction. The procedures are based on SW846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 8015D, 608, 608.3, 625, and 625.1.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Continuous Liquid/Liquid Extraction

2.1.1 A measured volume of sample (typically 1 liter, or 250 mL for reduced volume extraction requiring large volume injection) is placed into a continuous liquid/liquid extractor, adjusted if necessary, to a specific pH, and extracted with the appropriate solvent for 18-24 hours.

2.2. Concentration

2.2.1 Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read “Caution/Use Hood!” **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints, which can become stuck. Technicians must use Kevlar or

other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3520 Extraction Continuous Liquid/Liquid

5.9.1. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid-liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.9.2. Ensure all solvents are away from liquid-liquid extractor. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.9.3. Ensure all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	CLLE	Conc
pH Indicator paper, ranges: 0-14, 7.5-14, 0-6	√	
Graduated cylinder: Class A 1 liter. (other sizes may be used as needed)	√	√
Methylene chloride collection tank for waste disposal	√	
Initial volume template	√	
Chlorine Test Strips	√	
Solvent dispenser pump or 100 mL Class A graduated cylinder		√
Continuous liquid / liquid extractor	√	
Round or flat bottom flask: 250	√	
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	√	√
Cooling condensers	√	
Heating mantle: rheostat controlled	√	
Auto-timer for heating mantle	√	
Beakers: 250 & 400 mL, graduated	√	√
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control 90-98°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 / 2.5/ 40 mL capacity with Teflon®-lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√

EQUIPMENT AND SUPPLIES	CLLE	Conc
Culture tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette, syringe 1 mL	√	
Funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents For Extraction Procedures

7.1.1. All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	CLLE	Conc
Sodium hydroxide (NaOH), pellets: reagent grade	√	
Sodium hydroxide solution, 10 N: dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	
Sulfuric acid (H_2SO_4), concentrated: reagent grade	√	
Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	
Organic-free reagent water	√	
Sodium thiosulfate ($Na_2S_2O_3$): granular		
Sodium sulfate (Na_2SO_4), granular, anhydrous: purify by heating at 800°C a minimum of two hours	√	√
Extraction / exchange solvents: methylene chloride, hexane, acetone, pesticide quality or equivalent	√	√
Acetone, methylene chloride: used for cleaning	√	√
TCLP Fluid #1 : Made fresh daily in the Leachates department, or see SOP NC-OP-033 TCLP-SPLP	√	√

7.2. Standards

7.2.1. Stock Standards

7.2.1.1 Stock standards are purchased as certified solutions. Standards shall be stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

7.2.2.1 Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light or

stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

7.2.3.1 The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards.

7.2.3.2 Spiking solutions must be refrigerated and protected from light or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. The holding time for aqueous samples is seven days from sampling to extraction. If permitted by the state or responsible party, PCB samples have a 1 year holding time.

8.3.2. For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If

clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank (MB)

9.3.1. A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same level as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Aqueous MBs use 1000 mL (or 250 mL for reduced volume extraction) of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.3.3. TCLP MBs use 250 mL of leachate fluid spiked with the surrogates. SPLP MBs use 1000 mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The MB goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. LCSs are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.3.1 through 9.3.3), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. An MS/MSD is required for every 20 samples, otherwise a nonconformance memo must be included with associated samples.

9.7. QC requirements can be found in the various associated analytical SOPs.

9.8. Initial Demonstration of Capability

9.8.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.9. Control Limits

9.9.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.10. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs

9.11. Method Detection Limits (MDLs) and MDL Checks

9.11.1. MDLs and MDL Checks are established by the laboratory as described in SOP CA-Q-S-006.

9.11.2. MDLs are accessible via the LIMs.

9.12. Nonconformance and Corrective Action

9.12.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volumes of solvents into the appropriate sized glass culture tubes gravimetrically. The “standard” glass culture tubes are sealed, and the meniscus is noted by marking a line on the bottles. The glass

culture tubes containing the sample extracts are then compared against the "standard" glass culture tubes of the appropriate volume and solvent to ensure the volumes are consistent. The bottle top dispenser is calibrated quarterly and must be within ±5% of the target volume with an RSD ≤ 1%.

10.2. Calibration verification of the initial volume template is required annually. Bottle top dispensers are verified quarterly. See SOP NC-QA-004, current revision for details on volumetric equipment calibration.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo. Procedural variations are not allowed for Ohio VAP projects.

11.2. Continuous Liquid/Liquid Extraction from Water Samples

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2. Assemble the apparatus. Add approximately 250 mL of methylene chloride (or approximately 100 mL for reduced volume extractor bodies) to the extractor body. Add three to five boiling chips to the round-bottom distilling flask. Label the flask with a LIMS ID label.

11.2.3. Measure the initial sample pH with wide-range pH by inserting a disposable pipette into the sample, and placing a drop of sample onto the wide range pH paper. Record the pH on the extraction benchsheet. pH will be entered manually into the LIMS during level I review.

11.2.4. For 600 series tests, check for the presence of chlorine by inserting a disposable pipette into the sample and placing a drop of sample onto a chlorine test strip. If no chlorine is present, proceed to section 11.2.5. If chlorine is present in any amount, add 80 mg to 100 mg of sodium thiosulfate. Test the sample again. If chlorine is still present, add another 80 to 100 mg of sodium thiosulfate. Repeat sodium thiosulfate additions and testing until the chlorine is gone. Note the presence of chlorine in the appropriate column in the worksheet in the LIMS.

11.2.5. Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. Volumes will be entered manually into the LIMS during level I review. Prepare a MB, LCS, and MS/MSD for

each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. Use 1 L of reagent water for method blanks and LCS. If the sample cannot be prepared using continuous liquid/liquid extraction due to matrix, a waste dilution may be required. Refer to SOP NC-OP-043 for the waste dilution procedure.

Note: bottles that do not fit the volume template must be measured using a Class A graduated cylinder.

11.2.6. Use 250mL of leachate for TCLP semivolatiles and TCLP pesticides. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides. Dilute to about 1 liter with reagent water.

11.2.7. For a TCLP method blank and LCS, measure 250 mL of the buffer solution in a beaker and transfer to the continuous liquid/liquid extractor. Dilute to about 1 liter with reagent water. For an SPLP method blank and LCS, measure 1000 mL of the buffer solution using the volume template and transfer to the continuous liquid/liquid extractor. No dilution with reagent water is required.

11.2.8. Less than one liter of sample may be used for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event, dilute the sample to about 1 liter with reagent water. This must be documented with a Non-Conformance Memo.

11.2.9. Add reagent water to the extractor body until approximately 150 mL (approximately 50mL for reduced volume preps) of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Prime the extractor using reagent water.

11.2.10. The MB and samples are spiked with the surrogates, the LCS and matrix spikes with the surrogates, and matrix spiking solutions. All samples are spiked in the original sample bottle.

11.2.11. Pour the sample into the extraction vessel. Use 15 mL of Methylene Chloride to rinse the bottle and pour it directly into the extraction vessel. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible. Attach cold condenser (about 10^{o}C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours (24 hours required for 600 series).

Note: for BNA analyte Acrylamide there is **no** pH adjustment. Separate batch QC (without pH adjustment) must be prepared for Acrylamide.

11.2.12. If extraction at a secondary pH is required (see Table 1), turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample on the pH paper. Record the adjusted pH on the benchsheet. Re-attach the condenser, and turn on the heating mantle. Extract for 18-24 hours.

11.2.13. Turn off the heating mantle and allow the extractor to cool.

11.2.14. Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.4 for concentration.

11.3. Concentration

11.3.1. According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.3.2. Kuderna-Danish (KD) Method

11.3.2.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL K-D flask. Label the CT and K-D. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.2.2. Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.3.2.3. Place the KD apparatus on a water bath (90-98^{o}C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.2.4. Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes

will be lost.

11.3.2.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4. Nitrogen Evaporation to Final Concentration

11.4.1. Transfer the CT to the evaporation apparatus.

11.4.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3. Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.4.4. Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the “standard” glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.5.3. Record all sample and associated QC information in the LIMS. Level I and Level II reviews are performed in the LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each analyst must have initial and continuing demonstrations of capability data on file and the laboratory must maintain corresponding method detection limit files.

13.1.2. See SOP NC-QA-028, current revision, for details on demonstrations of capability. See SOP NC-QA-021, current revision, for details on MDLs.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental

Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride are disposed of in the Methylene chloride collection tank. This tank is then periodically rolled to the Tank Room, the pH is verified, contents are neutralized with sodium bicarbonate, pH re-verified, and Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.3. **Assorted flammable solvent waste from various rinses**: These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane- Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials:** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**: These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3520C, and 3580A

16.1.2. Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.3. EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608 and 608.3

16.1.4. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.5. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.6. Corporate Quality Management Plan (CQMP), current version

16.1.7. Revision History

Historical File:		Revision 1: 01/07/09 (NC-OP-032)		
(formerly CORP-OP-0001NC)		Revision 0: 03/03/11 (NC-OP-037)		
Revision 3.4: 10/16/98		Revision 1: 04/24/12		
Revision 3.5: 04/22/99		Revision 2: 02/05/13		
Revision 3.6: 05/13/99		Revision 3: 04/05/13		
Revision 3.7: 03/20/01		Revision 4: 06/04/14		
Revision 3.8: 05/23/01		Revision 5: 11/09/15		
Revision 3.9: 04/22/02		Revision 6: 11/07/17		
Revision 4.0: 02/04/03		4/2/19: Changed Logo. No change to revision		
Revision 4.1: 10/07/03		# or Date.		
Revision 4.2: 01/30/06				
Revision 0: 03/12/08 (NC-OP-032)				

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.6. Analysis of Pesticides and PCBs by EPA Methods 608 and 608.3, NC-GC-007

16.2.7. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.8. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and 8081B, NC-GC-042

16.2.9. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, NC-GC-043

16.2.10. Gas Chromatographic Analysis of PCBs Based on Methods 8082 and 8082A, NC-GC-045

16.2.11. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 and 608.3 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.1.3. Sodium Sulfate is heated for 1 hour at 800°C to purify. The reference method lists a minimum of 4 hours at 400°C.

17.1.4. The SW-846 method recommends that the water bath temperature for the concentration step be set at 10-20 °C above the solvent boiling point. The laboratory maintains the water bath temperatures at 90 – 98 °C.

17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA	Acid ext; 1-2 or Base ext; 11-12	Acid ext; 1-2 or Base ext; 11-12
BNA Acrylamide only[2]	NA	NA
Pesticide/PCB	5-9	None
TPH	1-2	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

[2] If the laboratory is analyzing for Acrylamide, the pH of the sample is not adjusted.

TABLE 2
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
BNA	N/A	2.0 mL
PCB	Approximately 18 mL hexane – water	5.0 for SPLP / TCLP 2.0 for regular aqueous and LVI*
Pesticides	Approximately 18 mL hexane	5.0 for SPLP2.0 for regular aqueous*
Pesticides/TCLP	Approximately 18 mL hexane	3.0 mL
BNA – SIM	N/A	2.0 mL 5.0 mL for reduced volume preps
TPH	N/A	5 mL

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3 Surrogate Spiking Solutions		
Analyte Group	**Surrogate Spike Solution ID**	**Volume (mL)**
BNA	20 ppm BNA Surrogate Spike	1.0 mL
PEST	0.2 ppm DCB/TCX	0.25 mL for 2 mL final, 0.5 mL for 5 mL final
TPH	40 ng/uL o-Terphenyl	1.0 mL
PCB	0.2 ppm DCB/TCX	0.25 mL for 2 mL final, 0.5 mL for 5 mL final
BNA – SIM	20 ppm BNA Surrogate Spike	0.1 mL

* Note: surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection.

TABLE 4 Matrix Spike and LCS Solutions		
Analyte Group	**Matrix Spike Solution ID**	**Volume (mL)**
PEST	1.0 ppm Pest NPDES Spike	0.25 mL for 2 mL final, 0.5 mL for 5 mL final
TPH	2500 ppm Diesel Spike	1.0 mL
PCB	10 ppm PCB Spike	0.25 mL for 2 mL final, 0.5 mL for 5 mL final
BNA – SIM	20 ppm BNA List 1	0.1 mL
BNA	20 ppm BNA All-Analyte Spike	1.0 mL

* Note: surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection.

TABLE 5

Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	20
	Nitrobenzene-d_5	20
	2-Fluorophenol	20
	Phenol-d_5	20
	2,4,6-Tribromophenol	20
	Terphenyl-d_{14}	20
Pesticides PCB	Decachlorobiphenyl	0.2
	Tetrachloro-m-xylene	0.2
TPH	o-Terphenyl	40.0

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	1,1'Biphenyl	20
	1,2,4,5-Tetrachlorobenzene	20
	1,2,4-Trichlorobenzene	20
	1,2-Dichlorobenzene	20
	1,2-Diphenylhydrazine	20
	1,3-Dichlorobenzene	20
	1,3-Dinitrobenzene	20
	1,4-Dichlorobenzene	20
	1,4-Dioxane	20
	1-Methylnaphthalene	20
	2,2'-oxybis[1-chloropropane]	20
	2,3,4,6-Tetrachlorophenol	20
	2,4,5-Trichlorophenol	20
	2,4,6-Trichlorophenol	20
	2,4-Dichlorophenol	20
	2,4-Dimethylphenol	20
	2,4-Dinitrophenol	40
	2,4-Dinitrotoluene	20
	2,6-Dichlorophenol	20
	2,6-Dinitrotoluene	20
	2-Chloronaphthalene	20
	2-Chlorophenol	20
	2-Methylnaphthalene	20
	2-Methylphenol	20
	2-Nitroanaline	20
	2-Nitrophenol	20
	3&4-Methylphenol	20
	3,3'-Dichlorobenzidine	40
	3-Methylphenol	10
	3-Nitroanaline	20
	4,6-Dinitro-2-methylphenol	40
	4-Bromophenyl phenyl ether	20
	4-Chloro-3-methylphenol	20
	4-Chloroanaline	20
	4-Chlorophenyl phenyl ether	20
	4-Methylphenol	10
	4-Nitroanaline	20
	4-Nitrophenol	40

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	20
	Acenaphthylene	20
	Acetophenone	20
	Aniline	20
	Anthracene	20
	Atrazine	40
	Azobenzene	20
	Benzaldehyde	40
	Benzidine	40
	Benzo[a]anthracene	20
	Benzo[a]pyrene	20
	Benzo[b]fluoranthene	20
	Benzo[g,h,i]perylene	20
	Benzo[k]fluoranthene	20
	Benzoic acid	40
	Benzyl alcohol	20
	Bis(2-chloroethoxy)methane	20
	Bis(2-chloroethyl)ether	20
	Bis(2-ethylhexyl)phthalate	20
	Butyl benzyl phthalate	20
	Caprolactam	40
	Carbazole	20
	Chrysene	20
	Dibenz(a,h)anthracene	20
	Dibenzofuran	20
	Diethyl phthalate	20
	Dimethyl phthalate	20
	Di-n-butyl phthalate	20
	Di-n-octyl phthalate	20
	Fluoranthene	20
	Fluorene	20
	Hexachlorobenzene	20
	Hexachlorobutadiene	20
	Hexachlorocyclopentadiene	20
	Hexachloroethane	20
	hexadecane	20
	Indene	40
	ideno[1,2,3-cd]pyrene	20
	isophorone	20
	Methyl phenols, Total	40
	Naphthalene	20
	n-Decane	20

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
	Nitrobenzene	20
	N-Nitrosodimethylamine	20
	N-Nitrosodi-n-propylamine	20
	N-Nitrosodiphenylamine	40
	n-Octadecane	20
	Pentachlorophenol	40
	Phenanthrene	20
	Phenol	20
	Pyrene	20
	Pyridine	20
Pesticides TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pesticides NPDES	Aldrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0
TPH	Diesel Fuel	2500 **µg/mL**

Note: *Other analytes may be added per program requirements. For OVAP, only analytes listed in the reference methods are certified.*

Title: SEPARATORY FUNNEL EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3510C AND 600 SERIES

[Methods: SW846 3510C and 600 Series]

Approvals (Signature/Date):

Technology Specialist	08/21/18 Date	Health & Safety Coordinator	08/21/18 Date
Quality Assurance Manager	10/15/18 Date	Technical Director	10/12/18 Date

This SOP was previously identified as SOP No. NC-OP-038, Rev 7, dated 3/19/18

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous and TCLP leachate matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/Mass Spectrometry (GC/MS) using Separatory (Sep) Funnel Extraction. The procedures are based on SW846 and 600 series methodologies and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 8015D, 608, 608.3, 625.1, and 625.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

2.1.1. A measured volume of sample, (typically one liter, or 250 mL for low volume initiative/ [LVI]) is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Concentration

2.2.1. Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3 - Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read “Caution/Use Hood!” **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of hazardous standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3510 Separatory Funnel

5.9.1. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting must be done immediately after the sample container has been sealed and inverted. Periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-

risk activity. The use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sep Fun.	Conc
Separatory funnel: 2 L	√	
Separatory funnel rack	√	
pH indicator paper, ranges: 0-14, 7.5-14, 0-6	√	
Chlorine test strips: 0-10 ppm		
Class A Graduated cylinder: 1 liter. (other sizes may be used as needed)	√	√
Centrifuge	√	
Methylene chloride collection tank	√	√
Initial volume template	√	
Solvent dispenser pump or 100 mL Class A graduated cylinder		√
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)		√
Beakers: 250 & 400 mL, graduated 450mL wide-mouth glass jars	√ √	√ √
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water Bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 mL, 2.5mL, and 40 mL capacity with Teflon®-lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√
Culture tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette, syringe 1 mL	√	
Glass wool	√	
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√
Paper towels	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

7.1.1. All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep Fun.	Conc
Sodium hydroxide (NaOH), pellets: reagent grade	√	
Sodium hydroxide solution, 10 N: dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	
Sulfuric acid (H_2SO_4), concentrated: reagent grade	√	
Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	
Hydrochloric acid (HCl)	√	
Sodium Thiosulfate: reagent grade	√	
Organic free reagent water	√	
Sodium Chloride (NaCl) crystals	√	
Sodium Chloride Solution: add NaCl crystals to DI water until the DI water is saturated.	√	
Sodium sulfate (Na_2SO_4), granular anhydrous: purify by heating at 800°C a minimum of one hour.	√	√
Extraction/exchange solvents: methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√
Acetone, methylene chloride: used for cleaning	√	√
TCLP Fluid #1: Made fresh daily in the Leachates department, or see SOP NC-OP-033 TCLP-SPLP	√	

7.2. Standards

7.2.1. Stock Standards

7.2.1.1. Stock standards are purchased as certified solutions. Semivolatile stock standards for all of the applicable methods are stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year from the time of preparation, if prepared in house, or from the time the ampoule is opened, if purchased. Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

7.2.2.1. Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are

purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced at least every six months, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

7.2.3.1. The same spiking solution is used for the matrix spike (MS) and the Laboratory Control Sample (LCS). Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced at least every six months, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4. See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. The holding time for aqueous samples is seven days from sampling to extraction.

8.3.2. For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time is seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix which are processed together, using the same procedures and reagents. The batch must contain a method blank (MB), an LCS, and a matrix spike/matrix spike

(MS/MSD) duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and un-spiked sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (MB, LCS, MS, and MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same concentration as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The MB is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated compound concentrations or false positive results.

9.3.2. Aqueous MBs use 1000 mL of reagent water (250 mL for LVI) spiked with the surrogates. The MB goes through the entire preparation procedure.

9.3.3. TCLP MBs use 250 mL of leachate fluid spiked with the surrogates. SPLP MBs use 1000 mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The MB goes through the entire preparation procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. LCSs are well-characterized laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the preparative and analytical processes, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire preparative and analytical procedure.

9.4.2. The LCS is made up in the same way as the MB (see Sections 9.3.1 through 9.3.3), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the applicable recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. An MS/MSD is required for every 20 samples, otherwise a nonconformance memo must be included with associated samples.

9.7. QC requirements can be found in the various associated analytical SOPs.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2. Laboratory control limits are internally generated and updated periodically, unless method specified. Control limits are easily accessible via the LIMs.

9.9. Method Detection Limits (MDLs) and MDL Checks

9.9.1. MDLs and MDL Checks are established by the laboratory as described in SOP CA-Q-S-006.

9.9.2. MDLs are accessible via the LIMs.

9.10. Nonconformance and Corrective Action

9.10.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volumes of solvents into the appropriately sized glass culture tubes, gravimetrically. The “standard” glass culture tubes are sealed, and the meniscus is noted. The glass culture tubes containing the sample extracts are then compared against the “standard” glass culture tubes of the appropriate volume and solvent to ensure the volumes are consistent. See Table 2 for final volumes.

11. PROCEDURE

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, Operations Supervisor, or designee, to accommodate variation in sample matrix, chemistry, sample size, or other conditions. Any variation in procedure must be completely documented using a Nonconformance memo, and approved by a supervisor. Procedural variations are not allowed for Ohio VAP projects.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and matrix spiking solutions from the refrigerator and allow to warm to room temperature.

11.2.2. Measure the initial sample pH by inserting a disposable pipette into the sample, and placing a drop of sample on the wide-range pH paper. Record the pH value in LIMS.

11.2.3. For 600 series tests, check for the presence of Chlorine by inserting a disposable pipette into the sample and placing a drop of sample onto a Chlorine test strip. If no Chlorine is present, proceed to section 11.2.4. If chlorine is present in any amount, add 80g to 100g of sodium thiosulfate. Test the sample again. If chlorine is still present, add another 80 to 100 g of sodium thiosulfate. Repeat sodium thiosulfate additions and testing until the chlorine is gone. Note the presence of chlorine in the appropriate column in the worksheet in the LIMS.

11.2.4. Measure the initial volume using the bottle volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. The normal sample volume is 1 liter (or 250 mL for LVI) Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for highly contaminated samples. If the sample cannot be prepared using a separatory funnel due to matrix issues, a waste dilution may be required. Refer to Section 11.3 for the waste dilution procedure.

11.2.5. Prepare a MB, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 1 L of reagent water for MBs and LCS. Use 1000 mL of sample for the MS/MSD. The LCS and MS/MSD are spiked with the surrogate and matrix spike solutions, the MB only with the surrogates. See Tables 3 and 4 for the appropriate amount of spike solution to use for each analytical method.

11.2.6. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles, measured in a beaker. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides measured in a beaker.

11.2.7. For a TCLP method blank and LCS, measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add approximately 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction, if needed, due to matrix. For an SPLP MB and LCS, measure 1000 mL of the buffer solution using the volume template and transfer to the separatory funnel.

11.2.8. Spike the samples with the appropriate surrogate and/or spike solutions. All samples are spiked in the original sample bottle with the exception of TCLP.

11.2.9. Pour the sample into a separatory funnel. Rinse the sample bottle with approximately 60 mL methylene chloride. Add the rinsate to the separatory funnel. Use approximately 60 mL of methylene chloride per sample (40mL for LVI).

11.2.10. Place a labeled collection jar under each separatory funnel. Place a small amount of glass wool into a funnel and fill with anhydrous sodium sulfate. Place one of these prepared funnels on each collection jar.

11.2.11. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record adjusted pH, spiking volumes, and LIMS standard ID numbers on the benchsheet. Return spiking solutions that require cold storage to the refrigerator as soon as possible.

11.2.12. Seal and shake or rotate the separatory funnel vigorously for two minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting must be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.13. Allow the organic layer to separate from the water phase until complete visible separation has been achieved. A minimum of 10 minutes is required for the organic layer to separate from the water phase for the first serial extraction. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in the continuous liquid-liquid extraction SOP NC-OP-037 (If this is done, the sample must be extracted as part of a valid CLLE batch).

*__Note__: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes must recover at least 50 mL of solvent.

11.2.14. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water, replace the existing sodium sulfate with fresh sodium sulfate. For 8270 acid fraction analysis, rinse the funnel immediately with a fresh aliquot (approximately 50 mL) of DCM.

11.2.15. Repeat the extraction process two more times using fresh approximately 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.16. If extraction at a secondary pH is required, replace the filtration funnel and adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record the adjusted pH on the benchsheet. Serially extract with three approximately 60 mL portions of methylene chloride (40 mL for reduced volume preps), as outlined in Steps 11.2.7 to 11.2.10. Collect these three extracts in the same container used for the previous fraction.

11.2.17. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.18. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.19. Cover with aluminum foil and refrigerate, if the extract is not going to be concentrated immediately. Refer to Section 11.3 for concentration.

11.3. Concentration

11.3.1. According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.3.2. Kuderna-Danish (KD) Method

11.3.2.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube (CT) to the 500 mL KD flask. Label the CT and KD with the sample ID. Transfer the sample to the labeled K-D flask, rinse the collection vessel with 20-30 mL DCM and transfer to the KD to complete the quantitative transfer.

11.3.2.2. Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly).

11.3.2.3. Place the KD apparatus on a water bath (90-98^{o}C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.2.4. Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.2.5. Remove the KD apparatus from the water bath and allow to cool and drain for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.3.3. Nitrogen Evaporation to Final Concentration

11.3.3.1. Transfer the CT to the evaporation apparatus.

11.3.3.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.3.3. Refer to Table 2 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.3.3.4. Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the “standard” glass tube noted in Section 10.1.

11.3.3.5. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.4. Analytical Documentation

11.4.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.4.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

Note: When making new standards, it is required that all information entered into TALS is reviewed by another analyst.

11.4.3. Record all sample and associated QC information directly into LIMS. Level I and Level II review is performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are prepared and analyzed using the same procedures used for the samples.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been

implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual
(CW-E-M-001) for “Waste Management and Pollution Prevention.”

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the tank room, the pH is verified, the contents are neutralized with sodium bicarbonate, the pH re-verified and the Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25-gallon solvent waste container located under the fume hood in extractions.

15.2.4. Methylene chloride waste from various rinses: These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. Hexane-Hexane waste: These samples are to be disposed in the flammable waste.

15.2.6. Waste Hexane in vials. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. Waste Methylene Chloride sample vials. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B and 3510C

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. The appropriate release of the Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Appendix A to part 136 – Methods for Organic Chemical Analysis of Municipal and Industrial Waste Water. Specific Methods are kept on file.

16.1.6. Revision History

Historical File:		Revision 1A: 04/24/12		
(formerly CORP-OP-0001NC)		Revision 2: 02/05/13		
Revision 3.4: 10/16/98		Revision 3: 05/15/13		
Revision 3.5: 04/22/99		Revision 4: 08/20/14		
Revision 3.6: 05/13/99		Revision 5: 11/25/15		
Revision 3.7: 03/20/01		Revision 6: 02/17/17		
Revision 3.8: 05/23/01		Revision: 8 03/19/18		
Revision 3.9: 04/22/02		4/2/19: Changed Logo. No Change		
Revision 4.0: 02/04/03		to revision # or date.		
Revision 4.1: 10/07/03				
Revision 4.2: 01/30/06				
Revision 0: 03/12/08 (NC-OP-032)				
Revision 1: 01/07/09 (NC-OP-032)				
Revision 0: 03/03/11 (NC-OP-038)				

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and 8081B, NC-GC-042

16.2.6. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, NC-GC-043

16.2.7. Gas Chromatographic Analysis of PCBs Based on Methods 8082 and 8082A, NC-GC-045

16.2.8. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.9. Analysis of Pesticides and PCBs by EPA Method 608 and 608.3, NC-GC-007

16.2.10. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.11. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 and 608.3 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.1.3. Sodium sulfate is heated for 1 hour at 800°C to purify. The reference method lists a minimum of 4 hours at 400°C.

17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA	Acid ext; 1-2 or Base ext; 11-12	Acid ext; 1-2 or Base ext; 11-12
Pesticide/PCB	5-9	None
TPH	As received	None

Note: If the laboratory has validated acid only 8270 extraction for the target compound list required, then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

TABLE 2
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
PCB	Approximately 18 mL Hexane	2.0 or 5.0
Pesticides	Approximately 18 mL Hexane	2.0 or 5.0
BNA –	N/A	2.0 mL 5.0 mL for reduced volume preps
TPH	N/A	5.0 mL
BNA-SIM	NA	2.0 mL

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Table 3

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Non-LVI Spike Volume	LVI Spike Volume
BNA	20 ppm BNA Surrogate Spike	1.0 mL	400 uL

PEST	0.2 ppm DCB/TCX	1 mL for 2 mL final, 1 mL for 5 mL final	NA
TPH	40 ng/uL o-Terphenyl	1.0 mL	200 uL
PCB	0.2 ppm DCB/TCX	1 mL for 2 mL final, 1 mL for 5 mL final	1 mL
BNA – SIM	20 ppm BNA Surrogate Spike	0.1 mL	0.4 mL (1,4-Dioxane)

* Note surrogate spiking levels adjusted for reduced volume preps which utilize large volume injection

Table 4

LCS and Matrix Spiking Solutions

Analyte Group	LCS Spike Solution ID	Non-LVI Spike Volume	LVI Spike Volume
BNA	20 ppm BNA Spike	1.0 mL	400 uL
PEST	1.0 ppm Pest NPDES Spike	0.25 mL for 2 mL final, 0.5 mL for 5 mL final	NA
TPH	2500 ppm Diesel Spike	1.0 mL	200 uL
PCB	10 ppm PCB Spike	0.25 mL for 2 mL final, 0.5 mL for 5 mL final	0.25 mL
BNA – SIM	20 ppm BNA Spike	0.1 mL	0.4 mL (1,4-Dioxane)

* Note surrogate spiking levels adjusted for reduced volume preps which utilize large volume injection

TABLE 5
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	20
	Nitrobenzene-d_5	20
	p-Terphenyl-d_{14}	20
	2-Fluorophenol	20
	Phenol-d_6	20

	2,4,6-Tribromophenol	20
	1,2-Dichlorobenzene-d_4	20
	2-Chlorophenol-d_4	20
Pesticides PCB	Decachlorobiphenyl	0.2
	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	1,1'Biphenyl	20
	1,2,4,5-Tetrachlorobenzene	20
	1,2,4-Trichlorobenzene	20
	1,2-Dichlorobenzene	20
	1,2-Diphenylhydrazine	20
	1,3-Dichlorobenzene	20
	1,3-Dinitrobenzene	20
	1,4-Dichlorobenzene	20
	1,4-Dioxane	20
	1-Methylnaphthalene	20
	2,2'-oxybis[1-chloropropane]	20
	2,3,4,6-Tetrachlorophenol	20
	2,4,5-Trichlorophenol	20
	2,4,6-Trichlorophenol	20
	2,4-Dichlorophenol	20
	2,4-Dimethylphenol	20
	2,4-Dinitrophenol	40
	2,4-Dinitrotoluene	20
	2,6-Dichlorophenol	20
	2,6-Dinitrotoluene	20
	2-Chloronaphthalene	20
	2-Chlorophenol	20
	2-Methylnaphthalene	20
	2-Methylphenol	20
	2-Nitroanaline	20
	2-Nitrophenol	20
	3&4-Methylphenol	20
	3,3'-Dichlorobenzidine	40
	3-Methylphenol	10
	3-Nitroanaline	20
	4,6-Dinitro-2-methylphenol	40

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	4-Bromophenyl phenyl ether	20
	4-Chloro-3-methylphenol	20
	4-Chloroanaline	20
	4-Chlorophenyl phenyl ether	20
	4-Methylphenol	10
	4-Nitroanaline	20
	4-Nitrophenol	40
	Acenaphthene	20
	Acenaphthylene	20
	Acetophenone	20
	Aniline	20
	Anthracene	20
	Azobenzene	20
	Benzidine	40
	Benzo[a]anthracene	20
	Benzo[a]pyrene	20
	Benzo[b]fluoranthene	20
	Benzo[g,h,i]perylene	20
	Benzo[k]fluoranthene	20
	Benzoic acid	40
	Benzyl alcohol	20
	Bis(2-chloroethoxy)methane	20
	Bis(2-chloroethyl)ether	20
	Bis(2-ethylhexyl)phthalate	20
	Butyl benzyl phthalate	20
	Carbazole	20
	Chrysene	20
	Dibenz(a,h)anthracene	20
	Dibenzofuran	20
	Diethyl phthalate	20
	Dimethyl phthalate	20
	Di-n-butyl phthalate	20
	Di-n-octyl phthalate	20
	Fluoranthene	20
	Fluorene	20
	Hexachlorobenzene	20
	Hexachlorobutadiene	20
	Hexachlorocyclopentadiene	20
	Hexachloroethane	20
	hexadecane	20

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
	Indene	40
	ideno[1,2,3-cd]pyrene	20
	isophorone	20
	Naphthalene	20
	n-Decane	20
	Nitrobenzene	20
	N-Nitrosodimethylamine	20
	N-Nitrosodi-n-propylamine	20
	N-Nitrosodiphenylamine	40
	n-Octadecane	20
	Pentachlorophenol	40
	Phenanthrene	20
	Phenol	20
	Pyrene	20
	Pyridine	20
Pesticides TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pesticides NPDES	Aldrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0
TPH	Diesel Fuel	2500 **µg/mL**

Title: SOXHLET (TRADITIONAL) EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3540C

[Method: SW846 3540C]

Approvals (Signature/Date):

Technology Specialist	05/14/18 Date	Health & Safety Coordinator	05/14/18 Date
Quality Assurance Manager	05/16/18 Date	Technical Director	05/14/18 Date

This SOP was previously identified as SOP No. NC-OP-040, Rev 5a, dated 5/25/17

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in soil, sediment, waste and wipe matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/Mass Spectrometry (GC/MS) using Soxhlet Extraction. The procedures are based on SW846 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, and 8015D.

1.1.2. The extraction procedures herein may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Soxhlet Extraction (Traditional)

2.1.1 A 30 g sample (10 g for Pesticides and PCBs) is mixed with anhydrous sodium sulfate until free flowing, or a 1 wipe sample is placed in an extraction thimble. They are extracted by refluxing with solvent.

2.2. Concentration

2.2.1 Procedures are presented for drying the extract and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate

Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of

methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sox	Conc
Graduated cylinder: 1 liter. (other sizes may be used as needed)		√
Erlenmeyer flask: 250 mL (other sizes optional)		√
Solvent dispenser pump or 100 mL graduated cylinder	√	√
Round or flat bottom: 250 mL	√	
Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	√	√
Cooling condensers	√	
Heating mantle: rheostat controlled or equivalent	√	
Auto-timer for heating mantle	√	
Soxgriddle or equivalent	√	
Beakers: 450mL wide-mouth glass jars		√
Balance: >100 g capacity, accurate to ±1.00 g	√	√
Soxhlet extractor	√	
Cellulose and glass thimbles	√	
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 mL and 40 mL capacity with Teflon®-lined screw-cap		√
Clean wipes for wipe matrix method blanks and laboratory control samples		
Nitrogen blowdown apparatus		√

EQUIPMENT AND SUPPLIES	Sox	Conc
Nitrogen: reagent grade.		√
Culture Tubes: 10 mL, 16 x100 mm		√
Microliter pipette and/or syringe 1 mL	√	
Glass wool	√	
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

7.2. All reagents must be ACS reagent grade or better, unless otherwise specified.

REAGENTS	Sox	Conc
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 800°C a minimum of one hour.	√	√
Magnesium sulfate	√	
Extraction Solvents (pesticide quality or equivalent): Methylene chloride, Methylene Chloride/acetone hexane/acetone,	√	√
Hexane/Acetone:, reagent grade: Used for cleaning glassware.	√	√

7.3. Standards

7.3.1. Stock Standards

7.3.1.1. Stock standards are purchased as certified solutions. S tock standards are stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened, if purchased). Standards that are cold stored must be allowed to come to room temperature before use.

7.3.2. Surrogate Spiking Standards

7.3.2.1. Prepare or purchase surrogate spiking standards at the concentrations listed in Table 2. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced every six months at a minimum, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards

7.3.3.1. The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 3. Spiking standards are purchased or prepared as dilutions of the stock standards.

7.3.3.2. Spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced every six months at a minimum, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 client samples and appropriate QC that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank (MB), an LCS, and a matrix spike/matrix spike duplicate (MS/MSD). (In some cases, at client request, it may be appropriate to process a matrix spike and un-spiked sample duplicate in place of the MS/MSD). If clients designate specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. See Policy QA-003 for further definition of the batch.

9.2. Method Blank (MB)

9.2.1. An MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same level as the samples. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.2.2. For a solid MB, use approximately 30 g of sodium sulfate spiked with the surrogates. For PCB and Pesticides, use approximately 10 g ± 0.5 g of sodium sulfate. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB goes through the entire analytical procedure.

9.2.3. For a wipe MB, use 1 clean wipe spiked with the surrogates. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB goes through the entire analytical procedure.

