Supplement A

Quality Assurance Project Plan

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Quality Assurance Project Plan

Failure Analyses of Underground Storage Tank Equipment in Biofuels Service II

Investigation of Corrosion Influencing Factors in Underground Storage Tanks with Ultra Low Sulfur Diesel Service

Version 2

Prepared for
U.S. EPA Office of Solid Waste and Emergency Response/
Office of Underground Storage Tanks

STREAMS II, Task Order 0016
Battelle Contract EP-C-11-038

Prepared by
Battelle Memorial Institute

January 16, 2015

A1 TITLE AND APPROVAL PAGE

Quality Assurance Project Plan

for

Investigation of Corrosion Influencing Factors in Underground Storage Tanks

with Ultra Low Sulfur Diesel Service

Version 2

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 U.S. EPA OSWER/OUST **Quality Assurance Manager**	**Date**
Anne Marie Gregg **Battelle** **Task Order Leader**	1/16/2015 **Date**
Elizabeth Cutié **Battelle** **Quality Assurance Manager**	1/16/2015 **Date**

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A3 LIST OF ABBREVIATIONS/ACRONYMS

ALS-CA	ALS Global laboratory in California
ALS-OH	ALS Global laboratory in Ohio
ASTM	ASTM International
CCV	continuing calibration verification
CFR	Code of Federal Regulations
COC	chain of custody
CRC	Coordinating Research Council
DQO	data quality objective
EPA	United States Environmental Protection Agency
ICFTL	Iowa Central Fuel Testing Laboratory
lpm	liters per minute
LRB	laboratory record book
LSD	low sulfur diesel
MIC	microbial influenced corrosion
NACE	National Association of Corrosion Engineers International
NIST	National Institute of Standards and Technology
NIOSH	National Institute for Occupational Safety and Health
%	percent
ppm	part per million
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RFS	Renewable Fuel Standard
RMO	Records Management Office
SOP	standard operating procedure
STP	submersible turbine pump
STREAMS	Scientific, Technical, Research, Engineering and Modeling Support
SwRI	Southwest Research Institute
TO	task order
TOCOR	Task Order Contracting Officer’s Representative
TOL	Task Order Leader
TSA	technical systems Audit
TVS	Tanknology Vacuum Sampler
ULSD	ultra low sulfur diesel
US	United States
UST	underground storage tank

A4 DISTRIBUTION LIST

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A5 PROJECT ORGANIZATION

Battelle will perform this project under the direction of the United States Environmental Protection Agency (EPA) through Scientific, Technical, Research, Engineering and Modeling Support (STREAMS) II Task Order (TO) 0016 of Contract EP-C-11-038. The organization chart in Figure 1 shows the individuals from Battelle, EPA, and Tanknology who will have responsibilities during this project. The specific responsibilities of these individuals are summarized below.

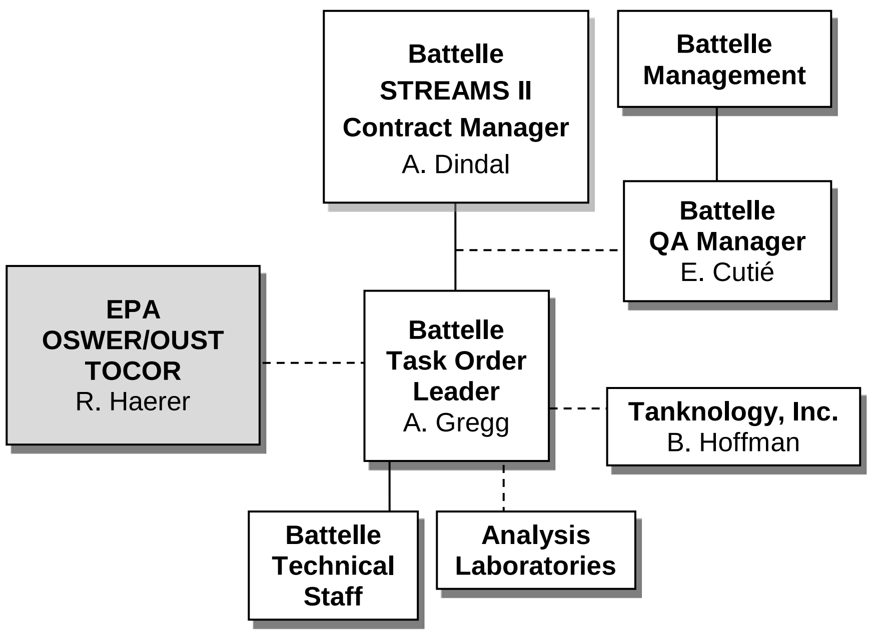

Figure 1. Project Organizational Chart

A5.1 Battelle

Ms. Anne Marie Gregg is the Battelle Task Order Leader (TOL) for this project. She will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the investigation are met, and that the procedures employed for the investigation are consistent with this quality assurance project plan (QAPP). Ms. Gregg will serve as the primary interface for EPA's Task Order Contracting Officer's Representative (TOCOR). Ms. Gregg's responsibilities are to:

- Prepare the QAPP (this document);

- Establish a budget and schedule for this project and direct the effort to ensure that the budget and schedule are met;
- Keep the Battelle STREAMS II Contract Manager informed of the progress and any difficulties in planning and conducting the investigation;
- Have responsibility for ensuring that this QAPP and any amendments are followed;
- Arrange for use of required facilities/laboratories;
- Arrange for the availability of qualified staff to conduct this project;
- Collect and review data generated during the project;
- Respond to any issues raised throughout the project, including instituting corrective action as necessary;
- Maintain all test records during the investigation and transfer them to permanent storage at the conclusion of the investigation;
- Maintain communication with the TOCOR throughout the project; and
- Prepare draft and final reports.

Ms. Amy Dindal is Battelle's STREAMS II Contract Manager. As such, Ms. Dindal will:

- Maintain communication with the EPA STREAMS II Project Officer on all aspects of the contract;
- Monitor adherence to budgets and schedules in this work;
- Provide the TOCOR with monthly technical and financial progress reports;
- Review the QAPP;
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the investigation;
- Support Ms. Gregg in responding to any issues that arise in assessment reports and audits;
- Issue a stop work order if audits indicate that data quality is being compromised; and
- Review the draft and final reports.

Ms. Elizabeth Cutié is Battelle's Quality Assurance (QA) Manager for the STREAMS II contract. Ms. Cutié will:

- Review and approve the QAPP;

- Ensure that all quality procedures specified in this QAPP are followed; and
- Perform a technical systems audit (TSA) and hold a verbal debrief of the observations and findings as soon as possible;
- Perform a data quality audit of at least 10 percent (%) of the data collected;
- Prepare and distribute assessment reports of the audits results;
- Verify implementation of any necessary corrective action;
- Notify Battelle's STREAMS II Contract Manager to issue a stop work order if internal audits indicate that data quality is being compromised. Notify the TOL if such an order is issued;
- Retain all QA records from the TO;
- Review the draft and final reports.

Several Battelle technical staff will support Ms. Gregg throughout this project. They will:

- Assist the TOL in the preparation of the QAPP;
- Assist the TOL in developing a schedule for the project;
- Work to carry out the test procedures specified in this QAPP;
- Update the TOL on progress and difficulties in planning and conducting the investigation;
- Accurately record all data and other information required by the QAPP and Battelle's policies and procedures; and
- Assist in preparing the draft and final reports.

A5.2 Tanknology, Inc.

Tanknology, Inc. is an underground storage tank (UST) inspection and testing company which will support Battelle in providing the UST site inspection, the internal video inspection, and fuel, water, and vapor sampling services during this project. Mr. Brad Hoffman is Tanknology's vice president in engineering, who will be overseeing the site inspection process for Tanknology. Mr. Hoffman will:

- Assist the TOL in the preparation of the QAPP;
- Assist the TOL in developing a schedule for the project;

- Work to ensure the test procedures are carried out as specified in the QAPP;
- Update the TOL on progress and difficulties in planning and conducting the investigations; and
- Assist the TOL in preparing the draft and final reports;

Tanknology field technicians will support Mr. Hoffman working in two-person teams and will:

- Conduct the on-site data and sample collection at each investigation site;
- Accurately record all data and other information required by this QAPP; and
- Update Mr. Hoffman on progress and difficulties in conducting the investigations.

A5.3 U.S. EPA Office of Solid Waste and Emergency Response/ Office of Underground Storage Tanks (OSWER/OUST)

Mr. Ryan Haerer, the U.S. EPA TOCOR for this project, will:

- Have overall responsibility for directing the project;
- Recruit and coordinate with owners participating in the study;
- Communicate with the TOL regularly to receive updates on the status of the project;
- Review the draft QAPP, distribute the QAPP for review and comment, and review and approve the final QAPP; and
- Review all versions of deliverables specified under this TO.

A5.4 Subcontracted Analytical Laboratories

The analytical laboratories performing various analyses are Iowa Central Fuel Testing Laboratory (ICFTL), Marathon Cattlesburg Control Laboratory, Southwest Research Institute (SwRI), Metrohm USA, Inc., TestAmerica, and ALS Global (ALS). The specific methods that will be performed by each laboratory are specified in Section B5 (Table 4). All participating laboratories will be required to meet the minimum requirements stated in Tables 5, 6, and 7 as appropriate for the sample matrices.

A6 PROBLEM DEFINITION/BACKGROUND

A6.1 Project Background and Objectives

Biofuels provide a significant contribution to the fuel supply in the United States (US). The contribution of ethanol and biodiesel for fuel blending has grown significantly through the past decade largely as a result of the of the enactment of the Renewable Fuel Standard (RFS) established by the Energy Policy Act of 2005 and amended by the Energy Independence and Security Act of 2007. These federal mandates have spurred a significant increase in biofuel production and an increase in the number of retail facilities storing and dispensing these fuels. The rapid expansion of biofuel storage in underground storage tank (UST) systems has created awareness about the different chemical properties these fuels possess and how those properties may affect how they react with the materials used in UST system construction. 40 Code of Federal Regulations (CFR) 280.32 requires that owners and operators use an UST system made of or lined with materials that are compatible with the substance stored in those UST systems. However, anecdotes from the field and laboratory testing of fuels and materials suggests that not all UST equipment in the ground today, especially in older USTs, is 100% compatible with some biofuel blends. EPA performed research on failed USTs that had been storing fuels blended with biofuels in an attempt to determine if incompatibility was the cause of those failures. However, definitively determining if incompatibility between fuels and the UST was the exact cause of failure of those UST systems proved to be difficult, because the dynamic nature of a UST environment creates difficulty in accurately identifying the chemical conditions inside of the UST prior to those failures when analyzed after the failure in a UST that has been exposed to an outside environment. Accurate analysis is also made more challenging because it is difficult to know the impacts on the failed UST of any fuel storage history prior to storing the biofuel blend.

Due to concerns over compatibility of biofuel blends with UST equipment, in June 2011, EPA issued guidance on compatibility of UST systems with biofuel blends. This document, "Guidance On Compatibility Of UST Systems With Ethanol Blends Greater Than 10 Percent And Biodiesel Blends Greater Than 20 Percent"[1], discusses how owners and operators of UST systems regulated under 40 CFR part 280 can demonstrate compliance with EPA's compatibility requirement when storing gasoline containing greater than 10 % ethanol or diesel containing greater than 20 % biodiesel. In the guidance, EPA lists the following UST system components to be critical for demonstrating compatibility:

- Tank or internal tank lining
- Piping
- Line leak detector
- Flexible connectors
- Drop tube
- Spill and overfill prevention equipment
- Submersible turbine pump (STP) and components
- Sealants (including pipe dope and thread sealant), fittings, gaskets, O-rings, bushings, couplings, and boots
- Containment sumps (including STP sumps and under dispenser containment)
- Release detection floats, sensors, and probes
- Fill and riser caps
- Product shear valve

These components are likely to be in direct contact with fuel, and if incompatible with the fuel, the failure of such components may lead to a release to the environment. For more background information regarding the compatibility of UST system components with biofuel blends, EPA's guidance can be found at: http://www.epa.gov/oust/altfuels/biofuelsguidance.htm.

In addition to the awareness of possibility material compatibility issues between biofuels and UST construction materials during the last decade, UST owners and operators around the country are continuing to report corrosion in USTs storing ultra-low sulfur diesel (ULSD). The reports of corrosion began around 2007 and generally correlate with the enactment of the RFS as well as the reduction of the maximum allowable sulfur content in highway use diesel from 500 parts per million (ppm) in low sulfur diesel (LSD) to 15 ppm in ULSD. EPA is hearing that corrosion can be severe and have a very rapid onset, and that sometimes in as little as six months after a new installation, STP shafts corroded to the extent that the shafts had to be replaced. Other metal components inside of the tank may be affected as well. Because this problem has been identified and reported only over the last seven years, there has been little investigation into the causes of corrosion or what to do about it, other than an initial hypotheses investigation by industry in 2012 based on results of examining six USTs. Verifiable data is limited, but anecdotes are revealing that experiences of the corrosion can be sporadic.

UST systems have several pieces of release prevention equipment, each of which must move freely or seal correctly as it was designed to do, so it performs the release prevention function correctly. However, EPA has heard from regulators and servicing companies, which perform required functionality testing in the field, that some of these components in USTs storing ULSD over the last few years are failing functionality testing at rates much higher than average; this could be associated with the increased reports of corrosion.

Figure 2. Example of Observed Corrosion on an STP

A previous industry study attempting to identify the most likely hypotheses on which to perform further research examined corrosion in six fiberglass USTs experiencing this corrosion. (One UST was to be a control tank free of corrosion, but after inspection corrosion was identified.)   The results of the hypotheses investigation suggested the corrosion is likely due to acetic acid distributed throughout the system. The hypothesis is that ethanol is contaminating the ULSD fuel supply (through cross or switch loading of fuel transportation trucks with no or inadequate cleanings between shipments or through other methods). The ethanol is being oxidized by *Acetobacter* bacteria living in the USTs. A byproduct of the microbial metabolism is acetic acid. The combination of these factors is the potential cause of the corrosion and is considered microbial influenced corrosion (MIC).[2] However, the results of the investigation also identified other possible hypotheses that could be tested based on findings of additional acids that usually result from a similar MIC pathway in which related bacteria species could oxidize glycerol present in ULSD, instead of ethanol contaminating the ULSD.

USTs storing ULSD may contain biodiesel blended up to 5 % by ASTM D975[3] standard for diesel fuel. This biodiesel in turn may contain glycerol, a byproduct of the production process. Most glycerol is removed after biodiesel production, but a small concentration is allowed to remain by the fuel standard. Glycerol may therefore be supplying the energy source for bacteria species closely related to *Acetobacter*, resulting in the production of more corrosive small molecular weight organic acids like highly corrosive propionic, lactic or glyceric acids. The bacterial metabolism may also result in a lowering of the pH of the UST environment and play a role in the rapid and severe episodes of corrosion being seen in ULSD tanks. This TO will evaluate the potential production of small molecular weight organic acids, including acetic acid, through MIC pathways.

This project aims to investigate multiple operational USTs (up to 42). The study will attempt to examine USTs both currently experiencing both rapid and severe corrosion incidents as well as tanks that are experiencing minimal or moderate corrosion, and will attempt to determine what chemical conditions or common maintenance, environmental, or UST historical factors may correlate with corrosion. Each UST will be evaluated against a set of standard criteria determined by industry to categorize the severity of corrosion in the UST. In addition, vapor, fuel, and water bottom (if present) samples from each UST will be collected and analyzed. This project will evaluate both ethanol and glycerol MIC pathways through chemical testing of samples collected from up to 42 USTs. The study will have an approximately even number of UST systems having steel and fiberglass tanks. Expanding on the previous industry study will result in a large set of data to assist EPA in better understanding the UST conditions and factors that may correlate with the presence of severe corrosion and possible increased risk of premature failure of metal components or indirect failure of operation of other components due to corrosion breakdown products in the UST.

A6.2 Schedule of Milestones and Deliverables

Table 1 lists the anticipated schedule and project activities for this study.

Table 1. Anticipated Testing Schedule

2015	Project Activities	Data Analysis and Reporting
January	• Preparation and gathering of supplies for field deployment	• Spreadsheet and database preparation
February-March	• Conduct up to 42 UST inspections • Perform TSA • Laboratory analysis of samples*	• Compile site inspection data (checklists, photos, in-tank videos) • Compile TSA results
April	• Incorporate all data into project database	• Data quality audit
May	• Write Draft Report	• Perform statistical analysis • Internal and QA reviews of draft report
June	• Draft Report under EPA review*	Not Applicable
July	• Final Report	• Revise report per review of draft report

*Assuming one month for receipt of data or comments.

A7 PROJECT DESCRIPTION

The objective of this project is to further the understanding of the conditions under which this severe and rapid corrosion develop by analyzing samples collected from a large number of USTs (up to 42) with both steel and fiberglass tanks to evaluate both ethanol and glycerol corrosion pathways hypotheses as identified by previous research. However, the project will also test for other possible contaminants, acids, or environmental conditions that may correlate with episodes of minimal or severe corrosion. Consequently, there are multiple analytical methods being used for the various sample matrices. The analyses cover the majority of the breakdown pathways from fuel, to water, to vapor, if applicable. If direct measurements cannot be performed, another known breakdown product will be used to identify the chemical relationship. For example, the glyceric acid has a low vapor pressure compared to acetic or formic acids. Therefore, it is not expected to be measureable in the ullage space. In this case, the other acids, specifically propionic acid, will be determined to investigate this breakdown pathway of glycerol.

The overall approach to the project is to develop and implement a procedure for inspecting, classifying, and sampling up to 42 USTs storing ULSD, analyze the samples, and use a statistical approach to evaluate the data (this document). This QAPP is prepared in accordance with the STREAMS II Quality Management Plan[4]. This approach ensures uniform inspections at all UST sites and a comprehensive analysis of samples and data. Ideally, the UST population will have approximately equal numbers of sites in three corrosion coverage categories; however realistically, this cannot be known before the inspections take place. The USTs for inspection were chosen to create a UST study population with approximately equal numbers of steel versus fiberglass tanks and to represent various geographic areas within the US. Site locations are at operational UST sites made available from federal and private volunteer partners. Upon confirmation of participation from the owners, EPA will contact Battelle and provide contact information for the tank owners or other individuals having knowledge of the facility. Detailed site history and general information on each UST will be collected to the extent possible (see Section B1.1 for more details).

UST investigations will be performed by Tanknology at these sites to visually inspect the inside of the tank with a camera. Following the Coordination Research Council's (CRC's) Diesel Performance Group corrosion coverage classification process (see Appendix E), Tanknology will determine the level of corrosion present by estimating the percent of corrosion coverage on the STP shaft. The three categories established are severe (>50% coverage), moderate (<50% and >5% coverage), or minimal (<5% coverage) corrosion coverage. Given that this process is subjective, at least three assessors will independently view the inspection videos and assess the corrosion coverage. If all three are in agreement then the classification identified by those three stands as the categorization level of the corrosion on that UST. If the assessors are not in agreement, the classification will be determined by the TOCOR with input from Battelle and Tanknology. After site selection and inspection scheduling, it was determined that some sites have suction systems without STP shafts and therefore cannot be assessed using the criteria established by CRC discussed above. In these cases, a proxy assessment of the level of corrosion will be determined by assessing the extent of corrosion coverage inside two riser pipes inside of the UST that are of similar material and vertical orientation across fuel and vapor space as the STP shaft in other USTs in the study. For some of the identified sites it has previously been determined from an internal video that the corrosion coverage is severe. In these cases, the video inspection may or may not be performed; if not performed, the UST will be categorized as "severe". It is at the discretion of the TOCOR as to what sites will and will not have internal video inspections. Fuel, ullage space, and water bottom (if present) will be sampled and analyzed for chemical parameters. In addition, an aliquot of the water bottom will be filtered and archived for potential biological analysis. It is expected that analysis of the resulting dataset will allow conclusions to be drawn to evaluate the likelihood of corrosion caused by ethanol and glycerol break-down pathways. The analyses may also point to other correlations that may identify causes that are yet to be determined. Future research for solutions can then be more efficiently targeted toward understanding and preventing corrosive tank conditions.

A8 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The data quality objective (DQO) of this study is to determine if the presence of biofuels, specifically ethanol or biodiesel, indirectly contributes to the corrosion or failure of functionality of UST equipment. The overall project will include three major components that involve making measurements: (1) sampling of fuel, headspace vapor, and water bottom (if present and obtainable) from USTs, (2) chemical analyses performed on those samples, and (3) analysis of the resulting data to identify correlations between objective measurement data and corrosion observed in the USTs. Most of the measurements will follow standard analytical methods that have been published and accepted by either the ASTM International (ASTM), National Association of Corrosion Engineers International (NACE), National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods or EPA. Detailed quality control (QC) requirements and method specific acceptance criteria are presented in Tables 5 and 6 for each method.

A9 SPECIAL TRAINING NEEDS/CERTIFICATION

The staff from Tanknology, Inc. who will be performing the site inspections and sampling will have documented training pertinent to their function in the inspection and sampling process. An inspection training session will be held via Live Meeting with the team leads for each field inspection team. Prior to inspection/sampling, each staff member will be required to review the applicable sampling methods and have experience or become adequately trained with the required sampling equipment. This training or experience will be documented in the project records. Analytical laboratories will be required to provide documented support for their proficiency in performing the required analyses in a thorough and safe manner with proper attention to QC samples and waste disposal. Laboratory compliance with the DQOs will be demonstrated by QC data provided by the laboratories performing the analyses.

A10 DOCUMENTS AND RECORDS

Project staff (staff members from Battelle, Tanknology, and analytical laboratories) will record all relevant aspects of this project on data sheets, in laboratory record books (LRBs), electronic files (both raw data produced by applicable analytical method and spreadsheets containing various statistical calculations), audit reports, and other project reports. Table 2 includes the records that each laboratory will include in their project records to be submitted to the TOL. The TOL will review all of these records within seven days of receipt and maintain them in her office or electronically during the project. At the conclusion of the project, the TOL will transfer the records to permanent storage at Battelle's Records Management Office (RMO). The Battelle QA Manager will maintain all quality records. All Battelle LRBs are stored indefinitely by Battelle's RMO. The TOL will distribute the final QAPP and any revisions to the distribution list given in Section A4. Section B10 further details the data recording practices and responsibilities.

Table 2. Project Records Submitted to TOL

Organization	Records	Submission Deadline
Battelle	LRBs, raw data spreadsheets	Within one week of completion of record generation
Tanknology	UST checklist and data forms, sample chain of custody (COC) forms, training documentation, inspection video and electronic photo files	Scanned copy of documents and video and photo files transferred to TOL within 10 days of record generation
Analytical laboratories	Raw data spreadsheets, QA and calibration data, COC forms, training documentation, and final analytical reports	Copies of all records transferred to TOL within two weeks of analysis

SECTION B
DATA GENERATION AND ACQUISITION

B1 EXPERIMENTAL DESIGN

The following section will guide the UST inspection, classification, and sampling that will be performed at each site. This section also includes the sample analysis methods, QC requirements, and statistical analysis approach to be performed on the samples collected from each UST.

B1.1 Information Gathering

Tanknology will perform inspections of up to 42 sites that will be selected by EPA. The list of sites will be finalized by the EPA in January 2015. A draft of the UST Inspection List (Appendix A) includes information about the selected USTs, such as location, material of tanks, and size of tanks. An effort will be made to collect as much UST-specific information as possible before visiting a site, which will be conducted in the manner specified in the agreements set in place with the site owners (see Appendix B for the questionnaire). This will allow for shorter inspections and for information gaps to be identified for potential collection during the inspection again, according to the established agreements. The information gathering and inspections will include visual documentation of the site (photos and video) and completion of an inspection checklist that includes: acquiring copies of applicable site records pertaining to equipment age and maintenance, fuel throughput and delivery, and UST and fuel maintenance practices. In addition, the field technicians will fill out a sample collection log sheet to document the QAPP was followed during sampling activities and to collect all pertinent information about the samples collected. Appendix C includes the inspection checklist and the sample collection log sheet to be used by Tanknology technicians during this work, and Appendix D includes a job safety analysis, which details all critical actions performed once Tanknology technicians arrive at the site and the possible hazards they may encounter.

