United States
Environmental Protection
Agency

EPA/600/R-10/122 October 2010

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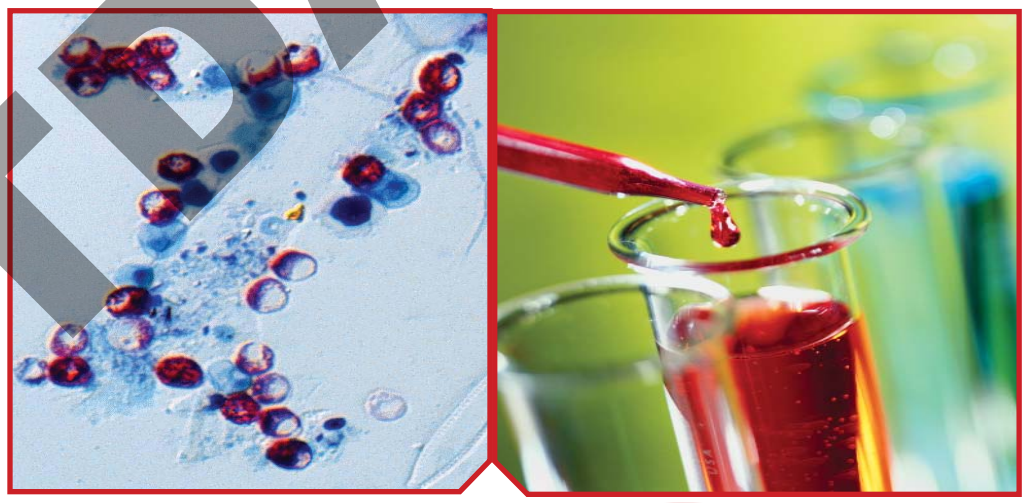
Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events – SAM 2010 **(Revision 6.0)**



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Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events – SAM 2010 (Revision 6.0)

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Cincinnati, OH 45268

Office of Research and Development
National Homeland Security Research Center

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The U.S. Environmental Protection Agency (EPA) through its Office of Research and Development funded and managed the research described here under Contract EP-W-06-046 to Computer Sciences Corporation (CSC). This document has been subjected to the Agency's review and has been approved for publication. Note that approval does not signify that the contents necessarily reflect the views of the Agency.

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Questions concerning this document or its application should be addressed to:

Kathy Hall
National Homeland Security Research Center
Office of Research and Development (NG16)
U.S. Environmental Protection Agency
26 West Martin Luther King Drive
Cincinnati, OH 45268
(513) 379-5260
hall.kathy@epa.gov

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Use of This Document

The information contained in this document represents the latest step in an ongoing effort of the Environmental Protection Agency's (EPA's) National Homeland Security Research Center (NHSRC) to provide standardized analytical methods for use by those laboratories tasked with performing confirmatory analyses of environmental samples in support of EPA restoration efforts following a homeland security incident. The information also can be found on the SAM Web site (www.epa.gov/sam), which provides searchable links to supporting information based on SAM analytes and the analytical methods listed.

Although at this time, some of the methods listed have not been fully validated for a particular analyte (e.g., analytes not explicitly identified in the method) or sample type, the methods are considered to contain the most appropriate currently available techniques. Unless a published method listed in this document states specific applicability to the analyte/sample type for which it has been selected, it should be assumed that method testing is needed, and adjustments may be required to accurately account for variations in analyte/sample type characteristics, environmental samples, analytical interferences, and data quality objectives (DQOs).

Many of the SAM analytes have only recently become an environmental concern. EPA is actively pursuing development and validation of Standard Analytical Protocols (SAPs) based on the methods listed, including optimization of procedures for measuring target analytes or agents. In those cases where method procedures are determined to be insufficient for a particular situation, EPA will provide guidelines regarding appropriate actions. EPA also is in the process of compiling information and preparing documents regarding field screening equipment, sample collection materials, rapid screening/ preliminary identification equipment, and disposal of samples corresponding to SAM analytes and sample types. This will be an ongoing process as EPA will strive to establish a consistent level of validation for all listed analytes.

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Foreword

Following the events of September 11, 2001, EPA's mission was expanded to account for critical needs related to homeland security. Presidential directives identified EPA as the primary federal agency responsible for the country's water supplies and for environmental decontamination following a chemical, biological, and/or radiological (CBR) attack. To provide scientific and technical support to help EPA meet this expanded role, EPA's National Homeland Security Research Center (NHSRC) was established. The NHSRC research program is focused on conducting research and delivering products that improve the capability of the Agency to carry out its homeland security responsibilities.

One focus area of NHSRC's research is to support the Environmental Response Laboratory Network (ERLN), a nationwide association of federal, state, local and commercial environmental laboratories, established by EPA. The ERLN can be deployed in response to a large-scale environmental disaster, providing consistent analytical capability, capacity, and data quality. Toward this end, NHSRC has worked with experts from across EPA and other federal agencies to undertake research designed to provide appropriate, effective, and verified technologies and methods. These methods and technologies can help the Agency determine the risks posed by CBR agents and enhance EPA's ability to detect, contain, and clean up following an incident involving such agents. This document provides a compendium of methods that have been selected for use when analytical laboratories must support an environmental restoration involving CBR contaminants.

This publication represents the sixth revision of this compendium. This information will continue to be revised as research progresses and new information becomes available. In addition, NHSRC will continue to provide a searchable form of this document which is available at http://www.epa.gov/sam. We value your comments as we move toward the development of an efficient process to manage environmental samples and move EPA one step closer to achieving its homeland security mission and its overall mission of protecting human health and the environment while supporting sustainable solutions.