9.3. Laboratory Control Sample (LCS)

9.3.1. LCSs are well-characterized laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.3.2. The LCS is made up in the same way as the MB (see Sections 9.2.1 through 9.2.3), but spiked with the LCS standard and the surrogates. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.4. Surrogates

9.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.4.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits of the applicable determinative method. See Table 2 for the appropriate amount of surrogate spike to use for each analytical method.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. An MS is an environmental sample to which known concentrations of target analytes have been added. An MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.6. QC requirements can be found in the various associated analytical SOPs.

9.7. Control Limits

9.7.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2. Laboratory control limits are internally generated and updated periodically, unless method specified. Control limits are easily accessible via the LIMs.

9.8. Method Detection Limits (MDLs) and MDL Checks

9.8.1. MDLs and MDL Checks are established and performed by the laboratory as described in SOPs CA-Q-S-006.

9.8.2. MDLs are easily accessible via the LIMs.

9.9. Nonconformance and Corrective Action

9.9.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volumes of solvents into the appropriate sized glass culture tubes gravimetrically. The “standard” glass culture tubes are sealed, and the meniscus is noted by marking a line on the tubes. The glass culture tubes containing the sample final extracts are then compared against the “standard” glass culture tubes of the appropriate volume and solvent to ensure the volumes are consistent. (See Table 1 for final volumes)The bottle top dispenser is calibrated quarterly and must be within ±5% of the target volume with an RSD ≤ 1%.

10.2. All labware, pipettes, and balances are calibrated according to SOPs NC-QA-004 and NC-QA-015.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2. Soxhlet

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator if cold stored, and allow to warm to room temperature.

11.2.2. If sample can be mixed easily in the sample jar, mix thoroughly by stirring with a clean plastic or wooden spoon or spatula. If the sample cannot be easily mixed (i.e., clay samples or samples of various and very different particle sizes), use the spoon or spatula to select enough separate portions from locations within the jar to produce a representative sample. Analyst judgment is important in determining how many portions and which locations are used to produce a representative aliquot. If the sample is uniform clay, at least 3 portions should be selected from different locations in the sample jar, if particle sizes or materials indicate a very non-homogenous sample, selection should be made carefully to collect an aliquot that represents the relative percentages of the various particle sizes and types in the sample jar.

11.2.3. Do not decant the water layer from sediment samples. The entire sample is used. A higher weight of sample portion must be weighed for sediment samples to account for the dry weight correction (see 11.2.3). Record and document in the LIMS if a water layer was present in the sample.

11.2.4. If the sample cannot be prepared using a Soxhlet due to matrix issues, a waste dilution may be required. Refer to SOP NC-OP-043 for the waste dilution procedure.

11.2.5. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30g ± 0.5 g of sample into a thimble or in a jar, recording the weight to the nearest 0.01g in LIMS. For PCB or Pesticides Extraction, weigh approximately 10 g of sample ± 0.5 g. Sample weights less than 30g, but over 1g, may be used if the appropriate reporting limits can be met. For sediment samples that contain excessive moisture, weigh 50 g ±0.5 g. For wipe samples, the wipe is placed in an extraction thimble. For concrete samples, weigh 5 g of sample ± 0.5g.

Note: Waste samples with difficult matrices (such as caulk) are extracted at 1g.

Note: Alternate sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the sample to reagent ration. All samples and standards must be processed similarly.

11.2.6. Prepare an MB, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate or a clean wipe as the matrix for the LCS and MB. The parent sample is used for the MS/MSD. The weight of sodium sulfate used must be approximately the weight of soil used for samples.

11.2.7. Add anhydrous sodium sulfate to each solid, sediment or waste sample and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the thimble tightly. The soxhlet extractor or extraction thimble must drain freely for the duration of the extraction period. Thimbles are only used for PCB and Pesticide extraction. A glass wool plug below the sample in the soxhlet extractor is used for other extractions.

11.2.8. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 2 and 3.

11.2.9. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS and TPH	1:1 v/v Methylene Chloride / Acetone
8270 (MS) Concrete	Methylene Chloride
PCB and Pesticides	1:1 v/v Hexane / Acetone
8082 Concrete	1:1 v/v Methylene Chloride/Acetone

11.2.10. Allow the extract to cool after the extraction is complete then disassemble by gently twisting the soxhlet from the flask.

11.2.11. The sample is now ready for the concentration step (Section 11.3).

11.2.12. Cover the extracts with aluminum foil and store at 4°C ± 2°C if the extract will not be concentrated immediately. Refer to Section 11.3 for concentration.

11.3. Concentration: According to the type of sample, different solvents and final volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.3.1. Kuderna-Danish (KD) Method:

11.3.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube (CT) to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask filtering the extracts through funnels filled with sodium sulfate. Rinse the sample flasks from the soxhlet setups with approximately 10 – 20 mL of methylene chloride Transfer the rinsate through the funnel and rinse

the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.1.2. Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. **Note:** It is important to wet with MeCl to ensure that the balls in the Snyder column do not stick, and the column will work properly.

11.3.1.3. Place the KD apparatus on a water bath (90-98^{o}C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.1.4. Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 1 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the CT joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4. Nitrogen Evaporation to Final Concentration

11.4.1. Transfer the CT to the evaporation apparatus.

11.4.2. Place the tube in a warm water bath that is at least 5^{o}C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40^{o}C
Acetone	56^{o}C
Hexane	69^{o}C
Acetonitrile	82^{o}C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3. Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.4.4. Rinse the CT and quantitatively transfer the extract with the rinsate to the appropriate final container, rinse the CT and transfer the rinsate to the final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.5.3. Record sample and associated QC information into LIMs. Level I and Level II technical reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each analyst must make an initial demonstration of capability (IDOC) for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all of the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.1.4. Method validation information (where applicable) in the form of analyst demonstrations of capabilities is maintained for this method in the analyst's training files

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.2. **Assorted flammable solvent waste from various rinses**: These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.3. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.4. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.5. **Waste Hexane in vials:** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.6. **Waste Methylene Chloride sample vials**: These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane:** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste: Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Sections 3500B, 3540C, and 3580A, current version

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version16.1.5 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.5. EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.6. Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		Revision 0: 03/24/11 (NC-OP-040)
		Revision 3.7: 03/20/01		Revision 1-A: 01/24/12
		Revision 3.8: 05/23/01		Revision 2: 08/28/13
		Revision 3.9: 04/22/02		Revision 3: 08/25/14
		Revision 4.0: 02/04/03		Revision 4: 01/18/16
		Revision 4.1: 10/07/03		Revision 5a: 05/25/17
		Revision 4.2: 01/30/06		

**4/19/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and 8081B, NC-GC-042

16.2.6. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, NC-GC-043

16.2.7. Gas Chromatographic Analysis of PCBs Based on Methods 8082 and 8082A, NC-GC-045

16.2.8. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.9. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.10. Analysis of Pesticides and PCBs by EPA Method 608.3, NC-GC-046

16.2.11. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Methods 625 and 625.1, NC-MS-003

16.2.12. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Sodium sulfate is heated for 1 hour at 800°C to purify. The reference method lists a minimum of 4 hours at 400°C.

17.2. Tables

TABLE 1

Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis*	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 36 mL Hexane	10.0
Pesticides	Approximately 18 mL Hexane	10.0
BNA – SIM	N/A	2.0 mL
TPH	N/A	5.0

Note: *PCBs and Pesticides only need the solvent exchange step when they are extracted in methylene chloride / acetone. If they are extracted in hexane / acetone, no solvent exchange is necessary.*
Note: *Different final volumes may be necessary to meet special client reporting limit requirements.*

TABLE 2

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	20 ppm BNA	1.0
BNA / SIM	20 ppm BNA	0.1
PEST	0.2 ppm DCB/TCX	1.0
TPH	o-Terphenyl	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 3
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	20 ppm BNA All-Analyte Spike	1.0
BNA / SIM	20 ppm BNA All-Analyte Spike	0.1
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	Diesel Spike	1.0

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Methods 8270C or 8270D.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP Nos. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270C*, current revision and ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270C or 8270D (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material.

Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in

the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent

- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent
- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is

reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/	2000ppm	10 ml (methanol)	100 ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
Std#9			
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D).

7.2.4 The preparation of all standards must be documented in TestAmerica LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an

LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing *BN only* extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing *AE only* extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as 'SIM" and the second vial as 'Total'. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3510C, Separatory Funnel Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS, SW846 Method 8270C*, current revision.

15.3 TestAmerica Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

15.4 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), current revision.

15.7 TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.9 TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)*

15.10 TestAmerica Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).*

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 03/26/2018

- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:

- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:

- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012

- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to TestAmerica Edison SOP No. ED-MSS-009 (*Semivolatile Organic Compounds by GC/MS, SW846 Method 8270C*D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10

- Section 3: revised to reference new location for definitions

- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270C*, current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Quality Assurance Manual Cover Page

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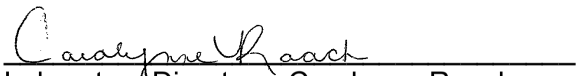
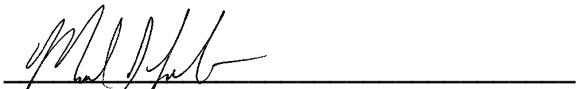


Laboratory Director – Carolynne Roach — Date: 03/29/19



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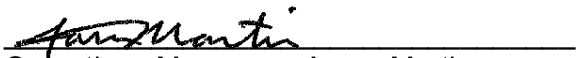

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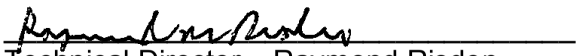

Technical Director – Raymond Risden — Date: 03/29/19

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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-QM-001	Policy on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Corporate Document Control and Archiving

REFERENCED LABORATORY SOPs

SOP Reference	Title
NC-QA-015	Balance and Thermometer Calibration, Container Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-019	Records Information Management
NC-QA-027	Preparation and Management of SOPs
NC-QA-028	Employee Orientation and Training
NC-QA-029	Nonconformance and Corrective Action System
NC-QA-030	Document Control
NC-SC-005	Sample Receiving and Sample Control
NC-SC-006	Sample Procurement Protocol
CA-Q-T-005	Laboratory Documentation
NC-QA-031	Internal Audits

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Canton's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2017. In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 2. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

Ohio VAP requirements are listed throughout the document.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control (NC-QA-030) and Updating Procedures (NC-QA-027).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Canton is a local operating unit of TestAmerica Laboratories, Inc. and includes various Service Centers. Service Centers under the direct authority of TestAmerica Canton include (but are not limited to) the Cambridge, Cincinnati, Columbus, and Dayton facilities in Ohio and the Grand Rapids and Brighton facilities in Michigan. The Brighton Michigan Service Center also houses a small laboratory that performs short hold analysis as needed. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent

operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Canton is presented in Figure 4-1. Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Canton laboratory.

Canton Laboratory Key Personnel

Name	Position
Rusty Vicinie	VP of Operations, Central
Carolynne M. Roach	Laboratory Director
Raymond Risden	Technical Director
Aaron M. Martin	Operations Manager
Mark J. Loeb	Quality Assurance Manager
Tom Stiller	GC/MS Volatiles Group Leader
Steve Jackson	Regional Safety Director, Waste Management Supervisor
Brittany Blythe	Extractions Group Leader
Will Cordell	Field Analytical Group Leader
Olguita Colon	GC Volatile/Semivolatiles Group Leader
Tom Hula	GC/MS Semivolatiles Group Leader
Joseph Weisend	General Chemistry Group Leader
Darren Miller	Maintenance
Karen Counts	Metals Group Leader
Patrick O'Meara	Project Management Group Leader

Name	Position
Nick Sutek	Sample Control Group Leader
Lance Hershman	Canton Shipping Hub Manager
Bonnie Bridwell	Cambridge Service Center Contact
Matt Barbieri	Cincinnati Service Center Contact
Gina Rivera	Columbus Service Center Contact
Theresa Daniels	Dayton Service Center Contact
Terri Harlin	Brighton Michigan Service Center / Laboratory Contact
Gary Wood	Grand Rapids Michigan Service Center Contact

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the quality standard and integrity program are met.

4.2.3 Chief Operation Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO is responsible for the operations of TestAmerica's subsidiary companies and the company's strategic growth.

4.2.4 Senior Vice President of Operations and Client Service

The SVP of Operations and Client Service leads the Client Service Organization (CSO); and oversees the operations of all TestAmerica laboratories, the Corporate Technical Services group and the Sales Opportunity Optimization efforts. The SVP provides direction to the VPs of Operations, Client Service Directors, Manager of Project Managers, Director of Technical Services and a Director of Sales. The SVP of Operations and Client Service reports directly to the President and CEO of TestAmerica.

4.2.5 Vice President of Operations (VPO)

Each VP of Operations (VPO) reports directly to the SVP of Operations and Clients Services. Each VPO is responsible for the overall administrative and operational management of their respective laboratories. The VPO's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VPO's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VPO's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Managers, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the analytical laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Managers development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; is responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents

(e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business' Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – i.e., the Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.15 Laboratory Director

TestAmerica Canton's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective VPO. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical directorss for the appropriate fields of testing. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.

- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves SOPs as directed by the Quality Assurance department prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director, and the Operations Manager as direct reports.

4.2.16 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.

- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager or designee will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, evaluate manual calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025**.**

4.2.17 Technical Director or Designee

The Technical Director reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.

4.2.18 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She assists the Technical Director in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Works with the Departmental Group Leaders to ensure that scheduled instrument maintenance is completed.

- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his/her substitute in the interim.

4.2.19 Environmental Health and Safety Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.20 Department Group Leaders

Department Group Leaders report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 17.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.21 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.

- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.22 Project Manager (PM)

The PM reports to the Manager of Project Management (MPM) and serves as the interface between the laboratory's technical departments and the laboratory's clients. There is an entire staff of Project Managers that makes up the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

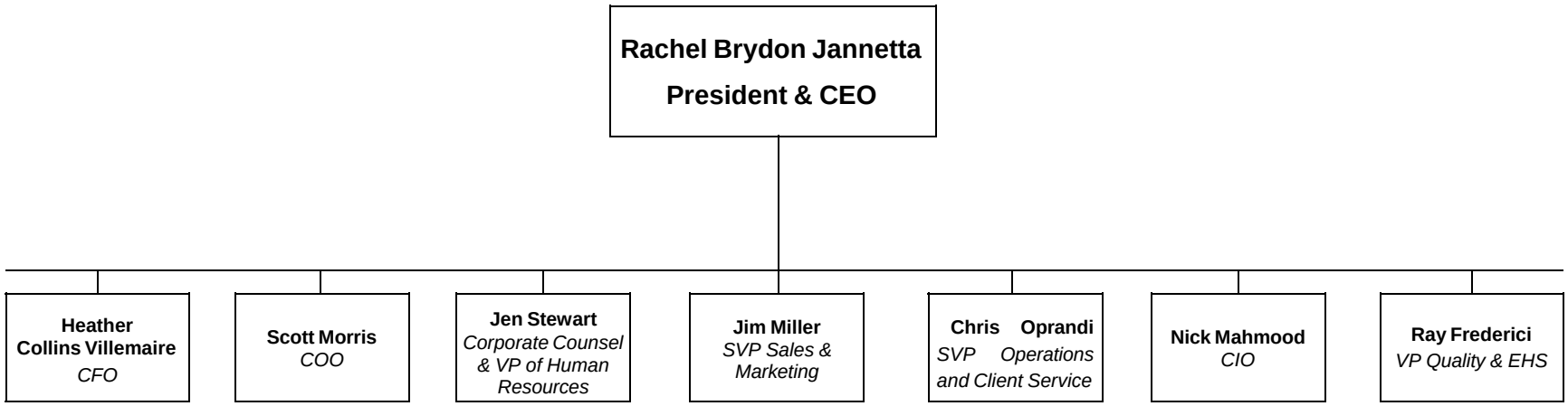
- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

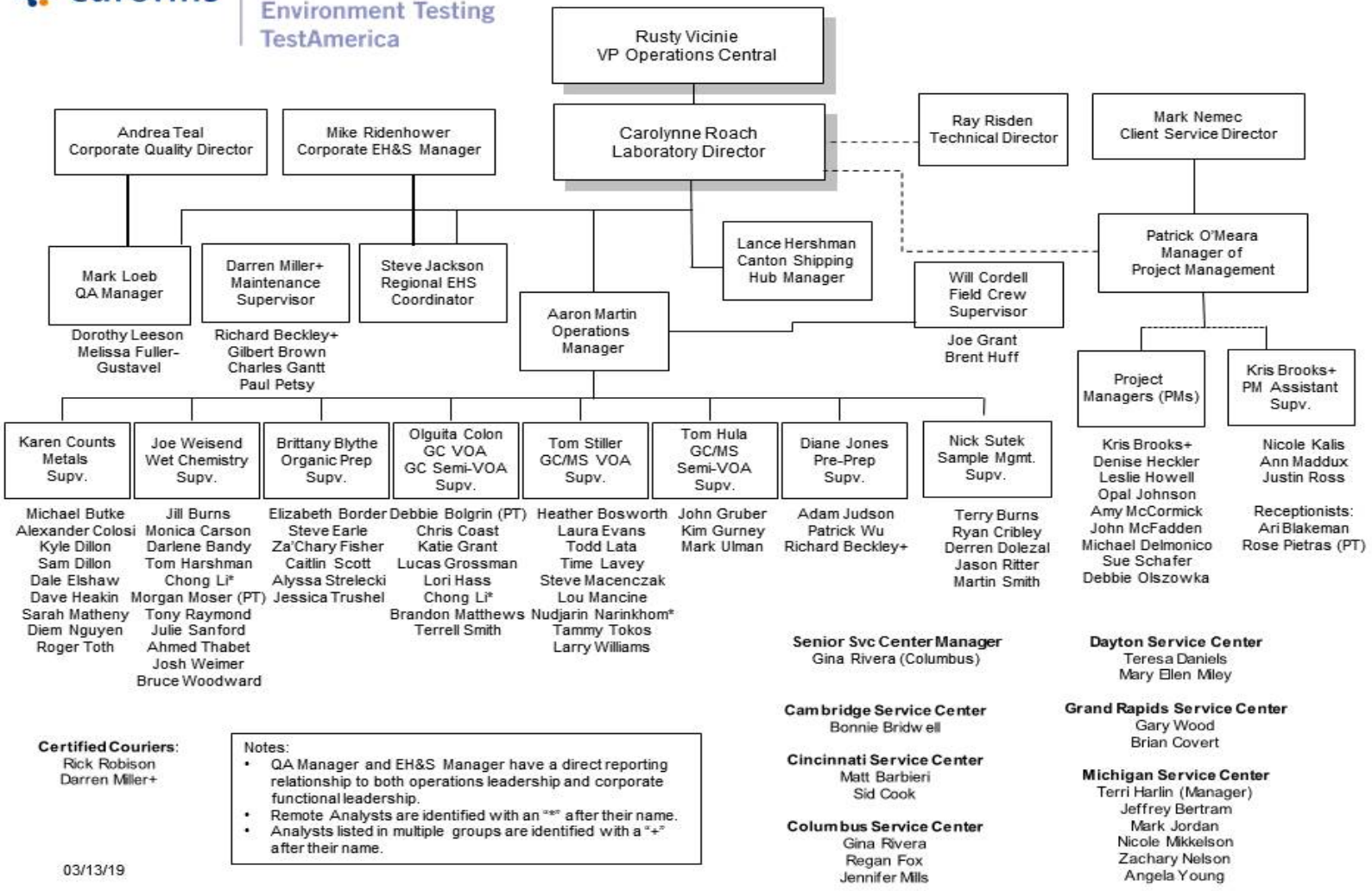
Key Personnel	Deputy
Laboratory Director	Technical Director QA Manager
Operations Manager	Technical Director Laboratory Director
Quality Assurance Manager	Quality Assurance Coordinator Laboratory Director
Technical Director	Operations Manager Laboratory Group Leaders
EHS Coordinator	EHS Manager Facilities Manager

Figure 4-1. Corporate and Laboratory Organization Charts
Note: *Organization Charts are subject to change without notice.*



Canton Laboratory Organization

eurofins | Environment Testing TestAmerica



Senior Svc Center Manager
Gina Rivera (Columbus)

Cambridge Service Center
Bonnie Bridwell

Cincinnati Service Center
Matt Barbieri
Sid Cook

Columbus Service Center
Gina Rivera
Regan Fox
Jennifer Mills

Dayton Service Center
Teresa Daniels
Mary Ellen Miley

Grand Rapids Service Center
Gary Wood
Brian Covert

Michigan Service Center
Terri Harlin (Manager)
Jeffrey Bertram
Mark Jordan
Nicole Mikkelson
Zachary Nelson
Angela Young

Certified Couriers:
Rick Robison
Darren Miller+

Notes:
- QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.
- Remote Analysts are identified with an "*" after their name.
- Analysts listed in multiple groups are identified with a "+" after their name.

03/13/19

SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2017 International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).

- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains tables, housed in TALS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into TALS. The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of TALS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the TALS analyte database. As sample results and the related QC are entered into TALS, the sample QC values are compared with the limits in TALS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

The laboratory's procedures for the creation of control charts are described in laboratory SOP No. NC-QA-018, "Statistical Evaluation of Data and Development of Control Charts." Control charts are created from data stored in the LIMS. The charts are evaluated by QA and/or technical staff to determine if limits need to be updated or to assess the need for corrective actions to improve method performance.

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP.

5.6.2 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms

- Corporate Policies and Procedures

The Corporate QA Department posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-030 Document Control and in SOP NC-QA-027 Preparation and Management of SOPs.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports *(however named)*. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed annually and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOPs NC-QA-027 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the SOP folder for the applicable revision.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP NC-QA-027 Preparation and Management of Standard Operating Procedures.. The SOPs identified above also define the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by the QA department in accordance with the procedures specified in laboratory SOP NC-QA-030.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP NC-QA-030.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel, and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below)

- Contract Administrator
- VP of Operations
- Laboratory Project Manager
- Laboratory Directors and/or Corporate Technical Managers
- Laboratory Directors and/or Corporate Information Technology Managers
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors

- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The ***Sales Director, Contract Administrator, Account Executive or Proposal Coordinator*** then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

7.3 Documentation

The Contracts Department maintains copies of all signed contracts. TestAmerica Canton Project Manager Assistants and/or PMs maintain electronic copies of signed contracts as needed.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Records are maintained electronically.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory during operations meetings, via emails, or directly. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Director. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

The Technical Director is available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs) or other responsible CSO members, for the Export Lab (i.e., the TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the

following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

8.2.1 When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

8.3 Oversight and Reporting

8.3.1 The status and performance of qualified subcontractors will be monitored by the Corporate Quality department, and includes an annual review process (see Subcontracting SOP CW-L-S-004). Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the

subcontracted laboratories.

- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

8.3.2 For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

Note: Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

8.3.3 All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the

final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Fixed Asset Acquisition, Retention and Safeguarding SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use. If the item is not in consignment, the analyst must provide the master item number, item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Operations Manager or Group Leader prior to placing the order. The Canton purchasing manager places the order.

9.3.2 Receiving

It is the responsibility of the warehouse manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It

is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on-file and available for review in the reagent module of the LIMS.

  Note: Some programs (such as OVAP) do not allow the use of verified standards and/or reagents.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and Technical Director must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained electronically in the LIMS or in binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, a unique identification name is assigned and added to the equipment list in the LIMS. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). This can be tracked by use of the New Equipment Tracking Form (WI-NC-091). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts, Group Leaders, and/or the Technical Director. The service providers that perform the services are approved by the Group Leaders or Operations Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP NC-QA-015. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOP NC-QA-029 Nonconformance and Corrective Action System. A copy of this procedure will be made available to any interested party on request.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP NC-QA-029.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The

lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Operations Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Director, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate*.* Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Director, QA Department, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. TestAmerica employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the TALS non-conformance memo (NCM) system. See Figure 12-1 for an example CAR.

Figure 12-1 Example CAR

Corrective Action Report

Laboratory: ***TestAmerica Canton***
Source of Issue: [redacted]
Date Initiated: ***9/22/2017***
Initiated By: [redacted]
Responsible for Investigation: ***Canton QA and Lab Staff***

Description of Problem:
Samples WWTP EFFLUENT COMP [redacted] and SITE COMP [redacted] were received on 8/30/2017 and logged into the database per sample IDs as listed on the chain of custody. When sample labels were printed, the labels were inadvertently switched between samples. Sample WWTP EFFLUENT COMP [redacted] was labeled using SITE COMP [redacted] labels and the samples SITE COMP [redacted] was labeled using WWTP EFFLUENT COMP [redacted] labels. The error was not discovered until the client requested review of the reported.

Investigation Summary & Root Cause Analysis:
The COC listed sample IDs "WWTP EFFLUENT COMP" and "SITE COMP". No sampling times were provided. The container IDs were listed as [redacted] and [redacted] The cooler receipt form noted that sample times were not listed, but did not note discrepancy in sample IDs from container to COC. The laboratory is currently performing a second level review for sample labels to insure containers are properly labeled. Unfortunately, this 2nd level review process did not catch the labeling error for this project.

Initial analytical results for sample site comp were above the permit limits. Per PM request, the both samples were pulled and results were verified. The labeling discrepancy was discovered during this review process. Once the labels were corrected, the results for site comp were not above the permit limit.

Corrective Action Plan:
Lab management was notified of the labeling error and the issue was discussed during a weekly process improvement meeting held October 3rd, 2017. Meeting attendees included the Laboratory Director, Operations Manager, Shipping/Receiving Department Manager and the QA Manager. The group was reminded of the importance of the attention to details needed to ensure client requirements are met with correctly reported results.

_____ 10/04/2017
QA Officer Date

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in TALS are also acceptable)

12.2.2 Corrective Actions Documented In the ICAT Database - Internal and external audit findings

- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Director, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Group Leader, Operations Manager, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Group Leaders and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager or designee reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager or designee and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Note: For the Ohio EPA Voluntary Action Program (VAP), please refer to the associated analytical SOPs for the acceptable criteria, corrective actions, and exceptions.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < RL. -See details in Method SOP	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Group Leaders)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Group Leaders*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewers)*	- % Recovery within limits documented in LIMS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
		the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewers)*	- % Recovery within limits specified in LIMS.	- -Check calculations and instrument performance - Re-analyze the LCS and if still outside of control limits, re-prepare and re-analyze all samples in the batch. It is acceptable to report data when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported. **Note:** If there is insufficient sample or the holding time cannot be met, contact the project manager for client instruction.
Surrogates *(Analyst, Data Reviewers)*	- % Recovery within limits specified in LIMS**.**	-Reprep and analyze the QC batch for MB surrogates below control limits -Report data with narrative if the surrogate is biased high and associated samples are < RL
Method Blank (MB) *(Analyst, Data Reviewers)*	< Reporting Limit with the exception of Common Laboratory Contaminants listed in QA-003 QC Policy.	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. -Results are acceptable if the same contaminants were not found in the associated samples.
Proficiency Testing (PT) Samples *(QA Department,, Analysts, Group Leaders)*	- Criteria supplied by PT Supplier.	- Any failures must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Department, Operations*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated system and necessary corrections must be made.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Manager, , Group Leaders, Laboratory Director)		
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Group Leaders, QA Manager, Corporate QA,)	SOP CW-Q-S-005, Data Recall	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or your lab's CA SOP.
Client Complaints *(Project Managers, Lab Director, QA Department, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint.
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director, *Group Leaders*)		- Corrective action is determined by the type of issue.
Health and Safety Violation (EH&S Coordinator, Lab Director, Operations Manager, Group Leaders)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For Ohio VAP, the method blank must be below the detection limit for all compounds of interest.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement
- Process for the preventive action or improvement
- Define the measurements of the effectiveness of the process once undertaken
- Execution of the preventive action or improvement
- Evaluation of the plan using the defined measurements
- Verification of the effectiveness of the preventive action or improvement

- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. The laboratory has a graded approach for managing change based on the Management Systems Review.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. Quality records are maintained by the QA department electronically. Electronic files are backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the individual analysts under the direct supervision of their group leader.

Table 14-1. Record Index[1]

Record Types [1]:	Retention Time:

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] *Record Types encompass hardcopy and electronic records.*
[2] *Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).*

** Exceptions listed in Table 14-2.*

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for approximately one month after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Michigan Department of Environmental Quality – all environmental data	10 years 12 years for Lead and Copper results
Ohio VAP	10 years and State contacted prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records

stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by TALS. The TestAmerica chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, electronic spreadsheets are used to record and file data, or the data is manually entered into TALS at the time of analysis. Standard and reagent information is recorded in logbooks or entered into TALS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink or entered directly into LIMS at the time the data is generated.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.

- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or

regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
- Method performance criteria including expected quality control requirements. These are indicated both in TALS and on specific analytical report formats**.**

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement
- sample identification, receipt, acceptance or rejection and login
- sample storage and tracking including shipping receipts, sample transmittal / COC forms
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy,

write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. All data are recorded sequentially within a series of sequential notebooks. Bench sheets, if used, are filed sequentially. Standards are maintained in TALS – no logbooks are required to be used to record that data**.** Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine

internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Director or Designee (Refer to CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Director or Designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency: Every 2 years
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to

identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: non-potable water and soil

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the lab's corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 12). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Group Leader of the department where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Director, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Director, QA Manager / Department, and Operations Manager) conducts a review annually of its quality systems and TALS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The TALS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to TALS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings
- Minutes from prior senior lab management meetings - Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources
    - Adequacy of policies and procedures
    - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan - Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity
- For labs analyzing radioactive samples, also include the following:
    - Radiation health and safety
    - Radioactive hazardous waste management
    - Radioactive materials management

- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operation and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Internal Investigations SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular

area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience

Specialty	Education	Experience
Group Leaders – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Group Leaders - **Microbiology**	Bachelor's degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Group Leader, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted

personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP NC-QA-028 Employee Orientation and Training.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy
- How and when to report ethical/data integrity issues / confidential reporting
- Record keeping
- Discussion regarding data integrity procedures
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring - investigations and data recalls
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 54,440 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered

sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide “spot power” where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and TALS are regulated to protect against raw data loss.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory’s Facility Addendum.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas
- Standard Methods, current Ed., 9020B, Sec. 2
- TNI V1M5, 1.7.3.7.a

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building key badges are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs),

reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP NC-QA-027 Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every 2 years (annually for the state of Kentucky), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Method 1630, Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap and CVAFS, August, 1998.*
- *Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-02-19, August 2002.*
- *Modified DRO, Method for Determining Diesel Range Organics, Wisconsin DNR, PUBL-SW-141, September 1995.*
- *Modified GRO, Method for Determining Gasoline Range Organics, Wisconsin DNR, PUBL-SW-140, September 1995.*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers.

Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP NC-QA-028, Employee Orientation and Training) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Group Leader and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

At least four aliquots must be prepared (including any applicable clean-up procedures) in the same fashion, and following all of the same procedures, as client samples, and analyzed according to the test method (either concurrently or over a period of days).

Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1 as an example) must be used to document the completion of each IDOC. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 Determination of Method Selectivity – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or

matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7 below. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ) – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of TestAmerica's procedure for conducting MDL studies are given in SOP # CA-Q-S-006).

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations two times or less than the selected LOQ, employing the complete analytical process.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte

used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below.
The laboratory is currently using the TestAmerica TALS LIMS system, which has been highly customized to meet the needs of the laboratory. It is referred to as TALS for the remainder of this section. More detailed descriptions of computer systems and associated controls given in the "IT Change Control Procedure Manual" (CW-I-M-001) policies and procedures posted on TestAmerica's intranet site, Oasis.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.

- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 **Ensure Information Availability** – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 **Maintain Confidentiality** – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Group Leader or alternate analyst once the data is uploaded or entered into TALS. The spreadsheets, or any other type of applicable documents, are initialed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to TALS, the results should be entered in TALS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the TALS System, the raw results and dilution factors are entered directly into TALS by the analyst, and the software calculates the final result for the analytical report. TALS has a defined significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the TALS, the raw results and dilution factors are transferred into TALS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the QA Manager or designee at the facility. The QA Manager or designee controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in several SOPs to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the TALS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and

reliability of sample results. The analyst transfers data into TALS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, verification of units vs matrix, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Director, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, reported units are appropriate for the sample matrix, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Projec Manager then electronically signs the final report which is sent out to the client.

19.15.4.8 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on expanded scale "before" and "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

GC Analyst Demonstration of Capability

TestAmerica Canton

Analyst:

DOC Run Date:

Preparation Method(s):

8151 Herbicide SOP: NC-GC-038	WI DRO SOP: NC-GC-013	8315 Formaldehyde SOP: NC-GC-035	WI GRO Prep/Analysis SOP: NC-GC-031	8082/608 PCBs SOP: NC-GC-007/NC-GC-038
8081/608 Pesticides SOP: NC-GC-038	8015 DRO SOP NC-GC-043	8015 GRO Prep/Analysis SOP: NC-GC-025	Aromatic Acids Analysis (solid and water), solid prep SOP: NC-GC-036	RSK-175 SOP: NC-GC-032
1630 MeHg	8011			

Prep/Analysis SOP: NC-GC-039	Prep/Analysis SOP: NC-GC-040			

Matrix: □Water □Solid

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at the facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).

2. The test method(s) was performed by the analyst identified on this certificate.

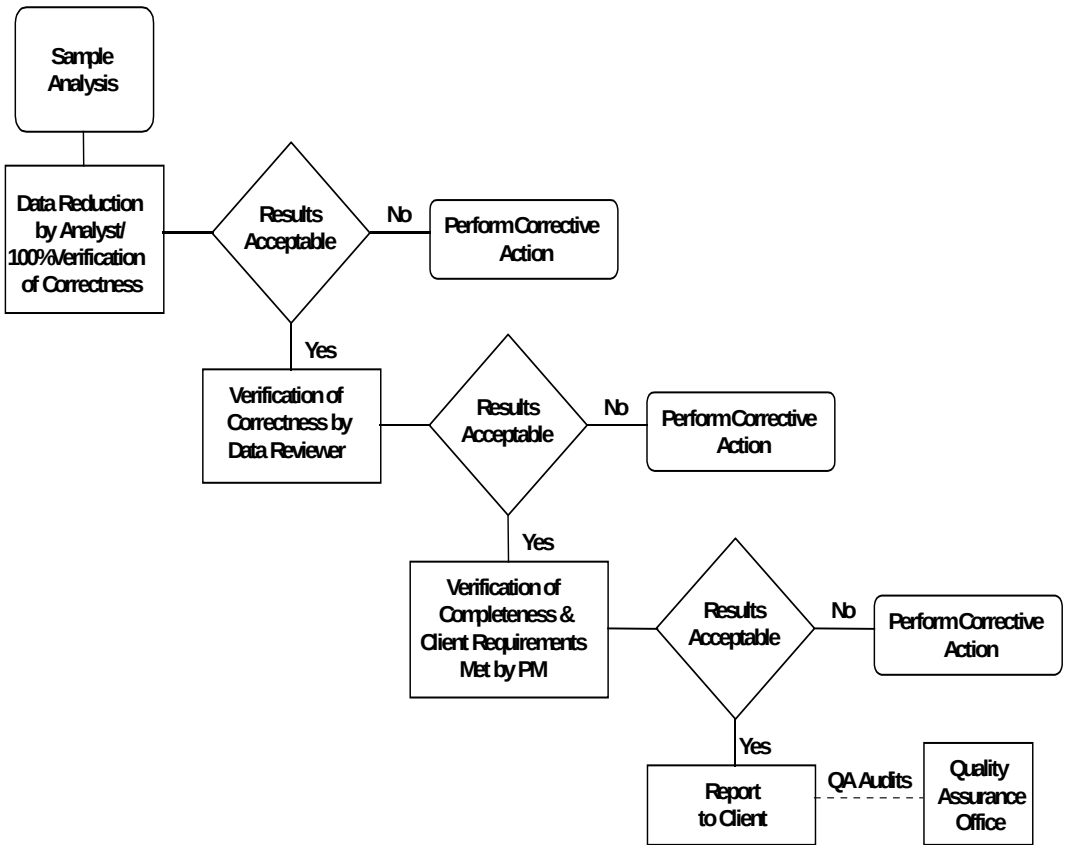
3. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory.

4. All raw data to reconstruct and validate these analyses have been retained at the facility.

5. The associated information is organized and available for review.

Department Supervisor	Signature	Date
Quality Assurance Officer	Signature	Date

Figure 19-2. Example: Work Flow



SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs and/or instrument manuals. (**Note:** for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Further details on balance calibrations and verifications can be found in SOP NC-QA-015 Balance and Thermometer Calibration, Container Verification.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All liquid in glass thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly.

The digital NIST thermometer is recalibrated every year (unless thermometer has been exposed to temperature extremes) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 0.01 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in spreadsheets, logsheets, and/or electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented on unit specific logsheets or in TALS. More information on this subject can be found in NC-QA-015 Balance and Thermometer Calibration, Container Verification.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer/continuous monitoring sensor for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

All of this information is documented on Daily Temperature Logsheets and/or electronically via TALS or the Temperature Guard System.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) and syringes are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy and considers the statement valid for the first six months. After six months, syringes that dispense volumes greater than 20 uL must be verified or replaced.