Tanknology will visually inspect and photograph the dispenser filters. Tanknology will also inspect the STP shaft to classify the corrosion coverage using a proprietary tank inspection camera. Corrosion classification will be based on a procedure developed by the CRC Diesel Performance Group to determine the extent of the observed corrosion (see Appendix E for the protocol).[5] The dispenser filters will be inspected for evidence of corrosion (which is often described as physically resembling coffee grounds in the filter). The tanks corrosion classification will be determined by visual inspection into three categories depending on corrosion observed on the STP shaft (or open riser(s)). The categories will be classified as follows:

- STP shaft corrosion coverage < 5% = minimal corrosion
- STP shaft corrosion coverage > 5% but < 50% = moderate corrosion
- STP shaft corrosion coverage > 50% = severe corrosion

The estimated percentage of corrosion on the STP shaft will be documented on the site inspection form.

B1.2 UST Database

A database will be compiled to summarize any relevant site information such as site location, point of contact, site history and field observations. The database will act as a central storage location that presents the site information in a clear, concise, understandable way. For those partners allowing detailed information gathering, as much pertinent information as possible will be collected for each site and compiled into a database in accordance with the agreement with the site owner. A detailed list of questions, Appendix B, has been prepared for this purpose and was also based on the CRC Diesel Performance Group procedure.[5] The database will be further amended to include analytical data as they become available.

B2 OBTAINING SAMPLES

As part of the site inspection, Tanknology field technicians will collect multiple samples. Initially, only one riser will be opened and a thermo-hygrometer probe (EW-03313-87, Cole Palmer or equivalent) will be deployed inside the ullage space (tank headspace) for triplicate humidity and temperature readings. Then vapor samples will be collected by connecting three tubes onto a ridged pipe with zip ties and lowered into the ullage space through the same riser. Pumping air through three sorbent cartridges simultaneously for 100 minutes will collect the vapor samples for analysis.

Once the vapor has been sampled, the liquid samples will be collected and may be collected from any riser or more than one riser, as needed. A fuel sample and a water sample using a closed-core type sampling thief (TL-3573, Gammon, Manasquan, New Jersey), or a Tanknology Vacuum Sampler (TVS) similar to the ones shown in Figure 3 will be used. The closed-core sampling thief is also known as the Bacon bomb sampler that is widely known and used in the industry for this purpose. The TVS is a sampler that was developed by Tanknology for the specific purpose of sampling water at the bottom of USTs. The TVS assembly includes a mason jar, a cap with two connections, fuel compatible tubing, and a vacuum pump. Tubing is connected to one compression fitting on the cap. A tee is connected to the other compression fitting on the cap. A pressure gauge and vacuum pump are connected to the tee. The tubing will be of sufficient length to reach to the bottom of the tank and the vacuum pump will draw liquid up into the sample jar. The tubing will be secured to the bottom of the tank by fixing brass fittings to the end of the tubing to weigh it down. A new sample jar and tubing will be used for each sample location. The brass fittings will be rinsed with deionized water and decontaminated with isopropyl alcohol, then allowed to air dry after each sample location.

a).  b). 

Figure 3. Closed-Core Sampling Thief (a) and Tanknology Vacuum Sampler (b)

Table 3. Sample Summary Information

Laboratory	Sample Volume and Containers for Analysis	Shipping and Storage Conditions	Maximum Hold Time Before Analysis
Ullage Vapor			
ALS-CA	• 100 L vapor sample on two SKC tubes 226-55 at 1 lpm •	• Ambient conditions	• 14 days
ALS-OH	• 100 L vapor sample on SKC tube 226-10-03 at 0.2 to 0.5 lpm	• Ambient conditions	• 21 days
Fuel			
ICFTL	• Minimum 750 mL amber glass or aluminum container	• Ambient conditions	• No limit
Marathon	• 1 L amber glass bottle	• Ambient conditions	• No limit
SwRI	• Minimum 600 mL in glass container	• Ambient conditions	• 6 months
Water Bottom			
Metrohm	• 125 mL glass or plastic container with 1% trisodium phosphate preservation • 125 mL glass or plastic container	• Ambient conditions	• No limit
TestAmerica	• 3 x 40 mL volatile organic analysis vials (unpreserved)	• Chilled (2-6°C)	• 14 days
Water Filter			
Battelle	• Field filtered aliquot for biological analysis	• Ship overnight on dry ice • Store in -65ºC freezer within 24 hours of sampling	• 36 months
Corrosion Scraping			
Battelle	• 50 mL (or larger) plastic conical tube	• Ship with water filter • Store at ambient conditions	• No limit

Inspection and sampling of USTs will be coordinated to attempt to avoid having to sample at sites where tanks are more than 50% full, but if not avoidable the information will be noted in the database. At or below 50% full allows for vapor space to be sampled as well as exposing the STP shaft for corrosion coverage classification. Samples will be drawn in the order described above and before the in-tank video inspection. Sampling will not take place through drop tubes or riser pipes that do not allow collection of a representative sample. It may be necessary to consolidate the water collected from multiple deployments of the sampler to obtain enough sample volume. Table 3 gives the type of sample to be collected, the sample volume, container, and storage conditions.

B2.1 Vapor Sampling Method

Vapor will be collected from the ullage space of the UST in three sorbent cartridges (two on SKC 226-55 and one on SCK 226-10-03). These samples will be used to characterize the vapor phase for acetic, formic, propionic, butyric, and lactic acids and will be collected by pumping UST ullage vapor through the sorbent cartridges (provided by ALS). ALS Method 102 and NIOSH 7903 will be followed for this sampling approach for the vapor analyses methods. The sampling flow rates will be as stated in Table 3 for the appropriate methods. Following sampling, the cartridges will be capped, bagged, and shipped to ALS. At a frequency of 10%, a field blank of an identical sorbent cartridge, will be taken on site following the ALS sampling method.

B2.2 Fuel and Water Sampling Methods

The fuel and water samples undergoing chemical analyses will be sampled following ASTM D7464[6]. This sampling method is specific to sampling for microbiological testing that is a higher standard of cleanliness than traditional sampling for chemical analyses. Aseptic sampling includes wearing sterile gloves, rinsing the sampling equipment first with sterile deionized water and then laboratory grade isopropyl alcohol before sampling and between sample locations (after sampling). Once rinsed with alcohol, allow the equipment to air dry. The step-by-step procedure for core thief bottom sampling is described in detail within Section 11.1.3.2 to 11.1.3.7of the ASTM sampling method. Additionally, this method provides specific direction about the cleanliness of the sampling equipment in Sections 8-10 of the ASTM sampling method. See Table 3 for sample volumes and containers for the fuel and water samples to be collected. The liquid samples will be packaged appropriately and shipped to the appropriate analytical laboratory for analysis.

An aliquot of the water sample will be filtered through a cellulose filter (Analytical Filter Unit, #130, Nalgene, Rochester, NY) until the filter clogs (~0.5 L). The filter is considered clogged when the pressure gauge holds steady for at least 5 minutes without water passing through the filter. The filters will be shipped on dry ice overnight to Battelle for immediate storage in a -65°C freezer for potential future biological analysis.

Temperature data loggers (3M TL30-KIT6 or equivalent) will be kept inside the coolers holding water bottom samples that are required to be kept within a specific temperature range. The logged data will confirm temperatures between 2 and 6°C are maintained throughout the duration of sample handling and shipment. The temperature will be checked by the field technician using the thermos-hygrometer before shipment and by the lab technician upon receipt. Both checks will be documented on the chain of custody (COC).

B2.3 Corrosion Sampling Methods

If available, a minimum of one corrosion scraping sample will be taken by using a stainless steel instrument cleaned with DI water and isopropyl alcohol and air dried. The scraping will be immediately placed into a 50 mL plastic conical tube or larger, the location where it came from documented on the sample collection log sheet and shipped to Battelle for potential compositional analysis.

B3 SAMPLE HANDLING AND CUSTODY

Each sample will be handled according to ASTM D7464-08[6] Section 16. All sample bottles and sorbent cartridge packages will be labeled with the UST identification, the date and time of sampling, the type of sample (fuel, water, etc.), and name of the sampling technician. Each cooler containing the samples will have a COC form that will be completed prior to shipment. The COC form will include the minimum requirements as stated in Battelle standard operating procedure (SOP) ENVS-6-055[7]. These items include unique sample identification, date and time of sampling, sample description, storage condition, and the date, time, and by whom the samples were relinquished to the shipping company. A copy of the COC form will by captured electronically by taking a photo. Upon receipt at the analytical laboratory, the integrity of the samples will be checked, documented, and receipt of the samples will be formally documented with a signature. Copies of all completed COC forms will be provided to the TOL.

B4 ANALYTICAL METHODS

Table 4 gives the analytical methods that will be used for this project. The table is separated by matrix (fuel, water and vapor) and includes the method title, standard method number (if applicable), and the laboratory performing the analysis. The standard methods are very detailed and will not be reiterated in this document. A few analyses require a non-standard method. In these cases, a summary of the method will be summarized in the results report. The laboratories will provide the QC data and instrument calibration records to establish method performance meets the requirements stated in Tables 5, 6, and 7.

Table 4. Analytical Methods by Matrix

Fuel Analysis Methods	Method Identifier	Determination of	Laboratory
Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration (Procedure B)	ASTM D6304[8]	Water Content	ICFTL
Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography	ASTM D4815[9]	Ethanol and Methanol Content	ICFTL
Determination of Density, Relative Density, and API Gravity of Liquids by Digital Density Meter	ASTM D4052[10]	Density	ICFTL
Acid Number of Petroleum Products by Potentiometric Titration	ASTM D664[11]	Total Acid Number	ICFTL
Determining Corrosive Properties of Cargoes in Petroleum Product Pipelines	NACE TM-172[12]	Corrosion Rating	SwRI
Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration	ASTM D6217[13]	Particulates	ICFTL
Determination of Biodiesel (FAME) Content in Diesel Fuel Oil Using Mid Infrared Spectroscopy (FITR-ATR-PLS Method)	ASTM D7371[14]	Biodiesel Content	ICFTL
Flash Point by Pensky-Martens Closed Cup Tester	ASTM D93[15]	Flashpoint	ICFTL
Determination of Free and Total Glycerin in Biodiesel Blends by Anion Exchange Chromatography	ASTM D7591[16]	Free and Total Glycerin	SwRI
GC-MS Full Scan	Lab In-House Method	Unknowns of Interest	ICFTL
Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence	ASTM D5453[17]	Sulfur Content	ICFTL
Electrical Conductivity of Aviation and Distillate Fuels	ASTM D2624[18]	Conductivity	Marathon
Determination of Short Chain Fatty Acids by Gas Chromatography-Mass Spectrometry (GC-MS)	Lab In-House Method	Acetate, Formate, Propionate, Lactate, Glycerate	Marathon
Water Bottom Analysis Methods	**Method Identifier**	**Determination of**	**Laboratory**
Ion Chromatography (IC) for short chain fatty acids	Modified EPA 300	Acetic, Formic, Propionic, Lactic Acids	Metrohm
IC Test for Free Glycerin	Lab In-House Method	Glycerin	Metrohm

Fuel Analysis Methods	Method Identifier	Determination of	Laboratory
Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography	ASTM D6919[19]	Cations (Sodium, Calcium, Magnesium, Potassium, Ammonium) and Anions (Chloride, Sulfate, Nitrate and Fluoride)	Metrohm
pH (Electric)	EPA 150.1[20]	pH	Metrohm
Conductance (Specific Conductance, umhos at 25°C)	EPA 120.1[21]	Conductivity	Metrohm
Nonhalogenated Organics Using GC/FID	SW846 8015B[22]	Ethanol and Methanol	TestAmerica
Vapor Analysis Methods	**Method Identifier**	**Determination of**	**Laboratory**
Ullage % Relative Humidity	Hygrometer used per manufacturer instructions	% relative humidity	Tanknology on-site
Carboxylic Acids in Ambient Air Using GC-MS	ALS Method 102	Acetic, Formic, Propionic, and Butyric Acids	ALS
Determination of Lactic Acid in Ambient Air	Modified NIOSH 7903	Lactic Acid	ALS

B5 QUALITY CONTROL REQUIREMENTS

Each method listed in Table 4 has QC procedures and samples that are required for analysis along with the field samples to ensure the quality of the measurements. A duplicate liquid sample will be collected for 10% of the samples collected. For the vapor samples, a field blank and a duplicate sample will be collected and submitted for analysis of 10% of the samples. In addition, analytical QC samples will be included by all of the laboratories to verify there is no cross-contamination or carry-over between samples during analysis. The QC procedures and acceptance criteria for fuel, water and vapor samples are presented in Tables 5, 6 and 7, respectively.

Table 5. Data Quality Objectives and Frequency of Instrument Calibration for Fuel Analysis Methods

Fuel Analysis Methods- Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Water Content	ASTM D6304	Metrohm Coulometer 899	Daily QC Check (10µL DI water) Quarterly Reference Check	10,000 ± 200 µg 253 ± 467 ppm	Fresh titrant
Ethanol and Methanol Content	ASTM D4815	Perkin Elmer Clarus 680	Multi-point Cal Semi-annual Daily QC Check Reference Check Quarterly	14.8 ± 1.1 mass %	New calibration curve
Density	ASTM D4052	Anton Paar DMA 35N	Daily QC Check Quarterly Water Reference Check	0.88 ± 0.0005 g/mL	Recalibrate
Total Acid Number	ASTM D664	Metrohm 836 Titrando	Daily QC Check Quarterly Reference Check	0.31 ± 0.11 mg KOH/g	Fresh titrant
Corrosion Rating	NACE TM0172	Lab-Line Instruments	Speed controller annually Thermometers every 6 months RPM by a speed gun daily Speed gun every 6 months	ASTM cross check samples 3 times per year	Rerun ASTM cross check sample if necessary
Particulates	ASTM D6217	Gast Vacuum pump and filtration apparatus	Semi-annual Manometer Check Semi-annual Balance Check	2.0 ±1.6 mg/L	Check vacuum connections Check balance
Biodiesel Content	ASTM D7371	Perkin Elmer Spectrum 100	Daily QC Check with Certified Reference Material	10.0 ± 1.2 vol %	Clean instrument and re-run
Flashpoint	ASTM D93	Petrolab PMA 4	Daily QC Check Semi-Annual Reference Check	140.7 ± 14.7 °C	Clean instrument and re-run
Free and Total Glycerin	ASTM D7591	Metrohm 871 Advanced Bioscan	Daily Calibration and/or QC check	Correlation >0.999 % RSD <3	Rerun calibration and/or make new standards Perform maintenance
Unknowns of Interest	Lab In-House Method	Perkin Elmer Clarus 500 GC with Clarus 560 MS	Qualitative Analysis Only	NA	Replace column Replace filaments
Sulfur Content	ASTM D5453	Mitsubishi TS-100V	Multi-point Cal Semi-annual Daily QC Check Reference Check Quarterly	1.19 ± 0.66 ppm	Clean instrument, new calibration curve
Conductivity	ASTM D2624	Emcee Electronics Model 1152	Manufacturer calibration Instrument-specific calibration involves daily zeroing	Internal check of metal probe conductivity <1% error	Rerun calibration
Acetate, Formate, Propionate, Lactate, Glycerate	Lab In-House Method	HP 6890 Series GC HP 5973 Mass Selective Detector	Auto-tune is performed on a monthly minimum.	Per tuning standard guidelines	Recalibrate

Table 6. Data Quality Objectives and Frequency of Instrument Calibration for Water Bottom Analysis Methods

Water Bottom Analysis Methods- Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Acetic, Formic, Propionic, Lactic Acids	Modified EPA 300	Metrohm 850 Professional IC	• Weekly calibration • QCs ran every day • QC ran every 10-15 samples	• Calibration curve acceptance with correlation coefficient of 0.995 or greater • 90-110% Recovery of QC samples	• Re-run current calibration; • Run new calibration • perform instrument maintenance
Glycerin	Lab In-House Method	Metrohm 850 Professional IC	• Weekly calibration • QCs ran every day • QC ran every 10-15 samples	• Calibration curve acceptance with correlation coefficient of 0.995 • 90-110% Recovery of QC samples	• Re-run current calibration; • Run new calibration • perform instrument maintenance
Cations and Anions	ASTM D6919	Metrohm 850 Professional IC	• Weekly calibration • QCs ran every day • QC ran every 10-15 samples	• Calibration curve acceptance with correlation coefficient of 0.995 • 90-110% Recovery of QC samples	• Re-run current calibration; • Run new calibration • perform instrument maintenance
pH	EPA 150.1	Hach LDO HQ20	• Daily calibration with 4.00, 7.00, and 10.00 pH buffers	• R_2 > 95%	• Repeat calibration • Have probe and/or meter serviced
Conductivity	EPA 120.1	VWR Symphony	• Daily calibration with 1413 µS/cm and 12.9 ms/cm standards	• Stable calibration readings equaling known concentrations	• Repeat calibration • Have probe and/or meter serviced
Ethanol and Methanol	SW846 8015B	Gas Chromatograph/ HP 6890	• Initial Calibration (five-point ICAL): as needed prior to sample analysis • Initial Calibration verification (ICV): Daily and before sample analysis • Continuing Calibration verification (CCV): Every 10 samples • Daily observation of chromatographic resolution and retention time checks	• Linear-mean RSD ≤ 20% • Value of second source for all analytes within ± 15% of expected • All analytes within ± 15% of expected value from the ICAL • Adequate resolution of peaks in accordance with method requirements or analyst judgment and Compound retention time within method requirements	• Repeat calibration • Rerun ICV one time, second failure requires recalibration • Re-inject CCV – if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate • Change column as needed and recalibrate

Table 7. Data Quality Objectives and Frequency of Instrument Calibration for Vapor Analysis Methods

Vapor Analysis Methods	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Ullage % Relative Humidity	Hygrometer	Commercially available brand	• Per manufacturer guidelines	• Per manufacturer guidelines	• Repeat calibration • Have probe and serviced
Acetic, Formic, Propionic, and Butyric Acids	ALS Method 102	Agilent 6890GC/ 5973MS	• Annual five point calibration curve with daily CCV	• Within control limits of routine QC check sample analyses	• Perform maintenance or run a new calibration curve
Lactic Acid	Modified NIOSH 7903	Dionex ICS 2000	• A 5 point initial calibration curve, CCV every 20 samples	• Within established control limits	• Reinject calibration standard • Perform instrument maintenance/ recalibrate

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The non-calibrated equipment needed for this project (samplers, sample containers, miscellaneous laboratory items, etc.) will be maintained and operated according to the quality requirements in this document and documentation of any applicable standard method or of the laboratory responsible for its use. Only properly functioning equipment will be used; any observed malfunctioning equipment will be documented and appropriate maintenance or replacement of malfunctioning equipment will be performed.

B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Some of the methods used during this project require calibration each day of analysis, but some require only a QC check sample to be analyzed to confirm the ongoing accuracy of calibration that is performed periodically (or possibly only by the manufacturer). Tables 5, 6, and 7 give the calibration frequency required for each method. Daily calibration checks will be performed by the field technicians on each of the sampling pumps and recorded on the sample collection log sheet (see Appendix C).

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All materials, supplies, and consumables to be used during this project will be ordered by the TOL or designee. Unless specifically noted, all other supplies required for the evaluation are expected to be standard laboratory supplies (e.g., beakers, racks, etc.) that will not be required to meet a customized set of specifications. When possible, National Institute of Standards and Technology (NIST) traceable materials will be used for preparation of calibration standards and check standards.

B9 NON-DIRECT MEASUREMENTS

Any secondary data required for this project will be collected from the site owners and operators and will be assumed to be accurate upon data gathering. The source of the data will be noted (i.e., verbal recollection, automatic tank gauge records, maintenance invoices, etc.). Such information may include tank volume, throughput, and additive information and will be collected in accordance with site owner agreement.

B10 DATA MANAGEMENT

All project staff will acquire and record data electronically or manually as described in Section A10. All handwritten entries will be recorded in ink, and corrections to the entry will be made with a single line so as not to obliterate the original entry; the corrections will be initialed and dated. An explanation will accompany all non-obvious corrections. Records received by or generated by any of the project staff during the project will be reviewed by the TOL or designee within two weeks of receipt or generation before the records are used to calculate, evaluate, or report results. The person performing the review will add his/her initials and date to the hard copy of the record being reviewed. In addition, all calculations, especially statistical calculations performed by project staff, will be spot-checked by the TOL or designee to ensure that calculations are performed correctly. All spreadsheets and word processing documents applicable to this project will be stored on the Battelle network server, which is backed up daily.

B11 STATISTICAL ANALYSIS APPROACH

This study is primarily exploratory in nature and seeks the most likely association and pathway between UST corrosion and the potential predictor variables collected. Due to the limited number of tanks to be sampled in comparison to the number of explanatory variables that can be used for analysis, and therefore resulting multiple testing, the quantitative assessments of significance represented by p-values from the various models described below are to serve as a heuristic guide to the most likely causes of corrosion.

The first step in the analysis will be to provide full descriptions of all variables collected, both corrosion and explanatory. Corrosion is classified uniformly across tanks on an ordered, categorical scale of minimal, moderate, or severe, and the frequency counts and percentages as well as counts of missing and non-missing assessments will be presented. Likewise, the same will be provided for all categorical variables such as region, tank material, and fuel source. For methods where more than 25% of the analyte concentrations are below the method’s detection limit, the data will be dichotomized as present or not and described as a categorical variable (rather than on the original continuous scale) to avoid biasing the estimated effect. For the continuous scale variables, the usual univariate descriptive statistics will be provided such as the mean, standard deviation, minimum, maximum and the 10th, 25th, 75th and 90th percentiles, as well as counts of missing and non-missing measurements. In addition, the data will then be examined with pairwise associations and correlations of all explanatory variables, including matrix scatter plots and tests of independence, as well as the identification of potential outliers.

The main analysis of corrosion will then start with a univariate analysis by examining how well corrosion is predicted by each variable on its own. As corrosion is assessed by a three category ordered scale, the natural, parametric model to use will be proportional odds logistic regression. These univariate assessments will provide intuition for expected marginal effects and a guide for adjusted multivariable models. Due to the limited number of corrosion assessments compared to the number of potential explanatory variables collected, it is anticipated not being very well powered to analyze moderately complex, adjusted models. Therefore should the available data be complete enough to allow it, consultations with Battelle and EPA experts will be held on plausible adjusted models in addition to standard algorithmic techniques such as forward/backward selection. Furthermore, in terms of classification and prediction accuracy, the machine learning technique known as random forests classification is particularly well suited to situations with much more covariates than observations. With a random forest for corrosion category, an associated measure of variable importance for prediction will be calculated for all variables. These two classification techniques will serve to provide a quantifiable ranking of variable association with corrosion, and aid in hypothesis generation.

SECTION C
ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Internal QC measures (e.g., QC check samples, regular review of raw data, spot-checking of calculations, etc.) described in this QAPP, implemented by the project staff and monitored by the TOL, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the TOL. Project staff have the responsibility of identifying problems that could affect data quality or the ability to use the data at the time the problem is discovered. Any problems that are identified will be reported to the TOL, who will work to resolve any issues and will notify the TOCOR of the problem and the corrective action taken or to be taken. Action will be taken to control the problem, identify a solution to the problem, minimize losses and correct data, where possible. Battelle will be responsible for ensuring that the following audits are conducted as part of this project.