Gregory D. Sayles, Acting Director
National Homeland Security Research Center

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Abbreviations and Acronyms

ACS	American Chemical Society
amp-ELISA	Amplified-enzyme-linked immunosorbent assay
APHA	American Public Health Association
APHL	Association of Public Health Laboratories
AOAC	AOAC International (formerly the Association of Official Analytical Chemists)
ASTM	ASTM International (formerly the American Society for Testing and Materials)
BHT	Butylated hydroxytoluene
BMBL	*Biosafety in Microbiological and Biomedical Laboratories*
BZ	Quinuclidinyl benzilate
°C	Degrees Celsius
CAS RN	Chemical Abstracts Service Registry Number
CBR	Chemical, biological, and/or radiological
CCID	Coordinating Center for Infectious Diseases
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CFSAN	Center for Food Safety and Applied Nutrition
CIEIA	Competitive inhibition enzyme immunoassay
CLLE	Continuous liquid-liquid extraction
CLP	Contract Laboratory Program
cps	Counts per second
CVAA	Cold vapor atomic absorption
2-CVAA	2-Chlorovinylarsonous acid
CVAFS	Cold vapor atomic fluorescence spectrometry
2,4-D	2,4-Dichlorophenoxyacetic acid
DAS	Diacetoxyscirpenol
DAS-HG-HSA	Diacetoxyscirpenol hemiglutarate human serum albumin
DAS-HS-HRP	Diacetoxyscirpenol hemisuccinate horseradish peroxidase conjugate
DB-1	100% Dimethylpolysiloxane
DBPR	Division of Bioterrorism Preparedness and Response
DHS	U.S. Department of Homeland Security
DIG-ELISA	Digoxigenin labeled enzyme-linked immunosorbent assay
DIMP	Diisopropyl methylphosphonate
2,4-DNPH	2,4-Dinitrophenylhydrazine
DoD	U.S. Department of Defense
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
DPD	N,N-Diethyl-*p*-phenylenediamine
DQO	Data quality objective
DTPA	Diethylenetriamine-pentaacetate
DVL	Detection verification level
EA2192	Diisopropylaminoethyl methylthiolophosphonate
ECD	Electron capture detector
e-CFR	Electronic Code of Federal Regulations
ECL	Electrochemiluminescence
ED	Ethyldichloroarsine
EDEA	N-Ethyldiethanolamine
EDL	Estimated detection limit
EDTA	Ethylenediaminetetraacetic acid
EDXA	Energy dispersive X-ray analysis
EIA	Enzyme immunoassay
ELISA	Enzyme-Linked Immunosorbent Assay

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EMC	Emission Measurement Center
EML	Environmental Measurements Laboratory
EMMI	Environmental Monitoring Methods Index
EMPA	Ethyl methylphosphonic acid
EMSL	Environmental Monitoring and Support Laboratory
EPA	U.S. Environmental Protection Agency
EQL	Estimated quantitation limit
ERLN	Environmental Response Laboratory Network
ESI	Electrospray ionization
ETV	Environmental Technology Verification
FBI	U.S. Federal Bureau of Investigation
FDA	U.S. Food and Drug Administration
FEMS	Federation of European Microbiological Societies
FGC-ECD	Fast gas chromatography with electron capture detection
FGI	Fluorescein derivative of *Conus geographus* α-conotoxin
FID	Flame ionization detector
FL	Fluorescence detector
FPD	Flame photometric detector
FRET	Fluorescence resonance energy transfer
FRMAC	Federal Radiological Monitoring and Assessment Center
GA	Tabun
GB	Sarin
GC	Gas chromatograph or Gas chromatography
GC-ECD	Gas chromatography – electron capture detector
GC-FID	Gas chromatography – flame ionization detector
GC-FPD	Gas chromatography – flame photometric detector
GC-MS	Gas chromatography – mass spectrometry
GC-MD	Gas chromatography – multi-detector
GC-NPD	Gas chromatography – nitrogen-phosphorus detector
GD	Soman
GE	1-Methylethyl ester ethylphosphonofluoridic acid
Ge	Germanium
Ge(Li)	Germanium (Lithium)
GESTIS	A German database (Gefahrstoffdaten banken) containing data and information on hazardous substances and products
GF	Cyclohexyl sarin
GFAA	Graphite furnace atomic absorption spectrophotometer or Graphite furnace atomic absorption spectrophotometry
HASL	Health and Safety Laboratory, currently known as Environmental Measurements Laboratory (EML)
HD	Sulfur mustard / mustard gas; bis(2-chloroethyl) sulfide
HHS	U.S. Health and Human Services
HMTD	Hexamethylenetriperoxidediamine
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HN-1	Nitrogen mustard 1; bis(2-chloroethyl)ethylamine
HN-2	Nitrogen mustard 2; 2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine
HN-3	Nitrogen mustard 3; tris(2-chloroethyl)amine
HP(Ge)	High purity Germanium
HPLC	High performance liquid chromatography
HPLC-FL	High performance liquid chromatography – fluorescence
HPLC-MS	High performance liquid chromatography – mass spectrometery
HPLC-MS-MS	High performance liquid chromatography tandem mass spectrometry

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HPLC-PDA	High performance liquid chromatography – photodiode array detector
HPLC-UV	High performance liquid chromatography – ultraviolet
HPLC-vis	High performance liquid chromatography – visible
HRP	Horseradish peroxidase
HV	High volume
IC	Ion chromatograph or Ion chromatography
IC 20	Inhibitory concentration – Concentration to inhibit 20%
IC 50	Inhibitory concentration – Concentration to inhibit 50%
ICP	Inductively coupled plasma
ICP-AES	Inductively coupled plasma – atomic emission spectrometry
ICP-MS	Inductively coupled plasma – mass spectrometry
IDL	Instrument detection limit
ILM	Inorganic Laboratory Method
IMPA	Isopropyl methylphosphonic acid
INCHEM	INCHEM is a means of rapid access to internationally peer reviewed information on chemicals commonly used throughout the world, which may also occur as contaminants in the environment and food. It consolidates information from a number of intergovernmental organizations whose goal it is to assist in the sound management of chemicals. http://www.inchem.org/
IO	Inorganic
i.p.	Intraperitoneally
IRIS	Integrated Risk Information System (EPA)
ISE	Ion specific electrode
ISG	Impregnated silica gel
ISO	International Organization for Standardization
KHP	Potassium hydrogen phthalate
L-1	Lewisite 1; 2-Chlorovinyldichloroarsine
L-2	Lewisite 2; bis(2-Chlorovinyl)chloroarsine
L-3	Lewisite 3; tris(2-Chlorovinyl)arsine
LC	Liquid chromatograph or Liquid chromatography
LC/APCI-MS	Liquid chromatography / atmospheric pressure chemical ionization – mass spectrometry
LC/ESI-MS	Liquid chromatography / electrospray ionization – mass spectrometry
LCMRL	Lowest common minimum reporting level
LC-MS	Liquid chromatography – mass spectrometry
LC-MS-MS	Liquid chromatography tandem mass spectrometry
LC-TSP	Liquid chromatography – thermospray
LFD	Lateral flow device
LLD	Lower limit of detection
LOD	Limit of detection
LOQ	Limit of quantitation
LRN	Laboratory Response Network
LSE	Liquid-solid extraction
Ltd.	A private company limited by shares
mAbs	Monoclonal antibodies
MALDI	Matrix-assisted laser-desorbtion ionization
MARLAP	*Multi-Agency Radiological Laboratory Analytical Protocols* (EPA/402/3-04/001A, B, C)
MDL	Method detection limit
MIC	Methyl isocyanate
MLD	Minimum lethal dose
MPA	Methylphosphonic acid
MRM	Multiple reaction monitoring
mRNA	Messenger ribonucleic acid
MS	Mass spectrometer or Mass spectrometry