20.3.6 Field Sampling Devices (ISCO Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated semiannually or as needed by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 20%, the procedure is repeated and if the result is still greater than 20%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to

determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable **reported based upon discussion and approval of the client** under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed

after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit. For Ohio VAP samples, results may not be reported when calibration verifications fail the lower limit criterion.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Further details are given in in policy memorandum CA-Q-QM-001, Policy on Tentatively Identified Compounds (TICs) – GC/MS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV UX2 MSD (screen)	Hewlett Packard	5972	US00029070	1992	NEW
MSV UX2 Concentrator	OI Analytical	4560	J615460591		
MSV UX2 Sampler	Varion	Archon	12175		
MSV HP6 MSD (screen)	Hewlett Packard	5973	US00005571	1998	NEW
MSV HP6 Concentrator	OI Analytical	4560	M943460127		
MSV HP6 Sampler	OI Analytical	4552	12258		

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV UX7 MSD (screen)	Hewlett Packard	5973	US00010937	1998	NEW
MSV UX7 Concentrator	OI Analytical	4560	N251460461		
MSV UX7 Sampler	Dynatech Precision Sampling Corp.	Archon 5100A	12019		
MSV UX8 MSD	Hewlett Packard	6890	US00027773	1999	NEW
MSV UX8 Concentrator	OI Analytical	Eclipse 4660	B444466152P		
MSV UX8 Sampler	OI Analytical	4552	14092		
MSV UX9 MSD	Hewlett Packard	5973	US00028329	2000	NEW
MSV UX9 Concentrator	OI Analytical	4560	M946460832		
MSV UX9 Sampler	Environmental Sample Technology, Inc.	Archon	13667		
MSV UX10 MSD	Hewlett Packard	5973	US00032072	2000	NEW
MSV UX10 Concentrator	OI Analytical	Eclipse 4660	BETA 6		
MSV UX10 Sampler	OI Analytical	4552	012058		
MSV UX11 MSD	Agilent	5973 Network	US00038093	2000	NEW
MSV UX11 Concentrator	OI Analytical	4560	K811460270		
MSV UX11 Sampler	OI Analytical	4552	13408		
MSV UX12 MSD	Agilent	5973 Network	US10202133	2002	NEW
MSV UX12 Concentrator	OI Analytical	4560	M041460393		
MSV UX12 Sampler	Varion	Archon	12151		
MSV UX14 MSD	Agilent	5973 Inert	CN10340027	2003	NEW
MSV UX14 Concentrator	OI Analytical	4660	D829466914P		
MSV UX14 Sampler	OI Analytical	4552	14092		
MSV UX15 MSD	Agilent	5973 Inert	CN10515062	2005	NEW
MSV UX15 Concentrator	OI Analytical	Eclipse 4660	C511466149F		
MSV UX15 Sampler	OI Analytical	4552	14368		
MSV UX16 MSD	Agilent	5973 Inert	CN10539865	2005	NEW
MSV UX16 Concentrator	OI Analytical	Eclipse 4660	D539466261P		
MSV UX16 Sampler	OI Analytical	4552	14519		
MSV UX17 MSD	Agilent	5975 Inert	US10831043	2012	NEW
MSV UX17 Concentrator	OI Analytical	Eclipse 4660	H224455292P		
MSV UX17 Sampler	OI Analytical	4552	US12160002		

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV UX18 MSD	Hewlett Packard	5973	US00020913	2013	NEW
MSV UX18 Concentrator	OI Analytical	4560	N213460621		
MSV UX18 Sampler	Varion	Archon	12174		
MSV UX19 MS	Agilent	5977B	US1813M021	2018	NEW
MSV UX19 GC	Agilent	7890B	CN1750321		
MSV UX19 Concentrator	OI Analytical	4760	A818447017		
MSV UX19 Sampler	OI Analytical	4100	D818410016		
MSS HP7 MSD	Hewlett-Packard	5973-6890	US71190756	1998	NEW
MSS HP7 Sampler	Hewlett-Packard	6890 Series	US64900400		
MSS HP7 Tower	Hewlett-Packard	6890 Series	US64900400		
MSS HP7 GC	Hewlett-Packard	G1530A	US00009247		
MSS HP9 MSD	Hewlett-Packard	5973-6890	US91422379	2000	NEW
MSS HP9 Sampler	Hewlett-Packard	7683	US94606476		
MSS HP9 Tower	Hewlett-Packard	7683	US93408794		
MSS HP9 GC	ALS	Ready	US00027943		
MSS HP10 MSD	Agilent	5973	US33220074	2003	NEW
MSS HP10 Sampler	Agilent	7683 Series	US83501650		
MSS HP10 Tower	Agilent	7683 Series	CN33832656		
MSS HP10 GC	Agilent	6890N	CN10340002		
MSS AG2 MSD	Agilent	5975C Inert XL	US71235692	2007	NEW
MSS AG2 Sampler	Agilent	7683B Series	US91204712		
MSS AG2 Tower	Agilent	7683B Series	CN53827833		
MSS AG2 GC	Agilent	7890A	CN10721110		
MSS AG3 MSD	Agilent	5977A	US12203016	2014 in use:2017	NEW
MSS AG3 Sampler	Agilent	7693	CN11120088		
MSS AG3 Tower	Agilent	7693	CN11120088		
MSS AG3 GC	Agilent	7890A	CN10501006		
GC A	Agilent	6890 FID	US10402056	2004	NEW
GC O	Hewlett Packard	6890 FID	US00007206	1997	NEW
GC Y	Hewlett Packard	6890 FID	US10337062	2003	NEW
GC Z	Agilent	6890 EPC & PDD/FID	10205072	2000	NEW
GC P1	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00023208	1998	NEW
GC P1	Hewlett Packard	Sampler	US83401589	1998	NEW
GC P1	Hewlett Packard	Tower	CN14422923	1998	NEW
GC P2 (Screening only)	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US83802337	1998	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
GC P2	Hewlett Packard	Sampler	US93806108	1998	NEW
GC P2	Hewlett Packard	Tower	US83602262	1998	NEW
GC P3	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00023674	1998	NEW
GC P3	Hewlett Packard	Sampler	US92205419	1998	NEW
GC P3	Hewlett Packard	Tower	CN42637504	1998	NEW
GC P4	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00029531	1999	NEW
GC P4	Hewlett Packard	Sampler	CN51232596	1999	NEW
GC P4	Hewlett Packard	Tower	US82401457	1999	NEW
GC P5	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00029508	2010	NEW
GC P5	Hewlett Packard	Sampler	CN33826455	2010	NEW
GC P5	Hewlett Packard	Tower	US92407745 US00311160	2010	NEW
GC P6	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00032848	2000	NEW
GC P6	Hewlett Packard	Sampler	CN43130187	2000	NEW
GC P6	Hewlett Packard	Tower	CN14422929 US82401457	2000	NEW
GC P9 (screening only)	Agilent	6890 EPC & Dual ECD Y-Splitter	US10205045	2005	NEW
GC P9	Agilent	Sampler	US01708111	2005	NEW
GC P9	Agilent	Tower	CN14523156	2005	NEW
GC P10	Agilent	6890 EPC & Dual ECD Y-Splitter	US91907177	1999	NEW
GC P10	Agilent	Sampler	CN14920067	1999	NEW
GC P10	Agilent	Tower	US83802337	1999	NEW
GC P11	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10517088	2004	NEW
GC P11	Agilent	Sampler	US12411936	2004	NEW
GC P11	Agilent	Tower	CN43220375	2004	NEW
GC P12	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10512025	2005	NEW
GC P12	Agilent	Sampler	US92205419	2005	NEW
GC P12	Agilent	Tower	CN51124095	2005	NEW
GC P13	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10435032	2004	NEW
GC P13	Agilent	Sampler	CN42429315	2004	NEW
GC P13	Agilent	Tower	CN51825000	2004	NEW
GC P14	Agilent	7890 EPC & Dual ECD Y-Splitter	CN10281044	2012	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
GC P14	Agilent	Sampler	CN10220022	2012	NEW
GC P14	Agilent	Tower - front	CN10290167	2012	NEW
GC P14	Agilent	Tower - rear	CN10290169	2012	NEW
GC P15	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10427010	2012	NEW
GC P15	Agilent	Sampler	US11911568	2012	NEW
GC P15	Agilent	Tower	CN43220375	2012	NEW
GC P16	Agilent	6890 EPC & Dual ECD Y-Splitter	US00025858	2014	Borrowed
GC P16	Agilent	Sampler	US83001373	2014	Borrowed
GC P16	Agilent	Tower	CN14422847	2014	Borrowed
GC P18	Agilent	6890 EPC & Dual ECD Y-Splitter	US00006438	2015	NEW
GC P18	Agilent	Sampler	US93806073	2015	NEW
GC P18	Agilent	Tower	CN53827833	2015	NEW
GC HPLC L2	Agilent	HPLC 1100	US82404153	1998	NEW
GC M	Tekran	Tekran 2700	25	2012	NEW
GC N	Agilent	7890 Atomic Fluorescence	CN10820009	2008	NEW
Metals I-12	Thermo	Trace Analyzer 6500 Duo Ash	ICP-20101711	2013	NEW
I-12 Sampler	Elemental Scientific	SC-0500-04	FST04-TSP-090815		
I-12 Pump	Elemental Scientific	FVA	FVA-100203		
Metals I-9	Thermo	Trace Analyzer 6500 Duo Ash	ICP 20102403	2010	NEW
I-9 Sampler	Elemental Scientific	SC-4DX	X4DX-HS-TSP-16-100210		
I-9 Pump	Elemental Scientific	FVA	FVA-090915		
Metals I-14	Agilent	7800	JP18131103	2018	NEW
I-14 Sampler	Agilent	SPS4	AU17494204		
Metals I-10	Agilent	7700 ICPMS Series	JP124521145	2013	NEW
I-10 Sampler	Agilent	ASX-500	US121435A520		
Metals H2	Leeman	Hydra II AA	5VV5ZQ1	2015	NEW
H2 Sampler	Teldyne Leeman	Hydra II	4090		
Metals H3	Leeman	Hydra II AA	17010003	2015	NEW
H3 Sampler	Teldyne Leeman	Hydra II	4055		
Metals H6	Leeman	Hydra AF Gold+	1012	2011	NEW
H6 Sampler	Leeman	Hydra II	0014		
H6 Pump	Leeman	Hydra II	0011		

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
H6 Pump	Leeman	Hydra II	2013		
Metals H7	Leeman	Hydra AF Gold+	2011	2012	NEW
H7 Sampler	Leeman	Hydra II	2070		
H7 Pump	Leeman	Hydra II	3023		
H7 Pump	Leeman	Hydra II	2009		
WC Autotitrator (Severus)	Mantech	PC-1000-102/E	MT-1G8-798	2018	NEW
WC Severus Pump	Mantech	PC-1300-475	MT-117-1019		
WC Severus Burette	Mantech	PC-1000-1040	MT-1G8-219		
WC Severus Sampler	Mantech	Automatic 73	191C8020		
WC BOD (Bugsy) Sampler	Mantech	AutoMax 122	261H3N236	2013	NEW
WC Bugsy Pump	Mantech	PC-1000-443	MT-1J3-182		
WC Bugsy Pump	Mantech	PC-1000-408	MT-1K3-508		
WC Bugsy Pump	Mantech	PC-1000-475	MT-1J3-280		
WC Bugsy Interface	Mantech	PB-10030	MT-1A4-169		
WC Spec (Oscar)	Genesys	Spectronic 20	3SGK137005	2016	NEW
WC Spec (Ernie)	Genesys	Spectronic 20	3SGL226006	2008	NEW
WC IC (Veronica)	Dionex	ICS 2100	12031443	2012	NEW
WC Veronica Sampler	Thermo Sci / Dionex	AS-AP	12050895		
WC IC (Thor)	Dionex	Dionex Integrion	16081629	2016	NEW
WC Thor Sampler	Thermo Sci / Dionex	AS-AP	16110203		
WC Discrete Analyzer (Maggie)	Systea	Easy Chem Plus	07004	2013	USED
WC TOC (Clark)	OI Analytical	1030W	P428730168/ PARA	2014	NEW
WC Clark Sampler	OI Analytical	1088 AS	E427788106		
WC Block Digester (Larry)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Moe)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Curly)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Carol)	Lachat	BD46 TKN	00000993	1992	NEW
WC Spec (Snuffleupagus)	Genesys	Spectronic 20	3SGU022007	2016	NEW
WC DO Meter	Mantech	YSI 1500	13D 100737	2013	NEW
WC Conductivity (Xavier)	Orion	Star A112	J14311	2017	NEW
WC Cyanide Analyzer (CNthia)	Astoria Pacific	rAPID-T	4600-1035	2017	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
WC Cyanide Hotblock (Phil)	Simple Dist Micro	C8000	2017MDISW137	2017	NEW
WC Cyanide Hotblock (Lil)	Simple Dist Micro	C8000	2017MDISW136	2017	NEW
WC Flashpoint (Whitey)	Herzog	HFP 339	073390084	2007	NEW
WC pH Meter (Randolph)	Orion	320	020032	2007	NEW
WC Turbidimeter (Gyarados)	Hach	2100Q Turbidimeter	16040C049659	2016	NEW
WC Sulfide Distillation	Westco	Easy Dist 483-B000-01	1193	2008	NEW
WC Solid Phase Extraction Unit (Earl)	Horizon	SPE-DEX 3000XL	14-1971	2014	NEW
WC Conductivity Screening Meter (Myron II)	Myron L Company	TechPro II	T3201412	2018	NEW
COD Block (W1)	Hach	16500-10	870512456	1998	USED
COD Block (W3)	Hach	45600	900201914	1998	USED
COD Block (W4)	Hach	16500-10	890512473	1998	USED
COD Block (W5)	Biosciences	100 003	COD-B0075	2000	NEW
COD Block (W6)	Hach	16500-10	880911479	2018	USED
MBAS Shaker	Eberbach	6010	049817	2015	USED

Note: Laboratory instrumentation, model numbers, and serial numbers are subject to change without notice.

Table 20-2. Example: Schedule of Routine Maintenance

(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations. Maintenance procedures below are examples only and are subject to frequent change)

ION CHROMATOGRAPH

As Needed	Daily	Weekly	Monthly
Check fuses when power problems occur.	Check plumbing/leaks	Check pump heads for leaks. Check Filter inlet	

As Needed	Daily	Weekly	Monthly
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure		
De-gas pump head when flow is erratic.	Check conductivity meter		

HIGH PRESSURE LIQUID CHROMATOGRAPH

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Back-flush column.

ICP AND ICP/MS

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		

Daily	Monthly or As Needed	Semi-Annually	Annually
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

GAS CHROMATOGRAPH

Daily *	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Clip off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Replace injection port liner when front portion of capillary column is clipped.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed. Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed

Daily *	As Needed
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance. Clip column leader when chromatography looks bad—not daily.	Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.

**No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.*

MASS SPECTROMETER

Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		

Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

ANALYTICAL/TOP LOADING BALANCES

Daily	Annually
Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration

REFRIGERATORS/WALK-IN COOLERS

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

OVENS

Daily	As Needed
Temperatures checked and logged	Electronics serviced

SPECIFIC DIGITAL ION ANALYZER

Daily	As Needed
Daily when used: Calibrate with check standards Inspect electrode daily, clean as needed Inspect electrode proper levels of filling solutions daily; fill as needed Clean probe after each use	Electronics serviced

TURBIDIMETER

Daily	Monthly	As Needed
Daily when used: Adjust linearity on varying levels of NTU standards. Standardize with NTU standards Inspect cells	Clean instrument housing	Electronics serviced

DISSOLVED OXYGEN METER

Daily	As Needed
Daily when used: Calibrate with saturated air Check probe membrane for deterioration Clean and replace membrane with HCl solution	Electronics serviced Clean and replace membrane with HCl solution

CONDUCTANCE METER

Daily	As Needed
Daily when used: Check probe and cables Inspect conductivity cell	Electronics serviced

CHEMICAL OXYGEN DEMAND (COD) REACTOR 1

Daily	As Needed
Daily when used: Calibrate with check standards	Electronics serviced

SPECTROPHOTOMETER

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

pH METER

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

TOTAL ORGANIC CARBON ANALYZER

Daily	As Needed	Weekly	Monthly
Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs	Check injection port septum Indicating drying tube NDIR zero, after change in color indicator Permeation tube, every 6 months of use	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

DIGESTION BLOCK

Annually
Check temperature with NIST thermometer

Flash Point Tester

Daily
Check tubing Clean sample cup each use
Clean flash assembly
Check stirrer

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware, quarterly accuracy checks are performed for all mechanical volumetric devices. Micro syringes used to dispense volumes greater than 20 uL are verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips,

acid etching or deformity (e.g., bent needle). If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique standard number and expiration date. All documentation received with the reference standard is retained as a QC record and references the standard number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in TALS or in binders in the laboratory departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to SOP NC-QA-017 Standards and Reagents.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIM TALS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)

- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID from TALS

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the analytical SOPs.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in each analytical SOP and in Subsampling SOP NC-OP-046.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested

- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections. SOP NC-SC-005, Sample Receiving, describes the laboratory's sample receipt procedure.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Cooler Receipt Form (CRF) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

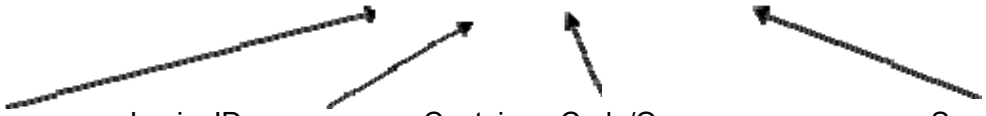
23.2.1.1 Unique Sample Identification

Note: Example sample **IDs from TALS.**

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):

Example: XXX - 9608 - A - 1



Location ID Number (3-digit # for your lab) | Login ID | Container Code/Occurrence | Sample

The above example states the TestAmerica Laboratory Location ID (Location XXX), the Login ID (9608) which is unique to a particular client/job occurrence, the container code (A) indicating the first container and Sample Number (1).

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005 Sample Receiving that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;

- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the TALS according SOP NC-SC-005.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in cold storage for 30 days after report generation, which meets or exceeds most sample holding times. After this time period, the samples are removed from the refrigerator

shelves and prepared for disposal. Special arrangements may be made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading “HAZARDOUS” or “FOREIGN SOIL” and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

See SOP NC-SC-019 Procedure of Acceptance and Handling of USDA Regulated Domestic and Foreign Soil for further information.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving). All procedures in the laboratory Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)

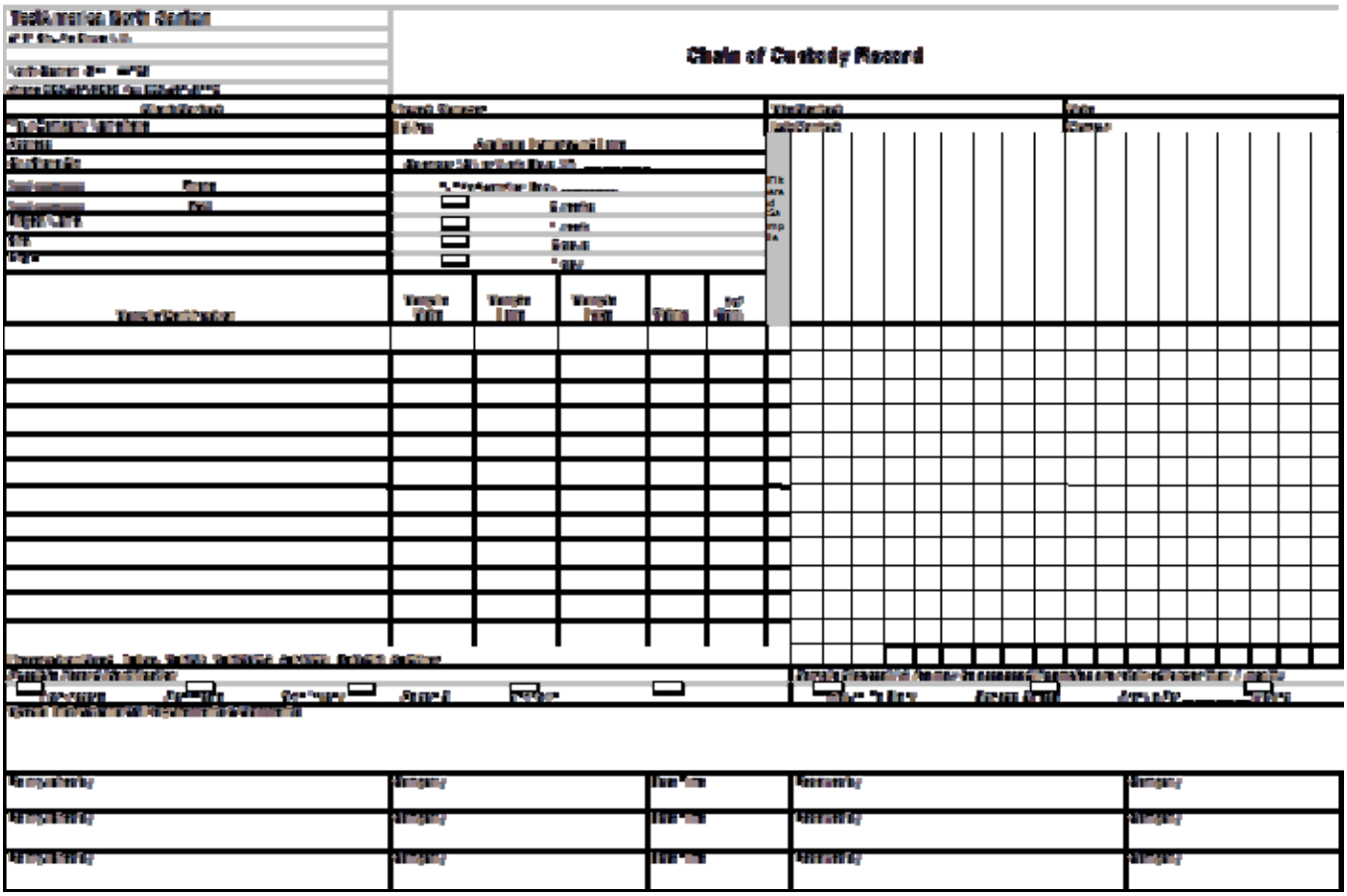


Figure 23-2. Example: Cooler Receipt Form (CRF)

TestAmerica Canton Sample Receipt Form/Narrative **Login # :_____**
Canton Facility

Client _____ Site Name_____ Cooler unpacked by:

Cooler Received on _____ Opened on_____

FedEx: 1st Grd Exp UPS FAS Clipper Client Drop Off TestAmerica Courier Other_____

Receipt After-hours: Drop-off Date/Time_____Storage Location_____

TestAmerica Cooler # _____ Foam Box Client Cooler Box Other _____

Packing material used: Bubble Wrap Foam Plastic Bag None Other _____

COOLANT: Wet Ice Blue Ice Dry Ice Water None

1. Cooler temperature upon receipt ☐ See Multiple Cooler Form

IR GUN# IR-8 (CF **+0** ºC) Observed Cooler Temp._____ºC Corrected Cooler Temp._____ºC

IR GUN #36 (CF **-0.3**°C) Observed Cooler Temp._____ºC Corrected Cooler Temp._____ºC

~~IR GUN # 627 (CF **-1.3**°C) Observed Cooler Temp._____ºC Corrected Cooler Temp._____ºC~~

2. Were tamper/custody seals on the outside of the cooler(s)? If Yes Quantity_____ Yes No
   - -Were the seals on the outside of the cooler(s) signed & dated? Yes No NA
   - -Were tamper/custody seals on the bottle(s) or bottle kits (LLHg/MeHg)? Yes No
   - -Were tamper/custody seals intact and uncompromised? Yes No NA
3. Shippers' packing slip attached to the cooler(s)? Yes No
4. Did custody papers accompany the sample(s)? Yes No
5. Were the custody papers relinquished & signed in the appropriate place? Yes No
6. Was/were the person(s) who collected the samples clearly identified on the COC? Yes No
7. Did all bottles arrive in good condition (Unbroken)? Yes No
8. Could all bottle labels be reconciled with the COC? Yes No
9. Were correct bottle(s) used for the test(s) indicated? Yes No
10. Sufficient quantity received to perform indicated analyses? Yes No
11. Are these work share samples? Yes No

    If yes, Questions 12-16 have been checked at the originating laboratory.
12. Were all preserved sample(s) at the correct pH upon receipt? Yes No NA pH Strip Lot# **HC740840**
13. Were VOAs on the COC? Yes No
14. Were air bubbles >6 mm in any VOA vials? ● ⬅ Larger than this. Yes No NA
15. Was a VOA trip blank present in the cooler(s)? Trip Blank Lot #_____ Yes No
16. Was a LL Hg or Me Hg trip blank present? _____ Yes No

Tests that are not checked for pH by Receiving:

VOAs
Oil and Grease
TOC

Contacted PM _____ Date _____ by _____ via Verbal Voice Mail Other

Concerning _____

17. CHAIN OF CUSTODY & SAMPLE DISCREPANCIES Samples processed by:

_____

_____

_____

_____

_____

18. SAMPLE CONDITION

Sample(s) _____were received after the recommended holding time had expired.

Sample(s) _____ were received in a broken container.
Sample(s) _____were received with bubble >6 mm in diameter. (Notify PM)

19. SAMPLE PRESERVATION

Sample(s) _____were further preserved in the laboratory.
Time preserved: _____Preservative(s) added/Lot number(s):_____.

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - *The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.*
   - Information must be legible

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink

- Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**

5) Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range.

   - Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.
   - If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
   - Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.
   - Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples. Note: Requirements for OVAP can be found in the OVAP specific SOPs.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	Used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the

laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.

Table 24-3. Sample Matrix Control

Control Type	Details	
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 200%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to NC-QA-018, Statistical Evaluation of data and Development of Control Charts for details.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Note: Ohio VAP does not allow the use of marginal exceedance. For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

Though marginal exceedances may be allowed by other programs, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., TestAmerica Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.)

25.2.11 Practical quantitation limits or reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND)

25.2.14 Sample results

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.23 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.24 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.26 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.27 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.2.28 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

25.2.29 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;
- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Canton offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by TestAmerica, the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting SOP No. CW-L-S-004.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

TestAmerica is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document, including cover letter.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R" The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "report re-issue" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

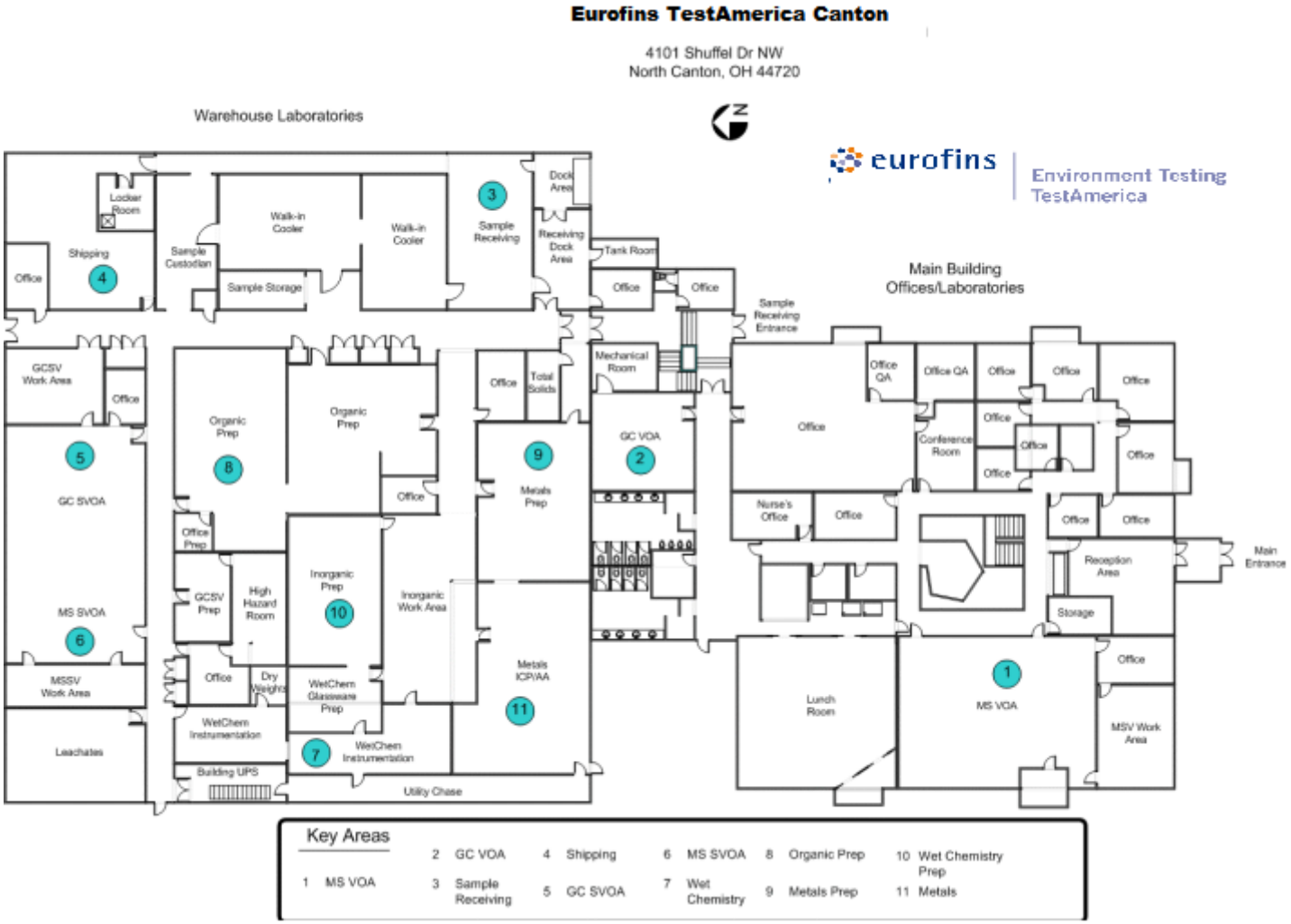
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate

EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Canton maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	2927	Nevada	OH-00048208A
Connecticut	PH-0590	New Jersey	OH001
Florida	E87225	New York	10975
Georgia	---	OVAP	CL0024
Illinois	004188	Pennsylvania	017
Kansas	E-10336	USDA (Dept. of Agriculture)	P330-08-00123
Kentucky Underground Storage Tank Program	112225	Washington	C971
Minnesota	039-999-348	West Virginia	210
Texas	T104704517-13-2 Changes based on the year and month of date of issue	Virginia	9448
Oregon	4062	Kentucky Wastewater	KY98016
Minnesota Petrofund	3506		

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

Title: Waste Dilution

[Method: SW846 Method 3580A]

Approvals (Signature/Date):

Technology Specialist	02/22/18 Date	Health & Safety Coordinator	02/26/18 Date
Quality Assurance Manager	05/10/18 Date	Technical Director	05/14/18 Date

This SOP was formerly known as NC-OP-043 Rev. 2, dated 2/22/18

Facility Distribution No. _____ Distributed To:_____

The controlled copies of this SOP are the SOP that is posted to the laboratory's SOP Directory and, where applicable, the copy of the SOP that is printed and placed in the General Chemistry SOP binder. Printed or electronic copies distributed outside of the facility are considered uncontrolled.

1. SCOPE AND APPLICATION

1.1. Method 3580A is a procedure for diluting nonvolatile and semivolatile organic compounds from organic, non-aqueous wastes that are **soluble** in the dilution solvent

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. The waste sample is weighed into a culture tube and diluted to an appropriate final volume using hexane or methylene chloride.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current edition.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. The use of high purity reagents and solvents helps minimize interference problems. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Test America associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the

Safety Data Sheets (SDS) maintained in the laboratory. The following specific hazards are known:

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Exposure to chemicals must be maintained **as low as reasonable achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of hazardous standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Test America associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Culture tubes with Teflon®-lined caps: 10 mL capacity

6.2. Balance: capable of accurately weighing ± 0.01g

6.3. Vortex mixer

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Extraction solvent is hexane or methylene chloride, pesticide quality or

equivalent.

7.2. Standards

7.2.1. Surrogate Standard

7.2.1.1. Prepare surrogate solutions at the appropriate concentration which allows detection of each compound within the linear range of the analysis (see Tables 1 and 3). Surrogate solutions are made in the solvent which is compatible with the dilution solvent. Store at 4°C ± 2° C.

7.2.2. Matrix Spike Standard

7.2.2.1. Prepare a matrix spike standard of the appropriate concentration for each target analyte group using the same procedures found in Section 7.2.1.1 (See Table 2).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are stored at 4°C ± 2°C.

8.2. Waste samples for PCB analysis, unless otherwise specified by regulatory or client programs, have a holding time of 1 year.

8.3. All other waste samples are extracted within fourteen days of the sampling date and the extracts analyzed within forty days of extraction.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, and MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of the appropriate dilution solvent containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated

analyte concentrations or false positive data. The MB should not contain any analyte of interest at or above the reporting limit.

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS consisting of the appropriate dilution solvent must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Summary

11.3.1. The waste sample is weighed into a culture tube and diluted to appropriate final volume using hexane or methylene chloride.

11.4. Sample Preparation Procedure

11.4.1. Sample Handling

11.4.1.1. Determine solubility of the waste in the appropriate dilution solvent.

Note: If the waste is not soluble in the dilution solvent do not proceed. The sample will have to be extracted as a solid waste. Alternately, the sample may be prepared as a water sample, dependent on the nature of the matrix.

Note: The phases of multiphase samples must be prepared separately.

11.4.1.2. Weigh approximately 1.0 g of the sample into the labeled and calibrated culture tube. Record the weight in the extraction log.

11.4.1.3. Add the appropriate volume of each required surrogate standard solution onto the sample. For the LCS and the sample in each analytical batch selected for matrix spiking, add the appropriate volume of the matrix spiking solution (see Section 9 for details on the surrogate standards and matrix spiking solutions).

11.4.1.4. Bring the sample to volume (10 mL) in the labeled and calibrated culture tube using the appropriate solvent. The dilution solvent for BNA and TPH analyses is methylene chloride. The dilution solvent for PCB and Pesticide analyses is hexane.

11.4.1.5. Mix thoroughly with vortex.

11.4.1.6. The sample is now ready for cleanup and analysis.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet and LIMs system, including the analytical data from standards, MBs, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not Applicable

13. METHOD PERFORMANCE

13.1. Each analyst must have initial demonstration of performance data on file and the laboratory must maintain corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with federal, state and local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in section 13 of the corporate environmental health and safety manual (cw-e-m-001) for “waste management and pollution prevention**.”**

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Sulfuric acid from acid stripping goes into Sulfuric Acid Waste #13.

15.2.2. TBA waste goes into Waste #26.

15.2.3. Extracts go into Vial Waste #4.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Method 3580A

16.1.2. Revision History

Historical File:		Revision 0: 08/19/13		
		Revision 1: 10/29/15		

**4/16/19: changed logo and copyright information. No changes were made to revision number or effective date.*

16.2. Associated SOPs

16.2.1. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and 8081B, NC-GC-042

16.2.2. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, NC-GC-043

16.2.3. Gas Chromatographic Analysis Based on Methods 8082 and 8082A, NC-GC-045

16.2.4. GC/MS Analysis Based on Methods 8270C and 8270D, NC-MS-018

16.2.5. Gas Chromatographic Analysis of Herbicides Based on Method 8151A, NC-GC-044

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. Refer to the individual methods listed in Table 4 for specific analyte reporting limits.