C1.1 Data Quality Audit

The Battelle QA Manager, or her designee, will audit at least 10% of the data acquired during the project. The Battelle QA Manager will trace the data from initial acquisition (reviewing at least 10% of raw data for each method), through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked. The Battelle QA Manager will prepare an audit report describing the results of the data quality audit.

C1.2 Technical Systems Audit

A TSA at one of the first field sites will be conducted by the Battelle QA Manager. The purpose of this audit is to ensure data collection, sampling, and sample shipment procedures are being performed in accordance with this QAPP. The TSA will be guided by a project-specific checklist based on this QAPP. Due to the time sensitive nature of the sampling activities, corrective actions will be immediately relayed verbally to all of the field technicians and to the

TOCOR. The results of the audit will be written up in a TSA report by the Battelle QA Manager and responded to by the TOL to document resolution to any issues identified in the TSA report.

C1.3 QA/QC Reporting

The audits will be documented in assessment reports and will include:

- Identification of any adverse findings or potential problems;
- Response to adverse findings or potential problems;
- Recommendations for resolving problems;
- Confirmation that solutions have been implemented and are effective; and
- Citation of any noteworthy practices that may be of use to others.

SECTION D
DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION

Data validity and usability will be assessed through review of QC check samples to assess accuracy and precision. The acceptance criteria for the QC objectives generally rely on the generation of routine QC check sample performance data. Data verification is accomplished by ensuring the accuracy and completeness of data transcribed from raw data to the results report. A comparison of raw data sheets or LRB comments against final data will be conducted to flag any suspect data and resolve any questions about apparent outliers. The data quality assessment, as described within Section C of this document, is designed to ensure the quality of these data.

D2 VALIDATION AND VERIFICATION METHODS

Data verification includes a visual inspection of hand written data to ensure that all entries were properly recorded and that any erroneous entries were properly noted, as described in Sections B10 and D1. Data validation efforts include the assessment of QC data and the performance of a data quality audit (Section C) to determine if the data collection and measurement procedures met the quality objectives defined in the QAPP. The Battelle QA Manager will conduct an audit of data quality to verify that data review and validation procedures were completed, and to assess the overall quality of the data.

D3 RECONCILIATION WITH USER REQUIREMENTS

The data obtained during this project will provide thorough documentation of the required measurements. The data review and validation procedures described in the previous sections will verify that data meet the quality objectives and are accurately presented in the report generated from this project. The data generated throughout this project will be compiled into a results database. The results report will present tables of the measured data and resulting data describing the results of the site inspections and required measurements. Any limitations to the data will be addressed and discussed in the results report.

SECTION E
REFERENCES

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4. Battelle, *Quality Management Plan for the Scientific, Technical, Research, Engineering, and Modeling Support II (STREAMS II), Version 4*. 2014.
5. Diesel Performance Group. *Coordinating Research Council Protocol for Selecting Sites with ULSD Systems Determined to Have Severe Corrosion*. CRC Project No DP-07-13. July 1, 2014.
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15. ASTM, *D93 Standard Test Method for Flash Point by Pensky-Martens Closed Cup Tester*. 2013.
16. ASTM, *D7591 Standard Test Method for Determination of Free and Total Glycerin in Biodiesel Blends by Anion Exchange Chromatography*. 2012.
17. ASTM, *D5453 Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence*. 2012.

18. ASTM, *D2624 Standard Test Methods for Electrical Conductivity of Aviation and Distillate Fuels*. February 2010.
19. ASTM, *D6919 Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography*. 2009.
20. EPA, *pH (Electronic), EPA Method 150.1*. 1982.
21. EPA, *Conductance (Specific Conductance, umhos at 25°C) , EPA Method 120.1*. 1982.
22. EPA, *SW846 8015B Nonhalogenated Organics Using GC/FID*. 1996.

Supplement A

Appendix A – Draft UST Inspection List

Appendix A
Draft UST Inspection List

UST #	Owner Identifier	City	State	Zip	UST Material	UST Capacity (gallons)
1	A	Waukegan	IL	60085	Fiberglass	12000
2	B	Vernon Hills	IL	60061	Fiberglass	6000
3	C	Chicago	IL	60699	Steel	20000
4	C	Chicago	IL	60699	Steel	20000
5	C	Gary	IN	46401	Steel	12000
6	D	Glenwood	IL	60425	Fiberglass	12000
7	C	South Bend	IN	46601	Steel	8000
8	C	Fort Wayne	IN	46802	Steel	6000
9	D	Fort Wayne	IN	46816	Fiberglass	12000
10	D	South Bend	IN	46628	Fiberglass	12000
11	D	Blue Springs	MO	64014	Fiberglass	12000
12	D	Topeka	KS	66604	Fiberglass	15000
13	D	Excelsior Springs	MO	66024	Fiberglass	12000
14	D	Houma	LA	70360	Fiberglass	12000
15	D	Morgan City	LA	70380	Steel	8000
16	D	Thibodaux	LA	70301	Fiberglass	6000
17	D	Houma	LA	70363	Fiberglass	12000
18	D	Knoxville	TN	37920	Fiberglass	12000
19	D	Knoxville	TN	37938	Fiberglass	12000
20	C	Knoxville	TN	37950	Steel	12000
21	D	Broomfield	CO	80020	Fiberglass	12000
22	D	Thornton	CO	80023	Fiberglass	12000
23	E	Commerce City	CO	80022	Steel	8000
24	G	Golden	CO	80403	Steel	6000
25	H	Sebastopol	CA	95472	Steel	12000
26	H	Forestville	CA	95436	Steel	10000
27	H	Santa Rosa	CA	95407	Fiberglass	20000
28	H	Santa Rosa	CA	95407	Fiberglass	6000
29	H	Rohnert Park	CA	94928	Steel Lined Interior	12000
30	I	Cutchogue	NY	11935	Fiberglass	12000
31	I	Mastic	NY	11950	Fiberglass	7000
32	C	Melville	NY	11747	Steel	12000
33	I	Eastport	NY		Steel	1000
34	I	Farmingdale	NY	11735	Fiberglass	6000
35	C	Garden City	NY	11599	Steel Lined Interior	15000
36	C	Flushing	NY	11351	Steel	5000
37	C	Bellmawr	NJ	08031	Steel	10000
38	C	Southeastern	PA	19399	Steel Coated	10000

UST #	Owner Identifier	City	State	Zip	UST Material	UST Capacity (gallons)
39	C	Reading	PA	19612	Steel Coated	10000
40	C	Capitol Heights	MD	20790	Steel	20000
41	J	Alexandria	VA	22303	Fiberglass	10000
42	J	Hybla Valley	VA	22306	Fiberglass	10000

Owner Identifier is a generic designation of the participating owners of the USTs

Supplement A

Appendix B – Owner/Operator Questions

Site Owner/Operator Questions

General Information:
Company
Company Point of Contact (POC) Name
Company POC Contact Information
Site Address
UST Identification Number on Site

Questions:	
1	Has this UST experienced or is currently experiencing corrosion problems since the introduction of ULSD in 2006? Please elaborate as necessary.
2	Has anyone ever noticed strange odors coming from the tank or filters? Vinegar type smell?
3	How long have you owned/operated this site?
4	How long has this UST been in operation? (Install Date)
5	Has this UST held HSD (high sulfur diesel), LSD (low sulfur diesel) before ULSD?
6	Was the UST converted from gasoline service to ULSD service? Are the vent lines common to both the diesel tank and the gasoline tanks?
7	What is the capacity (8,000, 10,000, gallons, etc.)?
	What is the material (fiberglass, steel) of the UST?
8	In which direction does the tank tilt? Toward the pump or drop tube end?
9	What is the approximate monthly ULSD throughput? How often is new fuel added to the UST? What is the turnover rate?
10	From what fuel terminal(s) is the ULSD received? If more than one, list and provide approximate percentage pulled from each.
11	What carrier(s) are being used for this site? Is the company a common carrier, jobber, company owned trucks, etc.?
12	What preventative maintenance procedures are used? At what frequency?
13	What water bottom practices (SOP or protocols) are used? At what frequency is water removed from the UST?
14	How often is the filter replaced? Is the filter inspected? Does the filter contain corrosion products?
15	Are additives added to the fuel before or after the fuel is dropped into the UST?
16	Is the fuel treated with biocides? If yes:
	· What type of biocide?
	· What frequency is the biocide added?
	· Is the treated fuel allowed to remain quiescent in the tank for a prescribed period of time?
17	Has the fuel been treated onsite?
18	Have any components on the tank/pump/filter system been replaced? Which one(s)? How often? When?
19	Has the UST ever been emptied and cleaned or resurfaced? If yes:
	· What was done to the UST?
	· When did the work occur?
20	Identify the records that are available for confidential review by checking the boxes. Please provide as many of these items when returning this form.
	Equipment maintenance and water bottom removal history from 2004 to present.
	Fuel throughput and delivery history data for the past 12 months.
	Water level data from tank for the past 12 months
	History of additives and system treatments used since 2004 for microbial mitigation.
	If the site has had corrosion issues, information corrective actions taken and the system response specific to this issue.

Supplement A

Appendix C – Inspection Checklist and Sample Collection Log

ULSD Site Inspection Field Form

Site Name/ID #: ______________________ Date: ______________

Address: ______________________ Time: ______________

City: ______________ ST: ______ Zip: ________ Technician: ______________

Contact: ______________ Phone: ______________ Signature: ______________

Tank and Piping Information and History				
Tank Identifier		Product:	ULSD	
How Water Monitored?	ATG or Stick			
Tank Capacity (gals)		Tank Diameter (inches):		
Tank Material		Single/Double Wall		
Tank Year of Installation				
Tank/Piping Manifolded?				
Over fill protection (type and observation)*				
STP Make/Model				□ PHOTO
STP Shaft Condition*	Minimal (<5%) or Moderate (5% to 50%) or Severe (>50%)			□ VIDEO

Observations	Fill Pipe	ATG	STP	Other ________
Riser Condition	□ PHOTO	□ PHOTO	□ PHOTO	□ PHOTO
Cap/Adapter Condition	□ PHOTO	□ PHOTO	□ PHOTO	□ PHOTO
Other Visible Corrosion?				
Product Level*				
Water Bottom Level				

Dispenser Info	Dsp # ________	Dsp # ________	Dsp # ________	Dsp # ________
Dispenser Make/Model				
Filter Make/Model*				
Filter Date Replaced*				
Filter and filter housing Condition*	□ PHOTO	□ PHOTO	□ PHOTO	□ PHOTO

Comments: ____

*Must be completed

DRAFT Sample Collection Log Sheet

Site ID:_______________ Technician Signature: _________________________

Date:______________

1. Check Sampling Pumps' Calibration.

Pump ID	Time	Target Flow Rate (LPM)	Flow Rate Average from Cal Burst of 10 (LPM)	Comments
		1.0		
		1.0		
		0.5		

2. Collect Temperature and Humidity Readings in triplicate.

Riser*	Time	Reading 1		Reading 2		Reading 3	
		%	Temp	%	Temp	%	Temp

*Fill, ATG, STP, Other

DRAFT Sample Collection Log Sheet

Site ID:_______________ Technician Signature: _________________________

Date:______________

3. Collect Vapor Samples for 100 Minutes.

Sample ID	Vapor Tube	Target Flow Rate (LPM)	Ship To	Ship	Riser	Pump ID	Flow Rate (LPM)	Start Time	Stop Time	Comments
-Va	226-55	1.0	ALS-CA	Ambient						
-Vb	226-55	1.0								
-Vc	226-10-03	0.5	ALS-OH							

V = vapor

a. Label and bag sample vapor tubes and place them in lab appropriate box/cooler with bubble wrap.

4. Prepare to Collect Samples

Step #	Description	Technician Initials
a	Don sterile gloves	
b	Prepare a clean space for sample handling	
c	Rinse the samplers, brass fitting on TVS sampler, and scraping instrument with **DI water**	
d	Rinse the samplers, brass fitting TVS sampler, and scraping instrument with **isopropyl alcohol**	
e	Allow for the samplers, brass fitting, and scraping instrument to air dry completely	

DRAFT Sample Collection Log Sheet

Site ID:_______________ Technician Signature: _________________________

Date:______________

5. Collect Liquid, Filter, and Corrosion Scraping Samples.

Collector's Initials	Time	Sample ID	Riser(s)	Sample Type	Sample Container	Shipping Conditions	Ship To	Comments
		-DFa		Fuel	1 L glass jar	Ambient	ICFTL	
		-DFb		Fuel	1 L glass jar	Ambient	Marathon	
		-DFc		Fuel	1 L glass jar	Ambient	SwRI	
		-Wa		Water	125 mL glass jar-with preservative	Ambient	Metrohm	
		-Wb		Water	125 mL glass jar	Ambient	Metrohm	
		-Wc		Water	3 each 40 mL vials	Chilled (2-6°C) with temp logger	Test America	Temp logger ID ________
		-F		Water Filter	Filter	Dry Ice	Battelle	Estimate water volume =___________
		-CS		Corrosion Scraping	50 mL plastic tube	Dry Ice	Battelle	

DF = Diesel Fuel, W = Water, F = Filter, CS = Corrosion Scraping

DRAFT Sample Collection Log Sheet

Site ID:________________ Technician Signature: ___________________________

Date:_______________

6. Store Samples and Clean Equipment

Step #	Description	Technician Initials	Photo taken
a	Wrap each sample bottle with bubble wrap and secure with tape		NA
b	Place samples in lab appropriate coolers		NA
c	Rinse the samplers, brass fitting on TVS sampler, and scraping instrument with **DI water**		
d	Rinse the samplers, brass fitting TVS sampler, and scraping instrument with **isopropyl alcohol**		
e	Allow for the samplers, brass fitting, and scraping instrument to air dry completely		
f	Place in storage bags for the next site		

Supplement A

Appendix D – Tanknology Job Hazard Analysis

Job Safety Analysis
ULSD SITE INSPECTION

MINIMUM REQUIRED PERSONAL PROTECTIVE EQUIPMENT (SEE CRITICAL ACTIONS FOR TASK-SPECIFIC REQUIREMENTS)			
☒ REFLECTIVE VEST ☐ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☒ GOGGLES ☐ FACE SHIELD ☐ HEARING PROTECTION ☒ SAFETY SHOES	☒ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING	☒ GLOVES ☒ Voltage Indicator ☐ OTHER

[1]JOB STEPS	[2]POTENTIAL HAZARDS	[3]CRITICAL ACTIONS
Arrival on site	Vehicle and Pedestrian Traffic. Forecourt Hazards Possible other contractors on site.	1- Wear PPE: Safety Vest, Steel toed boots, Safety Glasses, 100% cotton Tanknology uniform. 2- Contact MGR or site personal to explain job process and Safety Procedures. 3- Have Site Safety Checklist and CSE form filled out and ready to sign. 4- Conduct site safety meeting with any other contractors on site.
Position test vehicle	Vehicle and Pedestrian Traffic, Forecourt Hazards Unauthorized entry	1- Check all Forecourt and Pedestrian Traffic flow for test unit position 2- Deploy all Safety Equipment following Barricading procedures including Cones, Caution Tape, Flags and Fire Extinguishers
Open All Manhole Covers and Access Points at Tankfield	Vehicle and Pedestrian Traffic Unauthorized entry Tripping and Falling Lifting Exertion Hazardous Vapors	1- Maintain full barricade around tank pad. 2- Use proper lifting technique when opening turbine sump lids 3- Barricade open sumps or replace lids to avoid tripping or falling 4- Use LEL Meter and blower as necessary
Inspect Components at Tankfield	Vehicle and Pedestrian Traffic Unauthorized entry Sharp objects Insect Bites Possible product release	1- Maintain full barricade around tank pad. 2- Check for insects and spiders and other hazards after covers are removed 3- Use tools to remove any debris 4- Use proper tools to remove components 5- Use product-resistant gloves when handling wetted components
Remove STP and inspect internal components (if necessary)	Vehicle and Pedestrian Traffic Unauthorized entry Possible Hazardous Atmosphere Possible product release Electrical Hazard Over-Exertion	1- Maintain full barricade around tank pad. 2- Conduct Confined Space Entry procedures. 3- Check for stray voltage on/around STP 4- Perform Lock/out Tag/out & bag dispensers. 5- Verify product STP is disabled after Lockout/ Tagout completed. 6- Close product ball valve if present. Relieve excess pressure from line. Use absorbent cloth to collect any product release. 7- Spray STP bolts with WD-40 prior to removal. 8- Use tripod or lever to loosen STP prior to removal. 9- Use winch or two persons to assist in STP removal as necessary. 10- Replace O-rings, use proper lubrication, and reinstall STP after samples are taken.

Take Product/Vapor/Water Samples	Vehicle and Pedestrian Traffic Unauthorized entry Possible product release Possible hazardous atmosphere Possible electrical hazard Possible burn from dry ice Possible asphyxiation hazard from dry ice.	1- Maintain full barricade around tank pad. 2- Wear product resistant gloves 3- Use only hand pump, nitrogen-powered vacuum pump, or explosion-proof electric pump. 4- Connect any battery charger to GFCI in fresh atmosphere only. 5- Verify all hoses, chains, and/or control strings are secure when collecting samples. 6- Use absorbents to collect any product drips. 7- Use funnels and appropriate lab equipment to safely transfer and combine samples if necessary. 8- Secure all samples tightly to prevent product release. 9- Package samples per ASTM guidelines for safe shipment to laboratory. 10- Wear insulating gloves when handling dry ice to prevent burns. 11- To prevent accumulation of carbon dioxide (asphyxiant), don't keep dry ice inside vehicle or other enclosed area. Keep in back of truck.
Inspect Dispensers and related equipment	Vehicle and Pedestrian Traffic Unauthorized entry Possible product release Sharp objects Insect Bites	1- Establish barricade around all dispensers 2- Perform Lockout/Tagout & bag dispensers 3- Wear leather gloves when removing covers. 4- Check for insects and spiders and other hazards. 5- Trip shear valves and close ball valve if present. 6- Wear product resistant gloves when removing & handling filters. 7- Use absorbents to collect any product released while removing and inspecting filters. 8- Check to verify only 1 gasket is present when re-installing filter and tighten properly. 9- Remove Lockout/Tagout, open shear valves and ball valve. 10- Energize dispenser to check for leaks. 11- Conduct visual inspection with site manager.
Conduct TankCam® Inspection	Vehicle and Pedestrian Traffic Unauthorized entry Possible pinch or cut to hands while handling equipment Possible vapor exposure Possible hazardous atmosphere Possible electrical hazard	1- Maintain full barricade around tank pad. 2- Wear leather or cloth abrasion-resistant gloves. 3- Verify product level and adjust handle length to insert camera without immersion. 4- Verify camera is assembled properly and articulates properly. Secure low profile adapter to riser to hold TankCam control handle. 5- Connect interface box and monitor/recorder to GFCI and keep in area with fresh atmosphere. 6- Apply proper amount of nitrogen to inert camera housing prior to inspection. 7- Confirm operation of pressure gauge, shut-off switch and relief valve. 8- Wear proper vapor respirator if product vapors are present while operating camera. 9- Handle camera and hose carefully to avoid any damage to equipment.

Job Complete	Vehicle and Pedestrian Traffic Forecourt Hazards Driving hazards	1- Notify responsible person of any maintenance needs at location. 2- Complete Site Safety Checklist and all paperwork prior to leaving. 3- Place site back to original condition. 4- Remove all barricades. 5- Plan route first and then exit site avoiding distractions.

[1] Each Job or Operation consists of a set of tasks / steps. Be sure to list all the steps in the sequence that they are performed. Specify the equipment or other details to set the basis for the associated hazards in Column 2

[2] A hazard is a potential danger. How can someone get hurt? Consider, but do not limit, the analysis to: **Contact** - victim is struck by or strikes an object; **Caught** - victim is caught on, caught in or caught between objects; **Fall** - victim falls to ground or lower level (includes slips and trips); **Exertion** - excessive strain or stress / ergonomics / lifting techniques; **Exposure** - inhalation/skin hazards. Specify the hazards and do not limit the description to a single word such as "Caught"

[3] Aligning with the first two columns, describe what actions or procedures are necessary to eliminate or minimize the risk. Be clear, concise and specific. Use objective, observable and quantified terms. Avoid subjective general statements such as, "be careful" or "use as appropriate".

Change History

Process Owner : VP Engineering	Approved By : VP Engineering

Rev	Date Effective	Page(s) Changed	Change Description	Process Owner	Approval
A	10/30/2011	All	New inspection procedure	Brad Hoffman	Brad Hoffman
B	1/14/2015	2	Added section for TankCam inspection. Minor additions to other sections.	Brad Hoffman	Brad Hoffman

Last Review:	Reviewed by:	Brad Hoffman	Review date:	1/14/2015

Supplement A

Appendix E – CRC Protocol: Corrosion Coverage Classification

1 July, 2014

Corrosion in Systems Storing and Dispensing
Ultra Low Sulfur Diesel (ULSD)
(CRC Project No DP-07-13)
CRC Protocol for Selecting Sites with ULSD Systems Determined to Have Severe Corrosion

BACKGROUND

Severe and rapid corrosion has been observed in systems storing and dispensing ultra-low sulfur diesel (ULSD) since 2007. In addition, the corrosion is coating the majority of metallic equipment in both the wetted and un-wetted portions of ULSD underground storage tanks (USTs). To investigate this issue, multiple stakeholders in the diesel industry, through the Clean Diesel Fuel Alliance, sponsored a research study by Battelle Memorial Institute (hereafter termed "Battelle study"). That effort included the identification of retail fueling sites and the development of a protocol to ensure uniform and thorough sampling and inspections of fiberglass USTs. Fuel, water bottoms, vapor, bottom sediments, and scrape samples were taken from six sites across the country: one that was not supposed to have symptoms (but did to a much lesser degree) and five that were to have severe corrosion. Samples collected during the inspections were then analyzed for genetic material and chemical characteristics. These data, in combination with information on additives, allowed Battelle to draw conclusions with respect to three working hypotheses:

1) Aerobic and anaerobic microbes were producing by-products that established a corrosive environment in ULSD systems;
2) Aggressive chemical specie(s) (e.g., acetic acid) present in ULSD systems was (were) facilitating aggressive corrosion; and
3) Additives in the fuel were contributing to the corrosive environment in ULSD systems.

Based on the data collected, the Battelle study arrived at a final hypothesis that the ULSD stored in underground storage tanks was contaminated with ethanol, and the ethanol present in the systems was oxidized by *Acetobacter* into acetic acid which was diffused throughout the vapor and liquid space causing severe and rapid corrosion of metals associated with ULSD systems at retail stations. The study hypothesized that the presence of ethanol in the fiberglass USTs storing ULSD was the result of either tanker truck switch loading or legacy ventilation system connections to gasoline USTs allowing gasoline-ethanol vapors to back feed into the ULSD tank.

The Battelle study raises many questions that remain unanswered to date. For example, is accelerated corrosion only associated with fiberglass USTs or is it also present in steel USTs? Is the accelerated corrosion limited to the retail site level or is it present in upstream systems such as tanker truck, distribution terminal and associated equipment, pipeline, multi-product tanker ships or the refinery? Is a different contaminant (not ethanol) entering the fuel supply before the retail site? Is acetic acid the major contributor or are other organic and inorganic contaminants present in the UST bottom water that are reacting synergistically and resulting in accelerated corrosion? Is the formation of acetic acid exclusively due to the oxidation of fugitive ethanol or are there other source(s) of acetic acid present in the UST?