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MS-MS	Tandem mass spectrometry
MS/MSD	Matrix spike/Matrix spike duplicate
MSE	Microscale solvent extraction
MTBE	Methyl *tert*-butyl ether
MW	Molecular weight
NA	Not applicable
NaI(Tl)	Thallium-activated sodium iodide
NBD chloride	7-Chloro-4-nitrobenzo-2-oxa-1,3-diazole
NBD-F	7-Fluoro-4-nitro-2,1,3-benzoxadiazole
NCPDCID	National Center for the Prevention, Detection, and Control of Infectious Diseases
NCRP	National Council on Radiation Protection and Measurements
NEMI	National Environmental Methods Index
NERL	National Exposure Research Laboratory
NHSRC	EPA National Homeland Security Research Center
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
nM	Nanomolar
NMAM	NIOSH Manual of Analytical Methods
NNSA	National Nuclear Security Administration
NPD	Nitrogen-phosphorus detector
NRC	U.S. Nuclear Regulatory Commission
nS	nano Siemens
NTIS	National Technical Information Service
NTU	Nephelometric turbidity units
OAQPS	EPA Office of Air Quality Planning and Standards
OAR	EPA Office of Air and Radiation
ORAU	Oak Ridge Associated Universities
ORD	EPA Office of Research and Development
ORIA	Office of Radiation and Indoor Air
ORISE	Oak Ridge Institute for Science and Education
OSWER	EPA Office of Solid Waste and Emergency Response
OSHA	Occupational Safety and Health Administration
OVS	OSHA versatile sampler
OW	EPA Office of Water
PBS	Phosphate buffered saline
PCDDs	Polychlorinated dibenzo-*p*-dioxins
PCDFs	Polychlorinated dibenzofurans
PCR	Polymerase chain reaction
PDA	Photodiode array detector
PEL	Permissible exposure limit
PETN	Pentaerythritol tetranitrate
PFBHA	O-(2,3,4,5,6-pentafluorobenzyl)-hydroxylamine
PFE	Pressurized fluid extraction
PMPA	Pinacolyl methyl phosphonic acid
1,2-PP	1-(2-pyridyl)piperazine
PubMED	U.S. National Library of Medicine (http://www.pubmed.gov)
PUF	Polyurethane foam
PVC	Polyvinyl chloride
PVDH	Polyvinylidene fluoride
QA	Quality assurance
QAP	Quality assessment program
QC	Quality control
®	Registered trademark

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R 33	Methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester (VR)
RCRA	Resource Conservation and Recovery Act
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
RESL	Radiological and Environmental Sciences Laboratory
RLAB	Regional laboratory method
RNA	Ribonucleic acid
rpm	Revolutions per minute
RTECS	Registry of Toxic Effects of Chemical Substances
SAED	Select area electron diffraction
SAM	*Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events*
SAP	Standard Analytical Protocol
SEA	Staphylococcal enterotoxin type A
SEB	Staphylococcal enterotoxin type B
SEC	Staphylococcal enterotoxin type C
SIM	Selective ion monitoring
SM	*Standard Methods for the Examination of Water and Wastewater*
SPE	Solid-phase extraction
SRM	Single reaction monitoring
STEC	Shiga-toxigenic *E. coli*
STEL	Short term exposure limit
STX	Saxitoxin
Stx-1	Shiga toxin Type 1
Stx-2	Shiga toxin Type 2
SW	Solid Waste
TATP	Triacetone triperoxide
TBD	To be determined
TCLP	Toxicity Characteristic Leaching Procedure
TDG	Thiodiglycol
TEA	Triethanolamine
TEM	Transmission electron microscope or Transmission electron microscopy
TFA	Trifluoroacetic acid
™	Unregistered trademark
1,3,5-TNB	1,3,5-Trinitrobenzene
2,4,6-TNT	2,4,6-Trinitrotoluene
TO	Toxic Organic
TOFMS	Time-of-flight mass spectrometry
TOXNET	Toxicology Data Network
TRU	Transuranic
TSP	Thermospray
TSP-MS	Thermospray –mass spectrometry
TTN	Technical Transfer Network
TTX	Tetrodotoxin
U.S.	United States
USDA	U.S. Department of Agriculture
USGS	U.S. Geological Survey
UV	Ultraviolet
VCSB	Voluntary Consensus Standard Body
VE	Phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester
VG	Phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester
vis	Visible detector
VM	Phosphonothioic acid, methyl-,S-(2-(diethylamino)ethyl) O-ethyl ester
VOCs	Volatile organic compounds

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VR	Methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester (R 33)
VX	O-ethyl-S-(2-diisopropylaminoethyl)methylphosphonothiolate
WCIT	Water Contamination Information Tool
WEF	Water Environment Federation
WHO	World Health Organization
WSD	EPA Water Security Division

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Acknowledgments

Contributions of the following individuals and organizations to the development of SAM Revision 6.0 are gratefully acknowledged. Please refer to older versions of SAM for historical acknowledgments.