17.2. Tables

TABLE 1
Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA Waste Dilution	20 ppm BNA Surrogate Spike	2.5
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng o-Terphenyl	2.0
Herbicide	2 ppm 2,4-Dichlorophenylacetic Acid	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 2
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	20 ppm BNA Spike	0.5
BNA / SIM	20 ppm BNA Spike	0.1
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
Herbicide	Herbicide Standard Spike	1.0
TPH	Diesel	2.0

TABLE 3
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	20
	Nitrobenzene-d_5	20
	p-Terphenyl-d_{14}	20
	2-Fluorophenol	20
	Phenol-d_6	20
	2,4,6-Tribromophenol	20
	1,2-Dichlorobenzene-d_4	20
	2-Chlorophenol-d_4	20
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
Herbicide	2,4-Dichlorophenylacetic Acid	0.2
TPH	o-Terpphenyl	40.0

TABLE 4
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	1,1'Biphenyl	20
	1,2,4,5-Tetrachlorobenzene	20
	1,2,4-Trichlorobenzene	20
	1,2-Dichlorobenzene	20
	1,2-Diphenylhydrazine	20
	1,3-Dichlorobenzene	20
	1,3-Dinitrobenzene	20
	1,4-Dichlorobenzene	20
	1,4-Dioxane	20
	1-Methylnaphthalene	20
	2,2'-oxybis[1-chloropropane]	20
	2,3,4,6-Tetrachlorophenol	20
	2,4,5-Trichlorophenol	20
	2,4,6-Trichlorophenol	20
	2,4-Dichlorophenol	20
	2,4-Dimethylphenol	20
	2,4-Dinitrophenol	40
	2,4-Dinitrotoluene	20
	2,6-Dichlorophenol	20
	2,6-Dinitrotoluene	20
	2-Chloronaphthalene	20
	2-Chlorophenol	20
	2-Methylnaphthalene	20
	2-Methylphenol	20
	2-Nitroanaline	20
	2-Nitrophenol	20
	3&4-Methylphenol	20
	3,3'-Dichlorobenzidine	40
	3-Methylphenol	10
	3-Nitroanaline	20
	4,6-Dinitro-2-methylphenol	40
	4-Bromophenyl phenyl ether	20
	4-Chloro-3-methylphenol	20
	4-Chloroanaline	20
	4-Chlorophenyl phenyl ether	20
	4-Methylphenol	10
	4-Nitroanaline	20
	4-Nitrophenol	40
	Acenaphthene	20

TABLE 4
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthylene	20
	Acetophenone	20
	Aniline	20
	Anthracene	20
	Azobenzene	20
	Benzidine	40
	Benzo[a]anthracene	20
	Benzo[a]pyrene	20
	Benzo[b]fluoranthene	20
	Benzo[g,h,i]perylene	20
	Benzo[k]fluoranthene	20
	Benzoic acid	40
	Benzyl alcohol	20
	Bis(2-chloroethoxy)methane	20
	Bis(2-chloroethyl)ether	20
	Bis(2-ethylhexyl)phthalate	20
	Butyl benzyl phthalate	20
	Carbazole	20
	Chrysene	20
	Dibenz(a,h)anthracene	20
	Dibenzofuran	20
	Diethyl phthalate	20
	Dimethyl phthalate	20
	Di-n-butyl phthalate	20
	Di-n-octyl phthalate	20
	Fluoranthene	20
	Fluorene	20
	Hexachlorobenzene	20
	Hexachlorobutadiene	20
	Hexachlorocyclopentadiene	20
	Hexachloroethane	20
	hexadecane	20
	Indene	40
	ideno[1,2,3-cd]pyrene	20
	isophorone	20
	Naphthalene	20
	n-Decane	20
	Nitrobenzene	20
	N-Nitrosodimethylamine	20
	N-Nitrosodi-n-propylamine	20
	N-Nitrosodiphenylamine	40

TABLE 4
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
	n-Octadecane	20
	Pentachlorophenol	40
	Phenanthrene	20
	Phenol	20
	Pyrene	20
	Pyridine	20
Pesticides TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pesticides NPDES	Aldrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0
Herbicide	2,4,5-T	0.25
	2,4-D	1.0
	2,4-DB	1.0
	Dalapon	1.0
	Dicamba	0.5
	Dichlorprop	1.0
	Dinoseb	1.0
	MCPA	100
	MCPP	100
	Pentachlorophenol	0.25
	Picloram	1.0
	Silvex (2,4,5-TP)	0.25

TABLE 4
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
TPH	Diesel Fuel	2500 **µg/mL**

Title: ACID DIGESTION FOR SOLID SAMPLES

[Method: SW846 Method 3050B]

Approvals (Signature/Date):

Technology Specialist	08/30/18 Date	Health & Safety Coordinator	09/04/18 Date
Quality Assurance Manager	10/15/18 Date	Technical Director	10/12/18 Date

This SOP was previously identified as SOP No. NC-IP-010, Rev 7a, dated 7/19/17

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICPMS for the elements listed in Table 1 (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become “environmentally available”. By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, biological samples, and wipes.

1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICPMS analysis. The digestates are then filtered and diluted to 100 mL for subsequent analysis.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab-ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination, and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron from the glassware may leach into the sample solution during and following, sample processing. For critical low-level determinations of boron, only quartz and/or plastic lab-ware are recommended.

4.4. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., must be documented.

4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.

4.6. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5 The acidification of samples containing reactive materials may result in the release of toxic gases such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst must also be aware of the potential for a vigorous reaction.

5.6 Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8 Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned

up using appropriate spill kits.

5.10 Discard chipped or broken glassware to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath, or other heating source capable of maintaining a temperature of 91-99°C

6.2. Calibrated thermometer that covers a temperature range of 0-110°C

6.3. Vapor recovery device (Watch glasses, ribbed or other device)

6.4. Whatman No. 41 filter paper or equivalent

6.5. Funnels or equivalent filtration apparatus

6.6. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.7. Repeaters or suitable reagent dispensers

6.8. Calibrated automatic pipettes with corresponding pipette tips: 100uL, 500uL, 1mL-5mL

6.9. Class A volumetric flasks

6.10. Plastic digestate storage bottles, such as Corning Snap Seals™ (may be used if their accuracy is documented and is better than 2%)

6.11. Boiling Stones: Ultra Pure Polytetrafluoroethylene (PTFE) or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a DI water purification system that produces DI water approved for use in metals analysis. Reagent water must be free of the elements of interest as demonstrated through the analysis of method blanks (MB) as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in fluorinated ethylene propylene (FEP) fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for

up to one year from receipt and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP LCS/ MS solution: Prepare the ICP LCS/ MS working spike solutions from custom stock standards. Final concentrations are available in the LIMS. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS/MS solution must be made fresh every six months. The expiration date of the working ICP LCS/MS solution is the shorter of six months, the expiration date of the corresponding stock standard or when verification from an independent source indicates a problem. Refer to the reagent module in LIMS for details on standard preparation.

7.4. ICPMS LCS/MS solution: LCS/MS solutions are custom made. The final concentrations are available in the LIMS.

7.4.1. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional element(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.6. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid a violent reaction may occur.

7.7. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.8. 30% Hydrogen peroxide (H_2O_2), Ultrapure grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Solid samples are collected and stored in wide-mouth glass jars with PTFE-lined lids. A minimum of 10 g should be collected.

8.2. Sample holding time for metals included under the scope of this SOP is 180 days from the date of sample collection to the date of analysis.

8.3. Soil samples do not require preservation.

9. QUALITY CONTROL

9.1. Preparation Batch

9.1.1. A preparation batch consists of a group of up to 20 client samples (not counting the batch QC) that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain an MB, an LCS, and an MS/MSD. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated element concentrations or false positive data. Criteria for the acceptance of MB are contained within the individual analytical method SOPs. If the MB does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested.

9.2.2. The MB is prepared by weighing a 1g aliquot of PTFE boiling chips. The MB is then processed as described in Section 11.

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must contain all elements of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Refer to Section 7 for instructions on preparation of the LCS solutions.

9.3.2. The ICP LCS is prepared by spiking a 1g aliquot of PTFE boiling chips with 2 mL of the working LCS/ MS spike solution (Section 7). The ICPMS LCS is prepared by spiking a 1g aliquot of boiling chips with 1 mL of the LCS/MS solution (Section 7). The LCS is then processed as described in Section 11.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target elements have been added. An MSD is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. Tables 2 and 3 provide the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICPMS. Refer to Section 7 or the LIMS reagent module for instructions on preparation of the working matrix spike solutions.

9.4.3. The ICP MS/MSD is prepared by spiking a 1g aliquot of sample with 2 mL of the working LCS/MS spike solution (Section 7). The ICPMS MS/MSD is prepared by spiking a 1g aliquot of sample with 1 mL of the LCS/MS solution (Section 7). The MS/MSD is then processed as described in Section 11.

9.5. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.6. Control Limits

9.6.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.7.2. MDLs are accessible via the LIMs.

9.8. Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Hot block temperature must be verified daily for each unit used, and must be recorded in a hot block temperature log.

10.2. Laboratory support equipment is calibrated per SOPs NC-QA-004 and NC-QA-015.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo (NCM).

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Deviations are not allowed for Ohio VAP projects.

11.3. The heating procedures are carried out in a properly functioning hood.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. The LIMS provides sample labels to reduce transcription errors.

11.5. Samples are typically logged in as soils. Wastes, such as organic liquids or sludges and tissues (animal/vegetable), are usually logged in as solids. When initiating prep, examine the sample to see if the sample matches the matrix designation.

11.6. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. Refer to Appendix B for details.

11.8. Preparation of Soils, Sediments, and Sludges for Analysis by ICP and ICPMS

11.8.1. If sample can be mixed easily in the sample jar, mix thoroughly by stirring with a clean plastic or wooden spoon or spatula. If the sample cannot be easily mixed (i.e., clay samples or samples of various and very different particle sizes), use the spoon or spatula to select enough separate portions from locations within the jar to produce a representative sample. Analyst judgment is important in determining how many portions and which locations are used to produce a representative aliquot. If the sample is uniform clay, at least 3 portions should be selected from different locations in the sample

jar, if particle sizes or materials indicate a very non-homogenous sample, selection should be made carefully to collect an aliquot that represents the relative percentages of the various particle sizes and types in the sample jar.

11.8.2. For each digestion procedure required (i.e., ICP or ICPMS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.

Note: Wipe samples are not weighed. The entire wipe is used.

11.8.3. Measure additional aliquots of the designated sample(s) for the MS/ MSD analyses. MS/MSD samples must be weighed to the exact nominal weight due to a limitation of the LIMS system.

11.8.4. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.8.5. Heat sample to $95^{o} \pm 4°$ C and reflux for 10 minutes without boiling.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of element and the sample must be re-prepared.

11.8.6. Add 5 mL of concentrated HNO_3 to all samples and QC. Cover the sample containers with a watch glass or similar device.

11.8.7. Reflux at $95^{o} \pm 4°$ C for 30 minutes. (Add reagent water, as needed, to ensure that the volume of solution is not reduced to less than 5 mL.)

Note: If brown fumes are present, repeat steps 11.8.6 and 11.8.7 until brown fumes are no longer present.

11.8.8. Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.8.9. Continue adding 30% H_2O_2 in 1 mL aliquots until effervescence is minimal or sample appearance is unchanged. Make sure effervescence subsides before each addition of H_2O_2.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.8.10. Continue heating at $95^{o} \pm 4°$ C until the volume is reduced by cooking two hours or to approximately 5-10 mL.

11.8.11. Add 10 mL of concentrated HCL and reflux for an additional 15 minutes without boiling.

11.8.12. Allow the sample to cool.

11.8.13. Filter sample through Whatman 41 filter paper or equivalent, that has been rinsed with deionized water, into a measuring bottle (for example, Corning Snap Seals™). These may be used if their accuracy is documented and is better than ± 2%. Rinse sample container and filter paper with reagent water to ensure complete sample transfer.

11.8.14. Dilute sample to 100 mL with reagent water into a 120 mL graduated Snap Seal. The sample is now ready for analysis.

11.9. Incremental Sampling Method (ISM) Solid Preparation Procedure for Analysis by ICP and ICPMS

11.9.1. The laboratory will receive a single sample aliquot containing approximately 10 g from the Solids Lab. The Metals Prep department will divide this aliquot into two separate aliquots for the digestion procedure.

11.9.2. Two approximately 5 g aliquots of a sample are each digested in two separate 130 mL Environment Express tubes (or equivalent) and then combined and diluted to a final volume of 500mL. Because of this, reagents will be entered into TALS at 5X the usual amount, but only 2.5X of each reagent will be added to each tube since one sample is divided between two tubes.

11.9.3. To the two MB tubes, add exactly 5 g of Teflon® boiling chips each.

11.9.4. To the two LCS standard tubes, add exactly 5 g of Teflon® boiling chips each and the appropriate amount of the working standard.

11.9.5. To each sample and batch QC bottle carefully add 25 mL of 1:1 HNO3.

11.9.6. Heat the samples to 95° ±4° C and reflux for 10 minutes without boiling.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of element and the sample must be re-prepared.

11.9.7. Add 12.5 mL of concentrated HNO3 to each sample and batch QC digestion tube.

11.9.8. Continue heating at 95° ±4° C and reflux for 30 minutes.

Note: If brown fumes are present, repeat steps 11.9.7 and 11.9.8 until brown fumes are no longer present.

11.9.9. Add approximately 5 mL of reagent water and add 30% H2O2 in 1mL increments to each tube letting the effervescence subside between each addition until a total of 25mL has been added. It important to do this step carefully as the effervescence can be high.

11.9.10. Continue heating at 95° ±4° C and reflux for 2 hours.

11.9.11. Add 25mL of concentrated HCl to each tube. It is important to do this step carefully as the reaction of the hot sample with HCl can be high.

11.9.12. Continue heating at 95° ±4° C and reflux for 15 minutes.

11.9.13. Remove each sample and batch QC tube from the hotblock and allow the samples to cool.

11.9.14. Take each sample (2 digestate bottles per one sample) and pour the digestates into the appropriate labeled 500 mL plastic bottle. Rinse each digestion bottle twice with DI.

11.9.15. Using reagent water, bring the final volume up to the calibrated 500 mL mark and shake the sample vigorously to mix.

11.9.16. Filter approximately 50 mL to 60 mL of the sample from 11.9.15 through a Whatman #41 (or equivalent) filter that has been rinsed with reagent water into a 4 oz. snap seal container. The sample is now ready for analysis.

11.10. Analytical Documentation

11.10.1. Record all analytical information in LIMS including the analytical data from standards, blanks, LCSs, and MS/MSDs, Any corrective actions or modifications to the method must be noted in an NCM.

11.10.2. Record all standards and reagents in the LIMS Reagents module. All standards are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each analyst must make an initial demonstration of capability and yearly continuing demonstrations of capability for each individual element. This requires analysis of four QC Check samples.

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in a designated container labeled “Acid Waste”.

15.2.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled “Solid Waste”.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 3050B

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 1: 01/07/09		
(formerly CORP-IP0002NC)		Revision 2: 08/12/10		
Revision 2.1: 02/11/00		Revision 3: 11/23/12		
Revision 2.2: 09/25/01		Revision 4: 09/04/13		
Revision 2.3: 01/18/02		Revision 5: 09/30/14		
Revision 2.4: 02/19/03		Revision 6: 03/03/16		
Revision 2.5: 12/02/04		Revision 7a: 07/19/17		
Revision 2.6: 07/29/07				
Revision 0: 07/18/08 (NC-IP-010)				

**4/17/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Methods 6010B and 200.7, NC-MT-012

16.2.2. Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8, NC-MT-002

16.2.3. TestAmerica Canton Quality Control Program, QA-003

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Detection and Quantitation Limits CA-Q-S-006

16.2.7. Standards and Reagents, NC-QA-017

16.2.8. Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviations

17.1.1. The laboratory uses the same preparation procedure for ICP and ICPMS. Hydrochloric acid can be used for ICPMS due to the collision cell technology on newer instruments. Due to the potential chloride interferences, and possible inability to analyze for arsenic and tin, the laboratory must follow the instrument manufacturer guidelines pertaining to the use of HCL and ICP/MS analyses.

APPENDIX A: TABLES

TABLE1

Method 3050B Element List

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept clean.

Powdered gloves must not be used in the Metals Lab since the powder contains zinc, as well as other metallic elements.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic elements.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Appendix 2
Laboratory Reporting Limits and Method Detection Limits

Table 1

Laboratory Reporting Limits and Method Detection Limits - Soil
Former Cities Refinery
East Chicago, IN

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Soils	Percent Moisture	Moisture		Percent Solids	STL00234	0.100		%								
				Percent Moisture	STL00177	0.100		%								
Soils	Volatile Organic Compounds by GC/MS	8260C	5035A_FW	1,1,1-Trichloroethane	71-55-6	5.00	0.820	ug/Kg	60	126	40	27	131	40		
				1,1,2,2-Tetrachloroethane	79-34-5	5.00	1.43	ug/Kg	61	134	40	10	168	40		
				1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5.00	1.28	ug/Kg	58	144	40	30	145	40		
				1,1,2-Trichloroethane	79-00-5	5.00	1.13	ug/Kg	78	120	40	17	152	40		
				1,1-Dichloroethane	75-34-3	5.00	0.693	ug/Kg	69	120	40	35	129	40		
				1,1-Dichloroethene	75-35-4	5.00	0.903	ug/Kg	48	140	40	20	150	40		
				1,2,4-Trichlorobenzene	120-82-1	5.00	0.572	ug/Kg	56	120	40	10	120	40		
				1,2-Dibromo-3-Chloropropane	96-12-8	10.0	3.61	ug/Kg	35	137	40	10	135	40		
				Ethylene Dibromide	106-93-4	5.00	0.770	ug/Kg	73	126	40	24	138	40		
				1,2-Dichlorobenzene	95-50-1	5.00	1.11	ug/Kg	74	120	40	10	131	40		
				1,2-Dichloroethane	107-06-2	5.00	0.772	ug/Kg	66	120	40	33	130	40		
				1,2-Dichloropropane	78-87-5	5.00	0.851	ug/Kg	77	120	40	33	134	40		
				1,3-Dichlorobenzene	541-73-1	5.00	0.816	ug/Kg	74	120	40	10	131	40		
				1,4-Dichlorobenzene	106-46-7	5.00	0.882	ug/Kg	74	120	40	10	129	40		
				2-Butanone (MEK)	78-93-3	20.0	3.56	ug/Kg	61	131	40	31	148	40		
				2-Hexanone	591-78-6	20.0	4.08	ug/Kg	54	135	40	23	149	40		
				4-Methyl-2-pentanone (MIBK)	108-10-1	20.0	3.71	ug/Kg	56	124	40	29	140	40		
				Acetone	67-64-1	25.0	21.0	ug/Kg	47	157	40	18	167	40		
				Benzene	71-43-2	5.00	0.698	ug/Kg	75	120	40	32	131	40		
				Dichlorobromomethane	75-27-4	5.00	0.679	ug/Kg	63	121	40	18	125	40		
				Bromoform	75-25-2	5.00	2.40	ug/Kg	44	131	40	10	122	40		
				Bromomethane	74-83-9	5.00	0.988	ug/Kg	10	158	40	10	149	40		
				Carbon disulfide	75-15-0	5.00	1.16	ug/Kg	33	144	40	10	134	40		
				Carbon tetrachloride	56-23-5	5.00	3.25	ug/Kg	54	130	40	13	131	40		
				Chlorobenzene	108-90-7	5.00	0.916	ug/Kg	79	120	40	16	129	40		
				Chloroethane	75-00-3	5.00	1.22	ug/Kg	10	159	40	10	155	40		
				Chloroform	67-66-3	5.00	0.788	ug/Kg	74	120	40	38	129	40		
				Chloromethane	74-87-3	5.00	1.04	ug/Kg	40	127	40	20	140	40		
				cis-1,2-Dichloroethene	156-59-2	5.00	0.651	ug/Kg	76	120	40	35	130	40		
				cis-1,3-Dichloropropene	10061-01-5	5.00	1.44	ug/Kg	62	124	40	12	131	40		
				Cyclohexane	110-82-7	10.0	1.38	ug/Kg	57	126	40	17	133	40		
				1,4-Dioxane	123-91-1	250	22.2	ug/Kg	44	154	40	48	149	40		
				Chlorodibromomethane	124-48-1	5.00	2.78	ug/Kg	60	121	40	15	125	40		
				Dichlorodifluoromethane	75-71-8	5.00	0.943	ug/Kg	18	137	40	10	141	40		
				Ethylbenzene	100-41-4	5.00	1.05	ug/Kg	75	120	40	12	133	40		
				Isopropylbenzene	98-82-8	5.00	0.832	ug/Kg	74	120	40	10	135	40		
				Methyl acetate	79-20-9	25.0	3.40	ug/Kg	63	120	40	20	155	40		
				Methyl tert-butyl ether	1634-04-4	5.00	0.820	ug/Kg	66	120	40	42	127	40		
				Methylcyclohexane	108-87-2	10.0	1.23	ug/Kg	62	124	40	10	133	40		
				Methylene Chloride	75-09-2	25.0	12.0	ug/Kg	48	142	40	22	153	40		
				Styrene	100-42-5	5.00	1.16	ug/Kg	70	120	40	10	127	40		
				Tetrachloroethene	127-18-4	5.00	0.730	ug/Kg	75	124	40	13	144	40		
				Toluene	108-88-3	5.00	0.773	ug/Kg	76	120	40	20	141	40		
				trans-1,2-Dichloroethene	156-60-5	5.00	0.465	ug/Kg	74	125	40	31	138	40		
				trans-1,3-Dichloropropene	10061-02-6	5.00	1.03	ug/Kg	58	120	40	10	123	40		
				Trichloroethene	79-01-6	5.00	0.633	ug/Kg	75	123	40	10	162	40		
				Trichlorofluoromethane	75-69-4	5.00	1.08	ug/Kg	33	152	40	16	148	40		
				Vinyl chloride	75-01-4	5.00	0.837	ug/Kg	39	140	40	15	150	40		
				Xylenes, Total	1330-20-7	10.0	1.59	ug/Kg	77	120	40	10	134	40		
				m-Xylene & p-Xylene	179601-23-1	10.0	0.783	ug/Kg	76	120	40	10	132	40		
				o-Xylene	95-47-6	5.00	0.861	ug/Kg	76	120	40	11	134	40		
				Toluene-d8 (Surr)	2037-26-5	5.00		ug/Kg			40				64	124
				Dibromofluoromethane (Surr)	1868-53-7	5.00		ug/Kg			40				56	122
				4-Bromofluorobenzene (Surr)	460-00-4	5.00		ug/Kg			40				51	127
				1,2-Dichloroethane-d4 (Surr)	17060-07-0	5.00		ug/Kg			40				59	120
Soils	Volatile Organic Compounds by GC/MS	8260C	5035A_FM	1,1,1-Trichloroethane	71-55-6	250	14.0	ug/Kg	60	126	40	27	131	40		
				1,1,2,2-Tetrachloroethane	79-34-5	250	14.0	ug/Kg	61	134	40	10	168	40		
				1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	250	67.0	ug/Kg	58	144	40	30	145	40		
				1,1,2-Trichloroethane	79-00-5	250	13.0	ug/Kg	78	120	40	17	152	40		
				1,1-Dichloroethane	75-34-3	250	9.00	ug/Kg	69	120	40	35	129	40		
				1,1-Dichloroethene	75-35-4	250	15.0	ug/Kg	48	140	40	20	150	40		
				1,2,4-Trichlorobenzene	120-82-1	250	133	ug/Kg	56	120	40	10	120	40		
				1,2-Dibromo-3-Chloropropane	96-12-8	500	88.0	ug/Kg	35	137	40	10	135	40		
				Ethylene Dibromide	106-93-4	250	79.0	ug/Kg	73	126	40	24	138	40		
				1,2-Dichlorobenzene	95-50-1	250	21.0	ug/Kg	74	120	40	10	131	40		
				1,2-Dichloroethane	107-06-2	250	12.0	ug/Kg	66	120	40	33	130	40		
				1,2-Dichloropropane	78-87-5	250	8.00	ug/Kg	77	120	40	33	134	40		
				1,3-Dichlorobenzene	541-73-1	250	21.0	ug/Kg	74	120	40	10	131	40		
				1,4-Dichlorobenzene	106-46-7	250	17.0	ug/Kg	74	120	40	10	129	40		
				2-Butanone (MEK)	78-93-3	1000	157	ug/Kg	61	131	40	31	148	40		
				2-Hexanone	591-78-6	1000	263	ug/Kg	54	135	40	23	149	40		
				4-Methyl-2-pentanone (MIBK)	108-10-1	1000	238	ug/Kg	56	124	40	29	140	40		
				Acetone	67-64-1	1000	244	ug/Kg	47	157	40	18	167	40		
				Benzene	71-43-2	250	8.00	ug/Kg	75	120	40	32	131	40		
				Dichlorobromomethane	75-27-4	250	28.0	ug/Kg	63	121	40	18	125	40		
				Bromoform	75-25-2	250	106	ug/Kg	44	131	40	10	122	40		
				Bromomethane	74-83-9	250	166	ug/Kg	10	158	40	10	149	40		
				Carbon disulfide	75-15-0	250	108	ug/Kg	33	144	40	10	134	40		
				Carbon tetrachloride	56-23-5	250	18.0	ug/Kg	54	130	40	13	131	40		
				Chlorobenzene	108-90-7	250	35.0	ug/Kg	79	120	40	16	129	40		
				Chloroethane	75-00-3	250	150	ug/Kg	10	159	40	10	155	40		
				Chloroform	67-66-3	250	9.00	ug/Kg	74	120	40	38	129	40		
				Chloromethane	74-87-3	250	66.0	ug/Kg	40	127	40	20	140	40		
				cis-1,2-Dichloroethene	156-59-2	250	9.00	ug/Kg	76	120	40	35	130	40		

Table 1

Laboratory Reporting Limits and Method Detection Limits - Soil
Former Cities Refinery
East Chicago, IN

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
				cis-1,3-Dichloropropene	10061-01-5	250	37.0	ug/Kg	62	124	40	12	131	40		
				Cyclohexane	110-82-7	500	163	ug/Kg	57	126	40	17	133	40		
				1,4-Dioxane	123-91-1	12500	1090	ug/Kg	44	154	40	48	149	40		
				Chlorodibromomethane	124-48-1	250	117	ug/Kg	60	121	40	15	125	40		
				Dichlorodifluoromethane	75-71-8	250	53.0	ug/Kg	18	137	40	10	141	40		
				Ethylbenzene	100-41-4	250	12.0	ug/Kg	75	120	40	12	133	40		
				Isopropylbenzene	98-82-8	250	38.0	ug/Kg	74	120	40	10	135	40		
				Methyl acetate	79-20-9	1250	526	ug/Kg	63	120	40	20	155	40		
				Methyl tert-butyl ether	1634-04-4	250	37.0	ug/Kg	66	120	40	42	127	40		
				Methylcyclohexane	108-87-2	500	66.0	ug/Kg	62	124	40	10	133	40		
				Methylene Chloride	75-09-2	500	383	ug/Kg	48	142	40	22	153	40		
				Styrene	100-42-5	250	12.0	ug/Kg	70	120	40	10	127	40		
				Tetrachloroethene	127-18-4	250	15.0	ug/Kg	75	124	40	13	144	40		
				Toluene	108-88-3	250	45.0	ug/Kg	76	120	40	20	141	40		
				trans-1,2-Dichloroethene	156-60-5	250	15.0	ug/Kg	74	125	40	31	138	40		
				trans-1,3-Dichloropropene	10061-02-6	250	105	ug/Kg	58	120	40	10	123	40		
				Trichloroethene	79-01-6	250	13.0	ug/Kg	75	123	40	10	162	40		
				Trichlorofluoromethane	75-69-4	250	46.0	ug/Kg	33	152	40	16	148	40		
				Vinyl chloride	75-01-4	250	10.0	ug/Kg	39	140	40	15	150	40		
				Xylenes, Total	1330-20-7	500	23.0	ug/Kg	77	120	40	10	134	40		
				m-Xylene & p-Xylene	179601-23-1	250	53.0	ug/Kg	76	120	40	10	132	40		
				o-Xylene	95-47-6	250	11.0	ug/Kg	76	120	40	11	134	40		
				Toluene-d8 (Surr)	2037-26-5	250		ug/Kg			40				55	123
				Dibromofluoromethane (Surr)	1868-53-7	250		ug/Kg			40				49	122
				4-Bromofluorobenzene (Surr)	460-00-4	250		ug/Kg			40				51	124
				1,2-Dichloroethane-d4 (Surr)	17060-07-0	250		ug/Kg			40				47	136
Soils	Semivolatile Organic Compounds (GC/MS)	8270D	3540C	1,1'-Biphenyl	92-52-4	50.0	17.0	ug/Kg	43	120	40	38	120	32		
				bis (2-chloroisopropyl) ether	108-60-1	100	10.0	ug/Kg	29	120	40	27	120	40		
				2,4,5-Trichlorophenol	95-95-4	150	69.0	ug/Kg	28	120	40	22	120	40		
				2,4,6-Trichlorophenol	88-06-2	150	64.0	ug/Kg	14	120	40	15	120	34		
				2,4-Dichlorophenol	120-83-2	150	44.0	ug/Kg	40	120	40	30	120	40		
				2,4-Dimethylphenol	105-67-9	150	40.0	ug/Kg	31	120	40	25	120	36		
				2,4-Dinitrophenol	51-28-5	330	142	ug/Kg	10	120	40	10	120	40		
				2,4-Dinitrotoluene	121-14-2	200	62.0	ug/Kg	49	120	40	51	120	21		
				2,6-Dinitrotoluene	606-20-2	200	56.0	ug/Kg	49	120	40	51	120	20		
				2-Chloronaphthalene	91-58-7	50.0	14.0	ug/Kg	42	120	40	39	120	32		
				2-Chlorophenol	95-57-8	50.0	10.0	ug/Kg	42	120	40	30	120	40		
				2-Methylnaphthalene	91-57-6	15.0	1.96	ug/Kg	42	120	40	10	133	40		
				2-Methylphenol	95-48-7	200	31.0	ug/Kg	42	120	40	28	120	40		
				2-Nitroaniline	88-74-4	200	40.0	ug/Kg	44	120	40	49	120	19		
				2-Nitrophenol	88-75-5	50.0	13.0	ug/Kg	41	120	40	25	120	40		
				3,3'-Dichlorobenzidine	91-94-1	100	43.0	ug/Kg	29	120	40	10	120	40		
				3-Nitroaniline	99-09-2	200	49.0	ug/Kg	41	120	40	20	120	40		
				4,6-Dinitro-2-methylphenol	534-52-1	330	80.0	ug/Kg	27	120	40	10	123	40		
				4-Bromophenyl phenyl ether	101-55-3	50.0	14.0	ug/Kg	47	120	40	47	120	20		
				1,4-Dioxane	123-91-1	150	15.0	ug/Kg	10	120	40	10	120	40		
				4-Chloro-3-methylphenol	59-50-7	150	45.0	ug/Kg	39	120	40	33	120	40		
				4-Chloroaniline	106-47-8	150	30.0	ug/Kg	30	120	40	21	120	40		
				4-Chlorophenyl phenyl ether	7005-72-3	50.0	14.0	ug/Kg	46	120	40	47	120	21		
				4-Nitroaniline	100-01-6	200	60.0	ug/Kg	47	120	40	20	120	40		
				4-Nitrophenol	100-02-7	330	94.0	ug/Kg	29	120	40	14	125	36		
				Acenaphthene	83-32-9	15.0	2.86	ug/Kg	45	120	40	41	120	34		
				Acenaphthylene	208-96-8	15.0	4.01	ug/Kg	45	120	40	39	120	34		
				Acetophenone	98-86-2	100	11.0	ug/Kg	42	120	40	32	120	40		
				Anthracene	120-12-7	15.0	2.41	ug/Kg	52	120	40	43	106	32		
				Atrazine	1912-24-9	200	36.0	ug/Kg	54	120	40	50	120	22		
				Benzaldehyde	100-52-7	100	23.0	ug/Kg	38	120	40	18	120	40		
				Benzo[a]anthracene	56-55-3	15.0	3.41	ug/Kg	52	120	40	32	120	37		
				Benzo[a]pyrene	50-32-8	15.0	9.34	ug/Kg	50	120	40	35	120	38		
				Benzo[b]fluoranthene	205-99-2	15.0	6.50	ug/Kg	52	120	40	27	126	40		
				Benzo[g,h,i]perylene	191-24-2	15.0	7.10	ug/Kg	54	120	40	29	122	40		
				Benzo[k]fluoranthene	207-08-9	15.0	6.93	ug/Kg	54	120	40	39	120	37		
				Bis(2-chloroethoxy)methane	111-91-1	100	12.0	ug/Kg	43	120	40	36	120	39		
				Bis(2-chloroethyl)ether	111-44-4	100	12.0	ug/Kg	41	120	40	32	120	40		
				Bis(2-ethylhexyl) phthalate	117-81-7	70.0	51.0	ug/Kg	47	124	40	42	129	40		
				Butyl benzyl phthalate	85-68-7	70.0	22.0	ug/Kg	47	120	40	39	121	24		
				Caprolactam	105-60-2	330	75.0	ug/Kg	55	120	40	44	120	28		
				Carbazole	86-74-8	50.0	19.0	ug/Kg	51	120	40	46	120	28		
				Chrysene	218-01-9	15.0	1.49	ug/Kg	53	120	40	31	121	37		
				Dibenz(a,h)anthracene	53-70-3	15.0	6.92	ug/Kg	50	120	40	36	120	38		
				Dibenzofuran	132-64-9	50.0	13.0	ug/Kg	46	120	40	45	120	26		
				Diethyl phthalate	84-66-2	70.0	31.0	ug/Kg	45	120	40	45	120	19		
				Dimethyl phthalate	131-11-3	70.0	14.0	ug/Kg	47	120	40	47	120	20		
				Di-n-butyl phthalate	84-74-2	70.0	22.0	ug/Kg	50	120	40	46	120	21		
				Di-n-octyl phthalate	117-84-0	70.0	28.0	ug/Kg	38	122	40	44	120	25		
				Fluoranthene	206-44-0	15.0	4.45	ug/Kg	54	120	40	30	125	31		
				Fluorene	86-73-7	15.0	2.74	ug/Kg	48	120	40	44	120	32		
				Hexachlorobenzene	118-74-1	15.0	2.85	ug/Kg	45	120	40	47	120	23		
				Hexachlorobutadiene	87-68-3	50.0	12.0	ug/Kg	34	120	40	27	120	40		
				Hexachlorocyclopentadiene	77-47-4	330	62.0	ug/Kg	10	120	40	10	120	40		
				Hexachloroethane	67-72-1	50.0	9.00	ug/Kg	36	120	40	10	120	40		
				Indeno[1,2,3-cd]pyrene	193-39-5	15.0	7.36	ug/Kg	52	120	40	34	120	40		
				Isophorone	78-59-1	50.0	12.0	ug/Kg	42	120	40	32	120	40		
				N-Nitrosodi-n-propylamine	621-64-7	50.0	11.0	ug/Kg	39	120	40	30	120	40		
				N-Nitrosodiphenylamine	86-30-6	50.0	12.0	ug/Kg	50	120	40	46	120	24		
				Naphthalene	91-20-3	15.0	2.41	ug/Kg	39	120	40	30	120	40		
				Nitrobenzene	98-95-3	100	13.0	ug/Kg	42	120	40	32	120	40		

Table 1

Laboratory Reporting Limits and Method Detection Limits - Soil
Former Cities Refinery
East Chicago, IN

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
				Pentachlorophenol	87-86-5	150	58.0	ug/Kg	16	120	40	10	120	40		
				Phenanthrene	85-01-8	15.0	2.23	ug/Kg	50	120	40	31	120	35		
				Phenol	108-95-2	50.0	8.00	ug/Kg	39	120	40	25	120	40		
				Pyrene	129-00-0	15.0	2.14	ug/Kg	50	120	40	28	122	30		
				3 & 4 Methylphenol	15831-10-4	400	29.0	ug/Kg	43	120	40	34	120	40		
				Terphenyl-d14 (Surr)	1718-51-0	330		ug/Kg			40				39	120
				Phenol-d5 (Surr)	4165-62-2	330		ug/Kg			40				28	120
				Nitrobenzene-d5 (Surr)	4165-60-0	330		ug/Kg			40				28	120
				2-Fluorophenol (Surr)	367-12-4	330		ug/Kg			40				26	120
				2-Fluorobiphenyl (Surr)	321-60-8	330		ug/Kg			40				35	120
				2,4,6-Tribromophenol (Surr)	118-79-6	330		ug/Kg			40				10	120
Soils	Metals (ICP)	6010C	3050B	Aluminum	7429-90-5	20.0	5.33	mg/Kg	80	120	20	75	125	20		
				Silver	7440-22-4	1.00	0.0810	mg/Kg	80	120	20	75	125	20		
				Barium	7440-39-3	20.0	0.362	mg/Kg	80	120	20	75	125	20		
				Beryllium	7440-41-7	0.500	0.0540	mg/Kg	80	120	20	75	125	20		
				Calcium	7440-70-2	500	36.5	mg/Kg	80	120	20	75	125	20		
				Cadmium	7440-43-9	0.500	0.0480	mg/Kg	80	120	20	75	125	20		
				Cobalt	7440-48-4	1.00	0.200	mg/Kg	80	120	20	75	125	20		
				Chromium	7440-47-3	1.00	0.151	mg/Kg	80	120	20	75	125	20		
				Copper	7440-50-8	2.50	0.236	mg/Kg	80	120	20	75	125	20		
				Iron	7439-89-6	20.0	6.94	mg/Kg	80	120	20	75	125	20		
				Potassium	7440-09-7	500	36.1	mg/Kg	80	120	20	75	125	20		
				Magnesium	7439-95-4	500	46.1	mg/Kg	80	120	20	75	125	20		
				Manganese	7439-96-5	1.50	0.309	mg/Kg	80	120	20	75	125	20		
				Sodium	7440-23-5	500	62.8	mg/Kg	80	120	20	75	125	20		
				Nickel	7440-02-0	4.00	0.233	mg/Kg	80	120	20	75	125	20		
				Antimony	7440-36-0	2.00	0.359	mg/Kg	80	120	20	75	125	20		
				Vanadium	7440-62-2	5.00	0.822	mg/Kg	80	120	20	75	125	20		
				Zinc	7440-66-6	5.00	1.37	mg/Kg	80	120	20	75	125	20		
				Arsenic	7440-38-2	1.50	0.316	mg/Kg	80	120	20	75	125	20		
				Lead	7439-92-1	1.00	0.282	mg/Kg	80	120	20	75	125	20		
				Selenium	7782-49-2	2.00	0.469	mg/Kg	80	120	20	75	125	20		
				Thallium	7440-28-0	2.00	0.399	mg/Kg	80	120	20	75	125	20		
Soils	Mercury (CVAA)	7471A	7471A_Prep	Mercury	7439-97-6	0.100	0.0180	mg/Kg	80	120	20	80	120	20		