A panel comprised of members of the Clean Diesel Fuel Alliance (and other stakeholders) has been formed under the auspices of the Diesel Performance Group of the Coordinating Research Council (CRC) Performance Committee (hereafter termed "the CRC Corrosion Panel") to develop and carry out research to: (a) address these unanswered questions, (b) facilitate a better understanding of the causes of observed corrosion in UST systems storing and distributing ULSD, and (c) assist in developing remedies.

GENERAL GUIDELINES FOR SCREENING AND SELECTING SITES FOR FURTHER STUDY

The CRC Corrosion Panel has developed a set of general guidelines and equipment maintenance history questions for use by researchers interested in screening and selecting sites for further testing and evaluation of potential causes of observed corrosion in ULSD storage and distribution systems. These guidelines, provided in Tables 1 and 2 on the following pages, are based on the cumulative technical knowledge of the Corrosion Panel members based on field observations, and they are not represented to be analytically-derived pass/fail cut points for defining whether or not a particular piece of equipment is corroded. The latter is intended to be the outcome of the subsequent detailed study of the screened sites selected for sample analysis.

Note also that the guidelines provided in Table 1 focus on the screening of corroded equipment based only on visual inspection as well as analysis of fuel and water bottom samples collected at the sites in ULSD storage and distribution service. Since the guidelines are intended only for site screening purposes, the collection and analysis of vapor phase samples for corrosion has not been included in order to minimize both time and cost.

Table 1
General Guidelines for Screening Aggressive Corrosion Conditions in ULSD Storage and Distribution Systems

I. Screening criteria:
   a. Pull the dispenser filter and inspect for metal degradation in the filter as well as in the filter housing. (The filter must have been in service for a minimum of three months)
      i. Does the dispenser filter housing show any corrosion?
         1. If yes, then go to step III
         2. If no, then cut open the filter's metal housing and examine the metal components. If any corrosion observed, then go to step III

II. If the filter housing is seemingly clean
   a. Pull out the submersible turbine pump (or alternatively use video equipment in situ) and inspect the riser:
      i. Use NACE Test Method 0172 to measure corrosion on riser pipe surface. If $\geq$ 5% then go to step III

III. Definition of aggressive corrosion in UST systems
   a. Increased replacement of equipment
   b. Is submersible turbine pump riser more than 50% corroded? (Alternatively, use video equipment to inspect.)
      i. Yes, aggressive corrosion
   c. If aggressive corrosion is observed then pull the following equipment and determine:
      i. Is the drop tube and/or flapper valve corroded?
      ii. Are any brass, copper, and/or aluminum components corroded?
   d. Aggressive corrosion conditions -- Aggressive corrosion is considered if 3 or more of the following conditions are met based on analyses of fuel and water bottom samples:
      i. Fuel (test procedures … (all samples are to be obtained from the lower third of fuel volume)
         1. NACE of C or worse
         2. Haze >2
         3. Particulate (filtration using 0.8 micron with >10 mg/liter)
         4. Karl/Fischer water content >200 ppm (mg/kg)
      ii. Water bottom (test procedures …
         1. pH less than 5
         2. Microbial growth > 10,000 cfu/ml
         3. Presence of low molecular weight (C_1 – C_5) acids (via GCMS scan)

Table 2
Questionnaire for Operators of ULSD Storage and Distribution Systems
Screened & Selected for Further Study

1. What preventive maintenance procedures, if any, do you use? How often?
2. Do you record water levels in your fuel tank? Do you have a Veeder-Root system? Can we get any V-R records?
3. How often is water drained from the tank?
4. Is there a dispensing filter on the tank? How often is it changed?
5. When a dispensing filter is changed, does anyone inspect the old filter? Has anyone ever noticed corrosion products?
6. Is your fuel ever treated with biocides? Which one? How often? Is the treated fuel allowed to remain quiescent in the tank for a prescribed period of time?
7. Do you add any additives to the fuel before or after it goes in the storage tank?
8. Have any of the components on the tank/pump/filter system ever been replaced? Which one(s)? How often? When?
9. a) Has the tank ever been emptied and cleaned or resurfaced? When? What was done? b) Has water/tank bottoms ever been sucked out of the tank? This is sometimes inappropriately referred to as “cleaning”.
10. Did the tank ever hold anything but ULSD? What was it? When?
11. Is this tank connected to other tanks in any way? Manifolds, vents, etc.?
12. Has anyone ever noticed strange odors coming from the tank or filters? Vinegar type smell?
13. Does the same fuel supplier typically fill this tank?
14. How often is new fuel added to the tank (turnover rate)?
15. How long has the tank been in the ground?

Supplement B

Data Tables

Table B-1. General Information For Inspected ULSD USTs.

Site ID	State	Inspection Date	Geographic Cluster	Owner ID (a-i)	Tank Capacity (gallons)	Tank Diameter (inch)	Tank Material	Tank Wall	Tank Age At Inspection (# of years)	Is System Manifolded? (yes or no)	Overfill Protection Type	ULSD Level During Inspection (inch)	Ullage Space Height (inch)	Water Bottom Height (inches)
1-IL-FG	IL	26-Jan-15	Chicago/Northern Indiana	a	12,000	119	Fiberglass	Double	9	no	Drop tube flapper	59	60	0
2-IL-FG	IL	27-Jan-15	Chicago/Northern Indiana	b	6,000	92	Fiberglass	Double	20	no	Drop tube flapper	51	41	1.5
3-IL-ST	IL	28-Jan-15	Chicago/Northern Indiana	c	20,000	120	Steel (coated)	Double	22	no	External alarm	54	66	0
5-IL-FG	IL	2-Feb-15	Chicago/Northern Indiana	a	12,000	120	Fiberglass	Double	10	no	Drop tube flapper	46	74	0
6-IN-ST	IN	3-Feb-15	Chicago/Northern Indiana	c	8,000	92	Steel	Double	23	no	Drop tube flapper	40	52	0.5
7-IN-ST	IN	4-Feb-15	Ft. Wayne	c	6,000	96	Steel	Double	23	no	Drop tube flapper	5	91	1.0
8-IN-FG	IN	5-Feb-15	Ft. Wayne	a	12,000	120	Fiberglass	Double	9	no	Drop tube flapper	25	95	0
9-IN-FG	IN	3-Feb-15	Chicago/Northern Indiana	a	12,000	120	Fiberglass	Double	9	no	Drop tube flapper	26	94	0
10-MO-FG	MO	10-Feb-15	Kansas City	a	12,000	118	Fiberglass	Double	10	no	Drop tube flapper	41	77	0
11-KS-FG	KS	9-Feb-15	Kansas City	a	15,000	119	Fiberglass	Single	unknown	no	Ball float	26	93	0
12-MO-FG	MO	9-Feb-15	Kansas City	a	12,000	118	Fiberglass	Double	9	no	Drop tube flapper	40	78	0
13-LA-FG	LA	2-Feb-15	Louisiana	a	12,000	123	Fiberglass	Double	7	no	ATG	44	78	0.7
14-LA-ST	LA	3-Feb-15	Louisiana	a	12,000	119	Steel	Double	3	no	ATG	50	69	0
15-LA-FG	LA	4-Feb-15	Louisiana	a	6,000	90	Fiberglass	Double	15	no	ATG	58	32	0
16-TN-FG	TN	12-Feb-15	Knoxville	a	12,000	120	Fiberglass	Double	11	no	Drop tube flapper	34	85	0
17-TN-FG	TN	10-Feb-15	Knoxville	a	12,000	118	Fiberglass	Double	4	no	Ball float	49	69	0
18-TN-ST	TN	11-Feb-15	Knoxville	c	12,000	98	Steel	Double	29	no	Ball float	46	52	1.1
22-CO-FG	CO	2-Feb-15	Denver	a	12,000	118	Fiberglass	Double	3	no	Ball float	55	63	0
23-CO-FG	CO	3-Feb-15	Denver	a	12,000	118	Fiberglass	Double	1	no	Ball float	87	31	0
24-CO-ST	CO	4-Feb-15	Denver	d	8,000	120	Steel	Double	unknown	no	Drop tube flapper	81	39	0
26-CO-FG	CO	5-Feb-15	Denver	f	6,000	92	Fiberglass	Double	19	no	Drop tube flapper	59	33	0.5
27-CA-ST	CA	12-Feb-15	San Francisco	g	12,000	95	Steel	Double	17	no	Ball float	43	53	0
28-CA-ST	CA	10-Feb-15	San Francisco	g	10,000	112	Steel	Double	16	no	Drop tube flapper	61	51	0
29-CA-FG	CA	11-Feb-15	San Francisco	g	20,000	120	Fiberglass	Double	12	no	Drop tube flapper	57	64	0
30-CA-FG	CA	11-Feb-15	San Francisco	g	6,000	92	Fiberglass	Double	12	no	Drop tube flapper	53	40	0
31-CA-ST	CA	12-Feb-15	San Francisco	g	12,000	111	Steel (coated)	Double	28	no	Drop tube flapper	57	54	not recorded
32-NY-FG	NY	6-Feb-15	Long Island	h	12,000	120	Fiberglass	Single	8	no	Drop tube flapper	55	65	0
33-NY-FG	NY	3-Feb-15	Long Island	h	7,000	120	Fiberglass	Double	5	no	Drop tube flapper	64	56	0
34-NY-ST	NY	3-Feb-15	Long Island	c	12,000	96	Steel	Single	23	no	External alarm	31	65	0
35-NY-FG	NY	4-Feb-15	Long Island	h	6,000	92	Fiberglass	Single	29	no	Drop tube flapper	unknown	unknown	not recorded
36-NY-ST	NY	4-Feb-15	Long Island	c	15,000	120	Steel	Single	24	no	External alarm	unknown	unknown	not recorded
37-NY-ST	NY	5-Feb-15	Long Island	c	5,000	72	Steel	Double	24	yes	External alarm	43	29	1.0
39-NJ-ST	NJ	9-Feb-15	Southeast PA	c	10,000	96	Steel	Single	24	no	Drop tube flapper	35	61	0
40-PA-ST	PA	10-Feb-15	Southeast PA	c	10,000	96	Steel	Single	21	no	Drop tube flapper + External alarm	26	70	0
42-PA-ST	PA	11-Feb-15	Southeast PA	c	10,000	96	Steel	Double	21	no	Drop tube flapper + External alarm	62	34	0
43-MD-ST	MD	29-Jan-15	Washington, DC	c	20,000	126	Steel	Double	23	no	External alarm	45	81	0
44-VA-FG	VA	28-Jan-15	Washington, DC	i	10,000	92	Fiberglass	Double	unknown	no	Drop tube flapper	61	31	0
45-VA-FG	VA	29-Jan-15	Washington, DC	i	10,000	92	Fiberglass	Double	unknown	no	Drop tube flapper	58	34	0
46-IL-ST	IL	28-Jan-15	Chicago/Northern Indiana	c	20,000	120	Steel	Double	22	no	Drop tube flapper + External alarm	24	96	0
47-LA-FG	LA	5-Feb-15	Louisiana	a	12,000	119	Fiberglass	Double	13	no	Drop tube flapper	51	68	0
48-CO-FG	CO	4-Feb-15	Denver	a	12,000	118	Fiberglass	Double	3	no	Ball float	75	44	0
49-NY-ST	NY	6-Feb-15	Long Island	h	10,000	120	Steel	Single	5	no	Drop tube flapper	76	44	0

Table B-1. General Information For Inspected ULSD USTs Continued.

Battelle ID	What is the approximate monthly ULSD throughput in gallons? How many deliveries per month or what is the frequency of deliveries?	From what fuel terminal(s) is the ULSD received? If more than one, list and provide approximate percentage pulled from each.	What carrier(s) are being used for this site? Is the company a common carrier, jobber, company owned trucks, etc.?	Has this UST experienced or is currently experiencing corrosion problems since the introduction of ULSD in 2006?	Has anyone ever noticed strange odors coming from the tank or filters? Vinegar type smell.	How long have you owned/operated this site?	Has this UST held HSD (high sulfur diesel), LSD (low sulfur diesel) before ULSD?	Was the UST converted from gasoline service to ULSD service? Are the vent lines common to both the diesel tank and the gasoline tanks?
1-IL-FG	8,333	1095 - Forest View, IL	Chicago - Solar Transport	Unknown	Unknown	Since 2006	Unknown	No
5-IL-FG	8,066	884 Lockport, IL	Chicago - Solar Transport	Unknown	Unknown	Since 2005	Unknown	No
8-IN-FG	7,864	394 South Bend, IN	South Bend - KAG Group	Unknown	Unknown	Since 2006	Unknown	No
9-IN-FG	3,494	394 South Bend, IN	South Bend - KAG Group	Unknown	Unknown	Since 2006	Unknown	No
10-MO-FG	8,871	266 Kansas City, MO	Kansas City - Solar Transport	Unknown	Unknown	Since 2005	Unknown	No
11-KS-FG	18,441	266 Kansas City, MO	Wichita - United Petroleum Transport	Unknown	Unknown	Since 2011	Unknown	Unknown
12-MO-FG	23,509	266 Kansas City, MO	Kansas City - Solar Transport	Unknown	Unknown	Since 2006	Unknown	No
13-LA-FG	11,509	432 Pt Allen, LA	Chalmette - Dupre Transport	Unknown	Unknown	Since 2008	Unknown	No
14-LA-ST	20,797	189 Baton Rouge, LA	Baton Rouge - Dupre Transport	No	Unknown	Since 2012	Unknown	No
15-LA-FG	9,726	189 Baton Rouge, LA	Baton Rouge - Dupre Transport	Unknown	Unknown	Since 2000	Unknown	Yes converted. No common lines.
16-TN-FG	15,435	158 Knoxville, TN	Knoxville - Eagle Transport	Unknown	Unknown	Since 2004	Unknown	No
17-TN-FG	31,530	158 Knoxville, TN	Knoxville - Eagle Transport	Unknown	Unknown	Since 2011	Unknown	No
22-CO-FG	14,519	1023 Denver, CO	Denver - Offen Petroleum	Unknown	Unknown	Since 2012	Unknown	No
23-CO-FG	56,426	1023 Denver, CO	Denver - Offen Petroleum	Unknown	Unknown	Since 2014	Unknown	No

Table B-1. General Information For Inspected ULSD USTs Continued.

Battelle ID	What preventative maintenance procedures are used? At what frequency?	What water bottom practices (SOP or protocols) are used? At what frequency is water removed from the UST?	How often is the filter replaced? Is the filter inspected?	Are additives added to the fuel before or after the fuel is dropped into the UST?	Is the fuel treated with biocides? If yes: What type, how frequent, what process is followed?	Have any components on the tank/pump/filter system been replaced? Which one(s)? How often? When?	Has the UST ever been emptied and cleaned or resurfaced? If yes: What and when was done?
1-IL-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
5-IL-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
8-IN-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
9-IN-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
10-MO-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
11-KS-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	Unknown
12-MO-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
13-LA-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
14-LA-ST	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
15-LA-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
16-TN-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
17-TN-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
22-CO-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
23-CO-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No

Table B-1. General Information For Inspected ULSD USTs Continued.

Battelle ID	What is the approximate monthly ULSD throughput in gallons? How many deliveries per month or what is the frequency of deliveries?	From what fuel terminal(s) is the ULSD received? If more than one, list and provide approximate percentage pulled from each.	What carrier(s) are being used for this site? Is the company a common carrier, jobber, company owned trucks, etc.?	Has this UST experienced or is currently experiencing corrosion problems since the introduction of ULSD in 2006?	Has anyone ever noticed strange odors coming from the tank or filters? Vinegar type smell.	How long have you owned/operated this site?	Has this UST held HSD (high sulfur diesel), LSD (low sulfur diesel) before ULSD?	Was the UST converted from gasoline service to ULSD service? Are the vent lines common to both the diesel tank and the gasoline tanks?
24-CO-ST	2,500	Suncor	Offen	No	No	3.5 years	Unkown	No
26-CO-FG	350,000 per year	Suncor Denver	Manweiler Trans	Not answered	Not answered	Not answered	Not answered	Not answered
27-CA-ST	25,000-30,000	All 5 Bay area refineries	Robinson Oil carriers	None known about	Unknown	13 years	Yes	No
28-CA-ST	23,000	All 5 Bay area refineries	Robinson Oil carriers	None known about	Unknown	13 years	Yes	No
29-CA-FG	160,000	All 5 Bay area refineries	Robinson Oil carriers	None known about	Unknown	13 years	Yes	No
30-CA-FG	3,000	All 5 Bay area refineries	Robinson Oil carriers	None known about	Unknown	13 years	Yes	No
31-CA-ST	14,000	All 5 Bay area refineries	Robinson Oil carriers	Yes. Tank failed in 2013 and was lined with FRP	Unknown	30+ years	Yes	No
32-NY-FG	21,000	Northville	CBC (Common Carrier)	Yes. Clogged filters and valves	No	30 years	Yes	No
33-NY-FG	27,000	Northville	CBC (Common Carrier)	Yes. Clogged filters and valves	No	30 years	No	No
35-NY-FG	15,000	Northville 25%, Carbo 45%, Motiva 30%	CBC (Common Carrier)	Yes. Clogged filters and valves	No	30 years	Yes	No
47-LA-FG	14,924	432 Pt Allen, LA	Chalmette - Dupre Transport	Unknown	Unknown	Since 2002	Unknown	Unknown
48-CO-FG	34,075	1023 Denver, CO	Denver - Offen Petroleum	Unknown	Unknown	Since 2012	Unknown	No
49-NY-ST	40,000	Northville 85% Carbo 12% Motiva 3%	CBC (Common Carrier)	Yes. Clogged filters and valves	No	30 years	No	No

Table B-1. General Information For Inspected ULSD USTs Continued.

Battelle ID	What preventative maintenance procedures are used? At what frequency?	What water bottom practices (SOP or protocols) are used? At what frequency is water removed from the UST?	How often is the filter replaced? Is the filter inspected?	Are additives added to the fuel before or after the fuel is dropped into the UST?	Is the fuel treated with biocides? If yes: What type, how frequent, what process is followed?	Have any components on the tank/pump/filter system been replaced? Which one(s)? How often? When?	Has the UST ever been emptied and cleaned or resurfaced? If yes: What and when was done?
24-CO-ST	Kubat/monthly	When measured by water sensor	Inspected Monthly	Yes, Before	Unkown	As Needed	Unkown
26-CO-FG	Not answered	Not answered	Not answered	Not answered	Not answered	Not answered	Not answered
27-CA-ST	Tank polishing every 2 yrs	Removal as soon as possible once noted by monitoring system	Every 6 months	None	None	Dispenser filters	N/A
28-CA-ST	Tank polishing every 2 yrs	Removal as soon as possible once noted by monitoring system	Every 6 months	None	None	Dispenser filters	N/A
29-CA-FG	Tank polishing every 2 yrs.	Removal as soon as possible once noted by monitoring system	Every 6 months	None	None	Dispenser filters	N/A
30-CA-FG	Tank polishing every 2 yrs.	Removal as soon as possible once noted by monitoring system	Every 6 months	None	None	Dispenser filters	N/A
31-CA-ST	Tank polishing every 2 yrs.	Removal as soon as possible once noted by monitoring system	Every 6 months	None	None	Dispenser filters	April 2013 tank was emptied, sand blasted and lined with FRP
32-NY-FG	Used Bio Bore (Infrequent)	No practice/protocol	3-4 times a year (coffee ground substance in filters)	Yes, Before	No	Valves, Leak Detectors, Breakaways, Nozzles	No
33-NY-FG	Used Bio Bore (Infrequent)	no water	3-4 times a year (coffee ground substance in filters)	Yes, Before	No	Valves, Leak Detectors, Breakaways, Nozzles	No
35-NY-FG	Used Bio Bore (Infrequent)	no water	3-4 times a year (coffee ground substance in filters)	Yes, Before	No	Valves, Leak Detectors, Breakaways, Nozzles	No
47-LA-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
48-CO-FG	N/A	Removed at ≥1 inch	Only when slow flow issues are encountered	No	Only if tank is cleaned	Unknown	No
49-NY-ST	Used Bio Bore (Infrequent)	no water	3-4 times a year (coffee ground substance in filters)	Yes, Before	No	Valves, Leak Detectors, Breakaways, Nozzles	No

Table B-2. Analytical Results For Vapor Samples

Battelle ID	Site ID	1=Minimal 2=Moderate 3=Severe Corrosion Class	Acetic Acid (ppbv) ALS 102	Formic Acid (ppbv) ALS 102	Lactic Acid (ppbv) Mod NIOSH 7903	Propionic Acid (ppbv) ALS 102	Isobutyric Acid (ppbv) ALS 102	Butanoic Acid (Butyric) (ppbv) ALS 102	Pentanoic Acid (Valeric) (ppbv) ALS 102	Hexanoic Acid (Caproic) (ppbv) ALS 102	Octanoic Acid (Caprylic) (ppbv) ALS 102
1	1-IL-FG	2	200	320	ND	ND	ND	ND	ND	ND	ND
2	2-IL-FG	2	2700	140	ND	ND	ND	ND	ND	ND	ND
3	3-IL-ST	3	ND	960	ND	ND	ND	ND	ND	ND	ND
5	5-IL-FG	2	340	570	ND	ND	ND	ND	ND	ND	ND
6	6-IN-ST	3	ND	1500	ND	ND	ND	ND	ND	ND	ND
7	7-IN-ST	3	150	1700	ND	ND	ND	ND	ND	ND	ND
8	8-IN-FG	2	ND	210	ND	ND	ND	ND	ND	ND	ND
9	9-IN-FG	3	15	270	ND	ND	ND	ND	ND	ND	ND
10	10-MO-FG	2	28	76	ND	ND	ND	ND	ND	ND	ND
11	11-KS-FG	3	990	300	ND	ND	ND	ND	ND	ND	ND
12	12-MO-FG	2	88	85	ND	3.1	ND	ND	ND	ND	ND
13	13-LA-FG	3	6200	ND	ND	ND	ND	ND	ND	ND	ND
14	14-LA-ST	1	15	ND	ND	ND	ND	ND	ND	ND	ND
15	15-LA-FG	3	220	70	ND	ND	ND	ND	ND	ND	ND
16	16-TN-FG	3	1100	65	ND	ND	ND	ND	ND	ND	ND
17	17-TN-FG	2	ND	120	ND	ND	ND	ND	ND	ND	ND
18	18-TN-ST	2	ND	71	ND	ND	ND	ND	ND	ND	ND
22	22-CO-FG	1	100	ND	ND	3.5	ND	1.6	1	1.6	ND
23	23-CO-FG	2	230	64	ND	ND	ND	ND	ND	ND	ND
24	24-CO-ST	3	21	300	ND	ND	ND	ND	ND	ND	ND
26	26-CO-FG	3	28	ND	ND	ND	ND	ND	ND	ND	ND
27	27-CA-ST	2	87	74	ND	2.1	0.83	2.5	ND	0.63	ND
28	28-CA-ST	3	1900	69	ND	ND	ND	ND	ND	ND	ND
29	29-CA-FG	1	1500	86	ND	ND	ND	ND	ND	ND	ND
30	30-CA-FG	3	62	96	ND	1.4	1	1.3	ND	ND	ND
31	31-CA-ST	1	80	490	ND	ND	ND	ND	ND	ND	ND
32	32-NY-FG	3	400	150	ND	5.5	ND	ND	ND	ND	ND
33	33-NY-FG	3	81	330	ND	8	ND	ND	ND	ND	ND
34	34-NY-ST	2	230	250	ND	1	ND	0.73	ND	ND	ND
35	35-NY-FG	3	2600	110	ND	ND	ND	ND	ND	ND	ND
36	36-NY-ST	3	230	620	ND	1.2	ND	3.9	ND	ND	ND
37	37-NY-ST	2	39	160	ND	0.96	ND	0.91	ND	ND	ND
39	39-NJ-ST	2	1200	83	ND	ND	ND	ND	ND	ND	ND
40	40-PA-ST	2	31	100	ND	1.1	ND	ND	ND	ND	ND
42	42-PA-ST	1	150	76	ND	4.5	6.3	2.5	ND	ND	ND
43	43-MD-ST	3	1000	140	ND	3.8	3.5	5.8	ND	ND	ND
44	44-VA-FG	2	630	ND	ND	ND	ND	ND	ND	ND	0.77
45	45-VA-FG	1	29	650	ND	ND	ND	ND	ND	ND	ND
46	46-IL-ST	2	ND	2100	ND	ND	ND	ND	ND	ND	ND
47	47-LA-FG	3	2500	ND	ND	ND	ND	ND	ND	ND	ND
48	48-CO-FG	1	550	470	ND	ND	ND	ND	ND	ND	ND
49	49-NY-ST	2	14	840	ND	ND	ND	ND	ND	ND	ND