United States Environmental Protection Agency (EPA)

- **Office of Research and Development, National Homeland Security Research Center**
  Joan Bursey
  April Dupree
  Kathy Hall
  Romy Lee
  Alan Lindquist
  Matthew Magnuson
  Tonya Nichols
  Sarah Perkins
  Eugene Rice
  Frank Schaefer
  Sanjiv Shah
  Erin Silvestri
  Stuart Willison

- **Office of Research and Development, National Exposure Research Laboratory (NERL), Las Vegas**
  Thomas Hinners (Environmental Sciences Division)

- **Office of Research and Development, National Exposure Research Laboratory (NERL), Cincinnati**
  Gerard Stelma (Microbiological and Chemical Exposure Assessment Research Division)
  Jeanette Von Emon (Human Exposure & Atmospheric Sciences Division)

- **Office of Research and Development, National Health and Environmental Effects Research Laboratory (NHEERL)**
  Denise Macmillan

- **Office of Solid Waste and Emergency Response**
  Michele Burgess (Office of Emergency Management)
  Terry Smith (Office of Emergency Management)

- **Office of Air and Radiation, Office of Radiation and Indoor Air**
  Malek Chatila (National Air and Radiation Environmental Laboratory)
  George Dilbeck (National Air and Radiation Environmental Laboratory)
  John Griggs (National Air and Radiation Environmental Laboratory)
  Daniel Mackney (National Air and Radiation Environmental Laboratory)

- **Office of Water, Office of Ground Water and Drinking Water**
  Pamela Bernard (Water Security Division)
  Adrian Hanley (Water Security Division)
  Elizabeth Hedrick (Water Security Division)
  Karen Milam (Water Security Division)
  Malik Raynor (Water Security Division)
  John Sinclair (Technical Support Center)

OUTDATED

- **Office of Pesticide Programs**
  Christian Byrne (Stennis Space Center)
  Elizabeth Flynt (Stennis Space Center)
  Bill Reynolds (Ft. Meade)
  Charles Stafford (Ft. Meade)

- **EPA National Decontamination Team**
  Larry Kaelin

- **EPA Regions**
  Katie Adams (Region 10)
  Jack Berges (Region 9)
  Cynthia Caporale (Region 3)
  Gerald Dodo (Region 10)
  Diane Gregg (Region 6)
  Ted Haigh (Region 10)
  Stephanie Harris (Region 10)
  Johnson Matthew (Region 6)
  Ed O'Neill (Region 6)
  Steve Reimer (Region 10)
  Melvin Ritter (Region 6)
  Sue Warner (Region 3)
  Michael Wasko (Region 4)
  Adam Zachary (Region 5)
  Harold Zachary (Region 3)
  Larry Zintek (Region 5)

United States Department of Agriculture
Lou Blume (Food Safety and Inspection Service)
Mark Campbell (Food Safety and Inspection Service)
Marcus Head (Food Safety and Inspection Service)

United States Department of Commerce
Peter Moeller (National Oceanic & Atmospheric Administration)

United States Department of Defense
Linda Beck (U.S. Navy, Naval Surface Warfare Center Dahlgren Division)
Jon Davis (U.S. Army, Medical Research Institute of Infectious Diseases)
Bob Durgin (U.S. Army)
Eric Holwitt (U.S. Air Force)
Johnathan Kiel (U.S. Air Force)
David Norwood (.S. Army, Medical Research Institute of Infectious Diseases)
Elaine Strauss (U.S. Navy, Naval Surface Warfare Center Dahlgren Division)
Ronald Swatski (U.S. Army, Center for Health Promotion and Preventive Medicine)

United States Department of Energy
Steve Goldberg (New Brunswick Laboratory)

OUTDATED

United States Department of Health and Human Services

- **Centers for Disease Control and Prevention (CDC)**
  Clayton B'Hymer (National Institute for Occupational Safety and Health)
  Cheryl Bopp (National Enteric Laboratory Diagnostics and Outbreak Team)
  Vincent Hill (National Center for Zoonotic, Vector-Borne, and Enteric Diseases)
  Jennifer Links (National Center for Environmental Health)
  Stephen Morse (National Center for Preparedness, Detection and Control of Infectious Diseases)
  John Snawder (National Institute for Occupational Safety and Health)
  Laura Rose (National Center for Preparedness, Detection and Control of Infectious Diseases)
  Richard Wang (National Center for Environmental Health)

- **United States Food and Drug Administration**
  John Callahan
  Eric Garber
  Sherwood Hall
  Michael McLaughlin
  Shashi Sharma

United Stated Department of Homeland Security
Mark Whitmire (Chemical Security Analysis Center)

State Agencies
Jack Bennett (State of Connecticut Department of Public Health)
Sanwat Chaudhuri (Utah Unified State Laboratories)
William Draper (California Department of Public Health)
Eva King (Rhode Island Department of Health)
Marinea Mehrhoff (State Hygenic Laboratory at the University of Iowa)
William K. Nemeth (New Jersey Dept. of Health & Senior Services)
Bahman Parsa (New Jersey Dept. of Health & Senior Services)
Christopher Retarides (Virginia State Department of General Services)
David F. Tomkins (State Hygenic Laboratory at the University of Iowa)
Stephen E. Treimer (State Hygenic Laboratory at the University of Iowa)
Michael D. Wichman (State Hygenic Laboratory at the University of Iowa)

Municipalities
Gary Burlingame (Philadelphia Water Department)
John Consolvo (Philadelphia Water Department)
Earl Peterkin (Philadelphia Water Department)
David Nehrkorn (San Francisco Public Utilities Commission)
Tony Rattonetti **(**San Francisco Public Utility Commission)

National Laboratories
Carolyn Koester (Lawrence Livermore National Laboratory)
Donivan Porterfield (Los Alamos National Laboratory)

Universities
Tambra Dunams (Georgia State University)
Paul Zimba (Texas A&M University – Corpus Christi)

National Institute of Standards and Technology
Jayne Morrow

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Environmental Management Support, Inc.
Anna Berne

Computer Sciences Corporation
Eric Boring
Yildiz Chambers
John Chandler
Joan Cuddeback
Melody Jensen
Daniel Montgomery
Larry Umbaugh
Joshua Vinson

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Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events

DRAFT SAM 2010 (Revision 6.0)

October 2010

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Section 1.0: Introduction

After the terrorist attacks of September 11, 2001 and the anthrax attacks in the fall of 2001, federal and state personnel provided response, recovery, and remediation under trying circumstances, including unprecedented demand on their capabilities to analyze environmental samples. In reviewing these events, the Environmental Protection Agency (EPA) identified several areas where the country could better prepare itself in the event of future terrorist incidents. The need to improve the nation's laboratory capacity and capability to analyze environmental samples following a homeland security event (i.e., chemical, biological, and/or radiological [CBR] crime/attack) was one of the most important areas identified.