Table 2

Laboratory Reporting Limits and Method Detection Limits - Groundwater
Former Cities Refinery
East Chicago, Indiana

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Waters	1,4-Dioxane (GC/MS SIM)	8270D_SIM_14DX	3510C_LVI	1,4-Dioxane	123-91-1	0.400	0.174	ug/L	10	150	30	10	150	30		
				1,4-Dichlorobenzene-d4	3855-82-1			ug/L								
				Naphthalene-d8	1146-65-2			ug/L								
				DFTPP	5074-71-5			ug/L								
				Nitrobenzene-d5	4165-60-0			ug/L							41	144
Waters	Volatile Organic Compounds by GC/MS	8260C	5030C	1,1,1-Trichloroethane	71-55-6	1.00	0.240	ug/L	65	141	35	62	135	35		
				1,1,2,2-Tetrachloroethane	79-34-5	1.00	0.130	ug/L	45	151	35	37	149	35		
				1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1.00	0.410	ug/L	54	148	35	41	145	35		
				1,1,2-Trichloroethane	79-00-5	1.00	0.0900	ug/L	79	121	35	73	122	35		
				1,1-Dichloroethane	75-34-3	1.00	0.170	ug/L	74	126	35	71	121	35		
				1,1-Dichloroethene	75-35-4	1.00	0.190	ug/L	73	129	35	64	132	35		
				1,2,4-Trichlorobenzene	120-82-1	1.00	0.260	ug/L	47	120	35	37	120	25		
				1,2-Dichlorobenzene	95-50-1	1.00	0.150	ug/L	74	120	35	65	120	35		
				1,2-Dichloroethane	107-06-2	1.00	0.210	ug/L	66	129	35	65	127	35		
				1,2-Dichloropropane	78-87-5	1.00	0.150	ug/L	79	127	35	73	125	35		
				1,3-Dichlorobenzene	541-73-1	1.00	0.150	ug/L	74	120	35	64	120	35		
				1,4-Dichlorobenzene	106-46-7	1.00	0.160	ug/L	75	120	35	65	120	35		
				2-Butanone (MEK)	78-93-3	10.0	1.16	ug/L	41	151	35	40	147	35		
				2-Hexanone	591-78-6	10.0	0.540	ug/L	43	142	35	44	142	35		
				4-Methyl-2-pentanone (MIBK)	108-10-1	10.0	0.420	ug/L	43	145	35	40	147	35		
				Acetone	67-64-1	10.0	5.41	ug/L	33	155	35	32	157	35		
				Benzene	71-43-2	1.00	0.130	ug/L	77	123	35	70	121	35		
				Dichlorobromomethane	75-27-4	1.00	0.170	ug/L	73	122	35	69	120	35		
				Bromoform	75-25-2	1.00	0.760	ug/L	47	133	35	41	128	35		
				Bromomethane	74-83-9	1.00	0.420	ug/L	48	144	35	41	136	35		
				Carbon disulfide	75-15-0	1.00	0.280	ug/L	67	127	35	54	131	35		
				Carbon tetrachloride	56-23-5	1.00	0.260	ug/L	61	142	35	53	142	35		
				Chlorobenzene	108-90-7	1.00	0.140	ug/L	80	120	35	73	120	35		
				Chloroethane	75-00-3	1.00	0.830	ug/L	41	147	35	37	142	35		
				Chloroform	67-66-3	1.00	0.130	ug/L	74	127	35	66	126	35		
				Chloromethane	74-87-3	1.00	0.200	ug/L	46	148	35	33	146	35		
				cis-1,2-Dichloroethene	156-59-2	1.00	0.160	ug/L	75	124	35	68	121	35		
				cis-1,3-Dichloropropene	10061-01-5	1.00	0.610	ug/L	68	128	35	56	121	35		
				Cyclohexane	110-82-7	1.00	0.240	ug/L	69	133	35	48	136	35		
				Chlorodibromomethane	124-48-1	1.00	0.390	ug/L	75	120	35	66	120	35		
				Dichlorodifluoromethane	75-71-8	1.00	0.350	ug/L	35	137	35	27	132	35		
				Ethylbenzene	100-41-4	1.00	0.110	ug/L	80	120	35	66	122	35		
				Isopropylbenzene	98-82-8	1.00	0.0900	ug/L	73	123	35	56	122	20		
				Methyl acetate	79-20-9	10.0	1.72	ug/L	47	140	35	41	137	16		
				Methyl tert-butyl ether	1634-04-4	1.00	0.0700	ug/L	57	127	35	50	126	17		
				Methylcyclohexane	108-87-2	1.00	0.330	ug/L	64	123	35	40	125	35		
				Methylene Chloride	75-09-2	5.00	2.62	ug/L	63	134	35	59	127	32		
				Styrene	100-42-5	1.00	0.100	ug/L	75	121	35	57	126	35		
				Tetrachloroethene	127-18-4	1.00	0.150	ug/L	70	125	35	52	129	35		
				Toluene	108-88-3	1.00	0.140	ug/L	79	122	35	68	124	35		
				trans-1,2-Dichloroethene	156-60-5	1.00	0.190	ug/L	74	130	35	69	126	35		
				trans-1,3-Dichloropropene	10061-02-6	1.00	0.670	ug/L	64	120	35	53	120	35		
				Trichloroethene	79-01-6	1.00	0.100	ug/L	71	121	35	56	124	35		
				Trichlorofluoromethane	75-69-4	1.00	0.450	ug/L	52	148	35	45	152	35		
				Vinyl chloride	75-01-4	1.00	0.200	ug/L	61	134	35	49	136	35		
				Xylenes, Total	1330-20-7	2.00	0.150	ug/L	78	122	35	64	124	35		
				m-Xylene & p-Xylene	179601-23-1	2.00	0.0800	ug/L	79	121	35	65	123	35		
				o-Xylene	95-47-6	1.00	0.0900	ug/L	75	123	35	63	124	35		
				Toluene-d8 (Surr)	2037-26-5	1.00		ug/L			35				69	122
				Dibromofluoromethane (Surr)	1868-53-7	1.00		ug/L			35				78	129
				4-Bromofluorobenzene (Surr)	460-00-4	1.00		ug/L			35				47	134
				1,2-Dichloroethane-d4 (Surr)	17060-07-0	1.00		ug/L			35				75	130
Waters	Semivolatile Organic Compounds (GC/MS)	8270D	3510C_LVI_Acid	1,1'-Biphenyl	92-52-4	1.00	0.492	ug/L	61	120	35	36	120	35		
				bis (2-chloroisopropyl) ether	108-60-1	1.00	0.551	ug/L	43	120	35	46	120	31		
				2,4,5-Trichlorophenol	95-95-4	5.00	1.99	ug/L	60	120	35	52	120	25		
				2,4,6-Trichlorophenol	88-06-2	5.00	1.80	ug/L	63	120	35	58	120	24		
				2,4-Dichlorophenol	120-83-2	2.00	0.262	ug/L	61	120	35	59	120	27		
				2,4-Dimethylphenol	105-67-9	2.00	0.518	ug/L	55	120	35	46	120	35		
				2,4-Dinitrophenol	51-28-5	10.0	6.21	ug/L	13	120	35	18	120	31		
				2,4-Dinitrotoluene	121-14-2	5.00	2.07	ug/L	65	120	35	64	120	24		
				2,6-Dinitrotoluene	606-20-2	5.00	2.13	ug/L	67	120	35	62	120	25		
				2-Chloronaphthalene	91-58-7	1.00	0.483	ug/L	60	120	35	42	120	29		
				2-Chlorophenol	95-57-8	1.00	0.273	ug/L	59	120	35	62	120	31		
				2-Methylnaphthalene	91-57-6	0.200	0.111	ug/L	58	120	35	38	120	33		
				2-Methylphenol	95-48-7	1.00	0.209	ug/L	59	120	35	51	120	35		
				2-Nitroaniline	88-74-4	2.00	0.510	ug/L	63	120	35	57	120	24		
				2-Nitrophenol	88-75-5	2.00	0.564	ug/L	60	120	35	57	120	29		
				3,3'-Dichlorobenzidine	91-94-1	5.00	1.15	ug/L	57	120	35	16	120	30		
				3-Nitroaniline	99-09-2	2.00	0.566	ug/L	60	125	35	50	120	26		
				4,6-Dinitro-2-methylphenol	534-52-1	5.00	2.82	ug/L	48	120	35	47	120	26		
				4-Bromophenyl phenyl ether	101-55-3	2.00	0.499	ug/L	59	120	35	49	120	28		
				4-Chloro-3-methylphenol	59-50-7	2.00	0.296	ug/L	61	120	35	53	120	31		
				4-Chloroaniline	106-47-8	2.00	0.316	ug/L	10	120	35	10	120	35		
				4-Chlorophenyl phenyl ether	7005-72-3	2.00	0.551	ug/L	60	120	35	46	120	28		
				4-Nitroaniline	100-01-6	2.00	0.917	ug/L	59	150	35	41	154	29		
				4-Nitrophenol	100-02-7	10.0	2.17	ug/L	23	120	35	34	120	30		
				Acenaphthene	83-32-9	0.200	0.172	ug/L	60	120	35	38	120	25		
				Acenaphthylene	208-96-8	0.200	0.125	ug/L	62	120	35	48	120	25		
				Acetophenone	98-86-2	1.00	0.366	ug/L	60	120	35	57	120	33		
				Anthracene	120-12-7	0.200	0.135	ug/L	60	120	35	55	120	24		
				Atrazine	1912-24-9	2.00	0.952	ug/L	48	129	35	51	120	33		

Table 2

Laboratory Reporting Limits and Method Detection Limits - Groundwater
Former Cities Refinery
East Chicago, Indiana

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
				Benzaldehyde	100-52-7	2.00	0.759	ug/L	26	198	35	19	199	35		
				Benzo[a]anthracene	56-55-3	0.200	0.171	ug/L	62	120	35	58	120	23		
				Benzo[a]pyrene	50-32-8	0.200	0.173	ug/L	61	120	35	60	120	25		
				Benzo[b]fluoranthene	205-99-2	0.200	0.154	ug/L	64	120	35	62	120	26		
				Benzo[g,h,i]perylene	191-24-2	0.200	0.178	ug/L	63	120	35	58	120	26		
				Benzo[k]fluoranthene	207-08-9	0.200	0.140	ug/L	63	120	35	63	120	26		
				Bis(2-chloroethoxy)methane	111-91-1	1.00	0.455	ug/L	58	120	35	56	120	29		
				Bis(2-chloroethyl)ether	111-44-4	1.00	0.402	ug/L	52	120	35	50	120	29		
				Bis(2-ethylhexyl) phthalate	117-81-7	5.00	2.22	ug/L	63	120	35	59	120	31		
				Butyl benzyl phthalate	85-68-7	2.00	0.666	ug/L	63	120	35	59	120	26		
				Caprolactam	105-60-2	5.00	0.934	ug/L	10	120	35	10	120	35		
				Carbazole	86-74-8	1.00	0.490	ug/L	63	120	35	56	120	25		
				Chrysene	218-01-9	0.200	0.186	ug/L	61	120	35	59	120	25		
				Dibenz(a,h)anthracene	53-70-3	0.200	0.151	ug/L	62	120	35	58	120	25		
				Dibenzofuran	132-64-9	1.00	0.561	ug/L	60	120	35	48	120	25		
				Diethyl phthalate	84-66-2	5.00	3.82	ug/L	62	120	35	53	120	27		
				Dimethyl phthalate	131-11-3	2.00	0.515	ug/L	60	120	35	61	120	24		
				Di-n-butyl phthalate	84-74-2	5.00	1.80	ug/L	64	120	35	61	120	25		
				Di-n-octyl phthalate	117-84-0	2.00	0.821	ug/L	62	120	35	59	120	26		
				Fluoranthene	206-44-0	0.200	0.160	ug/L	62	120	35	58	120	26		
				Fluorene	86-73-7	0.200	0.169	ug/L	61	120	35	49	120	26		
				Hexachlorobenzene	118-74-1	0.200	0.161	ug/L	54	120	35	47	120	26		
				Hexachlorobutadiene	87-68-3	1.00	0.543	ug/L	45	120	35	15	120	35		
				Hexachlorocyclopentadiene	77-47-4	10.0	1.76	ug/L	38	120	35	10	120	35		
				Hexachloroethane	67-72-1	1.00	0.395	ug/L	48	120	35	25	120	35		
				Indeno[1,2,3-cd]pyrene	193-39-5	0.200	0.135	ug/L	64	120	35	59	120	26		
				Isophorone	78-59-1	1.00	0.324	ug/L	59	120	35	57	120	28		
				N-Nitrosodi-n-propylamine	621-64-7	1.00	0.253	ug/L	59	120	35	56	120	31		
				N-Nitrosodiphenylamine	86-30-6	1.00	0.440	ug/L	60	120	35	50	120	25		
				Naphthalene	91-20-3	0.200	0.109	ug/L	57	120	35	39	120	31		
				Nitrobenzene	98-95-3	1.00	0.514	ug/L	59	120	35	57	120	28		
				Pentachlorophenol	87-86-5	10.0	3.10	ug/L	20	120	35	14	120	35		
				Phenanthrene	85-01-8	0.200	0.167	ug/L	59	120	35	51	120	24		
				Phenol	108-95-2	1.00	0.128	ug/L	27	120	35	26	120	35		
				Pyrene	129-00-0	0.200	0.175	ug/L	61	120	35	58	120	24		
				3 & 4 Methylphenol	15831-10-4	2.00	0.191	ug/L	53	120	35	51	120	35		
				Terphenyl-d14 (Surr)	1718-51-0	2.00		ug/L			35				53	120
				Phenol-d5 (Surr)	4165-62-2	2.00		ug/L			35				23	120
				Nitrobenzene-d5 (Surr)	4165-60-0	2.00		ug/L			35				46	120
				2-Fluorophenol (Surr)	367-12-4	2.00		ug/L			35				27	123
				2-Fluorobiphenyl (Surr)	321-60-8	2.00		ug/L			35				46	120
				2,4,6-Tribromophenol (Surr)	118-79-6	2.00		ug/L			35				33	120
Waters	Metals (ICP)	6010C	3005A	Aluminum	7429-90-5	200	47.3	ug/L	80	120	20	75	125	20		
				Silver	7440-22-4	10.0	0.623	ug/L	80	120	20	75	125	20		
				Barium	7440-39-3	200	1.33	ug/L	80	120	20	75	125	20		
				Beryllium	7440-41-7	5.00	0.601	ug/L	80	120	20	75	125	20		
				Calcium	7440-70-2	5000	307	ug/L	80	120	20	75	125	20		
				Cadmium	7440-43-9	5.00	0.203	ug/L	80	120	20	75	125	20		
				Cobalt	7440-48-4	10.0	0.752	ug/L	80	120	20	75	125	20		
				Chromium	7440-47-3	10.0	0.625	ug/L	80	120	20	75	125	20		
				Copper	7440-50-8	25.0	3.55	ug/L	80	120	20	75	125	20		
				Iron	7439-89-6	200	26.0	ug/L	80	120	20	75	125	20		
				Potassium	7440-09-7	5000	557	ug/L	80	120	20	75	125	20		
				Magnesium	7439-95-4	5000	259	ug/L	80	120	20	75	125	20		
				Manganese	7439-96-5	15.0	2.12	ug/L	80	120	20	75	125	20		
				Sodium	7440-23-5	5000	560	ug/L	80	120	20	75	125	20		
				Nickel	7440-02-0	40.0	2.20	ug/L	80	120	20	75	125	20		
				Antimony	7440-36-0	20.0	7.46	ug/L	80	120	20	75	125	20		
				Vanadium	7440-62-2	50.0	5.56	ug/L	80	120	20	75	125	20		
				Zinc	7440-66-6	50.0	9.67	ug/L	80	120	20	75	125	20		
				Arsenic	7440-38-2	15.0	4.05	ug/L	80	120	20	75	125	20		
				Lead	7439-92-1	10.0	2.77	ug/L	80	120	20	75	125	20		
				Selenium	7782-49-2	20.0	5.96	ug/L	80	120	20	75	125	20		
				Thallium	7440-28-0	20.0	2.68	ug/L	80	120	20	75	125	20		
Waters	Mercury (CVAA)	7470A	7470A_Prep	Mercury	7439-97-6	0.200	0.130	ug/L	80	120	20	80	120	20		
Waters	EDB, DBCP, and 1,2,3-TCP (GC)	8011	8011_Prep	Ethylene Dibromide	106-93-4	0.0200	0.00600	ug/L	60	140	20	60	140	20		
				1,2-Dibromo-3-Chloropropane	96-12-8	0.0200	0.00800	ug/L	60	140	20	60	140	20		
				1,1,1,2-Tetrachloroethane	630-20-6			ug/L							60	140

Table 3

Laboratory Reporting Limits and Method Detection Limits - Waste
Former Cities Refinery
East Chicago, Indiana

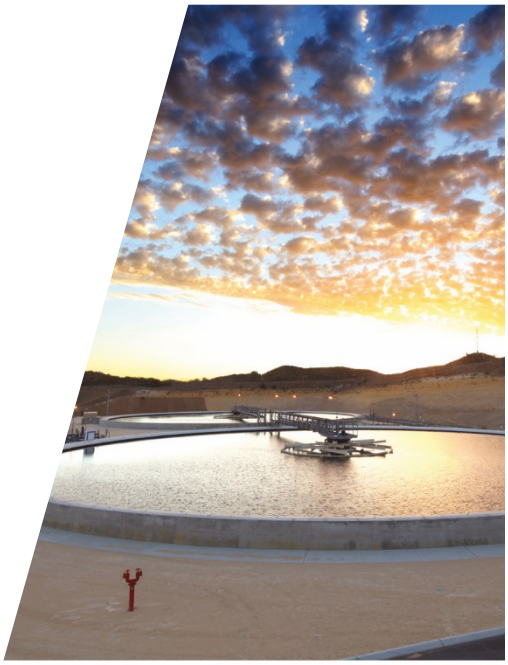
Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Wastes	Volatile Organic Compounds by GC/MS	8260C	5030C_H	1,1,1-Trichloroethane	71-55-6	1000	21.0	ug/Kg	60	126	40	27	131	40		
				1,1,2,2-Tetrachloroethane	79-34-5	1000	8.90	ug/Kg	61	134	40	10	168	40		
				1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2000	39.0	ug/Kg	58	144	40	30	145	40		
				1,1,2-Trichloroethane	79-00-5	1000	12.0	ug/Kg	78	120	40	17	152	40		
				1,1-Dichloroethane	75-34-3	1000	17.0	ug/Kg	69	120	40	35	129	40		
				1,1-Dichloroethene	75-35-4	1000	18.0	ug/Kg	48	140	40	20	150	40		
				1,2,4-Trichlorobenzene	120-82-1	1000	7.30	ug/Kg	56	120	40	10	120	40		
				1,2-Dibromo-3-Chloropropane	96-12-8	1000	50.0	ug/Kg	35	137	40	10	135	40		
				Ethylene Dibromide	106-93-4	1000	10.0	ug/Kg	73	126	40	24	138	40		
				1,2-Dichlorobenzene	95-50-1	1000	8.60	ug/Kg	74	120	40	10	131	40		
				1,2-Dichloroethane	107-06-2	1000	10.0	ug/Kg	66	120	40	33	130	40		
				1,2-Dichloropropane	78-87-5	1000	8.20	ug/Kg	77	120	40	33	134	40		
				1,3-Dichlorobenzene	541-73-1	1000	4.80	ug/Kg	74	120	40	10	131	40		
				1,4-Dichlorobenzene	106-46-7	1000	8.00	ug/Kg	74	120	40	10	129	40		
				2-Butanone (MEK)	78-93-3	4000	43.0	ug/Kg	61	131	40	31	148	40		
				2-Hexanone	591-78-6	4000	20.0	ug/Kg	54	135	40	23	149	40		
				4-Methyl-2-pentanone (MIBK)	108-10-1	4000	48.0	ug/Kg	56	124	40	29	140	40		
				Acetone	67-64-1	4000	170	ug/Kg	47	157	40	18	167	40		
				Benzene	71-43-2	1000	12.0	ug/Kg	75	120	40	32	131	40		
				Dichlorobromomethane	75-27-4	1000	9.90	ug/Kg	63	121	40	18	125	40		
				Bromoform	75-25-2	1000	19.0	ug/Kg	44	131	40	10	122	40		
				Bromomethane	74-83-9	2000	29.0	ug/Kg	10	158	40	10	149	40		
				Carbon disulfide	75-15-0	1000	12.0	ug/Kg	33	144	40	10	134	40		
				Carbon tetrachloride	56-23-5	1000	6.40	ug/Kg	54	130	40	13	131	40		
				Chlorobenzene	108-90-7	1000	6.40	ug/Kg	79	120	40	16	129	40		
				Chloroethane	75-00-3	2000	61.0	ug/Kg	10	159	40	10	155	40		
				Chloroform	67-66-3	1000	8.80	ug/Kg	74	120	40	38	129	40		
				Chloromethane	74-87-3	2000	14.0	ug/Kg	40	127	40	20	140	40		
				cis-1,2-Dichloroethene	156-59-2	1000	6.90	ug/Kg	76	120	40	35	130	40		
				cis-1,3-Dichloropropene	10061-01-5	1000	7.90	ug/Kg	62	124	40	12	131	40		
				Cyclohexane	110-82-7	2000	40.0	ug/Kg	57	126	40	17	133	40		
				Chlorodibromomethane	124-48-1	1000	12.0	ug/Kg	60	121	40	15	125	40		
				Dichlorodifluoromethane	75-71-8	2000	16.0	ug/Kg	18	137	40	10	141	40		
				Ethylbenzene	100-41-4	1000	5.40	ug/Kg	75	120	40	12	133	40		
				Isopropylbenzene	98-82-8	1000	6.50	ug/Kg	74	120	40	10	135	40		
				Methyl acetate	79-20-9	5000	25.0	ug/Kg	63	120	40	20	155	40		
				Methyl tert-butyl ether	1634-04-4	4000	7.10	ug/Kg	66	120	40	42	127	40		
				Methylcyclohexane	108-87-2	1000	12.0	ug/Kg	62	124	40	10	133	40		
				Methylene Chloride	75-09-2	1000	77.0	ug/Kg	48	142	40	22	153	40		
				Styrene	100-42-5	1000	5.60	ug/Kg	70	120	40	10	127	40		
				Tetrachloroethene	127-18-4	1000	12.0	ug/Kg	75	124	40	13	144	40		
				Toluene	108-88-3	1000	17.0	ug/Kg	76	120	40	20	141	40		
				trans-1,2-Dichloroethene	156-60-5	1000	9.20	ug/Kg	74	125	40	31	138	40		
				trans-1,3-Dichloropropene	10061-02-6	1000	20.0	ug/Kg	58	120	40	10	123	40		
				Trichloroethene	79-01-6	1000	9.70	ug/Kg	75	123	40	10	162	40		
				Trichlorofluoromethane	75-69-4	2000	16.0	ug/Kg	33	152	40	16	148	40		
				Vinyl chloride	75-01-4	2000	18.0	ug/Kg	39	140	40	15	150	40		
				Xylenes, Total	1330-20-7	2000	6.20	ug/Kg	77	120	40	10	134	40		
				m-Xylene & p-Xylene	179601-23-1	2000	6.20	ug/Kg	76	120	40	10	132	40		
				o-Xylene	95-47-6	1000	8.50	ug/Kg	76	120	40	11	134	40		
				Toluene-d8 (Surr)	2037-26-5			ug/Kg			40				64	124
				Dibromofluoromethane (Surr)	1868-53-7			ug/Kg			40				56	122
				4-Bromofluorobenzene (Surr)	460-00-4			ug/Kg			40				51	127
				1,2-Dichloroethane-d4 (Surr)	17060-07-0			ug/Kg			40				59	120
Wastes	Semivolatile Organic Compounds (GC/MS)	8270D	3580A	1,1'-Biphenyl	92-52-4	20000	432	ug/Kg	43	120	40	38	120	32		
				bis (2-chloroisopropyl) ether	108-60-1	20000	312	ug/Kg	29	120	40	27	120	40		
				2,4,5-Trichlorophenol	95-95-4	20000	294	ug/Kg	28	120	40	22	120	40		
				2,4,6-Trichlorophenol	88-06-2	20000	408	ug/Kg	14	120	40	15	120	34		
				2,4-Dichlorophenol	120-83-2	20000	318	ug/Kg	40	120	40	30	120	40		
				2,4-Dimethylphenol	105-67-9	20000	408	ug/Kg	31	120	40	25	120	36		
				2,4-Dinitrophenol	51-28-5	96000	2460	ug/Kg	10	120	40	10	120	40		
				2,4-Dinitrotoluene	121-14-2	20000	342	ug/Kg	49	120	40	51	120	21		
				2,6-Dinitrotoluene	606-20-2	20000	348	ug/Kg	49	120	40	51	120	20		
				2-Chloronaphthalene	91-58-7	20000	378	ug/Kg	42	120	40	39	120	32		
				2-Chlorophenol	95-57-8	20000	216	ug/Kg	42	120	40	30	120	40		
				2-Methylnaphthalene	91-57-6	20000	59.4	ug/Kg	42	120	40	10	133	40		
				2-Methylphenol	95-48-7	20000	402	ug/Kg	42	120	40	28	120	40		
				2-Nitroaniline	88-74-4	96000	306	ug/Kg	44	120	40	49	120	19		
				2-Nitrophenol	88-75-5	20000	204	ug/Kg	41	120	40	25	120	40		
				3,3'-Dichlorobenzidine	91-94-1	96000	294	ug/Kg	29	120	40	10	120	40		
				3-Nitroaniline	99-09-2	96000	192	ug/Kg	41	120	40	20	120	40		
				4,6-Dinitro-2-methylphenol	534-52-1	96000	2820	ug/Kg	27	120	40	10	123	40		
				4-Bromophenyl phenyl ether	101-55-3	20000	282	ug/Kg	47	120	40	47	120	20		
				1,4-Dioxane	123-91-1	20000	780	ug/Kg	10	120	40	10	120	40		
				4-Chloro-3-methylphenol	59-50-7	20000	3060	ug/Kg	39	120	40	33	120	40		
				4-Chloroaniline	106-47-8	20000	312	ug/Kg	30	120	40	21	120	40		
				4-Chlorophenyl phenyl ether	7005-72-3	20000	216	ug/Kg	46	120	40	47	120	21		
				4-Nitroaniline	100-01-6	96000	216	ug/Kg	47	120	40	20	120	40		
				4-Nitrophenol	100-02-7	96000	4860	ug/Kg	29	120	40	14	125	36		
				Acenaphthene	83-32-9	20000	55.2	ug/Kg	45	120	40	41	120	34		
				Acenaphthylene	208-96-8	20000	84.0	ug/Kg	45	120	40	39	120	34		
				Acetophenone	98-86-2	20000	330	ug/Kg	42	120	40	32	120	40		
				Anthracene	120-12-7	20000	138	ug/Kg	52	120	40	43	106	32		
				Atrazine	1912-24-9	20000	660	ug/Kg	54	120	40	50	120	22		
				Benzaldehyde	100-52-7	20000	414	ug/Kg	38	120	40	18	120	40		

Table 3

Laboratory Reporting Limits and Method Detection Limits - Waste
Former Cities Refinery
East Chicago, Indiana

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
				Benzo[a]anthracene	56-55-3	20000	84.0	ug/Kg	52	120	40	32	120	37		
				Benzo[a]pyrene	50-32-8	20000	138	ug/Kg	50	120	40	35	120	38		
				Benzo[b]fluoranthene	205-99-2	20000	138	ug/Kg	52	120	40	27	126	40		
				Benzo[g,h,i]perylene	191-24-2	20000	108	ug/Kg	54	120	40	29	122	40		
				Benzo[k]fluoranthene	207-08-9	20000	138	ug/Kg	54	120	40	39	120	37		
				Bis(2-chloroethoxy)methane	111-91-1	20000	960	ug/Kg	43	120	40	36	120	39		
				Bis(2-chloroethyl)ether	111-44-4	20000	246	ug/Kg	41	120	40	32	120	40		
				Bis(2-ethylhexyl) phthalate	117-81-7	20000	960	ug/Kg	47	124	40	42	129	40		
				Butyl benzyl phthalate	85-68-7	20000	222	ug/Kg	47	120	40	39	121	24		
				Caprolactam	105-60-2	20000	468	ug/Kg	55	120	40	44	120	28		
				Carbazole	86-74-8	20000	558	ug/Kg	51	120	40	46	120	28		
				Chrysene	218-01-9	20000	59.4	ug/Kg	53	120	40	31	121	37		
				Dibenz(a,h)anthracene	53-70-3	20000	90.0	ug/Kg	50	120	40	36	120	38		
				Dibenzofuran	132-64-9	20000	49.8	ug/Kg	46	120	40	45	120	26		
				Diethyl phthalate	84-66-2	20000	372	ug/Kg	45	120	40	45	120	19		
				Dimethyl phthalate	131-11-3	20000	384	ug/Kg	47	120	40	47	120	20		
				Di-n-butyl phthalate	84-74-2	20000	300	ug/Kg	50	120	40	46	120	21		
				Di-n-octyl phthalate	117-84-0	20000	660	ug/Kg	38	122	40	44	120	25		
				Fluoranthene	206-44-0	20000	55.8	ug/Kg	54	120	40	30	125	31		
				Fluorene	86-73-7	20000	78.0	ug/Kg	48	120	40	44	120	32		
				Hexachlorobenzene	118-74-1	20000	84.0	ug/Kg	45	120	40	47	120	23		
				Hexachlorobutadiene	87-68-3	20000	150	ug/Kg	34	120	40	27	120	40		
				Hexachlorocyclopentadiene	77-47-4	96000	174	ug/Kg	10	120	40	10	120	40		
				Hexachloroethane	67-72-1	20000	294	ug/Kg	36	120	40	10	120	40		
				Indeno[1,2,3-cd]pyrene	193-39-5	20000	120	ug/Kg	52	120	40	34	120	40		
				Isophorone	78-59-1	20000	210	ug/Kg	42	120	40	32	120	40		
				N-Nitrosodi-n-propylamine	621-64-7	20000	456	ug/Kg	39	120	40	30	120	40		
				N-Nitrosodiphenylamine	86-30-6	20000	246	ug/Kg	50	120	40	46	120	24		
				Naphthalene	91-20-3	20000	53.4	ug/Kg	39	120	40	30	120	40		
				Nitrobenzene	98-95-3	20000	384	ug/Kg	42	120	40	32	120	40		
				Pentachlorophenol	87-86-5	20000	2700	ug/Kg	16	120	40	10	120	40		
				Phenanthrene	85-01-8	20000	66.0	ug/Kg	50	120	40	31	120	35		
				Phenol	108-95-2	20000	342	ug/Kg	39	120	40	25	120	40		
				Pyrene	129-00-0	20000	60.0	ug/Kg	50	120	40	28	122	30		
				3 & 4 Methylphenol	15831-10-4	40000	1200	ug/Kg	43	120	40	34	120	40		
				Terphenyl-d14 (Surr)	1718-51-0	20000	0.0100	ug/Kg			40				39	120
				Phenol-d5 (Surr)	4165-62-2	20000	0.0100	ug/Kg			40				28	120
				Nitrobenzene-d5 (Surr)	4165-60-0	20000	0.0100	ug/Kg			40				28	120
				2-Fluorophenol (Surr)	367-12-4	20000	0.0100	ug/Kg			40				26	120
				2-Fluorobiphenyl (Surr)	321-60-8	20000	0.0100	ug/Kg			40				35	120
				2,4,6-Tribromophenol (Surr)	118-79-6	20000	0.0100	ug/Kg			40				10	120
Wastes	Mercury (CVAA)	7471A	7471A_Prep	Mercury	7439-97-6	0.100	0.0180	mg/Kg	80	120	20	80	120	20		
Wastes	Metals (ICP)	6010C	3050B	Aluminum	7429-90-5	20.0	5.33	mg/Kg	80	120	20	75	125	20		
				Silver	7440-22-4	1.00	0.0810	mg/Kg	80	120	20	75	125	20		
				Barium	7440-39-3	20.0	0.362	mg/Kg	80	120	20	75	125	20		
				Beryllium	7440-41-7	0.500	0.0540	mg/Kg	80	120	20	75	125	20		
				Calcium	7440-70-2	500	36.5	mg/Kg	80	120	20	75	125	20		
				Cadmium	7440-43-9	0.500	0.0480	mg/Kg	80	120	20	75	125	20		
				Cobalt	7440-48-4	1.00	0.200	mg/Kg	80	120	20	75	125	20		
				Chromium	7440-47-3	1.00	0.151	mg/Kg	80	120	20	75	125	20		
				Copper	7440-50-8	2.50	0.236	mg/Kg	80	120	20	75	125	20		
				Iron	7439-89-6	20.0	6.94	mg/Kg	80	120	20	75	125	20		
				Potassium	7440-09-7	500	36.1	mg/Kg	80	120	20	75	125	20		
				Magnesium	7439-95-4	500	46.1	mg/Kg	80	120	20	75	125	20		
				Manganese	7439-96-5	1.50	0.309	mg/Kg	80	120	20	75	125	20		
				Sodium	7440-23-5	500	62.8	mg/Kg	80	120	20	75	125	20		
				Nickel	7440-02-0	4.00	0.233	mg/Kg	80	120	20	75	125	20		
				Antimony	7440-36-0	2.00	0.359	mg/Kg	80	120	20	75	125	20		
				Vanadium	7440-62-2	5.00	0.822	mg/Kg	80	120	20	75	125	20		
				Zinc	7440-66-6	5.00	1.37	mg/Kg	80	120	20	75	125	20		
				Arsenic	7440-38-2	1.50	0.316	mg/Kg	80	120	20	75	125	20		
				Lead	7439-92-1	1.00	0.282	mg/Kg	80	120	20	75	125	20		
				Selenium	7782-49-2	2.00	0.469	mg/Kg	80	120	20	75	125	20		
				Thallium	7440-28-0	2.00	0.399	mg/Kg	80	120	20	75	125	20		

Appendix 3
GHD Laboratory SOP



Analytical Data Quality Assessment and Validation Standard Operating Procedure (SOP)

Proprietary Document

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Acronyms

A	Acid Fraction of SVOC
B/N	Base/Neutral Fractions of SVOC
CCAL	Continuing Calibration Standard
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain of Custody Form
CRDL	Contract Required Detection Limit
CVAA	Cold Vapor Atomic Absorption
DL	Dilution
%D	Percent Difference
DQO	Data Quality Objectives
DUP	Duplicate
EDD	Electronic Data Deliverables
EQL	Estimated Quantitation Limit
FDV	Full Data Validation
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HT	Holding Time
ICAL	Initial Calibration Standard
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IPC	Instrument Performance Check
IS	Internal Standard
LCS	Laboratory Control Spike
LCSD	Laboratory Control Spike Duplicate
MD	Matrix Duplicate
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
ND	Non-detect
NFG	National Functional Guidelines
PCB	Polychlorinated Biphenyls
QA	Quality Assurance
QC	Quality Control
QAPP	Quality Assurance Project Plan
QL	Quantitation Limit-defined as the sample-specific MDL where all individual sample factors are applied, i.e. dilution factors, sample weights and sample volumes
RDV	Reduced Data Validation
REG 5	USEPA Region 5
RF	Response Factor
RRF	Relative Response Factor
RL	Report Limit
RPD	Relative Percent Difference
%RSD	Percent Relative Standard Deviation
SAP	Sampling and Analysis Plan
R	Correlation Coefficient
R^2	Coefficient of Determination
SOP	Standard Operating Procedures
SVOC	Semi-Volatile Organic Compounds

TAL	Target Analyte List
TCL	Target Compound List
VOC	Volatile Organic Compounds
USEPA	United States Environmental Protection Agency

Definitions

Accuracy	A measure of overall agreement of a measurement to a known value. Accuracy is assessed by means of percent recoveries and reference samples.
Assessment	The evaluation process used to measure the performance or effectiveness of a system and its elements.
Action Level	The concentration level that is high enough to warrant action. Action levels are generally regulatory levels that are determined by a regulatory agency.
Analyte	The element or compound to be determined.
Blank	A purified sample matrix subjected to the usual analytical or measurement process. A blank should not contain analytes of interest. A blank is used to detect contamination during sampling, handling, preparation, and/or analysis.
Calibration Curve	The relationship between instrument response and analyte concentration. The "curve" may be linear or non-linear.
Calibration Factor	The response factor for external standard analyses expressed as peak area (or height) per nanogram of analyte injected.
Calibration Range	The concentration range bounded by the lowest and highest concentration calibration standards used in the equation for the calibration curve.
Continuing Calibration	The daily standard when it is used to update the response factors for sample quantitation.
Chain of Custody	An unbroken trail of accountability that ensures the physical security of samples, data, and records.
Comparability	A measure of the confidence with which one data set or method can be compared to another.
Completeness	A measure of the amount of valid data obtained from a measurement system.
Compliance Review	Compliance review is the assessment of data package completeness and compliance with project requirements as outlined in the project Analytical Scope of Work.
Daily Standard	A single calibration standard, normally at a concentration near the middle of the calibration range that is analysed at the beginning of each analysis shift.
Data Quality	A measure of the degree of acceptability or utility of data for a particular purpose.
Data Validation	Data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set.
Data Validation-Reduced	Reduced Data Validation (RDV) is assessment of data and quality control deliverables provided in a standard summary laboratory report. Qualification of sample results is based on sample holding time periods, sample preservation, laboratory batch quality control sample results (method blank, laboratory control samples, matrix spike/matrix spike duplicate or matrix spike/matrix duplicate, and surrogates) and field QC results in accordance with U.S. USEPA's National Functional Guidelines, the analytical methods, and project quality assurance documents.
Data Validation-Full	Full Data Validation (FDV) is assessment of data and quality control deliverables provided in an expanded laboratory report. Qualification of sample results is based on sample holding time periods, sample preservation, laboratory batch quality control sample results (method blank, laboratory control samples, matrix spike/matrix spike duplicate or matrix spike/matrix duplicate, and surrogates), field QC results, instrument calibration data, analyte identification and quantitation, additional method-specific quality control results (as appropriate), and raw data in accordance with U.S. USEPA's National Functional Guidelines, the analytical methods, and project quality assurance documents. FDV consists of reviewing and assessing all sample results and quality control data reported in summary tables and a 10 percent spot check of laboratory calculations and analyte identification from the raw data.