All other results were not detected for 2-Methylbutanoic acid, 3-Methylbutanoic acid, 2-Methylpentanoic acid, 3-Methylpentanoic acid, 4-Methylpentanoic acid, Heptanoic acid, 2-Ethylhexanoic acid, Benzoic acid, Cyclohexanecarboxylic acid, Nonanoic acid

Table B-3. Measured Relative Humidity and Temperature and Cluster Location Annual Average Ambient Relative Humidity and Temperature

UST System ID	Corrosion Class	Average Relative Humidity in Tank (%)	Average Temperature in Tank (°C)	Cluster Location Designation*	Cluster Location Annual Average Ambient Relative Humidity (%)**	Cluster Location Annual Temperature (°C)**
1-IL-FG	Moderate	41	4.3	Chicago/ Northern Indiana	70	10.0
2-IL-FG	Moderate	83	6.9	Chicago/ Northern Indiana	70	10.0
3-IL-ST	Severe	59	4.4	Chicago/ Northern Indiana	70	10.0
5-IL-FG	Moderate	33	5.4	Chicago/ Northern Indiana	70	10.0
6-IN-ST	Severe	80	6.2	Chicago/ Northern Indiana	70	10.0
7-IN-ST	Severe	86	6.3	Ft. Wayne	79***	9.9***
8-IN-FG	Moderate	65	5.9	Ft. Wayne	79***	9.9***
9-IN-FG	Severe	74	5.2	Chicago/ Northern Indiana	70	10.0
10-MO-FG	Moderate	88	9.9	Kansas City	68	14.0
11-KS-FG	Severe	79	11.3	Kansas City	68	14.0
12-MO-FG	Moderate	59	8.4	Kansas City	68	14.0
13-LA-FG	Severe	84	19.0	Louisiana	76	21.0
14-LA-ST	Minimal	73	16.7	Louisiana	76	21.0
15-LA-FG	Severe	95	15.6	Louisiana	76	21.0
16-TN-FG	Severe	67	11.5	Knoxville	76***	13.1***
17-TN-FG	Moderate	71	8.8	Knoxville	76***	13.1***
18-TN-ST	Moderate	70	11.3	Knoxville	76***	13.1***
22-CO-FG	Minimal	36	10.7	Denver	52	10.0
23-CO-FG	Moderate	52	11.8	Denver	52	10.0
24-CO-ST	Severe	72	2.6	Denver	52	10.0
26-CO-FG	Severe	35	15.2	Denver	52	10.0
27-CA-ST	Moderate	57	19.8	San Francisco	74	14.0
28-CA-ST	Severe	56	20.2	San Francisco	74	14.0
29-CA-FG	Minimal	62	10.7	San Francisco	74	14.0
30-CA-FG	Severe	61	18.1	San Francisco	74	14.0
31-CA-ST	Minimal	82	11.0	San Francisco	74	14.0
32-NY-FG	Severe	68	0.8	Long Island	63	13.0
33-NY-FG	Severe	83	3.7	Long Island	63	13.0
34-NY-ST	Moderate	98	4.6	Long Island	63	13.0
35-NY-FG	Severe	72	5.7	San Francisco	74	14.0
36-NY-ST	Severe	77	3.6	Long Island	63	13.0
37-NY-ST	Moderate	71	4.7	Long Island	63	13.0

Table B-3. Measured Relative Humidity and Temperature and Cluster Location Annual Average Ambient Relative Humidity and Temperature

UST System ID	Corrosion Class	Average Relative Humidity in Tank (%)	Average Temperature in Tank (°C)	Cluster Location Designation*	Cluster Location Annual Average Ambient Relative Humidity (%)**	Cluster Location Annual Temperature (°C)**
39-NJ-ST	Moderate	76	7.7	Southeast PA	67	13.0
40-PA-ST	Moderate	57	3.0	Southeast PA	67	13.0
42-PA-ST	Minimal	69	5.6	Southeast PA	67	13.0
43-MD-ST	Severe	66	5.1	Washington, D.C.	64	14.5
44-VA-FG	Moderate	83	8.5	Washington, D.C.	64	14.5
45-VA-FG	Minimal	82	7.9	Washington, D.C.	64	14.5
46-IL-ST	Moderate	55	4.2	Chicago/ Northern Indiana	70	10.0
47-LA-FG	Severe	61	16.5	Louisiana	76	21.0
48-CO-FG	Minimal	58	1.0	Denver	52	10.0
49-NY-ST	Moderate	77	0.9	Long Island	63	13.0

* Weather data for cluster location based on the following cities (Cluster name - city used for weather data): Louisiana - New Orleans, LA; Washington, D.C. - Washington, D.C.; San Francisco - San Francisco, CA; Denver - Denver, CO; Southeast PA - Philadelphia, PA; Ft. Wayne - Ft. Wayne, IN; Knoxville - Knoxville, TN; Chicago/Northern Indiana - Chicago, IL; Long Island - New York, NY; Kansas City - Kansas City, MO.
**Data obtained from http://www.currentresults.com/Weather/US/weather-averages-index.php unless otherwise noted.
*** Data obtained from http://www.usa.com/.

Table B-4. Analytical Results for Water Samples.

Site ID	1=Minimal 2=Moderate 3=Severe	pH	Conductivity (µS/cm)	Ethanol (ppm)	Methanol (ppm)	Acetic Acid (ppm)	Formic Acid (ppm)	Propionic Acid (ppm)	Glycerin (ppm)	Lactic Acid (ppm)
	Corrosion Class	EPA 150.1	EPA 120.1	SW846 8015B	SW846 8015B	Mod EPA 300	Mod EPA 300	Mod EPA 300	Mod EPA 300	Mod EPA 300
2-IL-FG	2	4.07	2,670	4100	100	13003	43.39	12.48	<0.10	28.05
6-IN-ST	3	4.86	2,540	230	ND	3745	269	32.09	<0.25	<2.56
7-IN-ST	3	5.04	7,530	4100	ND	19078	123	22.56	<0.10	<2.53
13-LA-FG	3	4.14	1,010	8400	110	8790	1.29	10.95	<0.025	17.39
18-TN-ST	2	6.24	5,280	71000	ND	1517	54.33	481	<0.50	38.18
26-CO-FG	3	4.49	8,610	2000	2400	19919	67.0	<0.26	2399	1464
35-NY-FG	3	5.43	6,390	5700	650	26971	73.97	4.08	496	345
37-NY-ST	2	4.14	3,700	210	140	6.98	<0.14	<0.14	<0.006	0.70
44-VA-FG	2	3.86	6,180	5100	440	17684	106	2.40	18322	599
45-VA-FG	1	3.37	4,450	320	33	5421	19.10	<0.26	2183	79.97
48-CO-FG	1	4.55	6,470	4800	3200	25157	196	<0.26	2590	1915

Site ID	1=Minimal 2=Moderate 3=Severe	F (ppm)	Cl (ppm)	NO_3 (ppm)	SO_4 (ppm)	Na (ppm)	NH_4 (ppm)	K (ppm)	Mg (ppm)	Ca (ppm)
	Corrosion Class	D 6919	D 6919	D 6919	D 6919	D 6919	D 6919	D 6919	D 6919	D 6919
2-IL-FG	2	30.91	720	<0.13	131	2378	<1.76	23.01	42.14	80.35
6-IN-ST	3	118	718	<0.13	4.27	1977	181	19.14	1.66	1.12
7-IN-ST	3	15.73	2139	<0.13	16.95	3043	224	640	89.58	399
13-LA-FG	3	<0.13	29.61	<0.13	32.57	1987	<1.91	3.30	2.97	33.19
18-TN-ST	2	20.67	1920	<0.13	24.25	4186	22.48	1112	627	4.51
26-CO-FG	3	<0.26	3468	<0.26	156	4687	<2.51	19.85	21.44	98.63
35-NY-FG	3	<0.25	1921	7.44	272	3658	404	307	85.50	253
37-NY-ST	2	<0.14	2.30	<0.14	1.72	176	<0.14	<0.14	<0.14	<0.14
44-VA-FG	2	88.34	2653	79.29	152	4224	14.42	46.72	208	230
45-VA-FG	1	36.29	1949	3.66	358	3326	<1.28	32.46	85.98	151
48-CO-FG	1	117	1321	11.35	83.20	3120	35.86	19.85	<1.25	98.63

Table B-5. Quantitative Analytical Results for Fuel Samples.

Site ID	1=Minimal 2=Moderate 3=Severe	Sulfur Content (ppm)	Density (g/mL)	Flashpoint (°C)	Total Acid Number (TAN) (mg KOH/g)	Particulates (mg/L)	Free Glycerin (wt. %)	Total Glycerin (wt. %)	Biodiesel Content (% volume)	NACE (A-E Ratings)	Water Content (ppm)	Conductivity (pS/m at 70°F)
	Corrosion Class	D 5453	D 4052	D 93	D 664	D 2617	D 7591	D 7591	D 7371	TM0172	D 6304	D2624
1-IL-FG	2	11.3	0.86	54.0	0.01	5.2	<0.001	<0.001	4.9	A	76	514
2-IL-FG	2	11.4	0.86	53.0	0.01	16.8	<0.001	<0.001	0.0	A	78	385
3-IL-ST	3	5.9	0.82	52.0	0.03	10.8	<0.001	<0.001	0.0	A	38	450
5-IL-FG	2	10.1	0.86	53.5	0.02	3.6	<0.001	0.004	11.0	A	83	477
6-IN-ST	3	6.8	0.86	69.5	0.04	19.2	<0.001	<0.001	0.0	A	105	290
7-IN-ST	3	7.8	0.84	46.5	0.01	9.6	<0.001	<0.001	0.0	A	68	70
8-IN-FG	2	12.6	0.85	56.0	0.02	9.6	<0.001	<0.001	0.0	A	40	120
9-IN-FG	3	7.5	0.85	61.5	0.04	285.6	<0.001	<0.001	0.0	A	202	489
10-MO-FG	2	8.2	0.84	57.0	0.01	13.2	0.028	0.021	4.4	A	172	425
11-KS-FG	3	8.0	0.84	62.0	0.06	158.0	0.001	0.006	4.3	A	127	464
12-MO-FG	2	8.2	0.84	56.0	0.01	6.8	<0.001	0.005	4.5	A	58	327
13-LA-FG	3	9.0	0.83	56.0	0.02	6.4	<0.001	<0.001	0.0	A	81	429
14-LA-ST	1	9.4	0.84	59.0	0.01	4.8	<0.001	<0.001	0.0	A	63	385
15-LA-FG	3	8.3	0.83	53.0	0.01	5.6	<0.001	<0.001	0.0	A	66	360
16-TN-FG	3	6.0	0.84	58.0	0.02	10.0	<0.001	<0.001	0.0	A	50	118
17-TN-FG	2	6.1	0.84	58.0	0.01	5.6	<0.001	<0.001	0.0	A	16	85
18-TN-ST	2	6.0	0.84	55.0	0.00	6.0	<0.001	<0.001	0.0	A	26	155
22-CO-FG	1	8.5	0.85	53.0	0.01	6.8	<0.001	0.001	2.0	A	67	407
23-CO-FG	2	8.2	0.85	66.0	0.01	4.8	<0.001	0.001	2.3	A	50	444
24-CO-ST	3	8.0	0.84	53.0	0.02	294.4	0.008	0.006	2.1	E	264	345
26-CO-FG	3	8.1	0.84	58.0	0.03	78.4	<0.001	<0.001	0.9	A	134	292
27-CA-ST	2	6.4	0.83	56.0	0.04	8.0	<0.001	0.001	2.1	A	58	589
28-CA-ST	3	3.8	0.83	54.0	0.03	112.0	<0.001	<0.001	0.8	E	107	319
29-CA-FG	1	3.2	0.84	58.0	0.03	3.6	<0.001	0.003	3.5	A	62	174
30-CA-FG	3	6.4	0.83	64.0	0.02	12.0	<0.001	0.002	3.3	A	94	603
31-CA-ST	1	4.0	0.83	55.0	0.00	4.0	<0.001	<0.001	0.6	A	46	430
32-NY-FG	3	6.3	0.84	54.0	0.04	10.8	<0.001	0.003	5.2	A	96	330
33-NY-FG	3	6.4	0.84	55.0	0.04	9.2	<0.001	0.003	5.3	A	84	382
34-NY-ST	2	7.2	0.84	55.0	0.03	5.6	<0.001	0.006	5.1	A	74	131
35-NY-FG	3	7.7	0.84	47.0	0.36	41.0	<0.001	0.003	3.4	A	186	209
36-NY-ST	3	8.0	0.84	52.0	0.01	2.0	<0.001	<0.001	0.1	A	41	768
37-NY-ST	2	7.6	0.84	56.0	0.01	63.0	<0.001	<0.001	1.18	A	154	680
39-NJ-ST	2	7.5	0.83	58.0	0.00	4.4	<0.001	<0.001	0.98	A	41	242
40-PA-ST	2	9.5	0.83	58.0	0.02	13.2	<0.001	0.002	2.4	A	218	350
42-PA-ST	1	7.9	0.84	55.0	0.03	3.2	<0.001	0.002	2.7	A	48	102
43-MD-ST	3	7.1	0.84	54.0	0.00	5.6	<0.001	<0.001	0.0	NA	41	74
44-VA-FG	2	7.9	0.84	54.0	0.03	51.6	0.004	0.001	0.3	E	72	128
45-VA-FG	1	7.6	0.84	53.0	0.01	5.2	<0.001	<0.001	0.1	A	76	138
46-IL-ST	2	14.4	0.82	57.0	0.00	85.2	<0.001	<0.001	0.7	A	32	1420
47-LA-FG	3	9.3	0.84	67.0	0.03	8.4	<0.001	0.003	1.7	A	58	488
48-CO-FG	1	7.2	0.85	63.0	0.28	103.2	0.068	0.109	2.0	E	322	321
49-NY-ST	2	6.1	0.84	56.0	0.04	5.2	<0.001	0.002	5.1	A	73	410

Table B-5. Qualitative Analytical Results for Fuel Samples.

	Site ID	1=Minimal 2=Moderate 3=Severe	FAME	Biodiesel biproducts	Ethanol	Isopropyl alcohol	Gas Contamination (C4-8)	1, 2, 3, and 4 Ring Aromatics	Water	Other(s)
Battelle ID		Corrosion Class	GC Scan	GC Scan	GC Scan	GC Scan	GC Scan	GC Scan	GC Scan	GC Scan
1	1-IL-FG	2	X	X	X		X	X	X	
2	2-IL-FG	2			X		X	X	X	
3	3-IL-ST	3			X	X	X	X	X	Phthalate (additive)
5	5-IL-FG	2	X	X	X	X	X	X	X	Diisooctylphthalate (additive)
6	6-IN-ST	3			X		X	X	X	
7	7-IN-ST	3			X		X	X	X	Diisooctylphthalate (additive), butoxyethanol
8	8-IN-FG	2			X	X	X	X	X	Dioctylisophthalate (additive)
9	9-IN-FG	3	X		X	X	X	X	X	
10	10-MO-FG	2	X	X	X	X	X	X	X	
11	11-KS-FG	3	X	X		X	X	X	X	Diisooctylphthalate (additive)
12	12-MO-FG	2	X	X	X	X	X	X	X	Diisooctylphthalate (additive)
13	13-LA-FG	3			X		X	X	X	
14	14-LA-ST	1			X		X	X	X	
15	15-LA-FG	3			X		X	X	X	
16	16-TN-FG	3	X		X		X	X	X	Diisooctlyphthalate
17	17-TN-FG	2			X		X	X	X	
18	18-TN-ST	2			X	X	X	X	X	
22	22-CO-FG	1	X	X	X	X	X	X	X	
23	23-CO-FG	2	X	X	X		X	X	X	
24	24-CO-ST	3	X	X	X		X	X	X	
26	26-CO-FG	3	X		X	X	X	X	X	
27	27-CA-ST	2	X	X	X		X	X	X	
28	28-CA-ST	3	X		X	X	X	X	X	
29	29-CA-FG	1	X	X	X		X	X	X	
30	30-CA-FG	3	X	X			X	X	X	
31	31-CA-ST	1	X		X		X	X	X	
32	32-NY-FG	3	X	X	X		X	X	X	
33	33-NY-FG	3	X	X	X	X	X	X	X	
34	34-NY-ST	2	X	X	X	X	X	X	X	
35	35-NY-FG	3	X	X	X		X	X	X	
36	36-NY-ST	3			X	X	X	X	X	
37	37-NY-ST	2					X	X	X	
39	39-NJ-ST	2			X		X	X	X	
40	40-PA-ST	2	X	X		X	X	X	X	
42	42-PA-ST	1	X	X	X	X	X	X	X	
43	43-MD-ST	3			X		X	X	X	
44	44-VA-FG	2			X		X	X	X	
45	45-VA-FG	1			X	X	X	X	X	
46	46-IL-ST	2			X		X	X	X	Phthalate (additive)
47	47-LA-FG	3	X	X	X		X	X	X	
48	48-CO-FG	1	X	X	X		X	X	X	
49	49-NY-ST	2	X	X	X	X	X	X	X	

X = Present

The following were not present in any of the samples: Methanol, Short Chain Fatty Acids (Acetic acid, Formic acid, Propionic acid, Butyric acid, Valeric acid), Ethylene glycol, Propylene glycol, heavy end contamination (>C18), Glyceric acid derivatives, Glycerin.

Supplement C

QA/QC Data Tables

Analytical QA Summary Tables by Laboratory And Analytical Method

ICFTL Method Summary Results

Method	Description	Average QC Value	Expected Value	ASTM Reproducibility	Units	Evaluation against QA Requirement (pass/fail)
D5453	Sulfur	1.22	1.13	0.64	ppm	pass
D93	Flash Point	132.9	137.2	14.7	° C	pass
D6217	Particulates	5.10	5.10	2.6	mg/L	pass
D7371	% Bidiesel	4.72	4.77	0.95	% volume	pass
D664	Acid Number	0.34	0.34	0.11	mg KOH/g	pass
D6304	KF Moisture	349%	346.60	14	ppm	pass
D4052	Density	0.8841	0.8841	0.0005	g/mL	pass

Method:	ASTM D5453	
Lab:	ICFTL	
Requirement:	Daily QC check (1.19 ±0.66 ppm)	
Date	Result (ppm)	QA Requirement (pass/fail)
2/5/2015	1.62	pass
2/9/2015	1.31	pass
2/10/2015	1.07	pass
2/13/2015	1.09	pass
2/18/2015	1.22	pass
2/24/2015	1.00	pass
Statistical Summary		
mean	1.22	
SD	0.23	
RSD	18.60%	
UCL	1.46	
LCL	0.98	

Method:	ASTM D93	
Lab:	ICFTL	
Requirement:	Daily QC Check (140.7 ±14.7 °C)	
Date	Result (°C)	QA Requirement (pass/fail)
2/4/2015	136.5	pass
2/9/2015	131	pass
2/10/2015	127	pass
2/11/2015	126.5	pass
2/12/2015	124.5	fail[1]
2/19/2015	142	pass
Statistical Summary		
mean	132.9	
SD	7.5	
RSD	5.60%	
UCL	139.8	
LCL	125.9	

1 Sample was not retested since it was so close to the to the lower range

Method:	D6217	
Lab:	ICFTL	
Requirement:	Daily QC Check (5.1 ± 2.6 mg/L)	
Date	Result (g/mL)	QA Requirement (pass/fail)
2/4/2015	4.4	pass
2/10/2015	4.8	pass
2/12/2015	5.6	pass
2/13/2015	6.8	pass
2/16/2015	4.8	pass
2/18/2015	5.2	pass
2/26/2015	4.4	pass
Statistical Summary		
mean	5.1	
SD	0.4	
RSD	8.10%	
UCL	5.9	
LCL	4.4	

Method:	D7371		
Lab:	ICFTL		
Requirement:	Daily QC Check with Certified Reference Material		
	(10.0± 1.2%; 5.0±0.95 %; 2.0 ± %)		
Date	Certified Reference Material Value (%)	Result (% volume)	QA Requirement (pass/fail)
2/4/2015	5	4.64	pass
2/4/2015	5	4.92	pass
2/10/2015	5	4.66	pass
2/10/2015	2	1.77	NA
2/10/2015	10	9.14	pass
2/10/2015	5	4.39	pass
2/18/2015	5	4.76	pass
2/18/2015	5	5.7	pass
2/18/2015	5	4.6	pass
2/18/2015	5	4.58	pass
2/18/2015	5	4.6	pass
Statistical Summary- for 5% reference			
mean	4.72		
SD	0.38		
RSD	8.10%		
UCL	4.99		
LCL	4.55		

NA=no QA criteria given for the 2% reference material. It was run to see how the instrument was running with lower % volumes.