In response, EPA formed the Homeland Security Laboratory Capacity Work Group to identify and implement opportunities for near-term improvements and to develop recommendations for addressing longer-term laboratory issues. The EPA Homeland Security Laboratory Capacity Work Group consists of representatives from the Office of Research and Development (ORD), Office of Air and Radiation (OAR), Office of Water (OW), Office of Solid Waste and Emergency Response (OSWER), Office of Environmental Information, Office of Chemical Safety and Pollution Prevention, and several EPA regional offices.

A critical area identified by the work group was the need for a list of analytical methods to be used by all laboratories when analyzing homeland security event samples and, in particular, when analysis of many samples is required over a short period of time. Having standardized methods would reduce confusion, permit sharing of sample load between laboratories, improve data comparability, and simplify the task of outsourcing analytical support to the commercial laboratory sector. Standardized methods would also improve the follow-up activities of validating results, evaluating data, and making decisions. To this end, work group members formed an Analytical Methods Subteam to address homeland security methods issues.

The Analytical Methods Subteam recognized that widely different analytical methods are required for various phases of environmental sample analyses in support of homeland security preparation and response: (1) ongoing surveillance and monitoring; (2) response and rapid screening for determining whether an event has occurred; (3) preliminary site characterizations to determine the extent and type of contamination; and (4) confirmatory laboratory analyses to plan, implement, and evaluate the effectiveness of site remediation. **Figure 1-1** represents these analytical phases. EPA's *Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events* (SAM) provides information for analytical methods to be applied during the "Site Remediation" phase.

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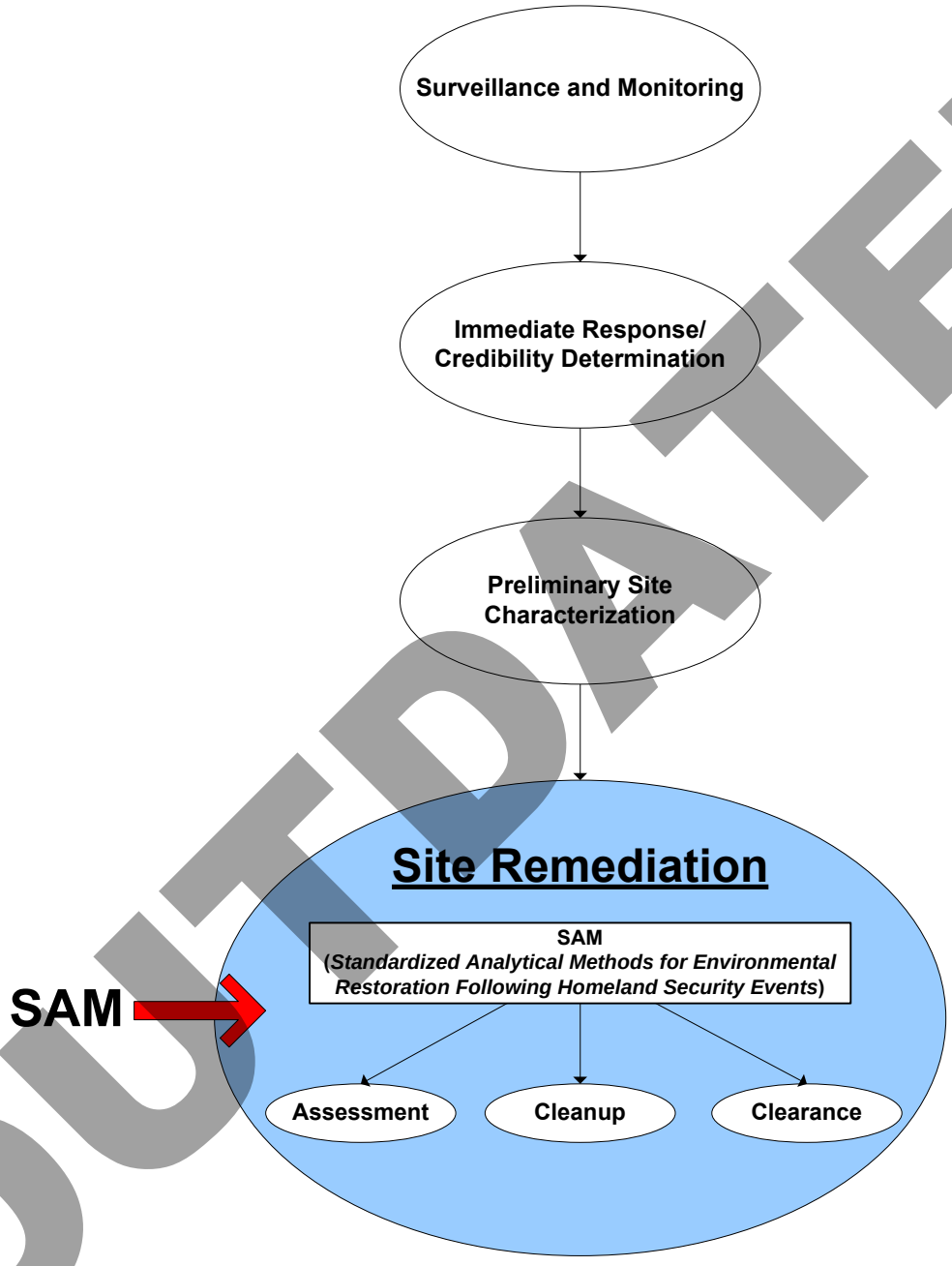
Figure 1-1. Environmental Evaluation Analytical Process Roadmap for Homeland Security Events