Data Validation-10/90	Validation level that consists of 10% full validation and 90% innovative validation.
Field Duplicate	A second aliquot of a sample collected in the field that is treated the same as the original in order to determine the precision of the sampling and analysis.
Field Blank (Trip, Rinsate)	A blank that measures contamination possibly introduced in the field or during transportation. It also includes any contamination introduced during sample handling, preparation and/or analysis.
Initial Calibration	The establishment of an analytical curve base on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentrations of acids as used in the sample preparation.
Initial Calibration Verification	The daily standard when it is used to verify that the calibration curve is still valid for sample quantitation (see also continuing calibration standard).
Inorganics	Inorganics as specified within this document are metals and cyanide.
Instrument Detection Limit	The minimum concentration of a target analyte, determined by various means, which can be measured above the instrument background noise.
Internal Standards	A non-target analyte added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance and are used in some methods for the quantitation of target compounds.
Laboratory Control Sample	An aliquot of a clean matrix, spiked with known quantities of selected analytes.
Laboratory Duplicate	Two separate aliquots of a single (routine) sample analysed as separate individual samples in order to determine the precision of the method.
Matrix	The predominant material of which the sample is composed. Matrix is not a synonymous phase.
Matrix Duplicate	A second aliquot of a sample that is treated the same as the original in order to determine the precision of the method.
Matrix Spike/Spike Duplicate	An aliquot of a sample spiked in duplicate with known quantities of selected analytes.
Method	A body of procedures and techniques for performing an activity, systematically presented in the order in which they are to be executed.
Method Blank	A blank prepared to represent the sample matrix as closely as possible and analyzed exactly like the calibration standards, samples, and QC samples. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the analytical procedure.
Method Detection Limit	The statistically derived lowest level of an analyte in a sample that will result in a signal different than zero as specified in 40 CFR 136, Appendix B.
Organics	Organics as specified within this document are volatile organic compounds, semi-volatile organic compounds, pesticides, and polychlorinated biphenyls.
Parent Sample	The sample from which an aliquot was taken by the laboratory to prepare a matrix spike or analyzed as a matrix duplicate. This also refers to the investigative sample and field duplicate relationship.
Percent (%) Difference	A parameter used to compare the daily standard to the initial calibration when a calibration or response factor is used, comparing the difference of the factors. It indicates both the direction and the magnitude of the comparison.
Percent (%) Drift	A parameter used to compare the daily standard to the initial calibration when a linear calibration curve is used, comparing the % difference of the determined concentrations. It indicates both the direction and the magnitude of the comparison.
Precision	A measure of agreement among repeated measurements of the same property under identical, of substantially similar, conditions. Precision is assessed by means of duplicate/replicate sample analysis.
Quality Assurance	A system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Control	A set of measures and activities that are used to fulfil the need for quality.
QC Sample	An uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra- laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
Quantitation	The degree to which the instrument measures the concentration of target analytes.

Quantitation Limit	The minimum concentration that a target analyte can be measured within specified limits of precision and accuracy. Quantitation limits are generally 5-10x the method detection limit.
Re-Analysis	The process of repeating a sample that includes both a new sample preparation and analysis.
Recovery	The act of determining whether or not the methodology measures all of the analyte contained in a sample.
Re-Extraction	The repeated extraction of a sample to fulfil a re-analysis.
Re-Injection	The process of repeating a sample that does not include a new sample preparation. It is a re-injection of an existing extract.
Relative Percent (%) Difference	Difference (between two results) divided by the Average (of the same tow results), multiplied by 100=RPD.
Reported Result	The concentration or a target analyte in a given sample that is reported by the laboratory on the analysis results form. A positive result that is below the report limit may or may not be a reported result depending on the client's request.
Report Limit	The detection limit or quantitation limit (depending on the client's request) for a target analyte in a given sample that is reported by the laboratory on the analysis results form. The value includes adjustment for any sample dilution or method factor.
Representativeness	The measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.
Sample	An environmental sample that is not a QC sample and any re-analysis or re-injection thereof.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest.
Spike	A substance that is added to an environmental sample to increase the concentration of the target analyte by a known amount. A laboratory control spike or matrix spike is used to measure accuracy. A spike duplicate is used to measure precision.
Split Samples	Two or more representative portions taken from one sample in the field or in the laboratory and analyzed by different analysts or laboratories. Split samples are quality control samples that are used to assess analytical variability and comparability.
Standard Operating Procedure	A document that details the method for an operation, analysis, or action which thoroughly describes the techniques and steps to be followed. It is officially approved as the method for performing certain routine or repetitive tasks.
Target Analyte	An analyte specifically reported for a given analysis.

1. Introduction

The purpose of this Standard Operating Procedure (SOP) is to ensure that all analytical data quality assessment and data validation is performed by GHD Services Inc. (GHD) in a consistent manner and in accordance with project-specific requirements. The purpose of data validation is to ensure that only those data that meet project-specific data quality objectives (DQO) are used for project decision making and reporting.

This SOP discusses data validation of the parameters associated with the following analytical techniques:

- Gas Chromatography/Mass Spectrometry (GC/MS) - volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs)
- GC - polychlorinated biphenyls (PCBs), chlorinated pesticides, herbicides, total petroleum hydrocarbons, etc.
- High Performance Liquid Chromatography (HPLC) – polynuclear aromatic hydrocarbons (PAH), carbamates, explosives, etc.
- Spectrometric - including inductively coupled plasma (ICP), inductively coupled plasma/mass spectrometer (ICP)/MS), cold vapor atomic absorption (CVAA)
- Spectrophotometry (Spec) – cyanide, sulfate, phenolics and other inorganics
- Ion Chromatography (IC) – inorganic anions
- Titrimetric – chloride, etc.

The data validation procedures detailed in this SOP are based on guidance and quality control procedures established by the United States Environmental Protection Agency (USEPA) including the "National Functional Guidelines for Superfund Organic Methods Data Review", EPA-540-R-2016-002, September 2016 and the "National Functional Guidelines for Inorganic Superfund Data Review", EPA 540-R-2016-001, September 2016, collectively referred to as NFGs throughout this SOP. In addition to the federal guidance, data review follows other project specific guidance such as regional SOPs, analytical methods, and laboratory SOPs as defined in project documentation (Quality Assurance Project Plan (QAPP), Sampling and Analysis Plan (SAP), Work Plan and Purchase Orders), and professional judgment.

If actual Contract Laboratory Program (CLP) data are to be validated, then the appropriate NFGs shall be followed as written.

2. Levels of Analytical Data Review and Assessment

GHD completes several types or levels of data review and assessment including compliance assessment and data validation. Descriptions of these and the information reviewed are discussed below.

As defined by the USEPA, data verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual compliance.

Data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set.

This SOP addresses four stages of data validation; application of each is dependent on project-defined data quality objectives. The specific elements addressed for each stage of review are presented in Table 2.1.

Table 2.1 - Data Review and Validation Levels

Item Reviewed	Stage 1 Compliance Check	Stage 2A Reduced Validation	Stage 2B Innovative Validation	Stage 3 Innovative Plus Recalculations*	Stage 4 Full Validation
General Report Deliverables					
Sample ID Check (COC versus Lab Deliverables)	x	x	x	x	x
Sample Dates/Times and Sample Receipt Date/Time	x	x	x	x	x
Sample Condition Upon Receipt	x	x	x	x	x
Methods/Procedures	x	x	x	x	x
Parameter List	x	x	x	x	x
Report/Detection Limits	x	x	x	x	x
Documentation/Deliverables	x	x	x	x	x
Sample Specific and Batch QC Results		x	x	x	x
Sample Preservation and Holding Times		x	x	x	x
Method Blanks		x	x	x	x
Field Blanks (Trip and Rinsate Blanks)		x	x	x	x
System Monitoring Compounds (Surrogates)		x	x	x	x
MS/MSD-Organics		x	x	x	x
MS/MSD, MS/MD-Inorganics		x	x	x	x
Laboratory Control Sample (LCS)		x	x	x	x
Serial Dilutions			x	x	x
Post Digestion Spikes		x	x	x	x
Field Duplicates		x	x	x	x
Expanded Data Elements					
Instrument Performance Check (GC/MS & ICP/MS)			x	x	x
Initial Calibration-Organics			x	x	x
Continuing Calibration-Organics			x	x	x
Initial Calibration Verification-Inorganics			x	x	x
Continuing Calibration Verification-Inorganics			x	x	x

Item Reviewed	Stage 1 Compliance Check	Stage 2A Reduced Validation	Stage 2B Innovative Validation	Stage 3 Innovative Plus Recalculations*	Stage 4 Full Validation
Internal Standards (GC/MS & ICP/MS)			x	x	x
Instrument Blanks-Inorganics			x	x	x
ICP/MS Internal Standards			x	x	x
ICP Interference Check Samples			x	x	x
Compound Identification					x
Chromatography					x
Compound/Analyte Quantitation (raw data)					x
Report Limit Verification			x	x	x
Recalculations				x	x

Notes:

* Raw data review including calculation checks

GC Gas Chromatograph

ICP Inductively Coupled Plasma

MS Mass Spectroscopy

For all samples sets, regardless of validation level employed, a Stage 1 assessment must be completed. A flat file, xtab, and sample summary is created by the project chemist for comparison with the project Simplified Scope of Work (SSOW) for all projects, except GSH projects. The Stage 1 assessment for GSH projects is performed by the database analyst (DBA).

Reduced data validation (RDV), also referred to Stage 2a validation, entails the review of sample preservation and holding time compliance, as well as the lab and field quality control results (blank, spikes, surrogates, etc.). It does not include the assessment of raw data, instrument calibration data, internal standard recoveries, inter element interference check standards, or serial dilutions.

Full data validation (FDV), also referred to as Stage 4 validation, includes all steps of the RDV process as well as the review of raw data associated with sample and quality control results and confirmation of analyte quantitation and identification (e.g., sample calculations, mass spectra, chromatograms, etc.) on a minimum of 10 percent of the laboratory data. FDV also includes the review of raw data and results for instrument calibration, (instrument performance, initial and continuing calibration), internal standards, inter element interference check standards, and serial dilutions.

A third stage of validation is identified in the Region III document "Innovative Approaches to Data Validation, 1995". This level of validation, referred to as innovative approach or Stage 2b falls between FDV and RDV; it does not include the review of raw data, but it does include the assessment of all sample and QC results, including instrument calibration data, internal standard recoveries, inter element interference check standards, and serial dilutions.

For clients requesting FDV (Stage 4) on 10 percent of their data and a lesser validation level on the remaining data packages, the innovative approach (Stage 2b) is used on the remaining 90 percent to ensure consistent qualifier application (this approach is referred to as 10/90).

The Stage 3 approach is similar to that used for projects requiring validation of New Jersey reduced deliverables.

Be aware that project-specific requirements may include data validation levels that differ from the main types outlined above.

3. Electronic Data Format and Coding

Data are received from the laboratory pdf format and electronically as an EQuIS® 4-file electronic data deliverable (EDD). The data are imported into the database by the laboratory and checked by the database analysts. For projects requiring data validation, the chemist generates an electronic output from the database (a flat file workbook) for their use in reviewing the data and for importing the data qualifications back to the database.

The flat file workbook consists of a sample summary worksheet, a flat file, and an xtab containing the laboratory qualifiers.

3.1 Sample Summary Worksheet

The sample summary provides a snapshot of the type and number of sample results imported into the database for that sample set. This spreadsheet indicates the number of analytes reported for each sample/test as shown in the example below:

Sample Name	MADEP-VPH	SW-6010R	SW-7470R	SW-8015V	SW-8021 BTEX	SW-8260 BMTBE
027626-032707-MW1					4	
027626-032707-MW11					4	
027626-032707-MW11 DUP					4	
027626-032707-MW12					4	
027626-032707-MW2					4	
027626-032707-MW4					4	
027626-032707-MW7					4	
027626-032707-MW8					4	
27626-051707-COMP1		7	1			
27626-051707-SB10 (10-12)				1		5
27626-051707-SB10 (14-15)				1		5
27626-051707-SB10 (8-10)				1		5
27626-051707-SB2 (10-12)				1		5
27626-051707-SB4 (14-15)				1		5
27626-051707-SB-4 (4-6)				1		5
27626-051707-SB-4 (8-10)				1		5
27626-051707-SB5 (10-12)				1		5

If the sample summary shows that there were 26 VOCs reported for all samples except for one sample that had 24, the discrepancy should be checked.

This spreadsheet will summarize the number of results flagged as "reportable" by the laboratory. If the laboratory performs a dilution or reanalysis, they are directed to include both results in the EDD, but one should be flagged as "Yes" for reportable and the other "No". If the lab has failed to flag data for dilutions or re analyses properly, the sample summary may show double the number of analytes

for one or more of the samples. This is rectified by changing the reportable flags in the flat file from "Yes" to "No" for the appropriate results.

The stage one checker add-in for Excel shall be used at this point to compare the type and number of analytes reported by the laboratory to what was requested on the Simplified Scope of Work (SSOW) document.

Open the appropriate project SSOW and go to the “SOW Rep Limits” tab at the bottom of the page. Click on the “Add-ins” tab in Excel, “SSOW Checker”, “Perform checks”. Select the appropriate flatfile workbook in the pop-up box and click on the “Compare SSOW” box and follow any additional instructions. A summary workbook will be generated. Review the green tabs and make any necessary corrections/edits.

3.2 Flat file Worksheet

The flat file worksheet is generated by the validating chemist by using the flat file generator website. Access to the website is provided to each chemist by database. If the project has requested their data be reported to specific reporting limits, then the "custom field_2" and "custom field_3" on the flat file shall be populated with the appropriate data, which can be any of the following:

i. Laboratory reporting limit (RL)

ii. Laboratory quantitation limit (QL), defined as the laboratory method detection limit adjusted for sample-specific factors, e.g. weights, volumes and dilution factors.

Note: Custom field_2 shall never be populated with the laboratory MDL. The QL is always the appropriate number when "MDL reporting" is requested. The QL is always the sample-specific adjusted MDL.

3.2.1 Flat file Fields

The following are a list of fields included in the flat file with descriptions.

parent sample code	"sys_sample_code" of parent sample for field duplicate (FD)/matrix spike (MS)/matrix spike duplicate (MSD)/laboratory replicate (LR)/laboratory control sample duplicate (LCSD)
lab sample id	laboratory sample
Field SDG	laboratory SDG
Matrix _Code	Sample Matrix
sample_type_code	Original (N), field duplicate (FD), Trip Blank (TB), etc.
result_type_code	Result type-"TRG" for a target or regular result, "TIC" for tentatively identified compounds, "SUR" for surrogates, "SC" for spiked compounds, or "IS" for internal standards (internal standards are not required in the deliverable)
loc_name	Sample location
start_depth	Initial sample depth
end_depth	Final sample depth
depth_unit	Sample depth unit
sample_date	Sample collection
sample_time	Sample collection
prep_date	Sample prep date
analysis_date	Analysis date
analysis_time	Analysis time

max_off_test_batch_id	Unique identifier for all lab batches
instrument_id	Instrument identifier
column_number	Both columns are required when testing for PCBs and pests
dilution_factor	Sample dilution factor
detect_flag	"Y" for positive detection, "N" for non-detects
result_test	Positive detection value (null if non-detect)
reporting_detection_limit	Laboratory reporting limit, adjusted for dilution factors, sample weights and sample volumes
quantitation_limit	sample-specific method detection limit (MDL), adjusted for dilution factors, sample weights, and sample volumes
method_detection_limit	MDL (unadjusted)-the unadjusted MDL is never used in custom_field_2 or custome_field_3
custom_field_2	Populated with associated RL or QL, depending on reporting requirements
custom_field_3	Populated with QL or RL, depending on reporting requirements
result_unit	Units
lab_qualifiers	Laboratory qualifiers, also known as laboratory flags
validator_qualifiers	Validation qualifiers
approval_a	Validation reason code
reportable_result	Indicates whether sample result is to be reported-"Yes" will allow for extraction, "No" will suppress
approval_b	DV guidance document code
approval_code**	Validation level code
subsample_amount	Amount of sample used for the test
subsample_amount_unit	Unit of measurement for "subsample_amount"final_volume
final_volume_unit	Unit of measure for "final_volume"
qc_original_conc	The concentration of the analyte in the original (unspiked)
sample qc_spike_added	The concentration of the analyte in the original (unspiked)
sample qc_spike_measured	The measured concentration of the analyte
qc_spike_recovery	The percent recovery for the spike
qc_dup_original_conc	The concentration of the analyte in the original (unspiked)
sample qc_dup_spike_added	The concentration of the analyte added to the original
sample qc_dup_spike measured	The measured concentration of the analyte in the duplicate
duplicate qc_dup_spike recovery	The duplicate percent recovery
qc_rpd	The relative percent difference between the spike and the spike duplicate
qc_spike_lcl	Lower control limit for spike recovery
qc_spike_ucl	Upper control limit for spike recovery
qc_rpd_cl	Relative percent difference control limit
qc_spike_status	Flag used to indicate whether the spike recovery was within control limits
qc_dup_spike status	Flag used to indicate whether duplicate spike recovery was within control
limit qc_rpd_status	Used to indicate whether the relative percent difference was within control
limits collection_quarter	Collection quarter
sampling_reason	Sample event identifier
Remark	Import metadata
original_result	Populated with original result

Fields that are color-coded are included for the chemist's information only. These fields should not be modified, as any changes will not be imported back into the database. If any changes are needed for these fields, please notify the database analyst.

The flat file can be made more manageable by "hiding" columns not needed. To do this, right click on the column letter to highlight the entire column select "hide". You can highlight one or several columns before hiding them. To unhide, highlight the two visible columns adjacent to the hidden columns, right click and select "unhide". To unhide all columns at once, click on the top left-corner of the .xls sheet, which highlights the entire sheet, and go to "format", "columns" and select "unhide".

3.2.2 Check Flat File Program

The Add-Ins/Check Flat file tab is located at the top of the flat file worksheet and includes macros to be used on the flat file including Convert Time, Run Flat file Completeness Checker (covered later in this section), and Run Qualifier Copy (J, B, U).

Convert Time - All fields are submitted as text and must remain as text (to preserve significant digits in the database) with the exception of the "analysis_time" field which can be converted to standard time format (for sorting purposes). Use the "convert time" command under the "Check Flat File" tab at the top of the spreadsheet.

Run Qualifier Copy - For data that are to be validated, the validator qualifier field must include all lab qualifiers that should be applied to the final data set. This includes any lab qualifiers that would be preserved through validation (e.g. "U"). Prior to adding validator qualifiers to the flat file, this program is used to copy pertinent lab qualifiers to the validator qualifier field as follows:

- U Qualifier - The program will ask "copy over U qualifiers?" If the user selects "yes", the program finds all instances of U in the "lab_qualifiers" field and inputs a "U" into the "validator_qualifier" field. The macro also inputs "PLU" (Preserve Lab U) into the "approval_code" field.

If the user selects "no", the program does not do anything with lab "U" qualifiers.

- J Qualifier - When the program finds a "J" in the "lab_qualifiers" field it copies "J" into the "validator_qualifiers" field, and copies "BRL" (Below Reporting Limit) into the "approval_code" field.
- B Qualifier –The B qualifier denotes blank contamination. The program will ask "copy inorganic B's over as J's?" Most labs now universally use "B" to denote blank contamination for organic and inorganic parameters. If this is the case, select "No". The "B" will not be copied over.

3.2.3 Data Qualification

Data qualifiers are entered into the "validator_qualifier" field. Each "validator_qualifier" must have an associated reason for the data qualification that is entered into the "approval_a" code field (see Section5.0 for more information on reason codes). The addition of new valid values for reason codes must be submitted to Julie Lidstone for review and inclusion in the "approval_a" reference table.

All preserved laboratory qualifiers in the "validator_qualifier" field must also have an associated "approval_a" code. The "approval_a" code for the preserved lab qualifier of "J" is "BRL". The "approval_a" code for preserved laboratory qualifier "U" is "PLU".

To qualify for blank contamination, the value in the "result_value" field is removed, the "detect_flag" field is changed from "Y" to "N", and the U qualifier is entered into the "validator_qualifiers" field. If the original sample concentration was an estimated value (e.g., 6J), the "reporting_detection_limit" field is not changed. If the project is set up to report to the QL (sample-specific MDL), then custom_field_2 will need to be raised to the detected value. If the original sample concentration was not an estimated value (e.g., >RL), the "reporting_detection_limit" field is changed. If custom_field_2 is populated, it will also need to be raised to the qualified value. The appropriate reason code is added to the "approval_a" field (e.g., MBK).

Samples may have more than one set of results due to dilutions and re-analyses completed by the laboratory. The EDD includes a field titled "reportable_result" which is populated with either "No" or "Yes" for each result. When multiple analyses are performed, the laboratory will designate which result they deem most usable based on quality control data. Any time there are multiple analyses for a sample, the data validator must determine which analysis best meets the project DQO. Consideration should be given to all quality control (QC) factors (e.g., surrogate recoveries, holding times, internal standard recoveries, etc.), during this determination. If the validator chooses to report a result other than that selected by the lab, the "reportable_result" field is changed from "Yes" to "No" for the value that the validator wishes not to report, and the "reportable_result" field is changed from "No" to "Yes" for the value to be reported.

Due to the fact that some validation guidance documents include different qualifiers and vary in the application of qualifiers, each result in the database must include documentation of the guidance used for data validation. This information is stored in the "approval_b" field (see Section 4.0 for more information on "approval_b" codes); this field must be populated for all results.

All data in the database must show the level of validation applied. The "approval_code" value represents the level of validation. The "approval_code" will be defaulted to "4" upon opening the spreadsheet and must be revised after validation to the "approval_code" field prior to submittal for import into the database. Approval codes are summarized in Table 3.1.

Table 3.1 - Data Validation Levels-"Approval_Codes"

Approval Code	Description
0	Validation Status Unknown
1	No Review Performed
2	Reduced Validation/Verification Performed
3	Full Validation Performed
4	Lab Qualified Result, in the process of Verification/Validation
5	Do not use
6	Compliance Check
8	Full Validation Performed by External Consultant
9	Reduced Validation/Verification Performed by External Consultant
10	Innovative Approach Validation (Forms Review includes NJ Reduced)

Non-detect reporting limits are normally reported from the "reporting detection limit" field of the flat file, which is typically the RL for organics and inorganics.

If it is required that reporting limits other than those stored in the "reporting_detection_limit" field are required (e.g., sample-specific MDLs be used for metals in place of RLs or sample-specific MDLs across the board for risk assessment or TRRP reporting), the reporting limits can be stored in an optional field called "custom_field_2". Prior to flat file generation, the validator will inform the database analyst about where reporting limits for non-detect data are to be obtained and these values will be imported into the "custom_field_2" field of the flat file.

If "custom_field_2" is used, when changing reporting limits due to method blank concentrations, the validator must be sure to change the values in the "custom_field_2", "reporting_detection_limit" and "quantitation_limit" fields as appropriate.

3.2.4 Flat file Completeness Checker

This program is located under the Add-ins Tab/Check Flat file tab and is used to check the final flat file for consistency and completeness. For example, the checker will identify inconsistencies like results that have "U" listed under "validator_qualifier" field, and a "Y" populated in the "detect_flag" field. This checker must be run and all errors addressed before the flat file is uploaded to database.

3.2.5 Saving the Updated Flat file

Upon completion of validation, the "L" in the file name is changed to a "V" and the validator's initials are added to the end of the file name. The validated FF is uploaded to the flat file website and a cross-tab is generated of the validated data using the “get xTab” option after the validated FF is uploaded. An e-mail shall be sent to the database analyst informing them that the flat file has been uploaded into the database and a cross-tab generated. The original flat file, validated flat file and cross-tab shall be attached to the email.

4. Review of Applicable Quality Control Documents and Standards

Prior to commencing data validation, all applicable QC documents must be reviewed and understood. Where applicable, this includes the QAPP, DQO, region-specific or project-specific validation guidelines, permits, SOPs, and project SAPs, etc.

Unless otherwise specified, data validation is performed using the principles outlined in the USEPA 2016 NFG, the QC criteria outlined in the analytical methods, the GHD data validation SOP, and professional judgment. Many elements of the 2016 NFG do not apply to the methods that are typically used for the analysis of numerous GHD projects. When this is the case, project-specific quality documents and professional judgment will take precedence over all other guidance documents for decision-making purposes during analytical data review and assessment.

The data validation guidance document used to perform the validation must be recorded in the database. A two-digit code has been assigned to each document (see Table 4.1) and the appropriate code must be entered into the "approval_b" field of the flat file.

Table 4.1 - Data Validation Guidance Documents-"Approval_b"

Guidance Code[1]	Data Validation Guidance Document(s)
01	National Functional Guidelines in conjunction with GHD SOP
02	GHD Analytical Data Quality Assessment and Validation SOP
03	NJDEP Guidance Documents in conjunction with GHD SOP
04	Region II Guidance Documents in conjunction with GHD SOP
05	Region III Guidance Documents in conjunction with GHD SOP
06	Region I Guidance Documents in conjunction with GHD SOP
07	Region 5 Guidance Documents in conjunction with GHD SOP
08	Texas Commission on Environmental Quality Regulatory Guidance Texas Risk Reduction Program (TRRP-13) in conjunction with GHD SOP
09	Canadian Validation Procedural Review
10	Guidance Document Unavailable (professional judgment used for data validation)
11	National functional Guidelines in conjunction with GHD SOP and QAPP
12	GHD Analytical Data Quality Assessment and Validation SOP
97	Unknown DV Guidance Document
98	Not applicable. Data not validated.
99	Predates population of approval_b field as of October 26, 2005

Notes:

[1]	Guidance codes are identified as Approval_b codes in the database and flat files
DV	Data Validation
NJDEP	New Jersey Department of Environmental Protection
SOP	Standard Operating Procedure

5. Reason Code Assignment

As data are qualified throughout the data validation processes, a reason code is assigned to document the reason for the qualification. A list of reason codes and definitions are presented in Table 5.1. These reason codes are stored in the database in the "approval_a" field. For all data qualifications, the appropriate reason code must be entered into the "approval_a" field of the flat file.

When a data value is qualified for multiple reasons, up to three reason codes (9 characters) can be assigned appending each to the primary qualifying code without separation. For example, if a data point is qualified for outlying surrogate and laboratory control sample (LCQ) recoveries, the result is qualified with "LCQSUR".

Table 5.1 - Data Validation Guidance Documents-Reason Codes

Reason Codes	Description
ABH	Analyte present, quantitation may be biased high due to presence of co-eluting target analyte(s)
BCL	Result reported is below calibration level; quantitation may not be accurate
BKR	Breakthrough of Aromatic Compounds into the Aliphatic Fraction
BRL	Below Reporting Limit (for preserving laboratory J)
BSQ	Qualified due to blank spike outlier
CAR	Radiochemistry – carrier outlier
CBK	Inorganics calibration blank contamination-initial or continuing
CCL	Continuing calibration outlier
CRA	CRA/Contract required detection limit outlier
DCD	Dual column discrepancy (for GC analyses only)
DEG	Qualified due to high degradation (GC pest analysis only)
DMC	Deuterated monitoring compound outliers
DUP	Laboratory duplicate variability
EXE	Exceeds calibration range
FBK	Field blank contamination
FCE	Field Collection Error
FDP	Field duplicate variability exceedance
FIL	Filter blank contamination
HIS	Professional judgement based on data non-comparability with respect to historical sample results
HSP	Headspace present in VOA vial used for analysis
HTQ	Holding time exceedance
IBA	Dioxin/Furan Ion Abundance Ratio Violation
ICL	Initial calibration outlier
ICS	ICP Calibration Stability (instrument drift)
IEM	Inadequate extraction mass
INT	Interference present (positive/negative)
IQC	Insufficient quality control performed
ISD	ICP Serial Dilution outlier
ISI	ICP Interference check sample outlier
IST	Internal standard outlier
IVS	Initial calibration verification standard outliers
LCQ	Laboratory control sample (LCS) percent recovery outlier
LCD	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCD) outlier
MAT	Matrix Interference impacted analyte quantitation and/or identification
MBK	Method blank contamination
MLD	Mass loss due to de-mineralization
MSA	Method of standard additions outliers (metals)
MSD	Matrix spike/matrix spike duplicate percent recovery outliers

Reason Codes	Description
MSQ	Matrix spike percent recovery outliers
NNQ	Nitrite results greater than combined nitrate/nitrite result
PCM	Poor chromatographic match to standard
PLQ	Preservation of laboratory qualifier
PLU	Preservation of laboratory U qualifier
PSP	Post digestion spike outlier
QUA	Analyte present, the reported value may not be accurate or precise due to poor chromatography. The sample chromatogram exhibits baseline interference that impacted sample quantitation.
RBK	Rinsate blank contamination
REP	Variability in replicate results
RSP	%RSD out for replicate aspirations (GFAA)
RTO	Retention Time Outlier
SIC	Suspected Instrumental Carryover
SLD	% Solids <30%
SPQ	Sample preparation/quantitation nonconformance (initial/final weight or volume is an approximate amount)
SPV	Sample preservation violation due to temperature, preservative and pH
SRN	Sample receipt Nonconformance
SUQ	QC sample surrogate outlier causing investigative sample qualification (specific states)
SUR	Surrogate outlier
TBK	Trip blank contamination
TIC	TIC qualifier
TOT	Total dioxins and/or furans are reported as estimated due to the nature of the quantitation
TUN	Tune nonconformance
TVD	Dissolved analyte result is significantly greater than the associated total analyte result
VCC	Validator's Choice of Columns for reporting GC results
VCD	Validator's Choice of Dilutions for when the dilution on the flat file is not the one you want to report
VCM	Validator's choice of method
VCR	Validator's choice of reanalysis

6. Data Validation Procedures

Data validation is performed in accordance with the USEPA 2016 NFGs (or other region- or project-specific validation guidance document) and any other project-specific quality control documents in conjunction with ***professional judgment***. The following sections detail the data validation procedures for each element.

During data validation, data qualifiers are applied to sample results to indicate potential biases in the data. The USEPA 2016 NFG data validation

qualifier definitions are presented in Table 6.1. Based on project specific requirements, directional bias indicators may or may not be needed.

Table 6.1 - Data Qualifiers and Definitions

Data Qualifier[1]	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample
J+	The result is an estimated quantity, but the result may be biased high
J-	The result is an estimated quantity, but the result may be biased low
NJ	The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value is the estimated concentration in the sample
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
C	The target pesticide or Aroclor analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS)
X	The target pesticide or Aroclor analyte identification was not confirmed when GC/MS analysis was performed

Notes:

[1] Qualifier codes identified are based on the following guidance documents "National Functional Guidelines for Superfund Organic Methods Data Review", EPA-540-R-2016-002, September 2016, and "National Functional Guidelines for Inorganic Superfund Methods Data Review", EPA-540-R-2016-001, September 2016.

6.1 Data Package Completeness and Compliance Review

Prior to data quality assessment and validation, the data package must be checked to ensure that all elements are present.

6.2 Sample Preservation and Holding Time Assessment

Holding times are defined as the amount of time that elapses between the collection of the appropriately preserved sample from the source in the field and the beginning of the analysis procedure (which may include extraction and a separate holding time period from extraction to analysis).

Sample holding times are verified by the Chain of Custody forms (COCs), laboratory custody records, and narrative to verify that samples were received at ≤6°C (if applicable), were properly preserved, and analyzed within the method or project-specific holding times. For exceedances, qualify data as indicated in Table 6.2, Sample Preservation and Holding Time (HT) Non-Compliance Action.

Table 6.2 - Sample Preservation and Holding Time Non-Compliance Action

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Approval Code
Organic Parameters - Hold Times [1]				
VOC Waters	Properly preserved and sample but HT>14 days	R	J	HTQ
VOC Soil	>48 hours to encore prep and not frozen >14 days for field preserved	No Qual R	No Qual J-	HTQ
SVOC/Pest Waters	>7 and <14 days (for extraction) >14 days (for extraction) >40 days (for analysis)	UJ R UJ	J- J- J-	HTQ
SVOC/Pest Soil	>14 and <28 days (for extraction) >28 days (for extraction) >40 days (for analysis)	UJ R R	J- J- J-	HTQ
Water PCBs (including congeners)	>1 year (for extraction) GHD >40 days (for analysis)	UJ UJ	J- J-	HTQ
Soil PCBs (including congeners)	>1 year (for extraction) GHD >40 days (for analysis)	UJ UJ	J- J-	HTQ
Water Dioxins/Furans	>1 year (for extraction) GHD >40 days (for analysis)	UJ UJ	J- J-	HTQ
Soil Dioxins/Furans	>1 year (for extraction) GHD >40 days (for analysis)	UJ UJ	J- J-	HTQ
Inorganic Parameters - Hold Times [1]				HTQ
Inorganic-Holding Time (all matrices)	Sample HT exceeds but <2x [2,3, and 4]	Prof. Judgement	J-	HTQ
Holding Time[1]	Sample HT >2x	UJ or R	J-	HTQ
Preservation				
Headspace (VOC Waters)	VOC vial contained headspace GHD (>6mm air bubble)	UJ	J-	SPV
VOC Preservation[1]	pH>2 and >7 days	R	J-	SPV or HTQ
Inorganic-Chemical Preservation (water only)	Not preserved in the field and pH not adjusted in the lab [5]	R	J-	SPV
Inappropriate Container	Sample collected in inappropriate container (GHD)	Prof. Judgment	Prof. Judgment	SPV
Sample/Cooler Temperature[6]	Ice present; >6 and ≤10	No Qual	No Qual	
	No Ice (GHD) and >6 and ≤10°C (GHD)	Prof. Judgment	Prof. Judgment	SPV
	No Ice (GHD) and >10 and ≤20°C (GHD)	UJ/R	J-	SPV
	No Ice (GHD) and >20°C (GHD)	R	J-	SPV

Notes:

[1] Holding time criteria will incorporate professional judgment for certain parameter/matrices.

[2] pH adjustments by the laboratory are acceptable for inorganics; use professional judgment for qualifications.

[3] If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low.