Method:	D664	
Lab:	ICFTL	
Requirement:	Daily QC Check (0.31 ±0.11 mg KOH/g)	
Date	Result (mg KOH/g)	QA Requirement (pass/fail)
2/4/2015	0.33	pass
2/9/2015	0.33	pass
2/12/2015	0.34	pass
2/16/2015	0.33	pass
2/16/2015	0.34	pass
2/26/2015	0.34	pass
Statistical Summary		
mean	0.34	
SD	0.01	
RSD	2.40%	
UCL	0.35	
LCL	0.33	

Method:	D6304	
Lab:	ICFTL	
Requirement:	Daily QC Check (253 ± 467 ppm)	
Date	Result (ppm)	QA Requirement (pass/fail)
2/3/2015	344	pass
2/3/2015	338	pass
2/10/2015	355	pass
2/10/2015	355	pass
2/16/2015	353	pass
2/20/2015	343	pass
2/26/2015	348	pass
Statistical Summary		
mean	349.3	
SD	7.1	
RSD	2.00%	
UCL	355.2	
LCL	343.3	

Method:	D6304	
Lab:	ICFTL	
Requirement:	Quarterly 10 µL DI water check (10,000 ± 200 ppm)	
Date	Trial	Result (ppm)
3/31/2015	1	9784
3/31/2015	2	9606
3/31/2015	3	9788
3/31/2015	4	9856
3/31/2015	5	9880
3/31/2015	6	9933
3/31/2015	7	9920
3/31/2015	8	9953
3/31/2015	9	10003
3/31/2015	10	9994
3/31/2015	11	10036
average		9887
QA requirement (pass/fail)		Pass

Method:	D4052	
Lab:	ICFTL	
Requirement:	Daily QC Check (0.8841 ±0.0005 g/mL)	
Date	Result (g/mL)	QA Requirement (pass/fail)
2/3/2015	0.8842	pass
2/3/2015	0.8841	pass
2/9/2015	0.8841	pass
2/12/2015	0.8843	pass
2/19/2015	0.8842	pass
2/26/2015	0.8842	pass
Statistical Summary		
mean	0.8841	
SD	0.0001	
RSD	0.01%	
UCL	0.8843	
LCL	0.884	

Method:	SW846 8015 B					
Lab:	Test America					
Requirement:	All analytes within ±15% of expected					
Date	**Sample Type**	**Analyte (mg/L)**			**Expected Amount (mg/L)**	**QA Requirement (pass/fail)**
		Ethanol	**Methanol**	**Isobutyl alcohol (IS)**		
2/24/2015	LCS	18.7	19	19.6	20	pass
2/24/2015	LCSD	18.1	19	19.8	20	pass
2/24/2015	MB	0.919	ND	19.6	0, 20	pass
2/10/2015	LCS	19.2	18.8	19.4	20	pass
2/10/2015	MB	ND	ND	19.4	0, 20	pass
2/10/2015	MS	8200	129	22.1	8120, 130, 20	pass
2/10/2015	MSD	7440	105	21	8120, 130 ,20	pass

LCS=Lab Control Sample; LCSD=Lab Control Sample Duplicate, MB=Method Blank; MS= Matrix Spike,

Method:	D6919-Anions				
Lab:	Metrohm				
Requirement:	Correlation coefficient of 0.995 or greater. 90-110% Recovery of QC samples				
Date	**Analyte**	**QC sample results (ppm)**	**QC Target (ppm)**	**% Recovery**	**QA Requirement (pass/fail)**
2/5/15, 2/6/2015, 3/26/2015	F	5.11	5.03	101.63	pass
	Cl	5.09	5.13	99.2	pass
	NO3	5.05	5.15	98	pass
	SO4	5.16	5.18	99.75	pass
2/12/2015, 2/17/2015, 2/18/2015, 3/12/2015	F	5.21	5.09	102.38	pass
	Cl	4.96	5.05	98.16	pass
	NO3	4.95	5.1	97.1	pass
	SO4	4.95	5.11	96.77	pass
2/5/2015, 2/6/2015	F	5.21	5.03	103.48	pass
	Cl	5.18	5.13	101.01	pass
	NO3	5.22	5.15	101.4	pass
	SO4	5.17	5.18	99.94	pass
2/12/2015, 2/17/2015, 3/27/2015	F	5.14	5.02	102.39	pass
	Cl	4.92	5.04	97.62	pass
	NO3	4.76	5.01	95.55	pass
	SO4	4.83	5.06	95.55	pass
2/13/2015, 2/18/2015, 3/28/2015	F	5.13	5.02	102.23	pass
	Cl	4.91	5.04	97.6	pass
	NO3	4.74	5.01	94.55	pass
	SO4	4.81	5.06	95.17	pass
2/13/2015, 3/27/2015	F	5.14	5.02	102.29	pass
	Cl	4.9	5.04	97.36	pass
	NO3	4.73	5.01	94.33	pass
	SO4	4.81	5.06	95.13	pass
3/27/2015, 3/28/2015	F	5.14	5.02	102.33	pass
	Cl	4.91	5.04	97.48	pass
	NO3	4.77	5.01	95.25	pass
	SO4	4.85	5.06	95.91	pass

Correlation Coefficient	
Date	**R-squared**
2/5/2015	0.999966
2/6/2015	0.999966
3/26/2015	0.999968
2/12/2015	0.999965
2/17/2015	0.999964
2/18/2015	0.999964
3/12/2015	0.999972
2/15/2015	0.999968
2/13/2015	0.999965
3/28/2015	0.999968
3/27/2015	0.999968

Method:	D6919-Cations				
Lab:	Metrohm				
Requirement:	90-110% Recovery of QC samples				
Dates	**Analyte**	**QC sample results (ppm)**	**QC Target (ppm)**	**% Recovery**	**QA Requirement (pass/fail)**
2/3/2015, 2/4/2015, 2/6/2015, 3/25/2015	Na	5.05	5.08	99.37	pass
	NH_4	4.99	5.01	99.68	pass
	K	5.8	5.84	99.43	pass
	Mg	4.99	5.03	99.22	pass
	Ca	4.95	4.99	99.22	pass
2/12/2015, 3/12/2015	Na	5.05	5.04	100.3	pass
	NH_4	5.14	5.22	98.37	pass
	K	4.96	5.09	97.47	pass
	Mg	5.01	5.09	98.39	pass
	Ca	5.05	5.21	96.89	pass
2/3/2015, 2/4/2015, 2/7/2015, 3/29/2015	Na	5.09	5.08	100.12	pass
	NH_4	5.04	5.01	100.68	pass
	K	5.84	8.84	100.07	pass
	Mg	5	5.03	99.34	pass
	Ca	4.94	4.99	98.96	pass
2/13/2015, 2/24/2015, 3/25/2015	Na	5.01	5.04	99.36	pass
	NH_4	5.13	5.22	98.28	pass
	K	4.94	5.09	96.94	pass
	Mg	4.93	5.09	96.99	pass
	Ca	5	5.21	96.01	pass
2/13/2015, 3/23/15	Na	5.11	5.06	100.91	pass
	NH_4	5.09	5.03	101.19	pass
	K	5.17	5.17	99.94	pass
	Mg	5.07	5.07	99.9	pass
	Ca	5.1	5.15	98.97	pass
2/13/2015, 3/26/2015	Na	5.14	5.06	101.42	pass
	NH_4	5.07	5.03	100.84	pass
	K	5.14	5.17	99.5	pass
	Mg	5	5.07	98.6	pass
	Ca	5.03	5.15	97.65	pass
3/26/2015, 3/27/2015, 3/28/2015	Na	5.13	5.06	101.28	pass
	NH_4	5.14	5.03	102.19	pass
	K	5.15	5.17	99.63	pass
	Mg	5.12	5.07	100.87	pass
	Ca	5.12	5.15	99.34	pass

Method:	D6919-Cations
Lab:	Metrohm
Requirement:	Correlation coefficient of 0.995 or greater.

Dates	**R-squared**
2/3/2015	0.999999
2/4/2015	0.999999
2/6/2015	0.99999
3/25/2015	1
2/12/2015	1
3/12/2015	0.999994
2/7/2015	0.99999
3/29/2015	0.999997
2/13/2015	1
2/24/2015	0.999966
3/27/2015	1
3/23/2015	0.999996
3/26/2015	1
3/28/2015	1

Method:	Modified EPA 300				
Lab:	Metrohm				
Requirement:	90-110% Recovery of QC samples				
Dates*	**Analyte**	**QC sample results (ppm)**	**QC Target (ppm)**	**% Recovery**	**QA Requirement (pass/fail)**
2/3/2015, 2/5/2015, 2/6/2015, 3/25/2015	Formic	5.18	5.07	102.23	pass
	Acetic	5.05	5.03	100.38	pass
	Propionic	5.17	5.01	103.23	pass
	Lactic	0.092	0.101	91.09	pass
2/12/15, 3/12/15, 3/16/2015	Formic	5.45	5.12	106.53	pass
	Acetic	5.36	5.11	104.79	pass
	Propionic	5.42	5.08	106.61	pass
	Lactic	0.1	0.1	100	pass
2/26/2015, 2/27/2015, 2/14/2015, 2/10/2015	Formic	5.19	5.07	102.49	pass
	Acetic	5.08	5.03	100.81	pass
	Propionic	5.13	5.01	102.37	pass
	Lactic	5.13	5.01	102.37	pass
2/13/2015, 3/27/2015	Formic	5.35	5.03	106.42	pass
	Acetic	5.44	5.04	107.86	pass
	Propionic	5.47	5.02	108.91	pass
	Lactic	0.1	0.1	97	pass
2/13/2015, 3/28/2015	Formic	5.32	5.03	105.87	pass
	Acetic	5.43	5.04	107.8	pass
	Propionic	5.07	5.02	100.94	pass
	Lactic	0.09	0.1	94	pass
2/13/2015, 3/26/2015	Formic	5.35	5.03	106.32	pass
	Acetic	5.39	5.04	106.87	pass
	Propionic	5.47	5.02	109.03	pass
	Lactic	0.1	0.1	100	pass
3/26/2015, 3/27/2015	Formic	5.4	5.03	107.32	pass
	Acetic	5.54	5.04	109.9	pass
	Propionic	5.48	5.02	109.21	pass
	Lactic	0.09	0.1	93	pass

Method:	Modified EPA 300		
Lab:	Metrohm		
Requirement:	Correlation coefficient of 0.995 or greater		
Formic, Acetic and Propionic		**Lactic**	
Date	**R-squared**	**Date**	**R-squared**
2/3/2015	0.99995	2/9/2015	0.999992
2/5/2015	0.99995	2/10/2015	0.999992
2/6/2015	0.99995	2/13/2015	0.999989
3/25/2015	0.999406	2/24/2015	0.999993
2/12/2015	0.999918	2/26/2015	0.999993
3/12/2015	0.999715	3/17/2015	0.999986
3/16/2015	0.999715	3/18/2015	0.999986
2/26/2015	0.999987	2/26/2015	0.999987
2/27/2015	0.999987	2/27/2015	0.999987
2/14/2015	0.999989	2/14/2015	0.999989
2/10/2015	0.999992	2/10/2015	0.999992
2/13/2015	0.999918	3/27/2015	0.999999
3/28/2015	0.999406	3/29/2015	0.999993
3/26/2015	0.999406		
3/27/2015	0.999406		

Method:	Lab-In House Method for Glycerin			
Lab:	Metrohm			
Requirement:	Correlation coefficient of 0.995 or greater. 90-110% Recovery of QC samples			
Dates	**QC sample results (ppm)**	**QC Target (ppm)**	**% Recovery**	**QA Requirement (pass/fail)**
2/6/2015, 2/9/2015, 2/10/2015, 3/27/2015, 3/28/2015	3.05	3.01	101.56	pass
2/13/2015, 3/17/2015	3.09	3	103.21	pass
2/6/2015, 2/9/2015, 2/10/2015, 3/29/2015, 3/3/2015	3.03	3.01	100.7	pass
2/14/2015, 3/29/2015	3.11	3.01	103.36	pass
2/14/2015, 2/23/2015, 3/17/2015, 3/29/2015	3.1	3.01	103.19	pass
2/14/2015, 2/23/2015, 3/27/2015, 3/29/2015, 3/30/2015, 3/31/2015	3.02	3.01	100.5	pass
2/23/2015, 3/27/2015, 3/29/2015, 3/30/2015	3.03	3.01	100.7	pass

Correlation Coefficient	
Date	**R-squared**
2/6/2015	0.999999
2/9/2015	0.999999
2/10/2015	0.999999
3/27/2015	0.99967
3/28/2015	0.999987
2/13/2015	0.999973
3/17/2015	1
3/29/2015	0.999994
3/30/2015	0.999982
2/14/2015	0.999973
2/23/2015	0.997309
3/27/2015	0.99967
3/29/2015	0.999994
3/31/2015	0.999982

Method	Modified NIOSH 7903			
Lab	ALS Environmental			
Requirement	Within established control limits of 70 - 130 % Recovery for Method Spike, ND for Method Blanks			
Sample ID	**Date**	**Lactic Acid QC sample Result (ppm)**	**% Recovery**	**QA Requirement (pass/fail)**
MBLK-26781-26781	2/10/2015	ND	NA	pass
LCS-26781-26781	2/10/2015	9.938	99%	pass
LCSD-26781-26781	2/10/2015	9.971	100%	pass
44-VA-FG-VC-VFB	2/11/2015	ND	NA	pass
26-CO-FG-VC-VFB	2/11/2015	ND	NA	pass
MBLK2-26890-26890	2/18/2015	ND	NA	pass
LCS-26890-26890	2/17/2015	10.04	100%	pass
LCS2-26890-26890	2/18/2015	8.231	82%	pass
LCSD-26890-26890	2/17/2015	10.36	104%	pass
LCSD2-26890-26890	2/18/2015	8.76	88%	pass
6-IN-ST-VC-VFB	2/18/2015	ND	NA	pass
MBLK2-26890-26890	2/18/2015	ND	NA	pass
39-NJ-ST-VC-VFB	2/19/2015	ND	NA	pass
MBLK-26891-26891	2/18/2015	ND	NA	pass
LCS-26891-26891	2/18/2015	10.59	106%	pass
LCSD-26891-26891	2/18/2015	10.54	105%	pass
17-TN-FG-VC-VFB	2/19/2015	ND	NA	pass
29-CA-FG-VC-VFB	2/19/2015	ND	NA	pass

Method	ALS Method 102			
Lab	ALS Environmental			
Requirement	Within established control limits of 50 - 150 % Recovery for Method Spike, ND for Method Blanks			
Sample ID	**Date**	**Formic Acid QC Sample Result (ppbv)**	**% Recovery**	**QA Requirement (pass/fail)**
P150209-MB	2/9/2015	ND	NA	pass
P150209-DLCS	2/9/2015	183	92%	pass
P150209-LCS	2/9/2015	183	92%	pass
44-VA-Fb-VFB	2/10/2015	ND	NA	pass
26-CO-FG-VFB	2/9/2015	ND	NA	pass
P150210-DLCS	2/10/2015	169	85%	pass
P150210-LCS	2/10/2015	172	86%	pass
P150210-MB	2/10/2015	ND	NA	pass
P150225-DLCS	2/25/2015	248	124%	pass
P150225-LCS	2/25/2015	243	122%	pass
P150225-MB	2/25/2015	ND	NA	pass

Method	ALS Method 102									
Lab	ALS Environmental									
Requirement	Within established control limits of the method (varies), ND for Method Blanks									
Analysis Date	**Acetic Acid (ppbv)**	**Propionic Acid (Propanoic) (ppbv)**	**2-Methylpropanoic Acid (Isobutyric) (ppbv)**	**Butanoic Acid (Butyric) (ppbv)**	**2-Methylbutanoic Acid (ppbv)**	**3-Methylbutanoic Acid (Isovaleric) (ppbv)**	**Pentanoic Acid (Valeric) (ppbv)**	**2-Methylpentanoic Acid (ppbv)**	**3-Methylpentanoic Acid (ppbv)**	**4-Methylpentanoic Acid (Isocaproic) (ppbv)**
2/5/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2/5/2015	22.3	8.56	8.99	9.38	9.98	9.83	9.54	10	10.4	10
2/5/2015	24.9	9.22	9.52	9.81	10.1	10	9.53	10.1	10.2	10
2/11/2015	27	9.98	10.1	10.3	10.7	10.6	10.1	10.4	10.7	10.5
2/11/2015	22	8.59	9.02	9.47	9.79	9.82	9.56	10	10.3	10.1
2/11/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2/23/2015	26.1	9.85	10.3	10.4	10.8	10.8	10.3	10.7	11	10.7
2/23/2015	26.4	9.45	10	10.2	10.6	10.4	10.1	10.6	10.8	10.6
2/23/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
QC Requirement (pass/fail)	pass	pass	pass	pass	pass	pass	pass	pass	pass	pass

Method	ALS Method 102						
Lab	ALS Environmental						
Requirement	Within established control limits of the method (varies), ND for Method Blanks						
Analysis Date	**Hexanoic Acid (Caproic) (ppbv)**	**Heptanoic Acid (Enanthoic) (ppbv)**	**2-Ethylhexanoic Acid (ppbv)**	**Cyclohexanecarboxylic Acid (ppbv)**	**Octanoic Acid (Caprylic) (ppbv)**	**Benzoic Acid (ppbv)**	**Nonanoic Acid (Pelargonic) (ppbv)**
2/5/2015	ND	ND	ND	ND	ND	ND	ND
2/5/2015	9.8	7.05	7.57	6.4	6.97	6.85	8.04
2/5/2015	9.76	6.87	7.36	6.28	6.85	6.35	8.05
2/11/2015	10.3	7.16	7.83	6.58	7.2	7.19	8.34
2/11/2015	9.85	6.72	7.35	6.2	6.7	6.91	7.95
2/11/2015	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND
2/23/2015	10.6	7.33	7.07	6.51	7.34	5.38	8.58
2/23/2015	10.4	7.19	7.12	6.24	7.19	5.46	8.51
2/23/2015	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND
2/23/2015	ND	ND	ND	ND	ND	ND	ND
QC Requirement (pass/fail)	pass	pass	pass	pass	pass	pass	pass

Supplement D

Statistics Codebook

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
1	SiteID	SiteID	Char	18	Non-Missing		42	1
					Missing		0	0
2	BattelleID	BattelleID	Num	8	Non-Missing		42	1
					Missing		0	0
3	Corrosion	Corrosion	Num	8	1	minimal	7	0.16667
					2	moderate	17	0.40476
					3	severe	18	0.42857
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
4	geo_cluster	Cluster	Char	16	denver		5	0.11905
					fort_wayne		2	0.04762
					kansas_city		3	0.07143
					knoxville		3	0.07143
					long_island		7	0.16667
					louisiana		4	0.09524
					northern_indiana		7	0.16667
					san_francisco		5	0.11905
					southeast_pa		3	0.07143
					washington_dc		3	0.07143
					Non-Missing		42	1
					Missing		0	0
5	tank_material	Tank Material (Fiberglass or Steel	Char	14	Fiberglass		24	0.57143
					Steel		16	0.38095
					Steel (coated)		2	0.04762
					Non-Missing		42	1
					Missing		0	0
6	tank_capacity	Capacity	Num	8	5000	5000	1	0.02381
					6000	6000	6	0.14286
					7000	7000	1	0.02381
					8000	8000	3	0.07143
					10000	10000	7	0.16667
					12000	12000	18	0.42857
					15000	15000	2	0.04762
					20000	20000	4	0.09524
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
7	stp_type	STP (Make/ Model)	Char	14	FE Petro		20	0.47619
					Red Jacket		16	0.38095
					Tokheim		1	0.02381
					suction system		5	0.11905
					Non-Missing		42	1
					Missing		0	0

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
8	owner	Unnamed Owner ID (a-i)	Char	1		a	16	0.38095
						b	1	0.02381
						c	12	0.28571
						d	1	0.02381
						f	1	0.02381
						g	5	0.11905
						h	4	0.09524
						i	2	0.04762
						Non-Missing	42	1
						Missing	0	0
9	sulfur_f	Sulfur Content (less than 15) Fuel 1.0-8000 ppm	Num	8		Minimum	3.2	.
						10th Percentile	6	.
						25th Percentile	6.4	.
						Median	7.75	.
						75th Percentile	8.3	.
						90th Percentile	10.1	.
						Maximum	14.4	.
						Mean	7.783	.
						Standard Deviation	2.129	.
						Standard Error of the Mean	0.328	.
						Non-Missing	42	1
						Missing	0	0
10	density_f	Density Fuel g/mL	Num	8		Minimum	0.825	.
						10th Percentile	0.829	.
						25th Percentile	0.835	.
						Median	0.839	.
						75th Percentile	0.845	.
						90th Percentile	0.853	.
						Maximum	0.859	.
						Mean	0.84	.
						Standard Deviation	0.008	.
						Standard Error of the Mean	0.001	.
						Non-Missing	42	1
						Missing	0	0
11	flashpoint_f	Flashpoint (38 min in cold mon, 52 min reg) Fuel 40-370 °C	Num	8		Minimum	46.5	.
						10th Percentile	53	.
						25th Percentile	54	.
						Median	56	.
						75th Percentile	58	.
						90th Percentile	63	.
						Maximum	69.5	.
						Mean	56.452	.
						Standard Deviation	4.655	.
						Standard Error of the Mean	0.718	.
						Non-Missing	42	1
						Missing	0	0

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
12	TAN_f	Total Acid Number (TAN) Fuel 0.01-150.0 mg KOH/g	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0.01	.
						Median	0.02	.
						75th Percentile	0.03	.
						90th Percentile	0.04	.
						Maximum	0.36	.
						Mean	0.035	.
						Standard Deviation	0.067	.
						Standard Error of the Mean	0.01	.
						Non-Missing	42	1
						Missing	0	0
13	particulates_f	Particulates (10 max) Fuel mg/L	Num	8		Minimum	2	.
						10th Percentile	4	.
						25th Percentile	5.2	.
						Median	8.8	.
						75th Percentile	19.2	.
						90th Percentile	103.2	.
						Maximum	294.4	.
						Mean	36.133	.
						Standard Deviation	67.292	.
						Standard Error of the Mean	10.383	.
						Non-Missing	42	1
						Missing	0	0
14	free_glycerin_f	Free Glycerin Fuel (wt. ppm)	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0.367	.
						75th Percentile	1.3	.
						90th Percentile	11.5	.
						Maximum	676.3	.
						Mean	26.451	.
						Standard Deviation	112.054	.
						Standard Error of the Mean	17.29	.
						Non-Missing	42	1
						Missing	0	0
15	total_glycerin_f	Total Glycerin Fuel (wt. ppm)	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0.551	.
						Median	7.4	.
						75th Percentile	28.2	.
						90th Percentile	56.6	.
						Maximum	1,088.50	.
						Mean	44.566	.
						Standard Deviation	168.745	.
						Standard Error of the Mean	26.038	.
						Non-Missing	42	1
						Missing	0	0

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
16	biodiesel_content_f	Biodiesel Content (less than 5%) Fuel 1.00-100 % Vol	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	1.08	.
						75th Percentile	3.4	.
						90th Percentile	5.1	.
						Maximum	11	.
						Mean	1.975	.
						Standard Deviation	2.337	.
						Standard Error of the Mean	0.361	.
						Non-Missing	42	1
						Missing	0	0
17	FAME_f	FAME (biodiesel) Fuel	Num	8	0	not present	17	0.40476
					1	present	25	0.59524
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
18	biodiesel_byproducts_f	Biodiesel biproducts Fuel	Num	8	0	not present	22	0.52381
					1	present	20	0.47619
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
19	NACE_f	NACE Fuel Rating	Char	2	A		37	0.88095
					E		4	0.09524
					Non-Missing		41	0.97619
					Missing		1	0.02381
20	water_content_f	Water Content (200 mg/kg max EN ISO) Fuel 10-25000 ppm	Num	8		Minimum	16	.
						10th Percentile	40	.
						25th Percentile	50	.
						Median	72.5	.
						75th Percentile	105	.
						90th Percentile	186	.
						Maximum	322	.
						Mean	91.595	.
						Standard Deviation	65.67	.
						Standard Error of the Mean	10.133	.
						Non-Missing	42	1
						Missing	0	0
21	conductivity_f	Conductivity (min 25 pS/m) Fuel pS/m at 70°F	Num	8		Minimum	70	.
						10th Percentile	118	.
						25th Percentile	174	.
						Median	355	.
						75th Percentile	450	.
						90th Percentile	589	.
						Maximum	1,420	.
						Mean	364.738	.
						Standard Deviation	238.283	.
						Standard Error of the Mean	36.768	.
						Non-Missing	42	1
						Missing	0	0
22	ethanol_f	Ethanol Fuel	Num	8	0	not present	4	0.09524
					1	present	38	0.90476
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
23	isopropyl_alcohol_f	Isopropyl alcohol Fuel	Num	8	0	not present	24	0.57143
					1	present	18	0.42857
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0
24	pH_w	pH Water pH	Num	8		Minimum	3.373	.
						10th Percentile	3.863	.