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Section 2.0: Background

In support of this document, EPA periodically assembles methods experts from within EPA and other federal agencies to review methods and, if necessary, revise the methods listed. SAM identifies a single method or method group per analyte/sample type to ensure a consistent analytical approach across multiple laboratories when analyzing environmental samples following an event. Method selection is based on consideration of specific criteria that emphasize method performance and include existing laboratory capabilities, laboratory capacity, method applicability to multiple environmental sample types, and method applicability to multiple SAM analytes. For some analytes, the preferred method is a clear choice; for others, competing criteria make the choice more difficult. Final method selections are based on technical recommendations from the SAM work groups. For analytes where limited laboratory testing/experience exists, such as chemical warfare agents, methods were selected based on their applicability to similar chemicals (e.g., nerve agents and some pesticides). In these cases, laboratory studies to test the ability of the selected method to measure the target analyte(s) are either underway or planned. **Figure 2-1** summarizes steps and provides the criteria used during the SAM method selection process. It is important to note that the method selection criteria included in this figure are listed in non-hierarchical order and, in some cases, only a subset of the criteria was considered.

Since 2004, EPA's National Homeland Security Research Center (NHSRC) has brought together experts from across EPA and its sister agencies to develop this compendium of analytical methods to be used when analyzing environmental samples, and to address site characterization, remediation and clearance following future homeland security events. Participants have included representatives from EPA program offices, EPA regions, EPA laboratories, Centers for Disease Control and Prevention (CDC), Food and Drug Administration (FDA), Department of Homeland Security (DHS), Federal Bureau of Investigation (FBI), Department of Defense (DoD), Department of Agriculture (USDA), and U.S. Geological Survey (USGS). Methodologies were considered for chemical and biological agents of concern in the types of environmental samples that would be anticipated. The primary objective of this effort was to identify appropriate SAM Analytical Methods Subteam consensus methods that represent a balance between providing existing, documented, determinative techniques and providing consistent and valid analytical results.

A survey of available confirmatory analytical methods for approximately 120 biological and chemical analytes was conducted using existing resources including the following:

- National Environmental Methods Index (NEMI) and NEMI for Chemical, Biological, and Radiological Methods (NEMI-CBR)
- Environmental Monitoring Method Index (EMMI)
- EPA Test Methods Index
- EPA Office of Solid Waste SW-846 Methods
- EPA Microbiological Methods
- National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods (NMAM)
- Occupational Safety and Health Administration (OSHA) Index of Sampling and Analytical Methods
- AOAC International
- ASTM International
- International Organization for Standardization (ISO) methods
- *Standard Methods for the Examination of Water and Wastewater*
- PubMED Literature Database

In September 2004, EPA published *Standardized Analytical Methods for Use During Homeland Security Events, Revision 1.0* (SAM, Revision 1.0, EPA/600/R-04/126), which provided a list of analytical and sample preparation methods that were selected for measurement of 82 chemical analytes in

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aqueous/liquid, solid, oily solid, and air samples, and 27 biological analytes in water, dust, and aerosol samples. During 2005, SAM was expanded to include radionuclides, several persistent chemical warfare agent degradation products, a drinking water sample type, methods for determination of the viability of biological organisms, and a separate section for biotoxin analytes. Where necessary, the methods included in SAM Revision 1.0 were updated to reflect more recent or appropriate methodologies. Similar efforts to those used for method selection during development of SAM Revision 1.0 were undertaken to select and include methods for measurement of radionuclides and chemical warfare agent degradation products in all sample types, for measurement of CBR analytes in drinking water, and to determine the viability of biological organisms. These additional analytes and the corresponding methods selected were included in SAM Revision 2.0.

During 2006, SAM was revised further to incorporate analytes included on updated federal agency lists, provide additional or more current method listings for target analytes, incorporate explosives into the chemical analytes listing, combine identification and viability methods information for pathogens, and address comments from EPA Science Advisory Board's Homeland Security Advisory Committee[1] to clarify the intended use of the document. These changes were included in SAM Revision 3.0 (*Standardized Analytical Methods for Environmental Restoration Following Homeland Security Events*, February 2007 / EPA/600/R-07/015). SAM Revision 3.0 included a new title to emphasize the intended use of SAM methods for analysis during environmental restoration activities. Following publication of SAM Revision 3.0, SAM work groups updated the document to include the addition of several chemical analytes, one radionuclide, and one biotoxin, along with corresponding selected methods, and provided the updated documents as SAM Revision 3.1 (November 2007 / EPA/600/R-07/136). In 2007, NHSRC also developed a Web-based version of the SAM document to allow users and other stakeholders to search for specific needs and to submit questions and comments regarding the information.

Since publication of SAM Revision 3.1 and its corresponding Web site, NHSRC has continued to convene technical work groups to evaluate and, if necessary, update the analytes and methods that are listed. During development of SAM Revision 4.0 (September 2008), work groups added a wipe sample type for chemical analytes and several polymerase chain reaction (PCR) methods for pathogens. A drinking water sample type was added during development of SAM Revision 5.0 (September 2009). SAM Revisions 4.0 and 5.0 also reflect the addition of several chemical, radiochemical, and biotoxin analytes. The current SAM Revision 6.0 (September 2010) reflects the addition of several more chemical, radiochemical, and biotoxin analytes along with removal of the non-aqueous/organic solid sample types in the chemistry methods section. SAM Revision 6.0 also reflects temporary removal of sections pertaining to pathogens methods for reformatting and further consideration. In the next revision of SAM, NHSRC and its partners plan to revise the document's title to better reflect its focus.

In addition to updating SAM analytes and methods, SAM work groups have identified four areas for development of SAM companion documents to provide information regarding field screening equipment, sample collection, rapid screening and preliminary analysis equipment, and sample disposal to supplement the analytical methods included in SAM. The information listed in these documents corresponds to the analytes and methods in SAM and will be updated periodically to reflect revisions to SAM. Currently available SAM companion documents are listed in Attachment 1.