[4] Due to limited information concerning hold times for soil, sediment, wipe, and filter samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria.

[5] Use professional judgment to qualify the sample based on the pH of the sample and the chemistry of the metal(s) of interest.

[6] Sample/cooler temperature evaluation will include other considerations, such as the presence of absence of ice in the cooler, the number of days in transit and the specific parameters requested. Same day delivery (less than 24 hours from collection) of samples is exempt from receipt temperature requirements.

6.3 Instrument Performance Checks

6.3.1 Instrument Performance Check (IPC) Organics by GC/MS and Metals by ICP/MS – Tune Checks

The instrument performance check is particular to the use of instruments having a Mass Spectrometer (MS) as the detector. The MS functions by bombarding the target analytes with electrons as they enter the analyzer. The electrons collide with target analyte molecules causing them to ionize. The analyzer then performs a count of the ion abundance of each ion created with the compound molecules. The software used in conjunction with the mass analyzer prepares a plot of abundance versus mass of the ions, called a mass spectrum. The relative abundance of the ions created and detected from the compound molecules is dependent upon the electrical and magnetic properties of the mass analyzer.

The following items are evaluated as part of the instrument performance check during FDV with calculations spot-checked at a 10 percent frequency:

- Check IPC summaries to ensure that instrument IPCs were performed every 12 hours (organics) and that all investigative samples are accounted for within those tune windows.
- Verify the tune checks were analyzed at least five times consecutively (ICP/MS only) and prior to every initial calibration. If tune checks have not been performed at the required frequency, use professional judgment to determine whether associated sample results are valid.
- Check that all abundances are normalized to the proper m/z.
- Verify the ICP/MS percent relative standard deviation (%RSD) values were within the specified criteria.
- Check that the proper number of significant figures have been reported.
- Spot-check for transcription errors between raw data and IPC summary form.
- Recalculate some of the relative ion abundance's to verify lab's calculations.
- Verify that spectra were obtained using accepted background subtraction techniques.
- Verify that appropriate (method- or QAPP-specific) ion abundance criteria were used.

If errors are noted, contact the laboratory and request a corrected re-submittal. If the lab cannot resubmit data, all associated data should be rejected (R).

Check all ion abundances against appropriate criteria (e.g., NFGs, SW-846 methods, or TO-15 for VOCs in SUMMA canisters). If outliers are noted, use professional judgment to determine whether

the data are usable. If data are judged to be usable, the data validation report must clearly state the nature of the tune non-conformances. If the IPC non-conformances are judged to impact data quality, qualify data as follows:

Table 6.3.1 - Instrument Performance Check Criteria and Validation Action

				Action	
Assessment Elements	Criteria	Failure	Non-Detects	Positive Results	Approval Code
VOC BFB Criteria (NFG-2016)	50 15.0-40.0% of 95 75 30.0-80.0% of 95 95 Base Peak (100%) 96 5.0-9.0% of 95 [1] 173 <2.0% of 174 174 50.0-120% of 95 175 5.0-9.0% of 174 176 95.0-101% of 174 177 5.0-9.0% of 176	*m/e non-compliant	UJ(GHD)	J(GHD)	TUN
		incorrect base mass assignment	R(GHD)	R (GHD)	TUN
		Analyzed >12 hours but ≤13 hours	UJ(GHD)	J(GHD)	TUN
		Analyzed >13 hours	R (NFG)	R(NFG)	TUN
VOCs by TO-15 BFB Criteria	50 8.0-40.0% of 95 75 30.0-66.0% of 95 95 Base Peak (100%) 96 5.0-9.0% of 95 173 <2.0% of 174 174 50.0-120.0% of 95 175 4.0-9.0% of 174 176 93.0-101.0% of 174 177 5.0-9.0 of 176	*m/e non-compliant	UJ(GHD)	J(GHD)	TUN
		incorrect base mass assignment	R(GHD)	R (GHD)	TUN
		Analyzed >24 hours but ≤25 hours	UJ(GHD)	J(GHD)	TUN
		Analyzed >25 hours	R (NFG)	R(NFG)	TUN
SVOC DFTPP Criteria (NFG-2016)	51 10.0-80.0% of 198 68 <2.0% of 69 69 Present 70 <2.0% of 69 127 10.0-80.0% of 198 197 <2.0% of 198 198 Base Peak (100%) [2] 199 5.0-9.0% of 198 275 10.0-60.0% of 198 365 >1.0% of 198 441 Present but <443 442 >50.0 of 198 443 15.0-24.0% of 442	*me/e non-compliant	UJ (GHD)	J (GHD)	TUN
		Incorrect base mass assignment	R (NFG)	R (NFG)	TUN
		Analyzed > 12 hours but ≤13 hours	UJ(GHD)	J(GHD)	TUN
		Analyzed >13 hours	R (NFG)	R (NFG)	TUN
Metals ICP/MS (NFG-2016)	Prior to calibration, analyze or scan the tuning solution at least 5 times consecutively. [3]	Tune not performed	R	R	TUN
	Tuning solution contains 10 µg/L Li, Co, In, and Tl-all required isotopes are present	Tune not performed properly	Prof Judgment	Prof Judgment	TUN
	The laboratory shall make any adjustments needed to bring the peak width within the manufacturer's specifications and adjust mass resolution to within 0.1 u over the range of 6-210 u.	Resolution of mass calibration not within 0.1u	UJ	J	TUN
	%RSD of the absolute signals for all analytes in the tuning solution must be <5%	%RSD>5%	UJ	J	TUN

[1] All ion abundances must not be normalized to mass to charge (m/z) 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

[2] All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 100% that of m/z 198.

[3] Four times for SW-846 method 6020 compliance.

6.3.2 Breakdown Evaluation – Pesticides

To monitor the inertness of the analytical system, the breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12 hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 20% for either compound.

Breakdown is calculated as follows:

$$\% \text{ Breakdown DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDD + DDE)}} \times 100$$

$$\% \text{ Breakdown Endrin} = \frac{\text{sum of degradation peak areas (aldehyde + ketone)}}{\text{sum of all peak areas (endrin + aldehyde + ketone)}} \times 100$$

If the performance evaluation standard was not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

- Spot check calculations (10 percent) for breakdown.

If errors are identified, contact the lab to have the data package corrected and resubmitted. If breakdown criteria are not met, qualify the data as follows:

Table 6.3.2- Organic Instrument Breakdown Check Criteria and Validation Action

				Action		
Method	Assessment Element	Criteria	Failure	Non-Detects	Positive Results	Reason Code
GC/ECD	PEM Frequency [1]	Once every 12 hours	Not performed	R	R	DEG
GC/ECD	PEM % Resolution	>90.0%	<90.0%	R	NJ	DEG
GC/ECD	PEM Breakdown 4,4' DDT	<20.0% Individual	4,4' DDT %Breakdown >20.0% and 4,4'-DDT is detected	None	J for 4,4' DDT, 4,4' DDD and 4,4' DDE	DEG
			4,4' DDT %Breakdown >20.0% and 4,4' DDT is not detected	R for 4,4' DDT	NJ for 4,4' DDD and 4,4' DDE	DEG
GC/ECD	PEM Breakdown Endrin	<20.0% Individual	Endrin %Breakdown >20.0% and Endrin is detected	None	J for Endrin, Endrin aldehyde, and endrin ketone	DEG

				Action		
Method	Assessment Element	Criteria	Failure	Non-Detects	Positive Results	Reason Code
			Endrin %Breakdown >20.0% and Endrin is not detected	R for Endrin	NJ for Endrin aldehyde and Endrin ketone	DEG
GC/ECD	PEM Combined Breakdown	<30% Combined	>30%	Apply qualifiers as described above considering degree of individual breakdown	Apply qualifiers as described above considering degree of individual breakdown	DEG

Notes:

[1] PEM analysis is not required as part of SW-846 8081 analyses. However, if provided by the laboratory, 2016 NFGs are utilized in the evaluation of pesticide performance evaluation mixture analyses.

6.4 Instrument Calibration - Organics

The following sections address initial and continuing instrument calibration for organic analyses. Instrument calibration data are reviewed during FDV only.

If CLP data are to be reviewed, the 2016 NFGs must be followed without exception.

6.4.1 Initial Instrument Calibration - Organics

Calibration is the establishment of a quantitative relationship between the response of the analytical procedure and the concentration of the target analyte. The initial calibration (ICAL) is the procedure that functions as the calibration curve for the target analytes. A necessary prerequisite is that a confident identification of the target analyte has already been established.

To assess ICAL data:

- Check lab data to ensure that initial calibration standards were analyzed at the proper frequency.
- Verify a minimum of five standards were utilized and were analyzed at the appropriate concentrations.
- Check that the ICAL was analyzed prior to all samples associated with it.
- Verify that the ICAL met the minimum average relative response factors (RRF) and %RSD criteria identified in the NFG unless alternate method criteria are specified.
- Check lab data to ensure that the compounds identified were within the retention time (RT) criteria.
- Verify that at least three peaks were used in the calibration of each Aroclor during PCB analysis.

If the calibration standards were not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

- Spot check calculations (10 percent) for RRF or response factors (RF) for methods using internal standards.
- Spot-check calculations (10 percent) of the calibration curve. If various calculation methods were used (e.g., average RRF, linear regression, quadratic, etc.), results reported using each calculation must be verified.
- For GC methods, spot-check the calculation of retention time windows.
- Spot-check %RSD, correlation coefficient (R^2) calculations if used.

If errors are identified, contact the lab to have the data package corrected and resubmitted.

- Check the linearity of the calibration curve; %RSDs ≤40.0% or ≤25.0% for the compounds listed in Tables 6.4.1 and 6.4.2; ≤20.0% for all other VOC and SVOC compounds (with the exception of 1,4-dioxane that has an advisory maximum criteria of 50.0% RSD); and %RSDs ≤20.0 % for GC (≤25.0% for alpha-BHC, delta-BHC and ≤30.0% for toxaphene), R^2 values ≥0.990).
- For methods using internal standards, check analyte sensitivity; average RRF must be ≥0.010 for the volatile target compounds listed in Table 6.4.1 with the exception of 1,4-dioxane that has an advisory RRF of ≥0.0050; the RRF for all other volatile target compounds must be ≥0.050.
- Verify that for the semi-volatile target compounds listed in Table 6.4.2, the initial calibration RRFs are > to 0.010, and for all other semi-volatile target compounds, RRFs are > to 0.050.

Table 6.4.1 - Volatile Compounds Exhibiting Poor Response – (RRF ≥0.010 and ≤40.0% RSD unless otherwise noted)

Compounds [1]	
Acetone	4-Methyl-2-pentanone (RRF ≥0.030 and ≤25.0%RSD)
2-Butanone	Trichlorofluoromethane
Chloroethane	1,1,2-Trichloro-1,2,2-trifluoroethane (RRF ≥0.050 and ≤25.0%RSD)
Chloromethane (<20.0%RSD)	Vinyl Chloride (<20.0%RSD)
Cyclohexane	Bromomethane
Dichlorodifluoromethane (<25.0%RSD)	
1,2-Dibromo-3-chloropropane (≤25.0%RSD)	
1,4-Dioxane	
Methyl acetate	
Methylene chloride	
Methylcyclohexane (RRF ≥0.050)	
Methyl tert-butyl ether (RRF >0.100)	

[1] Table 6.4.1 is the GHD interpretation of the 2016 NFGs RRF, %RSD, and %D Acceptance Criteria Tables.

If CLP data are to be reviewed, the 2016 NFGs must be followed without exception.

Table 6.4.2 – Semi-Volatile Compounds Exhibiting Poor Response – (RRF ≥0.010 and ≤40.0% RSD unless otherwise noted)

Compounds [1]	
2,2'-Oxybis-(1-chloropropane) (≤20.0%RSD)	Benzaldehyde (RRF≥0.100)
1,4-Dioxane	4-Chloro-3-Methylphenol (RRF≥0.040 and ≤20.0%RSD)
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene (RRF≥0.040 and ≤20.0%RSD)	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	Di-n-octylphthalate
3 3' Dichlorobenzidine	Pentachlorophenol
3-Nitroaniline (≤20.0%RSD)	Benzo(k)fluoranthene (≤20.0%RSD)
2,4-Dinitrophenol	Benzo(a)pyrene (≤20.0%RSD)
4-Nitrophenol	Indeno(1,2,3-cd)pyrene (≤20.0%RSD)
Caprolactam	2,3,4,6-Tetrachlorophenol (RRF≥0.040 and ≤20.0%RSD)
Atrazine	Dibenzo(a,h)anthracene (≤20.0%RSD)
2-Methylphenol (≤20.0%RSD)	
3-Methylphenol (≤20.0%RSD)	
4-Methylphenol (≤20.0%RSD)	
Benzo(g,h,i) perylene (≤20.0%RSD)	
Benzo(b)fluoranthene (≤20.0%RSD)	

[1] Table 6.4.2 is the GHD interpretation of the 2016 NFGs RRF, %RSD, and %D Acceptance Criteria Tables.

If CLP data are to be reviewed, the 2016 NFGs must be followed without exception.

If linearity or sensitivity criteria are not met, qualify data as follows:

Table 6.4.3 - Organic Initial Calibration Criteria and Validation Action

				Action		
Method	Assessment	Criteria	Failure	Non Detects	Positive Results	Reason Code
GC/MS (VOC & SVOC)	Minimum Average Relative Response Factor (RRF)	>0.05 or as specified in Table 6.4.1 & 6.4.2	<0.05 or as specified in Table 6.4.1 & 6.4.2	R	J+	ICL
		Must meet criteria for target compounds listed in Tables 6.4.1 & 6.4.2	<0.010 for target compounds listed in Tables 6.4.1 & 6.4.2	R	J+	ICL
		>0.0050 for 1,4-Dioxane	<0.0050 (advisory for 1,4-Dioxane)	R	J+	ICL
	Relative Standard Deviation (%RSD)	<40.0% for target compounds listed in Tables 6.4.1 & 6.4.2	>40.0% for target compounds listed in Tables 6.4.1 & 6.4.2	UJ (GHD)	J (GHD)	ICL
		<20.0% (all other compounds)	>20.0% (all other compounds)	UJ	J	ICL
		<50.0% (1,4-Dioxane)	>50.0% (1,4-dioxane)	UJ (GHD)	J (GHD)	ICL
	Quadratic Coefficient of Determination (R^2) (SW846)	≥0.99	<0.99	UJ (GHD)	J	ICL
	Linear Correlation Coefficient (R) (SW846)	≥0.995	<0.995	UJ (GHD)	J	ICL
GC & HPLC (PCB, Pest., VOCs, SVOCs)[1,4]	% Relative Standard Deviation (%RSD) (SW-846)	≤20.0%[2]	≥20.0% (SW-846)	UJ[3] (GHD)	J[3] (GHD)	ICL
	Linear Correlation Coefficient (R) (SW846)	≥0.995	<0.995	UJ[3] (GHD)	J[3] (GHD)	ICL
	Quadratic Coefficient of Determination (R^2) (SW846)	≥0.99	<0.99	UJ[3] (GHD)	J[3] (GHD)	ICL

Notes:

[1] Multi component analyte %RSD limits are based on average of all peaks used in calibration (GHD).

[2] 25% for alpha-BHC, delta-BHC; 30% toxaphene and surrogates

[3] GHD modification of NFG qualification:

- Aroclor-1016 Non-compliance is representative of Aroclor-1221 through 1248
- Aroclor-1260 Non-compliance is representative of Aroclor-1254 through 1268

[4] HPLC, SVOC and VOCs follow the method used

6.4.2 Continuing Instrument Calibration - Organics

The continuing calibration verification (CCAL) is used to verify that the initial calibration is maintained and correct while the instrument is used to process samples. The CCAL also serves to determine that the identification criteria are still being met. Valid sample results will always be

preceded by an acceptable CCAL analysis for GC/MS methods and are bracketed by acceptable CCAL analyses for most GC methods.

- Check lab data to ensure that continuing calibration standards were analyzed both at the beginning (opening CCV) and end (closing CCV, or as required by the individual methods) of each 12-hour analysis period.
- Verify that calibration standards were at the appropriate concentrations.
- Compare %Drift values to those compounds quantitated utilizing a quadratic curve.
- Check that the GC/MS CCAL met the minimum RRF and maximum percent difference (%D)/%Drift criteria.

If calibration standards were not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

If a CCV (opening and closing, or as required by the individual methods) was not run at the appropriate frequency, qualify all data as unusable "R"

- Spot-check calculations (10 percent) for RRF or RF for methods not using internal standards.
- Spot check calculations (10 percent) for RRF or RF, and %D/%Drift.
- For GC, verify that the analyte retention times fall within the established retention time windows.

If errors are identified, contact the lab to have the data package corrected and resubmitted.

- Check the linearity: %D/% Drift <25 percent (GC/MS), %D <25 percent for single component pesticides and surrogates in the PEM (CLP) and <20 percent for CCAL (GC), %D < 15 percent for PCBs

For methods using internal standards, check analyte sensitivity per Tables 6.4.1 and 6.4.2 under initial calibration. If linearity or sensitivity criteria are not met, qualify data as follows:

Table 6.4.4 - Organic Continuing Calibration Criteria and Validation Action

		Opening CCV	Closing CCV	Action		
Method	Assessment Element [1]			Non Detects	Positive Results	Reason Code
GC/MS (VOC & SVOC)	Minimum Average Relative Response Factor (RRF)	<0.010 or criteria listed Tables 6.4.1 & 6.4.2	<0.010 or criteria listed in Tables 6.4.1 & 6.4.2	R	J or R (NFG)2	CCL
		<0.050 (all other target compounds)	<0.050 (all other target compounds)	R	J or R (NFG)2	CCL
	Percent Difference (%D) or Percent Drift (SW-846)	+/- 50.0 (1,4-dioxane as a VOC)	>|50.0| (1,4-dioxane)	UJ	J	CCL
		+/- 40.0 or criteria listed in Tables 6.4.1 & 6.4.2	>|50.0| for target compounds listed in Tables 6.4.1 & 6.4.2	UJ	J	CCL
		+/- 20.0 for all other low/med VOC or SVOC target compounds	>|25.0| for all other low/med VOC or SVOC target compounds)	UJ	J (NFG)	CCL

		Opening CCV	Closing CCV	Action		
Method	Assessment Element [1]			Non Detects	Positive Results	Reason Code
GC & HPLC (PCB, Pest, VOCs, SVOCs) [4]	Percent Difference (%D) or Percent Drift (%Drift) (SW846)	>25.0% (Pest) >25.0% (PCB);<30.0% for surrogates >25.0% (Pest) >25.0% (PCB)	>25.0% (Pest) >50.0% (PCB) >25.0% (Pest) >50.0% (PCB)	UJ [3] (GHD) UJ [3] (GHD)	J [3] (GHD) J [3] (GHD)	CCL CCL
	Retention Time Window (SW846)	±0.07 Min of Standard (SW846)		Prof. Judgment (GHD)	Prof. Judgment (GHD)	CCL
	Time elapsed between opening CCV and closing CCV	exceeds 12 hours		Prof. Judgment (GHD)	Prof. Judgment (GHD)	CCL

Notes:

1 Multi-component analyte %D and % Drift limits are based on average of all peaks or concentrations used in calibration.

2 Based on mass spectral identification

3 GHD modification of NFG qualification:

– Aroclor-1016 Non-compliance is representative of Aroclor-1221 through 1248

– Aroclor-1260 Non-compliance is representative of Aroclor-1254 through 1268

4 HPLC, SVOCs, and VOCs follow the method used for analysis

6.5 Instrument Calibration – Inorganics

The following sections address initial and continuing instrument calibration for inorganic analyses. Instrument calibration data are reviewed during FDV only.

6.5.1 Initial Instrument Calibration – Inorganics

Calibration is the establishment of a quantitative relationship between the response of the analytical procedure and the concentration of the target analyte. The initial calibration (ICAL) is the procedure that functions as the calibration curve for the target analytes. A necessary prerequisite is that a confident identification of the target analyte has already been established.

The instrument must be successfully calibrated each time the instrument is set up and after continuing calibration verification (CCV) failure.

- The calibration time and date must be included with the raw data.
- The calibration for mercury and cyanide will consist of a blank and at least five calibration standards.
- The calibration for ICP/AES and ICP/MS will consist of a blank and at least one calibration standard.
- At least one of these standards must be at or below the CRQL (contract required quantitation level) for cyanide and mercury.

- All measurements must be within the instrument working range where the inter-element correction factors are valid.
- A minimum of three replicate exposures must be used for all ICP and ICP/MS standardization, QA/QC, and sample analysis.
- The calibration curve may only be created using linear regression or weighted linear regression.
- The curve must have a correlation coefficient $\underline{\geq}$ 0.995.

If calibration standards were not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

- Spot-check calculations for calibration curves at a frequency of 10 percent. Results using calibration curve must be verified.

Table 6.5.1 - Inorganic Initial Calibration Criteria and Validation Action

				Action[1]		
Method	Assessment Element	Criteria	Failure	Non Detects	Positive Results	Reason Code
ICP, ICP/MS, CVAA, Spec., IC	Correlation Coefficient (Metals, Hg, Gen. Chem.)	R ≥ 0.995	R< 0.995	Prof. Judgment	J	ICL
	%Difference for non-zero standards	<30%D of true value	>30%D	UJ	J	ICL
	Calibration	Calibration Complete	Calibration Incomplete	Prof. Judgment	Prof. Judgment	ICL
	Calibration	Calibrated every 24 hours (Hg & Cyanide)	Calibration not performed every 24 hours	R	R	ICL
ICP, ICP/MS (Metals)	Initial Calibration Verification (ICV)	±10% of true value	<90% but ≥ 75% <75% >110%	UJ R No Qual	J- J- J+	ICV ICV ICV
CVAA (Mercury)	ICV	±15% of true value	<85% but ≥ 70% <70% >115%	UJ R No Qual	J- J- J+	ICV ICV ICV
Spec & IC (General Chemistry)	ICV	±15% of true value	<85% but ≥ 70% <70% >115%	UJ R No Qual	J- J- J+	ICV ICV ICV

Notes:

[1] All samples prepared with the analytical batch are affected.

6.5.2 Continuing Instrument Calibration – Inorganic

The continuing calibration verification (CCV) is used to verify that the ICAL is maintained and correct while the instrument is used to process samples. The CCV also serves to determine that the identification criteria are still being met. Valid sample results will always be bracketed in time by acceptable CCV analyses.

- Check lab data to ensure that continuing calibration standards were analyzed at the proper frequency.
- Verify that calibration standards were at the appropriate concentrations.

If calibration standards were not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

Table 6.5.2 - Inorganic Continuing Calibration Criteria and Validation Action

				Action[1, 2]		
Method	Assessment Element	Criteria	Failure	Non Detects	Positive Results	Reason Code
ICP, ICP/MS-(Metals)	Continuing Calibration Verification (CCV)	After every 10 samples or every 2 hours & +/- 10% of true value	<90% but ≥ 75%	UJ	J-	CCL
			<75%	R	J-	CCL
			>110%	No Qual.	J+	CCL
CVAA (Mercury)	CCV	After every 10 samples or every hour & +/- 15% of true value	<85% but ≥ 70%	UJ	J-	CCL
			<70%	R	J-	CCL
			>115%	No Qual.	J+	CCL
Spec. & IC (Gen. Chem.)	CCV	After every 10 samples or every hour & +/- 15% of true value	<85% but ≥ 70%	UJ	J-	CCL
			<70%	R	J-	CCL
			>115%	No Qual.	J+	CCL

Notes:

[1] All samples between non-compliance CCVs within the analytical sequence are affected.

[2] Use professional judgment to qualify samples associated with CCVs that grossly exceed the upper control limits.

6.6 Blanks (Method Blanks, Instrument Blanks, Calibration Blanks, Cleanup Blanks and Field Blanks)

Blank samples are prepared, collected and analyzed to determine the existence or magnitude of contamination introduced during field or laboratory activities. Sources of sample contamination include the containers and equipment used to collect the samples, preservatives added to the samples, other samples in transport coolers and laboratory sample storage refrigerators, standards and solutions used to calibrate instruments, glassware and reagents used to process samples and the analytical instrument sample introduction equipment. No contaminants should be found in the blank(s). If issues are identified with any blank, all associated data must be thoroughly scrutinized to determine if inherent variability is present or if the occurrence is isolated. Each area of analysis has its own particular suite of common laboratory contaminants.

Method blanks apply to all investigative samples belonging to the same sample preparation batch, or in the case of VOC, all investigative samples analyzed on the same instrument on the same day. At least one method blank should be prepared and analyzed for each matrix, sample delivery group (SDG), or batch of no more than 20 samples, whichever is most frequent. The method blank consists of analyte-free matrix processed through the appropriate sample preparation and analysis procedure.

Inorganic initial calibration blanks (ICBs) apply to all samples analyzed after, while continuing calibration blanks (CCBs) apply to all samples analyzed immediately before and after. The ICB shall be analyzed after analytical standardization but not before the analysis of the ICV during the initial calibration of the instrument. The CCB shall be analyzed immediately following every CCV.

Field blanks (trip blanks, rinse blanks, equipment blanks, filter blanks, etc.) are intended to identify contamination introduced during sample collection and/or storage and should be applied only to those investigative samples collected on the same day as, and with the same equipment as the field blanks (see sample collection note, COC, sample key or field personnel to determine which field blanks apply to which samples). Trip blanks are applied to all VOC samples transported to the laboratory in the same shipping cooler as the trip blank.

If blanks were not analyzed at the proper frequency, potential contamination cannot be assessed for the corresponding investigative samples. This should be noted in the data validation report.

- Check the extraction logs, run-logs, and method blank summary forms to ensure that method blanks were prepared and analyzed with samples at the required frequency (typically one method blank per analytical batch or every 12 hours).
- Check the sample run-log to ensure that instrument blanks were analyzed after any sample having significantly high target analyte concentrations.
- Check the run-log to ensure that ICBs and CCBs were analyzed at the proper frequency for inorganic analyses (typically every 10 samples).
- Review blank raw data for target analytes.

If target analytes are detected in the blanks, detection of these analytes in associated investigative samples may reflect contamination, and must be qualified as indicated in the Table 6.6.1 below. For assessing contamination in soil samples, individual sample weights (dry or wet) must be applied to the blank result before comparing it to the sample result. Soil samples with associated field blank contamination less than the reporting limits will be documented in the validation text without qualification. Significant field blank contamination for soils will require qualification. Sample preparation and dilution factors for laboratory blanks (ICBs, CCBs, and method blanks) must also be applied to the blank results.

Sample preparation and dilution factors are NOT applied to field blanks.

To qualify for laboratory contamination of the blank, the value in the "result_value" field is removed, the "detect_flag" field is changed from "Y" to "N", the U qualifier is entered into the "validator_qualifiers" field, the "quantitation_limit" field (remember, this field contains the sample-specific detection limit) is raised to the detected value, and the appropriate reason code is added to the "approval_a" field (e.g., MBK). If the original sample concentration was an estimated value (e.g., 6J), the "reporting_detection_limit" field for the non-detect value will remain unchanged. If the analytes concentration was not an estimated value, the value in the "reporting_detection_limit" field must be elevated to the original sample concentration or the blank concentration.

If "custom_field_2" is used, the validator must be sure to change the values in the "custom_field_2" and the “QL field” as appropriate.

Table 6.6.1 - Blank Validation Action – Organic Parameters

Sample preparation and dilution factors are NOT applied to field blanks.

	Action[1]			
Blank Type	Blank Result	Positive Results (RL Reporting)	Positive Results (QL[3] Reporting)[GHD]	Reason Code
Method, Storage, Trip, Field, Rinse, or Filter Blank	<RL	<RL qualify U at RL ≥ RL (or ≥2x blank result for common lab contaminants[2]-VOCs Only) use prof. judgment	Sample result is similar to QL, qualify U at QL >QL (or ≥2x blank result for common lab contaminants[2]-VOCs ONLY) use prof. judgment	MBK, TBK, FBK, RBK, FIL
Method, Storage, Trip, Field, Rinse, or Filter Blank	≥ RL or ≥QL	<RL qualify U at RL ≥ RL but <blank result, qualify U at sample result ≥ RL and ≥blank result (or ≥2x blank result for common lab contaminants[2]-VOCs ONLY) use prof. judgment	<blank result, qualify U at blank result ≥blank result, use prof. judgment; qualify U at sample result ≥ QL and ≥blank result (or ≥2x blank result for common lab contaminants[2]-VOCs ONLY) use prof. judgment	MBK, TBK, FBK, RBK, FIL
Method, Storage, Trip, Field, Rinse, or Filter Blank	Gross Contamination	Report at sample result and qualify R or prof. judgment	Report at sample result and qualify R or prof judgment	MBK, TBK, FBK, RBK, FIL

Notes:

1 Hierarchy of qualifying data due to blank contamination is MBK, CBK, TBK, FBK, FIL [(GHD)]
2 Common laboratory contaminants: VOCs-acetone, 2-butanone and methylene chloride.
3 QL refers to the sample-specific MDL

Table 6.6.2 - Blank Validation Action – Inorganic Parameters

Sample preparation and dilution factors are NOT applied to field blanks.

	Action[1]			
Blank Type	Blank Result	Positive Results (RL Reporting)	Positive Results (QL[2]) Reporting [GHD]	Reason Code
Method Blank (MBK)	<RL	<RL, qualify U at RL >RL, use prof. judgment	Sample result is similar to MBK, qualify U at sample result	MBK
Method Blank (MBK)	>RL or >QL	≤ RL, qualify U at RL >RL but <10x MBK result, report at MBK and use prof. judgment to qualify results as estimated high (J+) or unusable (R)	Sample result is similar to MBK, qualify U at sample result	MBK
ICB/CCB[3]	≤RL Absolute value is ≥ QL but ≤RL	≤ RL, qualify U at RL >RL, use prof. judgment	Sample result is similar to ICB/CCB, qualify U at sample result	CBK CBK
ICB/CCB[3] ICB/CCB[3]	>RL or >QL >RL or >QL	≤ RL, qualify U at RL >RL but < blank result, qualify U at ICB/CCB result	<ICB/CCB result, qualify U at ICB/CCB result	CBK CBK

	Action[1]			
Blank Type	Blank Result	Positive Results (RL Reporting)	Positive Results (QL[2]) Reporting GHD	Reason Code
		≥Blank result, use prof. judgment to qualify results	≥ Blank result, use prof. judgment to qualify results	CBK
ICB/CCB[3]	≤ (-QL) but ≥ (-RL)	>RL or non-detect, use prof. judgment	>QL or non-detect use prof. judgment	CBK
ICB/CCB[3]	≤ (-RL) or ≤ (-QL)	Qualify detects J- Use prof. judgment to qualify non-detects, UJ or R	Qualify detects J- Use prof. judgment to qualify non-detects, UJ or R	CBK
Field, Rinse, Filter Blanks	>RL or >QL	≥ QL but <RL qualify U at RL >RL, use prof. judgment to qualify sample result	≥ QL but < blank qualify U at sample result >QL, use prof judgment to qualify sample result	FBK, RBK, FIL FBK, RBK, FIL

Notes:

[1] Hierarchy of qualifying data due to blank contamination is MBK, CBK, TBK, FBK, FIL (GHD)
[2] QL refers to the sample-specific MDL
[3] ICB contamination affects all samples analyzed in the analytical run, CCB contamination affects only samples before and after the CCB with values reported.

6.7 System Monitoring Compounds

Surrogates are non-target compounds added to every sample at the beginning of the sample preparation to monitor the success of the sample preparation on an individual sample basis. If the samples are being analyzed and reported by CLP methodology, consult the NFGs for guidance evaluating the deuterated monitoring compounds (DMC).

For PCB analysis tetrachloro-m-xylene (TCMX) the more volatile of the two surrogates, is more volatile than any of the target analytes of this method. The recoveries of TCMX are linked to the lowest volume to which the sample extract was concentrated and target compounds such as Aroclor-1221 and Aroclor-1232. Samples with higher weight PCBs contain substantial portions of decachlorobiphenyl (DCB), which will affect the calculated recovery of this surrogate when these analytes are present in the sample.

- Check surrogate summary forms to verify that all investigative sample have been accounted for.
- Spot-check raw data to verify the recoveries on the surrogate summary form
- Spot-check calculations of the surrogate recoveries
- Check that the control limits used are the appropriate limits for the project
- Check surrogate recoveries against the laboratory control limits

If any errors are found in the surrogate summaries, or are determined in the calculations, contact the laboratory and request the forms and any affected data pages are resubmitted with corrections.

If outliers are noted, verify that samples were re-analyzed. If surrogate recoveries for extractable parameters are still outside of the control limits, verify that the laboratory re-extracted the samples to confirm matrix interference. When there are unacceptable surrogate recoveries, followed by acceptable re-analyses, verify that the designated successful analysis was reported in the flat file.

Sample data with outlying surrogate recoveries should be qualified as summarized below. SVOC surrogate recoveries are assessed by fraction (base/neutral or acid) and only those analytes within that fraction affected by outlying surrogate recoveries are qualified.

Interferences and/or sample dilutions of 5 times and greater can prohibit surrogate recovery assessment. In such cases, it should be noted in the data validation report that surrogate recoveries could not be assessed.

Table 6.7.1 - Surrogate Criteria and Validation Action

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
GC, GC/MS & HPLC	If one or more surrogates: %R>UCL	None	J+	SUR
	%R≥ 10% but <LCL	UJ	J-	SUR
	%R<10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
GC/MS SVOC Surrogate Recovery	1 out in a fraction > 10%	None	None	SUR
	≥ 2 out of a fraction %R>UCL	None	J+	SUR
	≥ 2 out of a fraction %R≥ 10% but <LCL	UJ	J-	SUR
	One %R<10% per fraction	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
PCBs[1] & Pesticides	Retention time outside of established window TCMX +/- 0.05 min DCB +/- 0.10 min	Prof. Judgment	Prof. Judgment	SUR
TPH-GRO/DRO/ORO	If one or more surrogates: %R > UCL	None	J+	SUR
	%R≥ 10% but <LCL	UJ	J-	SUR
	%R<10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
TPH-VPH with target analytes	If surrogate analyzed with PID detector (i.e. surrogate for target analytes and <C10 (Aromatic) fractions): %R > UCL	None	J+	SUR
	%R ≥ 10% but <LCL	UJ	J-	SUR
	%R<10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
	If surrogate analyzed with FID detector (i.e. surrogate for <C12 (Aliphatic) fractions):			
	%R > UCL	None	J+	SUR
	%R ≥ 10% but <LCL	UJ	J-	SUR
	%R < 10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
TPH-EPH with target analytes	If o-terphenyl (i.e. surrogate for target analytes and Aromatic fractions):			
	%R > UCL	None	J+	SUR
	%R≥ 10% but <LCL	UJ	J-	SUR
	%R<10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
	If 1-chlorooctadecane (i.e. surrogate for Aliphatic fractions):			
	%R > UCL	None	J+	SUR
	%R≥ 10% but <LCL	UJ	J-	SUR
	%R<10%	R	J-	SUR
	Diluted (≥ 1:5) (GHD)	None	None	None
	For 2-fluorobiphenyl and 2-bromonaphthalene (i.e. fractionation surrogates):			
	If both are out-qualify as above	--	--	SUR
	If one is out	Prof. Judgment	Prof. Judgment	Prof. Judgment

[1] PCB Surrogates-TCMX non-compliance associated with Aroclor-1221, 1232, 1242, 1248 and 1016

-DCB non-compliance associated with Aroclor-1254, 1260, 1262 and 1268

If the surrogates in method blanks and/or laboratory control samples (LCS) are as low as sample surrogate recoveries, matrix effects cannot be assumed and the overall extraction efficiency is in question. The problem should be noted in the data validation notes, and the laboratory should be contacted to provide an explanation and corrective actions for future analyses.

6.8 Laboratory Control Spike/Laboratory Control Spike Duplicate Samples

An LCS consists of a portion of analyte-free water or solid phase sample that is spiked with target analytes at a known concentration. The LCS is processed through the entire method procedure with each sample batch; the results are examined for target analyte recovery. In analytical batches where an MS/MSD sample is not available the LCS may be analyzed in duplicate. The LCS/laboratory control sample duplicate (LCSD) can be used by the laboratory in cases where the MS/MSD failed to achieve acceptable recovery and/or precision. If the LCS/LCSD fails to generate acceptable results, this should cause concern about the validity of the results for all samples in the batch.

- Verify that LCS or LCS/LCSD were prepared and analyzed at the proper frequency (one per batch of 20 samples).

If LCS or LCS/LCSD were not prepared and analyzed at the proper frequency, note this in the data validation narrative.

- Spot-check LCS or LCS/LCSD recovery calculations from raw data (FDV only).
- Check that the control limits used are the appropriate limits for the project.

If errors are determined in the control limits or calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.

- Assess recoveries against established laboratory control limits.