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
						25th Percentile	4.066	.
						Median	4.491	.
						75th Percentile	5.036	.
						90th Percentile	5.426	.
						Maximum	6.243	.
						Mean	4.563	.
						Standard Deviation	0.8	.
						Standard Error of the Mean	0.241	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
25	conductivity_w	Conductivity Water mS/cm	Num	8		Minimum	1.006	.
						10th Percentile	2.542	.
						25th Percentile	2.67	.
						Median	5.275	.
						75th Percentile	6.474	.
						90th Percentile	7.533	.
						Maximum	8.612	.
						Mean	4.984	.
						Standard Deviation	2.334	.
						Standard Error of the Mean	0.704	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
26	ethanol_w	Ethanol Water mg/L	Num	8		Minimum	210	.
						10th Percentile	230	.
						25th Percentile	320	.
						Median	4,100	.
						75th Percentile	5,700	.
						90th Percentile	8,400	.
						Maximum	71,000	.
						Mean	9,632.73	.
						Standard Deviation	20,518.86	.
						Standard Error of the Mean	6,186.67	.
						Non-Missing	11	0.2619
						Missing	31	0.7381

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
27	methanol_w	Methanol Water mg/L	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	110	.
						75th Percentile	650	.
						90th Percentile	2,400	.
						Maximum	3,200	.
						Mean	643	.
						Standard Deviation	1,100.50	.
						Standard Error of the Mean	331.813	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
28	acetic_w	Acetic Water ppm	Num	8		Minimum	6.983	.
						10th Percentile	1,517.49	.
						25th Percentile	3,745.06	.
						Median	13,002.94	.
						75th Percentile	19,919.44	.
						90th Percentile	25,157.17	.
						Maximum	26,971.33	.
						Mean	12,844.81	.
						Standard Deviation	9,544.50	.
						Standard Error of the Mean	2,877.77	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
29	formic_w	Formic Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	1.293	.
						25th Percentile	19.103	.
						Median	67	.
						75th Percentile	122.627	.
						90th Percentile	195.527	.
						Maximum	269.224	.
						Mean	86.614	.
						Standard Deviation	83.462	.
						Standard Error of the Mean	25.165	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
30	propionic_w	Propionic Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	0.26	.
						25th Percentile	0.26	.
						Median	4.084	.
						75th Percentile	22.558	.
						90th Percentile	32.085	.
						Maximum	480.991	.
						Mean	51.497	.
						Standard Deviation	142.835	.
						Standard Error of the Mean	43.066	.
						Non-Missing	11	0.2619
						Missing	31	0.7381

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
31	glycerin_w	Glycerin Water ppm	Num	8		Minimum	0.006	.
						10th Percentile	0.025	.
						25th Percentile	0.1	.
						Median	0.5	.
						75th Percentile	2,398.81	.
						90th Percentile	2,590.41	.
						Maximum	18,322.34	.
						Mean	2,362.89	.
						Standard Deviation	5,401.57	.
						Standard Error of the Mean	1,628.64	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
32	lactic_w	Lactic Water ppm	Num	8		Minimum	0.702	.
						10th Percentile	2.53	.
						25th Percentile	2.56	.
						Median	38.18	.
						75th Percentile	598.593	.
						90th Percentile	1,463.83	.
						Maximum	1,915.01	.
						Mean	408.374	.
						Standard Deviation	667.989	.
						Standard Error of the Mean	201.406	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
33	F_w	F Water ppm	Num	8		Minimum	0.13	.
						10th Percentile	0.14	.
						25th Percentile	0.25	.
						Median	20.674	.
						75th Percentile	88.337	.
						90th Percentile	117.075	.
						Maximum	117.891	.
						Mean	38.88	.
						Standard Deviation	46.612	.
						Standard Error of the Mean	14.054	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
34	Cl_w	Cl Water ppm	Num	8		Minimum	2.297	.
						10th Percentile	29.612	.
						25th Percentile	718.35	.
						Median	1,920.37	.
						75th Percentile	2,139.30	.
						90th Percentile	2,652.71	.
						Maximum	3,467.89	.
						Mean	1,531.10	.
						Standard Deviation	1,086.82	.
						Standard Error of the Mean	327.69	.
						Non-Missing	11	0.2619
						Missing	31	0.7381

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
35	NO3_w	NO3 Water ppm	Num	8		Minimum	0.13	.
						10th Percentile	0.13	.
						25th Percentile	0.13	.
						Median	0.14	.
						75th Percentile	7.435	.
						90th Percentile	11.354	.
						Maximum	79.285	.
						Mean	9.344	.
						Standard Deviation	23.502	.
						Standard Error of the Mean	7.086	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
36	SO4_w	SO4 Water ppm	Num	8		Minimum	1.719	.
						10th Percentile	4.272	.
						25th Percentile	16.949	.
						Median	83.204	.
						75th Percentile	155.672	.
						90th Percentile	271.575	.
						Maximum	357.562	.
						Mean	111.919	.
						Standard Deviation	117.175	.
						Standard Error of the Mean	35.33	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
37	Na_w	Na Water ppm	Num	8		Minimum	176.12	.
						10th Percentile	1,977.21	.
						25th Percentile	1,986.77	.
						Median	3,120.47	.
						75th Percentile	4,186.28	.
						90th Percentile	4,224.39	.
						Maximum	4,686.74	.
						Mean	2,978.45	.
						Standard Deviation	1,293.66	.
						Standard Error of the Mean	390.054	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
38	NH4_w	NH4 Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	1.28	.
						25th Percentile	1.76	.
						Median	14.422	.
						75th Percentile	180.797	.
						90th Percentile	224.26	.
						Maximum	404.083	.
						Mean	80.863	.
						Standard Deviation	132.797	.
						Standard Error of the Mean	40.04	.
						Non-Missing	11	0.2619
						Missing	31	0.7381

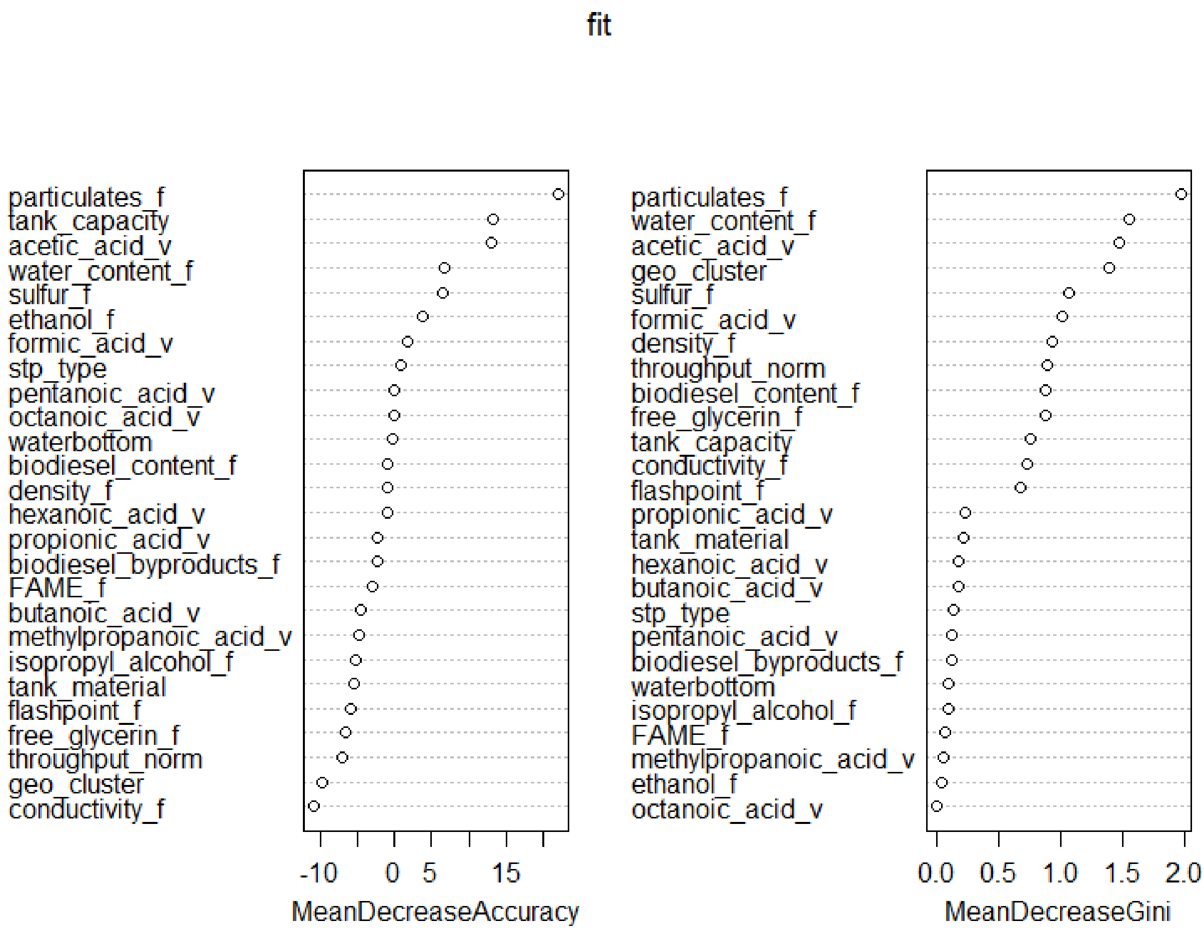
Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
39	K_w	K Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	3.299	.
						25th Percentile	19.14	.
						Median	23.014	.
						75th Percentile	306.73	.
						90th Percentile	640.008	.
						Maximum	1,111.55	.
						Mean	202.069	.
						Standard Deviation	359.862	.
						Standard Error of the Mean	108.503	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
40	Mg_w	Mg Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	1.25	.
						25th Percentile	1.662	.
						Median	42.137	.
						75th Percentile	89.582	.
						90th Percentile	208.31	.
						Maximum	626.981	.
						Mean	105.996	.
						Standard Deviation	183.817	.
						Standard Error of the Mean	55.423	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
41	Ca_w	Ca Water ppm	Num	8		Minimum	0.14	.
						10th Percentile	1.122	.
						25th Percentile	4.514	.
						Median	98.631	.
						75th Percentile	229.889	.
						90th Percentile	253.077	.
						Maximum	398.936	.
						Mean	122.674	.
						Standard Deviation	126.619	.
						Standard Error of the Mean	38.177	.
						Non-Missing	11	0.2619
						Missing	31	0.7381
42	acetic_acid_v	Acetic Acid Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	28	.
						Median	125	.
						75th Percentile	630	.
						90th Percentile	1,900	.
						Maximum	6,200	.
						Mean	612.81	.
						Standard Deviation	1,155.82	.
						Standard Error of the Mean	178.347	.
						Non-Missing	42	1
						Missing	0	0

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
43	formic_acid_v	Formic Acid Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	71	.
						Median	130	.
						75th Percentile	330	.
						90th Percentile	840	.
						Maximum	2,100	.
						Mean	326.548	.
						Standard Deviation	469.997	.
						Standard Error of the Mean	72.522	.
						Non-Missing	42	1
						Missing	0	0
44	propionic_acid_v	Propionic Acid (Propanoic) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	1	.
						90th Percentile	3.5	.
						Maximum	8	.
						Mean	0.861	.
						Standard Deviation	1.785	.
						Standard Error of the Mean	0.275	.
						Non-Missing	42	1
						Missing	0	0
45	methylpropanoic_acid_v	2-Methylpropanoic Acid (Isobutyric) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	0	.
						90th Percentile	0	.
						Maximum	6.3	.
						Mean	0.277	.
						Standard Deviation	1.109	.
						Standard Error of the Mean	0.171	.
						Non-Missing	42	1
						Missing	0	0
46	butanoic_acid_v	Butanoic Acid (Butyric) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	0	.
						90th Percentile	1.6	.
						Maximum	5.8	.
						Mean	0.458	.
						Standard Deviation	1.191	.
						Standard Error of the Mean	0.184	.
						Non-Missing	42	1
						Missing	0	0

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
47	pentanoic_acid_v	Pentanoic Acid (Valeric) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	0	.
						90th Percentile	0	.
						Maximum	1	.
						Mean	0.024	.
						Standard Deviation	0.154	.
						Standard Error of the Mean	0.024	.
						Non-Missing	42	1
						Missing	0	0
48	hexanoic_acid_v	Hexanoic Acid (Caproic) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	0	.
						90th Percentile	0	.
						Maximum	1.6	.
						Mean	0.053	.
						Standard Deviation	0.263	.
						Standard Error of the Mean	0.041	.
						Non-Missing	42	1
						Missing	0	0
49	octanoic_acid_v	Octanoic Acid (Caprylic) Vapor ppbv	Num	8		Minimum	0	.
						10th Percentile	0	.
						25th Percentile	0	.
						Median	0	.
						75th Percentile	0	.
						90th Percentile	0	.
						Maximum	0.77	.
						Mean	0.018	.
						Standard Deviation	0.119	.
						Standard Error of the Mean	0.018	.
						Non-Missing	42	1
						Missing	0	0
50	throughput	Monthly Throughput (gallons)	Num	8		Minimum	3,000	.
						10th Percentile	7,864	.
						25th Percentile	9,725.77	.
						Median	16,937.81	.
						75th Percentile	29,167	.
						90th Percentile	40,000	.
						Maximum	160,000	.
						Mean	24,987.89	.
						Standard Deviation	30,130.31	.
						Standard Error of the Mean	5,909.04	.
						Non-Missing	26	0.61905
						Missing	16	0.38095

Order of Variable	Variable Name	Variable Label	Variable Type	Variable Length	Response Category	Response Category Description	Number of Observations / Distribution Summary	Percent of Population
51	throughput_norm	Normalized Throughput (gallons)	Num	8		Minimum	29.114	.
						10th Percentile	65.533	.
						25th Percentile	95.908	.
						Median	168.548	.
						75th Percentile	262.75	.
						90th Percentile	470.217	.
						Maximum	800	.
						Mean	218.325	.
						Standard Deviation	174.852	.
						Standard Error of the Mean	34.291	.
						Non-Missing	26	0.61905
						Missing	16	0.38095
52	waterbottom	Water Bottom	Num	8	0	not present	31	0.7381
					1	present	11	0.2619
					Non-Missing	Non-Missing	42	1
					.	Missing	0	0

The same analysis approach that was described in report Section III E was completed when the data set included a throughput variable but was restricted to the 26 UST systems with throughput data available. The OOB estimate of error rate is 57.69%. This does a reasonably better job than chance at predictive accuracy and again finds particulates and water content near the top of the variable importance scores, with acetic acid measurements from the vapor the third most important by both criteria. The results of the variable importance results and the univariate, ordered logistic regression are presented below.



Corrosion Class	Predicted Class			Error in Predicting Class
	Minimal	Moderate	Severe	
Minimal	0	2	3	1
Moderate	1	4	4	0.556
Severe	1	4	7	0.417

Analysis Results Of Each Variable Evaluated Individually According To Three Corrosion Categories For 26 UST Systems With Throughput Data

P-value Rankings From The Ordered Logistic Regression For UST Systems Including The Variable Fuel Throughput

Variable	P-value	Log Odds Estimate
tank_capacity_g	0.006	
acetic_acid_v	0.107	0.0011
tank_material_g	0.110	
free_glycerin_f	0.138	-0.0055
hexanoic_acid_v	0.143	-2.425
particulates_f	0.171	0.0180
throughput_norm	0.199	-0.0029
formic_acid_v	0.250	-0.0020
owner_g	0.254	
total_glycerin_f	0.278	-0.0047
waterbottom	0.378	1.105
biodiesel_byproducts_f	0.409	-0.6526
propionic_acid_v	0.469	0.1518
butanoic_acid_v	0.472	-0.4066
density_f	0.553	-26.82
geo_cluster_g	0.630	
water_content_f	0.645	0.0033
methylpropanoic_acid_v	0.653	0.6781
NACE_f_g	0.667	
isopropyl_alcohol_f	0.691	0.2941
TAN_f	0.742	1.772
biodiesel_content_f	0.751	-0.0421
conductivity_f	0.769	0.0008
FAME_f	0.804	0.2280
stp_type_g	0.814	
flashpoint_f	0.954	0.0050
sulfur_f	0.981	-0.0041
ethanol_f	0.993	-16.03
pentanoic_acid_v	0.995	-17.38

Supplement E

Quality Assessment and Oversight

Supplement E

Quality Assessment And Oversight

Assessments And Response Actions

Internal quality control measures (e.g., QC check samples, regular review of raw data, spot-checking of calculations) were implemented by the project staff and monitored by the Task Order Leader (TOL) to ensure data quality during data collection. Any issues that could affect data quality or the ability to use the data (e.g., missing quality control data) were identified in real-time and forwarded to the TOL for corrective action. If necessary, the analytical laboratories were contacted to provide additional data and information to support the reported results. In the event these issues were not in accordance with the Quality Assurance Project Plan (QAPP) for the *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, a deviation report was prepared to evaluate if there was an impact to the data quality. The deviation reports prepared for this project are included here as Attachment 1. In addition, Battelle conducted the following audits as part of this project.

Technical Systems Audit

A Technical Systems Audit (TSA) was conducted by the Battelle QA Manager during the second day of data collection at one of the field sites. The purpose of this audit was to ensure data collection, sampling, video recording, and sample shipment procedures were being performed in accordance with the approved QAPP. As a guide for the audit, a checklist was developed to cover the technical specifications referenced in the QAPP, as well as other aspects of the field activities.

The following activities were observed: Mobilization, vapor pump calibration, temperature and humidity data collection, vapor sample collection, water bottom sample collection, water filter sample collection, dispenser filter and filter housing inspections, fuel sample collection, QC sample collection, tank video cam inspection, STP shaft corrosion assessment, riser and cap/adaptor condition assessments, site photographs, sample labeling, equipment decontamination, chain-of-custody (COC) preparation, and demobilization. All TSA observations were detailed in a TSA checklist. Due to the time sensitive nature of the sampling activities, corrective actions were immediately relayed verbally to all of the field technicians and to EPA. The results of the audit were written up in a TSA report by the Battelle QA Manager which identified observations and recommended actions to resolve any issues. The TOL responded to the observations and after the QA Manager confirmed that the responses were implemented and effective, the TSA report was approved. The TSA report is included in Attachment 2.

Data Quality Audit

The Battelle QA Manager audited at least 10% of the reported sample results acquired during the project and 100% of the associated QC data. The Battelle QA Manager traced the data from initial acquisition (reviewing at least 10% of raw data for each method), through reduction and statistical comparisons, to final reporting. All calculations performed on the data were checked. The results of the audit were written up in an Audit of Data Quality (ADQ) report by the Battelle QA Manager which identified observations and recommended actions to resolve any issues. The TOL responded to the observations and after the QA Manager confirmed that the responses were implemented and effective, the ADQ report was approved. The ADQ report is included in Attachment 3.

Data Review, Validation, And Verification

Data validity and usability was assessed by the QA Manager through review of QC check sample results against the data quality objectives (DQOs) outlined in QAPP tables 5 through 7. The DQOs included the frequency of instrument calibration, QC check samples, acceptance criterial and corrective action. Data verification was accomplished by ensuring the accuracy and completeness of data transcribed from raw data to the report tables. A comparison of raw data against reported data was conducted to flag any suspect data and resolve any observations of apparent outliers. The data quality assessment, as described in Section C1.2 was designed to ensure data quality.

Validation And Verification Methods

Data verification included a visual inspection of hand written data to ensure that all entries were properly recorded and that any erroneous entries were properly noted, as described in the QAPP. Data validation included the assessment of QC data and the performance of a data quality audit (Section C1.2) to determine if the data collection and measurement procedures met the data quality objectives defined in the QAPP.

Reconciliation With User Requirements

The data obtained during the project were reviewed to ensure that results were legally defensible and reproducible. The data review and validation procedures described previously verified that the data meet the data quality objectives and are accurately presented in the report. The data generated throughout the project were compiled into a results database so that measurement data and site inspection details could be easily summarized for reporting purposes.

Supplement E

Attachment 1 – Deviation Reports

QAPP DEVIATION REPORT

QAPP TITLE AND DATE: Quality Assurance Project Plan (QAPP) *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015

DEVIATION NUMBER: 1 – Nitrile gloves

DATE OF DEVIATION: 1/26/15

DESCRIPTION OF DEVIATION:
QAPP Section B2.2 states that aseptic sampling technique including the wearing of sterile gloves will be followed for collection of fuel and water bottom samples. The gloves being used in the field are clean, nitrile as opposed to sterile.

CAUSE OF DEVIATION:
During the technical systems audit it was noted that sampling staff were decontaminating all sampling equipment, wearing new, nitrile gloves for all sampling and changing gloves between matrices. However, this is not considered aseptic/sterile technique.

IMPACT OF DEVIATION ON THE TEST:
There is no anticipated negative impact on data quality when sampling using nitrile gloves instead of sterile gloves. Achieving a sterile, aseptic environment in the field is not practical.

CORRECTIVE ACTION: Using new, clean gloves and replacing when they get dirty was discussed with the teams to ensure a clean environment for sampling. Also discussed was the need to properly decontaminate the equipment before and after use and to setup a clean workspace for the filtering apparatus.

DEVIATION NUMBER: 2 – Removing D4815 for a fuel analysis method

DATE OF DEVIATION: 2/2/2015

DESCRIPTION OF DEVIATION:
The QAPP Table 4, pg. 25 states that ASTM D4815 "Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography" will be used to analyze fuel for ethanol and methanol content. This method is not appropriate for diesel fuel.

CAUSE OF DEVIATION:
ASTM D4815 is a method specified for gasoline fuels and is not appropriate for diesel fuel. This analysis will not be performed on the diesel fuel collected in the field. Identifying ethanol and methanol in the sample will be done using the GC-MS Full Scan method.

IMPACT OF DEVIATION ON THE TEST:
The potential impact on the data is that the alternate method of the GC-MS Full Scan is not quantitative. The intensities of the signal from one sample to another may be compared; however, the actual concentrations will be unknown.

CORRECTIVE ACTION: ASTM D4815 was removed from the fuel method analysis list. Ethanol and methanol will be monitored from the GC-MS full scan already listed in QAPP Table 4.

DEVIATION NUMBER: 3 – Sampling order clarification

DATE OF DEVIATION: 1/26/15

DESCRIPTION OF DEVIATION:
The QAPP (Section B.2, pg. 22) states that "samples will be drawn in the order described above and before the in-tank video inspection". The statement is referencing the sample order outlined in Table 3 in which samples would be collected in the following order, vapor, fuel, water bottom, water filter, and corrosion scraping. It is important to collect the vapor samples first and before the rest of the inspection; however, the order of performing the other sample collection and video inspection does not need to be specified and can happen in any order.

CAUSE OF DEVIATION:
To clarify the language on the order of the sampling and the video inspection.

IMPACT OF DEVIATION ON THE TEST:
There is no anticipated negative impact on data quality if the liquid samples are taken before or after the video inspection.

CORRECTIVE ACTION: Prepared this deviation for clarification and discussed the order of inspection events with sampling teams stressing the need for the vapor samples to be taken first and without any other disturbances to the tank.

ORIGINATED BY:

Anne M. Gregg

(Name)

2/2/15
DATE

APPROVED BY:

Amy Dindal	Ealcuttor
Battelle Program Manager	Battelle Quality Assurance Manager
2/2/15	2/2/15
DATE	DATE

QAPP DEVIATION REPORT

QAPP TITLE AND DATE: Quality Assurance Project Plan (QAPP) *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015

DEVIATION NUMBER: 4 – Acceptance Criteria for ASTM D4052 and D6217

DATE OF DEVIATION: February 2015

DESCRIPTION OF DEVIATION:

Table 5 in the QAPP (Section B5, page 27) states the data quality objectives and acceptance criteria for the fuel analysis methods. After reviewing the data package from Iowa Central Fuel Testing Laboratory (ICFTL), it was determined that the acceptance criteria that were actually used for ASTM D4052 and D6217 differed from the criteria in the QAPP. See Table 1, snip of QAPP Table 5.