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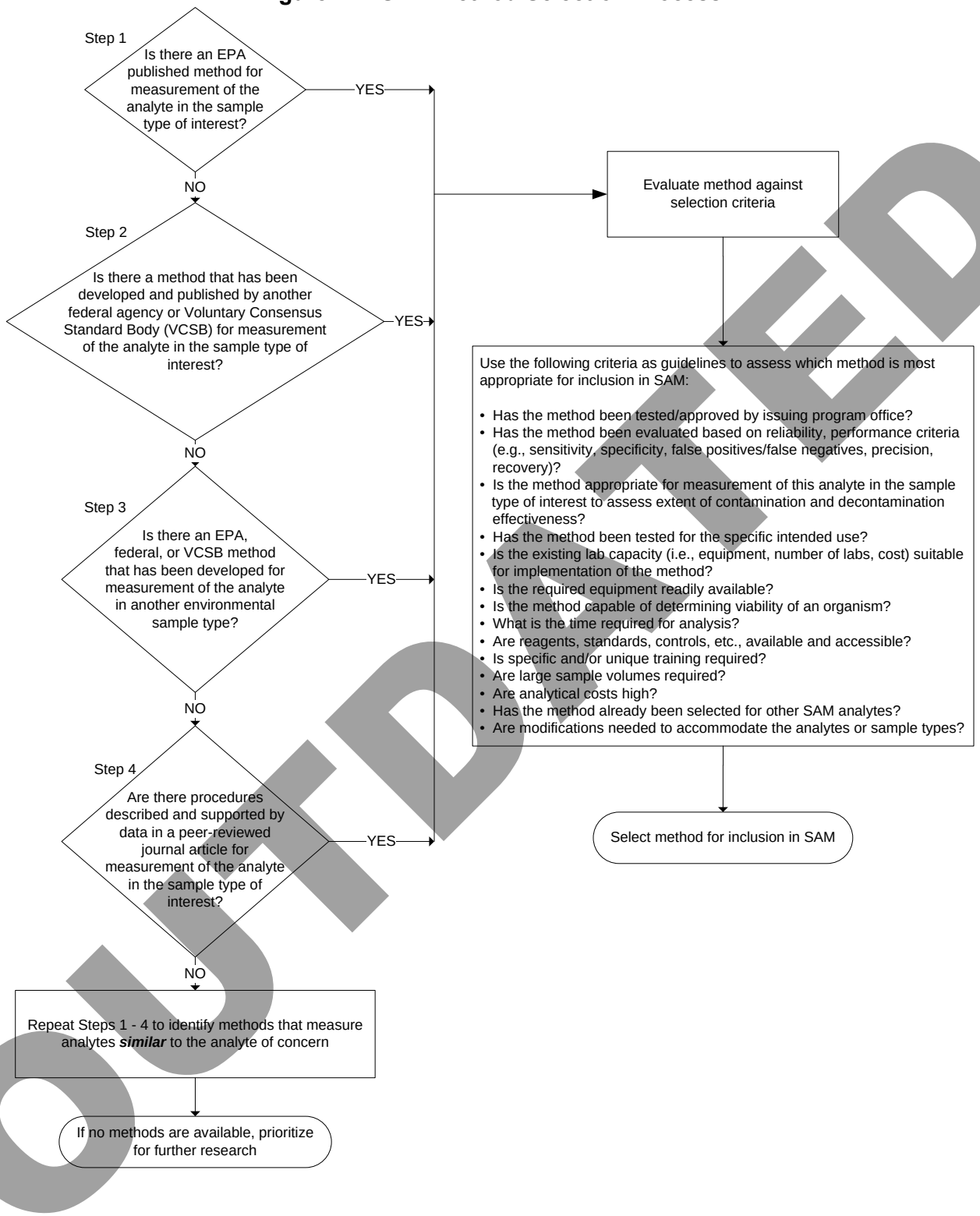
[1]EPA Science Advisory Board's Homeland Security Advisory Committee: http://yosemite.epa.gov/sab/sabpeople.nsf/WebCommittees/BOARD

Figure 2-1. SAM Method Selection Process



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Section 3.0: Scope and Application

The premise and purpose of this document is to standardize the analytical methods that will be used in cases when multiple laboratories are called on to analyze environmental samples following a homeland security event (i.e., CBR crime/attack). The document also is intended as a tool that will be available to assist state and local laboratories in planning for and analyzing environmental samples following a homeland security event. The methods presented in this document should be used to:

- Determine the extent of site contamination (assumes early responders have identified contaminants prior to EPA's remediation effort), and
- Confirm effectiveness of decontamination in support of site clearance decisions.

The methods provided are limited to those that would be used to determine, to the extent possible within analytical limitations, the presence of chemical, radiochemical, pathogen, and biotoxin analytes of concern and their concentrations and viability, when applicable, in environmental media. The methods include detailed laboratory procedures for confirming the identification of analytes and determining their concentrations in environmental samples. The methods, therefore, are not designed to be used for rapid or immediate response or for conducting an initial evaluation (triage or screening) of suspected material to determine if it poses an immediate danger or should be analyzed in specially designed, highly secure facilities. This document also is not intended to provide information regarding sample collection activities or equipment. Methods for addressing these needs are and will be the subject of other efforts. In conjunction with SAM, NHSRC is developing SAM companion documents that are intended to provide information regarding field screening equipment, sample collection, laboratory rapid screening/preliminary identification equipment, and sample disposal in support of the confirmatory methods and analytes listed in SAM. Currently available SAM companion documents are listed in Attachment 1.

Methods are provided in this document as corresponding to specific analyte/sample type combinations that are listed in Appendices A (chemical), B (radiochemical), C (pathogen), and D (biotoxin). Summaries of each method are provided in numerical order by the developing agency, throughout Sections 5.2 (chemical methods), 6.2 (radiochemical methods), and 8.2 (biotoxin methods).

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It is important to note that, in some cases, the methods included in this document have not been fully validated for the analyte/sample type combination(s) for which they have been selected. The information contained in this document represents the latest step in an ongoing effort by EPA's NHSRC to provide standardized analytical methods for use by those laboratories tasked with performing confirmatory analyses on environmental samples in support of EPA restoration efforts following a homeland security incident. The information also can be found on the SAM Web site (www.epa.gov/sam), which provides searchable links to supporting information based on SAM analytes and the analytical methods listed.