If outlying analyte recoveries are identified, qualify all associated sample results in that sample batch as summarized below. If the LCS or LCSD recoveries indicate a systematic error (e.g., recoveries for all spiking compounds are low), qualifications of all compounds (spiking and non-spiking compounds) can be performed.

Table 6.8.1 - LCS and/or LCSD Validation Action-Organics

		Action[2]		
Assessment Element	Failure[1]	Non-Detects	Positive Results	Reason Code[3]
LCS or LCSD Accuracy	%R>UCL	None	J+	LCQ or LCD
	%R≥ 10% but<LCL	UJ	J-	
	%R<10% (GHD)	R	J-	
LCS/LCSD Precision	RPD> lab control limit	NA	J	LCD

Notes:

[1] Organic LCS Criteria - List of target compounds the laboratory will utilize are presented with each sample delivery group. The criteria used in validation will be those limits statistically generated by the laboratory, and may change periodically. Therefore the limits used for validation are referred to as the upper control limit (UCL) or lower control limit (LCL).

[2] GHD Modification to NFG Qualifications:

- Aroclor-1016 Non-compliance is representative of Aroclor-1221 through 1248
- Aroclor-1260 Non-compliance is representative of Aroclor-1254 through 1268

[3] Use LCD reason code for any data qualifications related to LCS/LCSD accuracy and/or precision. Use LCQ reason code for any data qualifications related to accuracy when LCS only is analyzed.

Table 6.8.2 - LCS and/or LCSD Validation Action-Inorganics

			Action		
Assessment Element	Criteria	Failure[1]	Non-Detects	Positive Results	Reason Code
LCS or LCSD Accuracy	70-130%R	<40%R	R	J-	LCQ or LCD
		40-69%R	UJ	J-	
		>130%R	None	J+	
	50-150%R for Ag and Sb	<20%R for Ag & Sb	R	J-	LCQ or LCD
		20-69%R for Ag & Sb	UJ	J-	
		>150%R (ICP/AES)	None	J+	
LCS/LCSD Precision	≤20% RPD	RPD>20%	NA	J	LCD

Notes:
[1] Inorganic LCS Criteria - The criteria used in validation may be those limits statistically generated by the laboratory, and may change periodically. Therefore the limits used for validation are referred to as the upper control limit (UCL) or lower control limit (LCL).
[2] Use LCD reason code for any data qualifications related to LCS/LCSD accuracy and/or precision. Use LCQ reason code for any data qualifications related to accuracy when LCS only is analyzed.

6.9 Laboratory Duplicate and Matrix Spike Samples – Organic & Inorganic Analyses

The matrix (or laboratory) duplicate and matrix spike samples analyses are designed to demonstrate and provide information about method precision and/or accuracy.

Only project specific matrix (laboratory) duplicate or matrix spike samples are considered when evaluating and qualifying samples. Samples identified as blanks or performance evaluation (PE) samples cannot be used.

- Verify that the DUP, MS, or MS/MSD were prepared and analyzed at the proper frequency (typically one per sample batch).
- If the DUP, MS, or MS/MSD were not prepared and analyzed at the proper frequency, note this in the data validation notes.
- Spot-check MS recovery calculations from raw data (FDV only).
- Spot-check RPD calculations for the DUP (FDV only).

If errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be revised and resubmitted.

Assess recoveries against control limits.

If outlying analyte recoveries are observed, qualify results for all associated samples (samples prepared in the same analytical batch) if the samples are sufficiently similar. Professional judgment will need to be exercised to determine sample similarity. The determination of similarity will include, but not be limited to, the review of the following information: site and sampling documentation, field test data, and laboratory data for other parameters.

6.9.1 Matrix (or Laboratory) Duplicate Samples

The matrix (or laboratory) duplicate (DUP) demonstrates/assesses method precision by the laboratory at the time of the analysis. It also provides a means to generate data that determines the long-term precision as it applies to various matrices.

Table 6.9.1 - Matrix (or Laboratory) Duplicate Validation Action

		Action		
Assessment Element	Failure[1]	Non-Detects	Positive Results	Reason Code[2]
Precision	RPD (if both values are >5x RL) Waters >20%, Soils>35%	UJ	J	DUP
	RPD>100	Prof. Judgment	Prof. Judgment	DUP
	RL (if either value is <5x RL), Waters>1x RL value (GHD) Soils >2x RL value (GHD)	UJ	J	DUP

Notes:

1 Laboratory may develop specific criteria.

2 Use the DUP reason code for any data qualifications related to precision when MD is analyzed.

6.9.2 Matrix Spike/Matrix Spike Duplicates – Organic Analyses

An MS consists of a sample fortified with a known amount of a target analyte and is typically analyzed in duplicate (MS/MSD). Analysis of the MS/MSD and comparison with the unspiked sample result provides the ability to assess accuracy and precision of the method on a given sample matrix. MS/MSD recoveries are used for a qualitative indication of accuracy due to matrix effects. The relative percent difference (RPD) between the recoveries is used for a qualitative indication of precision.

Only project-specific MS/MSD results are considered when evaluating and qualifying sample results. Samples identified as field blanks or performance evaluation (PE) samples cannot be used for MS/MSD spiking.

- Verify that MS/MSDs were prepared and analyzed at the proper frequency (typically one per sample batch, up to 20 samples).
- If MS/MSDs were not prepared and analyzed at the proper frequency, note this in the data validation report.
- Spot-check MS/MSD recovery calculations from raw data.
- Spot check RPD calculations.

- If errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.
- Assess recoveries against established control limits.

If outlying analyte recoveries are identified, qualify all associated results for that sample only as summarized below. MS/MSD outliers observed in the organic analyses are associated with the parent sample only. Outlying results for multiple MS/MSD sample recoveries can indicate a systematic error (e.g., recoveries for multiple MS/MSDs show similar biases). In such cases, results for all associated samples in that batch may be qualified.

In cases where the original sample concentrations are significantly greater than the spiking concentrations (>4x), matrix spike recoveries cannot be assessed. This should be noted in the data validation report, and the associated samples are not qualified.

Sample dilutions of 5 times and greater (with the exception of volatile analysis) can prohibit MS/MSD recovery assessment. In such cases, it should be noted in the validation report that the MS/MSD recoveries and RPDs could not be assessed.

- Assess RPD values against laboratory established control limits.

If outlying MS/MSD percent recoveries or RPDs are identified, qualify all associated positive results for that sample only as follows:

Table 6.9.2 - MS/MSD-Organics Validation Action

		Action[2]		
Assessment Element	Criteria[1]	Non-Detects	Positive Results	Reason Code
VOC/Pest/PCBs MS/MSD Accuracy (Recovery)	%R> UCL (Both MS & MSD)	None	J	MSD
	%R≤ LCL but > 20% (Both MS & MSD) (GHD)	UJ	J	
	%R<20% (Either MS or MSD) (GHD)	R	J	
SVOCs MS/MSD Accuracy (Recovery)	%R> UCL (Both MS & MSD)	None	J	MSD
	%R≤ LCL but > 10% (Both MS & MSD) (GHD)	UJ	J	
	%R<10% (Either MS or MSD) (GHD)	R	J	
All Organics-MS/MSD Precision	RPD>UCL	None	J	

Notes:

[1] List of target compounds the laboratory will utilize are presented with each sample delivery group. The criteria used in validation will be those limits statistically generated by the laboratory, and may change periodically. Therefore the limits used for validation are referred to as the upper control limit (UCL) or lower control limit (LCL).

[2] GHD Modification to NFG Qualifications:
- Aroclor-1016 Non-compliance is representative of Aroclor-1221 through 1248
- Aroclor-1260 Non-compliance is representative of Aroclor-1254 through 1268

6.9.3 Matrix Spike/Matrix Spike Duplicates/Post Digestion Spike – Inorganic Analyses

A spiked sample is a sample fortified with a known amount of a target analyte. Analysis of the spiked sample and comparison with the unspiked sample results provides the ability to assess accuracy of the method on a given sample matrix.

If the spike is added to the sample before the digestion (i.e. prior to reagent addition), it is referred to as the matrix spike (MS). If the spike is added to the sample after completion of the digestion, it is referred to as the post-digestion spike (PS).

Precision may also be assessed through analysis of a matrix spike duplicate (MSD). MS/MSD recoveries are used for a qualitative indication of accuracy (bias) due to matrix effects. The RPD between the recoveries (MS/MSD) is used to assess precision.

In cases where the original sample concentrations are significantly greater than the spiking concentrations (>4x), MS recoveries cannot be assessed. Since the accuracy cannot be adequately determined, accuracy is considered not calculable (NC).

- Matrix spikes are not required for wipe or filter samples
- When the MS recovery falls outside of the control limits and the sample result is < four times the spike added, a PS shall be performed for those analytes that do not meet the specified criteria. An aliquot of the unspiked sample shall be spiked at two times the sample level or the CRQL, whichever is greater.
- PS samples are not required for silver.

If MS, PS, or MS/MSD data do not meet the technical criteria, qualify all associated positive results as indicated in the following table. Professional judgment should be used to determine sample similarity. Use should be made of all available data when considering similarity in a matrix. It may be determined that only some of the samples (or none of the samples) are sufficiently similar to the MS to be qualified based on the MS and the supporting PS results.

Table 6.9.3 - MS/MSD/PS-Inorganics Validation Action

		Action		
Assessment Element	Failure[1]	Non-Detects	Positive Results >QL	Reason Code[2]
Metals-ICP/AES and ICP/MS (MS/PS Recovery)	MS %R<30% (MS and MSD) PS %R<75%	R	J-	MSQ
	MS %R<30% PS %R≥ 75%	UJ	J	MSQ
	MS %R=30-74% PS %R<75%	UJ	J-	MSQ
	MS %R=30-74% PS %R≥ 75%	UJ	J	MSQ
	MS %R>125% PS %R>125%	None	J+	MSQ
	MS %R>125% PS %R<125%	None	J	MSQ
	MS-%R<30% (MS OR MSD) PS None analyzed	R	J-	MSQ

		Action		
Assessment Element	Failure[1]	Non-Detects	Positive Results >QL	Reason Code[2]
	MS %R=30-74% PS None analyzed	UJ	J-	MSQ
	MS %R>125% PS None analyzed	None	J+	MSQ
Metals-Mercury (Recovery)	MS %R<30%	R	J-	MSQ
	MS %R=30-74%	UJ	J-	MSQ
	MS %R>125%	None	J+	MSQ
General Chemistry MS and/or Post-Distillation (PS) Spike	MS %R<30% PS %R<75%	R	J-	MSQ
	MS %R<30% PS %R≥ 75%	UJ	J	MSQ
	MS %R=30-74% PS %R<75%	UJ	J-	MSQ
	MS %R=30-74% PS %R≥ 75%	UJ	J	MSQ
	MS %R>125% PS %R>125%	None	J+	MSQ
	MS %R>125% PS %R<125%	None	J	MSQ
	MS %R<30% PS None	R	J-	MSQ
General Chemistry MS and/or Post-Distillation (PS) Spike	MS %R=30-74% PS None	UJ	J-	MSQ
	MS %R>125% PS None	None	J+	MSQ
Metals MS/MSD or MD Precision	RPD (if both values are >5x RL) Waters>20%, Soils>35%	None	J	MSD
	RL (if either value is <5x RL), Waters>1xRL value, Soils>2xRL value	None	J	MSD
General Chemistry MS/MSD or MD Precision	RPD (if both values are >5xRL) Waters>20%, Soils>35%	None	J	MSD

Notes:
[1] Laboratory may develop specific ICP/MS criteria.
[2] Use MSD reason code for any data qualifications related to MS/MSD accuracy and/or precision. Use the MSQ reason code for any data qualifications related to accuracy when MS only is analyzed. Use the DUP reason code for any data qualifications related to precision when MD is analyzed.

6.10 Internal Standards

Internal standards (IS), assessed during FDV only, are used as the quantitation and relative retention time standard for the target analytes analyzed by MS or methods that require internal standardization. Low area counts for the internal standards translate into falsely high reported values for target analytes in the samples. Any reported value for an analyte is an estimate when high or low IS area counts are reported.

- Verify that all samples are accounted for on the IS review forms.
- Spot check for transcription errors between the IS area counts reported on the summary form, and the raw data from calibration and sample analyses.
- Spot check the IS area range calculations.
- Spot check IS recoveries for the samples.
- Spot check that the retention times (RT) are within criteria for all samples and blanks.

If there are errors in the IS summaries, or errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.

- Check IS recoveries against the control limits in Table 6.10.1. If outliers are noted, verify that samples were re-injected. Where there are unacceptable IS recoveries, followed by acceptable re-analyses, verify that the designated successful analysis was reported in the flat file.
- Verify that all internal standards were recovered.
- Check that the IS brackets the masses of target analytes (ICP/MS).
- Verify when outliers are observed during ICP/MS analysis, a twofold dilution is performed and a reanalysis of the calibration blank is performed.
- For IS recoveries outside of the control limits, only results for those analytes (contact the laboratory for compounds associated to each internal standard) calculated from the outlying ISs will be affected as follows:

Table 6.10.1 - Internal Standard Criteria and Validation Action – Organics

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
Trace VOCs, Low/Med VOCs, SVOCs IS Recovery	<20%	R	J+	IST
	≥20% but <50% [1]	UJ	J+	IST
	>200%	None	J-	IST
	RT shift >10.0 seconds between sample and 12-hour standard	R	R	IST
Pesticides & PCBs [2] IS Recovery	<20%	R	J+	IST
	≥20% but <50% [1]	UJ	J+	IST
	>200%	None	J-	IST
	RT shift >10.0 seconds between sample and 12-hour standard	R	R	IST

Notes:

[1] Detects should not need to be qualified as unusable "R" if the mass spectral criteria are met; for area counts in the range of 20-50%, non-detected compounds may be qualified as UJ based on further evaluations on the data

[2] Pesticide and PCB internal standard criteria are not included in the 2016 NFGs. The 2016 NFG criteria will be adopted for SW-846 methods for consistency sake.

Table 6.10.2 - Internal Standard Criteria and Validation Action – Inorganics

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
ICP/AES IS Recovery [1]	<30%	R	J	IST
	>125%	Prof. Judgment	J	IST
ICP/MS IS Recovery	<60% or >125% and original sample reanalyzed at a 2-fold dilution	UJ	J	IST
	Original Sample not reanalyzed at a 2-fold dilution after IS failure	UJ or R (Prof. judgement)	J	IST
Presence of Internal Standards (ICP-MS)	IS not present or <5 of the required internal standards	R	R	IST
	Target analyte not associated with an IS	R	R	IST
	No IS	R	R	IST

Notes:

[1] Advisory limits based on SW-846 Method 6010

6.11 Interference Check Sample-Metals

The interference check samples ICS (ICSA and ICSAB) assessed during FDV only, are analyzed at the completion of daily calibration and at the end of operation (minimum of two analyses per 8 hours of run time). The ICS data should be carefully checked for positive and negative target analyte results greater than the absolute value of the IDL for elements not included in the ICSA or ICSAB solutions. Positive results are an indication of either laboratory contamination of the ICS test solutions or an improperly generated interelement correction factor for that element. The latter situation can lead to false positive results on samples.

Aqueous samples that exhibit levels of the four ICSA interferents (Al, Ca, Fe and Mg) at least as high as those present in the ICSAB (500, 500, 200, and 500 milligrams per liter [mg/L], respectively) can be examined for potential false positive and false negative results based on interference correction factor deficiencies. Application of these guidelines to soil samples requires that reduced sample size and dilution effects upon the levels of the four ICSA elements be taken into account. These calculate to be 100,000 milligrams per kilogram (mg/kg) for Al, Ca and Mg, and 40,000 mg/kg for Fe. If the low-level concentrations are used for the ICSA, then the guidelines apply to all recoveries, regardless of the sample concentrations of the interfering elements. The guidelines, which apply, are noted below.

- Check lab data to ensure that the interference check samples were analyzed at the proper frequency.
- Spot check for transcription errors between the ICS recoveries reported on the summary form, and the raw data. Recalculate from the raw data one or more of the analyte percent recoveries and compare to the laboratory reported recovery on the summary form.
- Evaluate the ICS raw data for results with an absolute value greater than the IDL for those analytes that are not present in the ICS solution. If results greater than the IDL are observed an

evaluation of the associated sample data for the affected elements should be made. If aluminum (Al), calcium (Ca), iron (Fe), and magnesium (Mg) equals ICS levels, a potential for false positive results exist and professional judgment should be used.

- Evaluate recovery and indicate whether the evaluation was performed based on the NFG criteria or laboratory specific criteria.

If interference check samples were not analyzed at the proper frequency, use professional judgment to determine the usability of the data.

Table 6.11.1 - ICSA/ICSAB Criteria and Validation Action

		Action[1]		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
ICS Recovery (ICP/AES and ICP/MS)	ICSAB <50%R	R	J-	ISI
	ICS 50-79%R (or ICS <true value - 2x CRDL), whichever is lower	R	J-	ISI
	ICS >120%R (or ICS>true value +2x CRDL), whichever is greater	None	J+	ISI
	ICS >150%R	Prof. Judgment	Prof. Judgment	ISI
	Potential false positives in field samples w/interferents	NA	J+	ISI
	Potential false negatives in field samples w/interferents	UJ	J-[2]	ISI
	ICS not analyzed	R	R	ISI
	ICS not analyzed in proper sequence	Prof. Judgment	Prof. Judgment	ISI

Notes:

[1] Action is based on the fact that Al, Ca, Fe, or Mg are present at least as high as ICSAB solution Al, Ca, Mg (500 mg/L or 100,000 mg/kg) and Fe (200 mg/L or 40,000 mg/kg)

[2] Results $\geq$ MDL but < 10 x (negative value)

6.12 Serial Dilution Sample – Inorganics

The serial dilution of samples quantitated by ICP and ICP/MS determines whether or not significant physical or chemical interferences exist due to sample matrix. A serial dilution of 1:5 must be performed on at least one sample from every batch of analyses by ICP and ICP/MS to determine if physical or chemical interferences exist in the analyte determinations. Field blanks cannot be used to fulfill the serial dilution requirement. If the analyte in the sample is at least 50 times the value of the MDL for ICP and ICP/MS respectively, then the percent difference between the value obtained from the 1:5 dilution and the undiluted value must be within 10 percent. Serial dilution data assessment is completed during FDV only.

- Check lab data to ensure that the serial dilution samples were analyzed at the proper frequency.
- Check the raw data and recalculate the percent difference (%D). Verify that the serial dilution analysis results and the calculated %D agree with the values reported on the summary form.

- Check the raw data for any evidence of negative interference (results from the diluted sample that are significantly higher than the original sample), possibly due to high levels of dissolved solids in the sample, etc.

Table 6.12.1 - Serial Dilution Criteria and Validation Action

		Action[1]		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
% Difference	%Difference > 10% (Detects >50xMDL)	UJ	J	ISD
Interferences	Interferences present	Prof. Judgment	Prof. Judgment	ISD

6.13 Tentatively Identified Compounds-GC/MS

Tentatively identified compounds (TICs) are only provided upon request and are reviewed during FDV only. TICs are found in the samples that elute from the GC/MS as defined peaks, yet are not calibrated analytes. Up to 30 peaks (10 per VOC and 20 per SVOC fractions) greater than 10 percent in area or height of the nearest internal standard are candidate TICs. The following cautions are appropriate when considering spectral matches from these libraries:

- Library spectra used may not be obtained from mass spectrometers tuned to meet BFB/DFTPP acceptance criteria.
- The source of the compound/spectra in the library may be of questionable integrity (identifications are simply wrong).
- Some of the spectra may have been background corrected to remove artifact signals and others have not.
- In the interest of conservation of storage space, the spectra have been reduced to eliminate many of the small signals, leaving just the major signals.
- Check regional regulations for state specific criteria or the NFGs for additional CLP guidelines.
- Check that all TICs include a raw spectra, an enhanced spectra and library matches. For TICs that are not reported as "unknown":
- Review mass spectra for each TIC identified and compare to the library match selected by the laboratory.
- Ensure that major ions are present in the sample mass spectra.
- Spot-check relative intensities of major ions to verify that they are within 20 percent of the expected values.
- Check to see that all positive identifications have library matches greater than 85 percent.

If there are questions regarding compound identification, contact the laboratory to discuss and obtain corrected forms if needed.

If TICs are identified in blanks at concentrations similar to those in the samples, these TICs should be considered contamination and identified by the laboratory as such.

If any target analytes from another fraction are identified as TICs (e.g., naphthalene in the VOC analysis), check to see that the compound was reported as a target analyte with the proper fraction. If the analyte was reported with the proper fraction, disregard it as a TIC in the other fraction; do not report the analyte in both fractions.

The following TICs are common laboratory artifacts or analytical by-products. If these compounds are not of specific interest to the site, the presence of these compounds should be noted in the data validation memo and they should be disregarded as TICs.

- Common laboratory contaminants: CO_2, siloxanes, diethyl ether, hexane, certain freons, and low level phthalates.
- Solvent preservatives such as cyclohexane and its related by-products (cyclohexanone, cyclohexanol, chlorocyclohexane and chlorocyclohexanol).
- Aldol condensation products (4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-pentene-2-one, and 5,5-dimethyl-2(5H)-furanone).

All TIC data must be qualified as estimated (J). TICs that are identified by the laboratory with a specific compound name, a Chemical Abstracts Service Registry Number (CASRN), and meet the review criteria above are qualified with "NJ", indicating that presumptive evidence of the compound exists and the reported concentration is estimated.

It should be noted that the laboratory may have a more extensive list of calibrated compounds than the requested compound list, and these calibrated compounds may be reported as TICs. Non-target, calibrated compounds reported as TICs are qualified as estimated (J) because they have not been reviewed in the same manner as the target compounds. The data validation report should include a discussion and tabular summary (if appropriate) of these TICs.

Table 6.13.1 - Tentatively Identified Compounds and Validation Action

Assessment Element	Reported Compound	Action Positive Results	Reason Code
TIC-calibrated	TIC	NJ	TIC
TIC-non-calibrated	TIC	J	TIC
TIC Contaminants	TIC-Common Lab Contaminants	Disregard (Reportable NO)-Note in DV Memo	NA

6.14 Analyte Identification - Organics

Spot check positive sample results to ensure that retention times are within the established retention time windows at a 10 percent frequency.

For GC/MS, spot-check mass spectra at a 10 percent frequency during FDV as follows:

- Perform a visual comparison of sample mass spectra against reference spectra to ensure a general match.
- Verify that all major ions are present and their intensities are within 20 percent of the expected values.
- Review ions in the sample with >10 percent abundance that are not in the reference spectra.

If there are any discrepancies in analyte identification, the laboratory must be contacted for resolution and corrected forms submitted if necessary. Qualification of data due to analyte identification discrepancies are summarized below.

Table 6.14.1 - Analyte Identification Approval Codes

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
Second Column Confirmation	%RPD>40%≤80% (GHD)	NA	J	DCD
	%RPD>80% (GHD)	NA	NJ	DCD
Chromatography	Poor chromatography	Prof. Judgment	Prof. Judgment	QUA
Carry over contamination	Suspected instrumental carryover	Prof. Judgment	Prof. Judgment	SIC

6.15 Analyte Quantitation

Analyte quantitation is verified at a 10 percent frequency during FDV with the following checks:

- Spot check sample report limits for non-detected analytes (for organics and general chemistry analytes, use the lowest calibration standard to verify the quantitation limit; for metals, use the MDL to verify the report limit), making sure that report limits reflect individual original sample weights/volumes, final volumes, percent moisture (where applicable), dilutions, etc.
- Spot-check random positive sample results.
- Note in the data validation text whether values below the report limit were reported and qualified as estimated (lab "J" flagged values). These analytes are qualified as J values with the BRL reason code.
- Review replicate or quadruplicate results as specified in methods.
- Note in the data validation text whether soil results were reported on a dry-or wet-weight basis.
- If dilutions were performed, ensure that values above the calibration range (E values) are not used unless absolutely necessary (these values should be qualified as estimated). Evaluate original and diluted results to ensure they are comparable.

If there are any discrepancies in analyte quantitation, the laboratory must be contacted for resolution and corrected forms submitted if necessary. Qualification of data based on analyte quantitation discrepancies observed are summarized below.

Table 6.15.1 - Analyte Quantitation Approval Codes

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
Exceeds Calibration Range	Sample results exceeds instrument calibration range	NA	J	EXE
Method of Standard Addition	Percent recovery outside method criteria	UJ	J	MSA

		Action		
Assessment Element	Failure	Non-Detects	Positive Results	Reason Code
Variability	Variability in replicate results	Prof. Judgment	Prof. Judgment	REP
Precision-Metals	%RSD outside criteria for replicate aspiration	Prof. Judgment	Prof. Judgment	RSP
Percent Solids[1]	Percent solids less than 30%	UJ	J	SLD
Variability in Sample Results	Dissolved analyte result significantly greater than total (>20%D)	UJ	J	TVD
Dilutions	Validator's choice of dilution	Prof. Judgment	Prof. Judgment	VCD
Reanalysis	Validator's choice of reanalysis	Prof. Judgment	Prof. Judgment	VCR

Notes:

[1] Percent solids qualification applies to soil samples only. Sediment samples do not require qualifications based on percent solids; however, low solids should be noted in the validation memo.

6.16 Field Duplicates

Overall precision for the sampling event and laboratory procedures is monitored through the collection and analysis of field duplicate sample sets.

- Compare results for original sample and field duplicate and calculate the RPD. If one or more results are non-detect, use the report limit or MDL value to calculate the RPD.
- Compare the RPDs to the project control limits. If limits have not been established, use default limits of 50 percent for water and 100 percent for soil samples if the sample results are greater than five times the RL. If sample results are less than five times the RL, precision is assessed by comparing the difference between the two results to a control limit of plus or minus one time the RL value for water samples and two times the RL value for soil samples.

If outlying RPDs are identified, qualify all associated positive results for that sample and it's duplicate as summarized below. If the field duplicate results show drastic differences, check with the sampler to ensure proper field duplicate identification, then have the laboratory verify proper sample labeling and analyte quantitation.

Table 6.16.1 - Field Duplicate Sample Assessment

		Action		
Assessment Element	Failure	Non-Detects	Sample Detections	Reason Code
Precision Sample or duplicate concentrations > 5xRL	Water sample RPD >50%	UJ	J (GHD)	FDP
	Air sample RPD >50% (Advisory)	UJ	J (GHD)	FDP
	Soil sample RPD >100%	UJ	J (GHD)	FDP
Sample or duplicate concentrations <5xRL	Water sample difference >1xRL	UJ	J (GHD)	FDP
	Air sample difference >1xRL	UJ	J (GHD)	FDP
	Soil sample difference >2xRL	UJ	J (GHD)	FDP

Note:

If one sample is non-detect, use the reporting limit (RL or QL) for calculation of RPD/difference if the second sample is >RL.

7. Lab Resubmission Documentation

Corrections to laboratory reports must be requested through email and the request should be sent to email filing. The laboratory must provide the entire revised report in PDF form through email. If the revised report is too big to email, arrangements can be made to download it from the laboratory's website, or it can be provided via mailed disk.

The revised package must be saved electronically to the project file either in email filing, in WP or in Laserfiche.

If a complete package resubmission is requested, the obsolete package must be marked "obsolete" and the new package include a summary of the changes in the report narrative.

8. Flat File Checks

Prior to extracting an analytical table from the flat file, the following checks must be performed:

i) Ensure for each sample that there is only one result designated "reportable" for all associated analyte

ii) Verify that acceptance codes for all results reflect the level of data review performed by the validator

iii) Ensure that all lab qualifiers have been addressed and converted if necessary to data validation qualifiers (e.g., B, E, *, etc.)

iv) Ensure that all validation qualifications have appropriate Reason Codes

v) Ensure that all results qualified "U" or "UJ" have "N" in the detected field.

To automatically do this, there is a program located under the Add-Ins/Check Flat file tab, labeled "flat file completeness checker", that quickly checks the final flat file for consistency and completeness. For example, the checker will identify inconsistencies like results that have "U" listed under "validator_qualifier" field, and a "Y" populated in the "detect_flag" field. This checker must be run and all errors addressed before uploading the flat file to database.

9. Report Format

Data validation memo/report should include the following:

i) An introduction detailing the objective of the sampling and analysis program; general sample collection information; the analytical methods used; and a reference of all pertinent quality documents

ii) Text describing the elements of the data package that were reviewed, the findings for each, and the impact each finding had on the overall data usability

iii) A sampling and analysis summary table

iv) Tables summarizing all data qualification and the rationale for each

Example validation memos, reports and table templates are located in the chemistry peer to peer section of the portal.

10. References

- "National Functional Guidelines for Superfund Organic Methods Data Review", USEPA-540-R-2016-002, September 2016.
- "National Functional Guidelines for Inorganic Superfund Methods Data Review", USEPA-540-R-2016-001, September 2016.
- USEPA Guidance on Environmental Data Verification and Data Validation, USEPA QA/G-8, November 2002.
- USEPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, USEAP-540-R-05-005, 13 January 2009.

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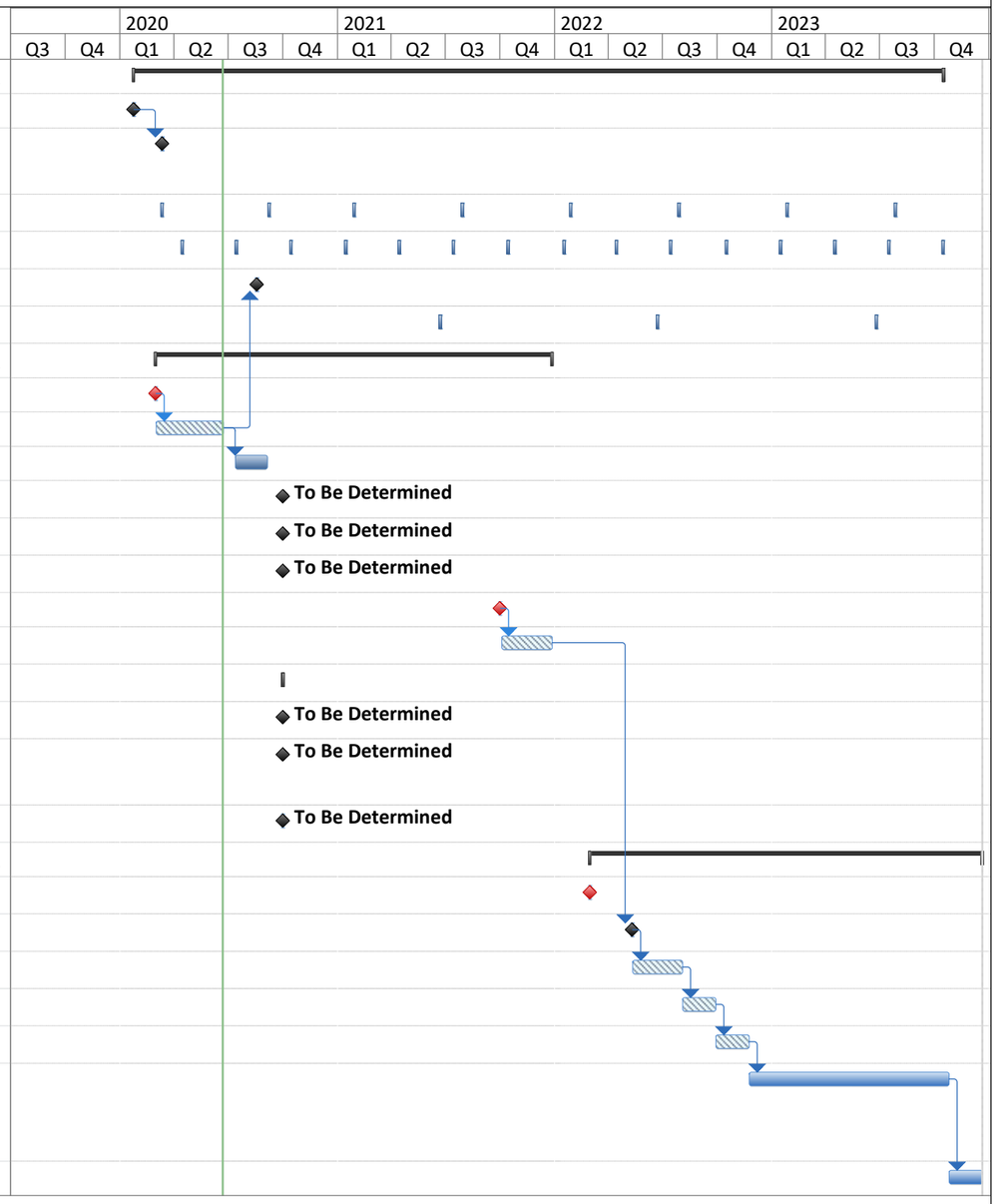
Appendix C
Conceptual Schedule

Draft Project Schedule - AOC
Former Cities Refinery
East Chicago, IN

ID	Task Name	Duration	Start	Finish
1	**Administrative**	**965 days**	**Fri 1/24/20**	**Mon 10/16/23**
2	Effective Date of AOC	1 day	Fri 1/24/20	Fri 1/24/20
3	Met with U.S. EPA to discuss objectives, expectations, and timelines and provide to EPA for approval a draft CAF (Unless otherwise agreed to by the parties, within 45 Days of the effective date of the Order)	1 day	Thu 3/12/20	Thu 3/12/20
4	Semi-Annual Meetings - Meeting with EPA either by phone or in person on at least a semi-annual basis	874 days	Thu 3/12/20	Fri 7/28/23
13	Quarterly Reports - Fifteenth day of the month after the end of each quarter	906 days	Wed 4/15/20	Mon 10/16/23
29	Financial Assurance - Within 60 days of EPA approval of the RFI workplan (initial estimated cost of work)	1 day	Tue 8/18/20	Tue 8/18/20
30	Financial Assurance Update (Updated annually and/or with subsequent work plans)	523 days	Wed 6/23/21	Mon 6/26/23
34	**RFI**	**469 days**	**Sun 3/1/20**	**Mon 12/27/21**
35	Submit RFI Workplan (With SAP/QAPP) - No later than March 1, 2020	1 day	Sun 3/1/20	Sun 3/1/20
36	EPA Review and Approval of RFI Work Plan	4 mons	Mon 3/2/20	Mon 6/22/20
37	RFI Field Activities	2 mons	Mon 7/13/20	Fri 9/4/20
38	Additional RFI Work Plan(s), as needed	1 day	Thu 10/1/20	Thu 10/1/20
39	EPA Review and Approval of RFI Work Plan(s), as needed	1 day	Thu 10/1/20	Thu 10/1/20
40	RFI Field Activities, as needed	1 day	Thu 10/1/20	Thu 10/1/20
41	Submit RFI Report - No later than October 1, 2021	1 day	Fri 10/1/21	Fri 10/1/21
42	EPA Review and Approval of the RFI Report	3 mons	Mon 10/4/21	Mon 12/27/21
43	**Interim Corrective Measures, as needed**	**1 day**	**Thu 10/1/20**	**Thu 10/1/20**
44	Interim Corrective Measures Work Plan(s), as needed	1 day	Thu 10/1/20	Thu 10/1/20
45	Interim Corrective Measures Implementation, as needed (90 days following submittal of Interim Corrective Measures Work Plan and Schedule)	1 day	Thu 10/1/20	Thu 10/1/20
46	Interim Corrective Measures Report (Either prior to or as part of the Environmental Indicators Report)	1 day	Thu 10/1/20	Thu 10/1/20
47	**Final Corrective Measures**	**472 days**	**Tue 3/1/22**	**Wed 12/20/23**
48	Environmental Indicators Report - No later than March 1, 2022 (unless EPA agrees to extend that deadline)	1 day	Tue 3/1/22	Tue 3/1/22
49	Final Corrective Measures Proposal (Including schedule) - Within 135 days of EPA's approval of the RFI report	1 day	Wed 5/11/22	Wed 5/11/22
50	EPA review and comments, supplemental information / investigation request	3 mons	Thu 5/12/22	Wed 8/3/22
51	Public review and comment	2 mons	Thu 8/4/22	Wed 9/28/22
52	EPA Final Decision	2 mons	Thu 9/29/22	Wed 11/23/22
53	Final Corrective Measures Implementation - (According to the schedule in the Final Decision, i.e. much of the construction completed within one year, or within a reasonable period of time after EPA selects the final corrective measures)	12 mons	Thu 11/24/22	Wed 10/25/23
54	Final Remedy Construction Completion Report (Including an Operation and Maintenance Plan)	2 mons	Thu 10/26/23	Wed 12/20/23



Project: Former Cities Refinery, East Chicago, IN
Date: Mon 6/22/204:05 PM

Expected Timeframe
Contingent Timeframe
Expected Date
Fixed Date
Summary

Note: Per AOC, 'days' refers to calendar days
Schedule subject to change based on regulatory reviews/approvals, weather, and other factors beyond Respondent's control

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DRAFT

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