Table 1. Snip from Data Quality Objectives and Frequency of Instrument Calibration for Fuel Analysis Methods (QAPP Table 5)

Fuel Analysis Methods-Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Density	ASTM D4052	Anton Paar DMA 35N	Daily QC Check Quarterly Water Reference Check	0.88 ± 0.0005 g/mL	Recalibrate
Particulates	ASTM D6217	Gast Vacuum pump and filtration apparatus	Semi-annual Manometer Check Semi-annual Balance Check	2.0 ±1.6 mg/L	Check vacuum connections Check balance

Table 2 below shows the acceptance criteria that were used in practice by ICFTL.

Table 2. Actual Acceptance Criteria Used for D4052 and D6217

Fuel Analysis Methods-Determination of	Method Identifier	Acceptance Criteria
Density	ASTM D4052	0.8841 ± 0.0005 g/mL
Particulates	ASTM D6217	5.1 ± 2.6 mg/L

CAUSE OF DEVIATION:
For D4052, the ASTM provided acceptance criteria that only have two significant figures (0.80-0.88), if rounded 0.8841 falls within ASTM criteria. This deviation is a clarification as to the actual values utilized by the laboratory.

For D6217, the ASTM method provides several repeatability and reproducibility values for various QC target values, including 5.0 ± 2.6 mg/L. ICFTL utilized a different target QC value than originally stated in the QAPP; however, it still follows the ASTM method.

IMPACT OF DEVIATION ON THE TEST:
There is no anticipated negative impact on data quality.

CORRECTIVE ACTION: Confirmed the acceptance criteria with the laboratory following data review. Prepared this deviation for clarification of the acceptance criteria used by ICFTL for ASTM D4052 and D6217.

ORIGINATED BY:

[signature]
Task Order Manager

4/21/15
DATE

APPROVED BY:

[signature]
Battelle Program Manager

4/21/15
DATE

[signature]
Battelle Quality Assurance Manager

4/21/15
DATE

QAPP DEVIATION REPORT

QAPP TITLE AND DATE: Quality Assurance Project Plan (QAPP) *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015

DEVIATION NUMBER: 5 – Acceptance Criteria for NACE TM0172

DATE OF DEVIATION: February 2015

DESCRIPTION OF DEVIATION:

Table 5 in the QAPP (Section B5, page 27) states the data quality objectives and acceptance criteria for the fuel analysis methods. After reviewing the data package from Southwest Research Institute (SwRI), it was determined that the frequency of calibration and acceptance criteria that were actually used for NACE TM0172 differed from the criteria in the QAPP. See Table 1, snip of QAPP Table 5.

Table 1. Snip from Data Quality Objectives and Frequency of Instrument Calibration for Fuel Analysis Methods (QAPP Table 5)

Fuel Analysis Methods-Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Corrosion Rating	NACE TM 0172	Lab-Line Instruments	Speed controller annually Thermometers every 6 months RPM by a speed gun daily Speed gun every 6 months	ASTM cross check samples 3 times per year	Rerun ASTM cross check sample if necessary

Table 2 below shows the acceptance criteria that were used in practice by ICFTL.

Table 2. Actual Acceptance Criteria Used for NACE TM0172

Fuel Analysis Methods-Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Corrosion Rating	NACE TM 0172	Lab-Line Instruments	Speed controller annually Thermometers annually Speed gun every 6 months	ISO certification	Request maintenance, if needed

CAUSE OF DEVIATION:
During QAPP preparation, SwRI supplied thermometer calibration frequency and acceptance criteria that did not match their standard operating procedures which lead to discrepancies in the quality assurance requirements that were outlined in the QAPP. The lab has ISO certifications for the method instead of using an ASTM cross check sample.

IMPACT OF DEVIATION ON THE TEST:
There is no anticipated negative impact on data quality.

CORRECTIVE ACTION: Confirmed the acceptance criteria with the laboratory following data review. Prepared this deviation for clarification of the acceptance criteria used by SwRI for NACE TM0172.

ORIGINATED BY:

Anne M. Gregg
____
Task Order Manager

5/11/15_______________________
DATE

APPROVED BY:

Amy Dindal
____
Battelle Program Manager

5/11/15_______________________
DATE

____
Battelle Quality Assurance Manager

5/11/15_______________________
DATE

QAPP DEVIATION REPORT

QAPP TITLE AND DATE: Quality Assurance Project Plan (QAPP) *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015

DEVIATION NUMBER: 6 – EPA 120.1 Conductivity Standards

DATE OF DEVIATION: February 2015

DESCRIPTION OF DEVIATION:

Table 6 in the QAPP (Section B5, page 28) states that daily calibrations with 1413 µS/cm and 12.9 ms/cm standards will be conducted. After reviewing the data package from Metrohm it was determined that the calibration was completed with a 9.986 mS/cm standard. See Table 1, snip of QAPP Table 6.

Table 1. Snip from Data Quality Objectives and Frequency of Instrument Calibration for Water Analysis Methods (QAPP Table 6)

Water Bottom Analysis Methods-Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Conductivity	EPA 120.1	VWR Symphony	• Daily calibration with 1413 µS/cm and 12.9 ms/cm standards	• Stable calibration readings equaling known concentrations	• Repeat calibration • Have probe and/or meter serviced

Table 2 below shows the acceptance criteria that were used in practice by Metrohm.

Table 2. Actual Acceptance Criteria Used for Conductivity (EPA 120.1)

Water Bottom Analysis Methods-Determination of	Method Identifier	Instrument Make/Model	Frequency of Instrument Calibration and QC Procedures	Acceptance Criteria	Corrective Action
Conductivity	EPA 120.1	VWR Symphony	• Daily calibration with 9.986 mS/cm standard	• Stable calibration readings equaling known concentrations	• Repeat calibration • Have probe and/or meter serviced

CAUSE OF DEVIATION:
During QAPP preparation, Metrohm supplied the information regarding the calibration standards that would be utilized, but this information did not match their standard operating procedures which led to discrepancies in the quality assurance requirements that were outlined in the QAPP. The lab was able to obtain stable readings equaling the known conductivity of 9.986 mS/cm.

IMPACT OF DEVIATION ON THE TEST:
There is no anticipated negative impact on data quality. The laboratory used a cell constant to measure conductivity rather than a calibration curve. The calibration standard was compared to the determined cell constant to verify that the instrument was working properly. The calibration standard was within ± 10% of the known value.

CORRECTIVE ACTION: Confirmed the standards used with the laboratory following data review. Prepared this deviation for clarification of the standards used by Metrohm for conductivity analysis.

ORIGINATED BY:

Anne M. Gregg

Task Order Leader

5/21/15
DATE

APPROVED BY:

Amy Dindal	ZAlvarez
Battelle Program Manager	Battelle Quality Assurance Manager
5/22/15	5/21/15
DATE	DATE

Supplement E

Attachment 2 – Technical Systems Audit Report

TECHNICAL SYSTEM AUDIT REPORT

Failure Analysis of Underground Storage Tank Equipment in Biofuels Service

Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel

Audit Performed by:
Betsy Cutié, Battelle

Audit Date: January 27, 2015

Audit Report Date: January 29, 2015

I. INTRODUCTION

A technical systems audit (TSA) was performed on the project entitled, *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*. This project is being performed under the direction of the United States Environmental Protection Agency (EPA) through Scientific, Technical, Research, Engineering and Modeling Support (STREAMS) II Task Order (TO) 0016 of Contract EP-C-11-038. The audit was conducted by Betsy Cutié, Quality Assurance Manager for the STREAMS II Contract.

The intent of the audit was to verify adherence to the approved Quality Assurance Project Plan (QAPP) entitled, *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015.

II. PERSONNEL INTERVIEWED and AUDIT CONDUCT

The following personnel were interviewed as part of this technical system audit:

John Pollack, QC Manager and Sampling Lead, Tanknology
Greg Ford, Sampling Technician and Video Camera Operator, Tanknology

The TSA was conducted by Betsy Cutie, at UST Site 2, located in Vernon Hills, Illinois on January 27, 2015. The focus of this assessment was data collection and collection of vapor, fuel, water bottom in the field. As a guide for the audit, a checklist was developed to cover the technical specifications referenced in the QAPP, as well as other aspects of the field activities.

The following activities were observed: Mobilization, vapor pump calibration, temperature and humidity data collection, vapor sample collection, water bottom sample collection, water filter sample collection, dispenser filter and filter housing inspections, fuel sample collection, quality control (QC) sample collection, tank video cam inspection, STP shaft corrosion assessment, riser and cap/adaptor condition assessments, site photographs, sample labeling, equipment decontamination, chain-of-custody (COC) preparation, and demobilization. Observations are detailed in the TSA Checklist (Attachment 1).

III. DEFINITIONS

For this TSA report the following definitions are provided.

Finding: *A deficiency at the program or project level that may or will have a significant adverse effect on quality.*

Observation: *A deficiency at the program or project level that will not have a significant adverse effect on quality.*

Recommendation: *Recommendation made to help verify that project QA requirements will be met.*

IV. AUDIT RESULTS

The audit identified 0 Findings and 1 Observation and 2 Recommendations as detailed below.

OBSERVATIONS

1. QAPP Section B2.2 states that aseptic sampling technique including the wearing of sterile gloves will be followed for collection of fuel and water bottom samples. Sampling staff were properly decontaminating all sampling equipment, wearing new, nitrile gloves for all sampling and changing gloves between matrices. This is not considered aseptic/sterile technique.

   Recommended Action: Prepare a QAPP deviation to clarify that aseptic technique does not apply to the field sample collection.

   Response: A QAPP deviation is prepared to address this change.

RECOMMENDATIONS

1. Staff noted that it would be helpful to have clean, decontaminated tweezers on site to remove the water filter from the filter housing unit. Recommend adding that item to the sampling equipment available for the field teams.

   Response: The request will be made that the teams purchase tweezers for this purpose.

2. Staff noted from past experience that the temperature/humidity probe can take up to five minutes to equilibrate in order to take the three readings. Recommend relaying that information to the field teams.

   Response: This will be relayed to the team leads during the training meeting set for January 30.

Audit Response Review/ Approval	Signature	Date
Anne Gregg Task Order Leader		*1/30/2015*
B. Cutié QA Manager		*1/30/2015*
Amy Dindal Program Manager		*1/30/2015*

Completed Technical System Audit Checklist

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Currie

Component	Yes/ No	Comment
1.0 Documentation and Records		
1.1 Is the approved Quality Assurance Project Plan (QAPP) available for reference by the sampling team?	Yes	
1.2 Are activities adequately recorded in project notebooks or data sheets to allow for reconstruction of the verification data? Can the form entries be linked to sampling personnel? Are the initials of each data recorder present in the records?	Yes	
1.3 Are entries made in ink, legible, with notations of entry date, description of the data being recorded, and any units applicable to recorded data?	Yes	
1.4 Are corrections to records made by drawing a single line through the entry with the initials and date of the person making the correction?	Yes	
1.5 Are training records present for all sampling personnel? Note sampling personnel, responsibilities, and documented training.	Yes	John Pollack - Data recording & oversight of sampling, sample collection Greg Ford - Sample collection & Tank Cam.
1.6 Are the sampling staff using the ULSD Site Inspection Field Form, Sample Collection Log Sheet and QA Sample Collection Log Sheet (if applicable) to collect data?	Yes	
1.7 Review data collection forms. Is the documentation complete? Note any issues or discrepancies vs. the QAPP.	Yes	SCLS Step 1a states to don sterile gloves. Staff are using new 0.7 ml nitrile gloves.
2.0 General		
2.1 Are staff wearing the proper PPE according to QAPP Appendix D (Job Safety Analysis)?	Yes	
3.0 Vapor Sampling		Pump # Rate Actual
3.1 Are the pumps for vapor sampling calibrated before the tank is opened using a Bios DryCal DC-Lite flow rate calibrator? Note calibrator and pump IDs. Was an average of 10 readings recorded on the log sheet?	Yes	B18738B 1.0 l/min 0.9282 B20299B 1.0 0.9975 B18648B 0.5 0.5439 B20307B 0.5 0.4956
3.2 Are the triplicate humidity and temperature readings being taken from one riser? Note the riser and riser length.	Yes	Logger # TLBBA68327 - Per John it can take up to 5 minutes ⑨ ~~6.8~~ 6.9 °C, 83% RH

SCLS - Sample Collection Log Sheet

⑨ Illegible entry Ecc 1/27/15

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Cutie

Component	Yes/ No	Comment
3.3 Are the vapor samples being collected from the same riser as the T/H readings? Is the sampling start and end time recorded?	Yes	Yes, using the ATG. Start = 9.03 am Stop = 10:43 am
3.4 Was the SKC sampling pump calibrated at 1.0 L/min using a CAL SKC 226-55 calibration tube prior to sample collection?	Yes	
3.5 Were two vapor samples collected into SKC 226-55 tubes for 100 minutes at 1.0 L/min?	Yes	
3.6 Was the SKC sampling pump calibrated at 0.5 L/min using a CAL SKC 226-10-03 calibration tube prior to sample collection?	Yes	
3.7 Was one vapor sample collected into an SKC 226-10-03 tube for 100 minutes at 0.5 L/min?	Yes	
3.8 Did all other risers remain closed during vapor sampling?	Yes	
3.9 Were vapor QC samples collected in a similar manner during this sampling event?	Yes	Vapor QC sample collected at 0.5 L/min with low-flow manifold in line.
3.10 Check all vapor sample tube labels against QAPP Table 3.	Yes	Checked all labels
4.0 Observations, Photos and Video		
4.1 Has the STP Shaft make/model been recorded on the ULSD Site Inspection Field Form and has the STP been photographed?	Yes	STP is an FE Retro model. STP photographed with the tank cam.
4.2 Has the riser entry for videotaping been recorded?	Yes	
4.3 Has the STP shaft corrosion coverage been classified according to the QAPP (e.g. minimal, moderate, or severe)? Note who performed the classification.	Yes	John stated during the tank cam videotaping that it appeared to be moderate corrosion.
4.4 Have the observations of the riser condition, cap/adapter condition and other corrosion been recorded? Have photos of the components been taken?	Yes	Dispenser filters are newer (Dec 2014) and looked clean.
4.5 Has the product level been recorded from the ATG monitoring system?	Yes	Observed the print-out.

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Cutié

Component	Yes/ No	Comment
4.6 Has the water bottom level been recorded from the ATG monitoring system?	Yes	
4.7 Has the dispenser filter make and model been recorded?	Yes	
4.8 Have the filter and filter housing condition been recorded and photographed? ✓	Yes	Filters are newer (Dec 2014) and looked clean.
5.0 Fuel, Water Bottom and Filter Water Sampling		
5.1 Has the sampling equipment been cleaned according to the QAPP and sample collection log sheet? Note the fuel sampling equipment (e.g. Bacon bomb, TVS). ✓	Yes	TVS used for water bottom and fuel samples
5.2 Are a new sample jars and tubing with clean brass fittings being used to collect liquid samples?	Yes	
5.3 Are the sampling personnel wearing sterile gloves? NO	NO	They are using new 0.7 mil Nitrile gloves between matrices. Sterile gloves would be supplied in one-use, sterile packs
5.4 Were fuel samples collected in the appropriate containers and volumes according to ASTM Method 7464 and QAPP Table 3?	Yes	
5.5 Were water bottom samples collected in the appropriate containers and volumes according to ASTM Method 7464 and QAPP Table 3? ✓ Were containers filled with zero headspace? ✓	Yes	
5.6 Were fuel and/or water bottom QC samples collected in a similar manner during this sampling event?	Yes	Checked all QC sample labels. Filled after primary samples
5.7 Was an aliquot of the water sample filtered through a cellulose filter (e.g. Nalgene Analytical Filter Unit, #130) until the filter clogged (~0.5 L)? The filter is considered clogged when the pressure gauge holds steady for at least 5 minutes without water passing through the filter.	Yes	Greg and John applied pressure (hand pump) for at least 5 minutes. Very little liquid passed through. Pressure remained steady
5.8 Was the filter placed in the appropriate container according to QAPP Table 3?	Yes	Field team could use a clean tweezers to lift filter. They tapped the filter into the bag.
5.9 Check all fuel and water bottom sample labels against QAPP Table 3.	—	Checked labels
6.0 Corrosion Sampling		
6.1 Has the sampling scraper been cleaned according to the QAPP and Sample Collection Log Sheet?	Yes	

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Currie

Component	Yes/ No	Comment
6.2 Was the scraping placed into a 50 mL plastic conical tube or larger, and the location where it came from documented on the sample collection log sheet?	No	There was no corrosion material available to collect a scraping. No sample collected.
7.0 Sample Labeling and Chain-of-Custody		
7.1 Are all sample bottles and sorbent cartridge packages labeled with the UST identification, the date and time of sampling, the type of sample (fuel, water, etc.), and name of the sampling technician?	Yes	
7.2 Does each cooler that is ready for shipment, have a chain-of-custody (COC) form that lists all samples?	— ①	Reviewed the COC forms, but did not observe them in the coolers.
7.3 Does the COC form include the unique sample identification numbers that match the labels of the samples?	Yes	
7.4 Does the COC form also include the date/time of sampling, sample descriptions, storage conditions, and the signature, date, and time, of the person who is relinquishing samples to the shipping company?	Yes	I didn't observe the signing of the COC forms. John packed the samples and took them with him to ship from home.
7.5 Were photo(s) taken of the COC forms?	— ①	
7.6 Was the data logger allowed to equilibrate in the cooler before the cooler temperature was taken and recorded on the COC?	— ①	① Did not observe. These steps were performed after de-mobilization at the site.

Staff: John Pollack
Greg Ford

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Currie

Narrative

Arrived on site at 8:00 am. Greg had area marked off with cones and table set up. Site has two dispenser stands labeled 1/2 and 3/4. Greg had access cover removed.

Observed pump calibrations using the BIOS Dry Cal DC Lite Calibrator. All pumps needed minor adjustments with a screwdriver to get within range. An average of 10 readings was recorded for all pumps. Used cal tubes SKC 226-55 for 1.0 L/min & SKC 226-10-03 (0.5 L/min)

Observed John collecting the temperature and humidity readings through the ATG. John noted that it can take up to 5 minutes for the sensor to equilibrate versus 1 minute in the training session slides.

Observed the set up and collection of vapor samples. ~~Also~~ ① Checked tube ID's - using SKC 226-55 for the 1.0 L/min samples, and SKC 226-10-03 for the 0.5 L/min sample and the QC sample. Greg cut new tubing for all samples. Samples collected for 100 minutes. John kept the sample tube caps in the warm truck cab so that they would be easier to re-cap. The 0.5 L/min tubes are about twice as long as the 1.0 L/min tubes.

Observed water bottom sample collection. ~~As~~ ① The ATG printout showed water layer in the tank. Staff wore new gloves and collected samples with a TVS. Samples poured directly into containers using disposible paper funnels. All containers were filled with zero headspace. The 3×40 mL vials were filled first, then the other water samples and a full set of water QC samples.

① spelling error. EAC 1/27/15

Technical System Audit Checklist

Project: Failure Analysis of UST Equipment in Biofuels Service II

Date of Audit: 1/27/15 Site: 2 Auditor: Betsy Cutié

Narrative

② ~~Staff~~ John and Greg donned new gloves, assembled the filter housing with filter and attached it to a hand pump. The remaining water from the TVS was added to the housing and pressure was applied for at least 5 minutes. A small amount of water layer passed through the filter. Filter was tipped into sample bag, bag was sealed, wrapped and placed on dry ice in cooler.

Observed Greg opening the dispensers and removing the filters. John inspected the filters and housing and took pictures. Filters look new and records indicate they were changed in Dec. 2014.

Observed fuel sample collection using the TVS. Staff donned new gloves. Fuel was sampled from the ATG and fill ~~pipe~~ ② and poured directly into sample containers. Free QC samples were also collected.

Observed Greg performing the tank cam video inspection. John noted during the inspection that he would consider the STP to have moderate corrosion. ~~Thee~~ ③ There was no corrosion in the riser, so no corrosion scraping was collected.

Sampling equipment was cleaned with DI water and alcohol and placed into ziploc bags. Tubing was discarded. The UST dispensing system was restored to full working order.

Reviewed all data collection forms, sample labels and site safety checklist with John. No discrepancies were noted. Clarified with Anne Gregg by phone that the vapor field blanks are to be collected at one site per week.

—END—

③ Spelling error Eac 1/29/15 ② wrong wording Eac 1/27/15

Supplement E

Attachment 3 – Audit Of Data Quality Report

DATA QUALITY AUDIT REPORT

Failure Analysis of Underground Storage Tank Equipment in Biofuels Service

Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel

Audit Performed by:
Betsy Cutié, Battelle

Audit Date: May 20-21, 2015

Audit Report Date: May 21, 2015

I. INTRODUCTION

An audit of data quality (ADQ) was performed on the project entitled, *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*. This project is being performed under the direction of the United States Environmental Protection Agency (EPA) through Scientific, Technical, Research, Engineering and Modeling Support (STREAMS) II Task Order (TO) 0016 of Contract EP-C-11-038. The audit was conducted by Betsy Cutié, Quality Assurance Manager for the STREAMS II Contract.

The intent of the audit was to verify adherence to the approved Quality Assurance Project Plan (QAPP) entitled, *Failure Analysis of Underground Storage Tank Equipment in Biofuels Service, Investigation of Corrosion Influencing Factors in UST Tanks with Ultra Low Sulfur Diesel Fuel*, dated January 16, 2015.

II. AUDIT CONDUCT

Data from all participating laboratories were audited. At least 10% of the sample results and 100% of the associated quality control (QC) data were reviewed. The data were traced from initial acquisition through reduction to final reporting. All calculations performed on the data were reviewed.

III. DEFINITIONS

For this ADQ report the following definitions are provided.

Finding: *A deficiency at the program or project level that may or will have a significant adverse effect on quality.*

Observation: *A deficiency at the program or project level that will not have a significant adverse effect on quality.*

IV. AUDIT RESULTS

The audit identified 0 Findings and 5 Observations as detailed below.

OBSERVATIONS

1. SwRI provided fuel sample results for corrosion rating (NACE TM0172) but did not provide the method QC results. QAPP Table 5 states that an ASTM cross-check sample will be run 3 times per year.

   Recommended Action: Request QC results from the laboratory.

   Response: This was addressed in Deviation #5.

2. Marathon provided fuel sample results for short chain acids (In-house Method) but did not provide the method QC results. QAPP Table 5 states that an auto-tune is performed monthly at a minimum.

Recommended Action: Request QC results from the laboratory.

Response: The laboratory confirmed that an instrument auto-tune is performed monthly.

3. Marathon provided fuel sample results for conductivity (ASTM D2624) but did not provide the method QC results. QAPP Table 5 states that an internal check of the probe will be conducted.

   Recommended Action: Request QC results from the laboratory.

   Response: The laboratory confirmed that an internal check of the probe was conducted by the manufacturer and the instrument was zeroed daily prior to use.

4. Metrohm provided water bottom sample results for conductivity (EPA 120.1) however the daily QC standard reported with each batch was 9.986 mS/cm. QAPP Table 6 states that daily QC standards will be 1413 µS/cm and 12.9 mS/cm.

   Recommended Action: Address the change in QC standard in a QAPP deviation report.

   Response: This was addressed with Deviation #6.

5. SwRI shipped water bottom samples to Test America under ambient conditions. Test America report (#J11284) states that the water samples were received at 18 ^{0}C. QAPP Table 3 states that water bottom samples for Method 8015B (ethanol and methanol) should be shipped chilled (2-6^{0}C).

   Recommended Action: Address the sample shipping conditions in a QAPP deviation report.

   Response: The additional water samples are outside of the scope of the QAPP. If these data are reported, the sample shipment conditions will be noted in a footnote.

Audit Response Review/ Approval	Signature	Date
A. Gregg Task Order Leader	Anne M. Gregg	*5/29/2015*
B. Cutie Quality Assurance Manager	[signature]	*5/29/2015*
A. Dindal Program Manager	Amy Dindal	*5/29/15*