Although at this time, some of the methods listed have not been fully validated for a particular analyte (e.g., analytes not explicitly identified in the method) or sample type, the methods are considered to contain the most appropriate currently available techniques. Unless a published method listed in this document states specific applicability to the analyte/sample type combination for which it has been selected, it should be assumed that method testing is needed, and adjustments may be required to accurately account for variations in analyte characteristics, environmental samples, analytical interferences, and target risk levels.

Many of the SAM analytes have only recently become an environmental concern. EPA is actively pursuing development and validation of Standard Analytical Protocols (SAPs) based on the methods listed, including optimization of procedures for measuring target analytes or agents. In those cases where method procedures are determined to be insufficient for a particular situation, EPA will provide guidelines regarding appropriate actions. This will be an ongoing process as EPA will strive to establish a consistent level of validation for all listed analytes.

EPA recognizes that specification of a single method may limit laboratory capacity and techniques that may be needed to evaluate difficult samples. In those cases where method procedures are determined to be insufficient for a particular situation, EPA will provide guidelines regarding appropriate actions (see list of contacts in Section 4). Where further development and testing are necessary, EPA is developing and validating SAPs based on the methods that are listed in this document. Once validation is complete, data regarding the resulting method performance and data quality objectives will be available. The SAM document and corresponding SAPs will be reviewed frequently. EPA plans to continue to update the SAM document to address the needs of homeland security, to reflect improvements in analytical methodology and new technologies, and to incorporate changes in analytes based on needs. EPA also anticipates that addenda may be generated to provide guidelines regarding issues that currently are not addressed by this document. Any deviations from the methods referenced in this document should be coordinated with the appropriate point(s) of contact identified in Section 4.

Participants in the chemical, radiochemical, pathogen, and biotoxin work groups, including representatives from the EPA, CDC, FDA, DHS, FBI, DoD, USDA, and USGS evaluated the suitability of existing methodologies and selected this set of methods for use by those laboratories that support EPA environmental restoration efforts in an emergency. EPA recognizes that this advanced selection of such methods may pose potential risks, including the following:

- Selecting technologies that may not be the most cost-effective technologies currently available for addressing the particular situation at hand;
- Selecting methodologies that may not be appropriate for use in responding to a particular emergency because EPA did not anticipate having to analyze for a particular analyte or analyte/sample type combination; and
- Preventing development and adoption of new and better measurement technologies.

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To address these potential risks as soon as possible, EPA plans to take several steps. These include the following:

- Developing and specifying measurement quality objectives for all analyte/sample type combinations listed in this document. This includes required minimum standards of accuracy (bias and precision) and sensitivity for the analysis of samples that support the data quality needs of the particular stage of the emergency response/recovery process);
- Specifying guidelines for ensuring the analytical methods listed provide results that are consistent with and support their intended use as indicated in SAM;
- Working with other government agencies and the private sector to establish a laboratory network to ensure that laboratories, selected to assist EPA and its federal, state, and local partners in responding to homeland security events, have the requisite expertise and systems to perform this type of testing; and
- Continuing to work with multiple agencies and stakeholders to update SAM and supporting documents periodically.

Public officials must accurately assess all of the activities that are needed concerning site contamination following an emergency situation. These activities include initial assessment of potential site contamination for determination of immediate public and environmental risk, determination of the extent of contamination, and full remediation of the site. EPA recognizes that having data of known and documented quality is critical in making proper decisions during each of these activities. Data quality objectives (DQOs) must be established for each response activity[2]. These DQOs are based upon needs for both quality and response time. During initial assessments, time is of utmost importance and DQOs must be established that weigh the need for rapid analytical response (e.g., using screening methods) against the need for very high quality data (confirmational methods such as those listed in SAM). Many of the methods listed in this document include quality control (QC) requirements for collecting and analyzing samples. EPA will assess these QC requirements to ensure analytical data quality supports decisions concerning site remediation and release. These QC requirements may be adjusted as necessary to maximize data and decision quality. Specific QC considerations and recommendations for analysis of samples for chemical, radiochemical, pathogen, and biotoxin analytes are provided in each corresponding section of this document (i.e., Sections 5.1.2, 6.1.2, 7.1.2, and 8.1.2, respectively).

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[2] Information regarding EPA's DQO process, considerations, and planning is available at: http://www.epa.gov/QUALITY/dqos.html.

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Section 4.0: Points of Contact

Questions concerning this document, or the methods identified in this document, should be addressed to the appropriate point(s) of contact identified below. These contacts should be consulted regarding any method deviations or modifications, sample problems or interferences, QC requirements, the use of potential alternative methods, or the need to address analytes or sample types other than those listed in SAM. As previously indicated, any deviations from the recommended method(s) should be reported immediately to ensure data comparability is maintained when responding to homeland security events. In addition, general questions and comments can be submitted via the SAM Web site (www.epa.gov/sam).

General	
Kathy Hall - *Primary* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 379-5260 hall.kathy@epa.gov	Romy Lee - *Alternate* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 569-7016 lee.romy@epa.gov
Chemical Methods	
Steve Reimer - *Primary* U.S. EPA Region 10 - Manchester Laboratory 7411 Beach Drive East Port Orchard, WA 98366 (360) 871-8718 reimer.steve@epa.gov	Matthew Magnuson - *Alternate* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 569-7321 magnuson.matthew@epa.gov
Radiochemical Methods	
John Griggs - *Primary* U.S. EPA Office of Radiation and Indoor Air Environmental Laboratory 540 South Morris Avenue Montgomery, AL 36115-2601 (334) 270-3450 griggs.john@epa.gov	Kathy Hall - *Alternate* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 379-5260 hall.kathy@epa.gov
Pathogen Methods	
Sanjiv Shah - *Primary* National Homeland Security Research Center U.S. EPA ORD - 8801RR 1200 Pennsylvania Avenue, NW Washington, DC 20460 (202) 564-9522 shah.sanjiv@epa.gov	Erin Silvestri - *Alternate* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 569-7619 silvestri.erin@epa.gov
Biotoxins Methods	
Matthew Magnuson - *Primary* National Homeland Security Research Center U.S. EPA ORD (NG16) 26 West Martin Luther King Jr. Drive Cincinnati, OH 45268 (513) 569-7321 magnuson.matthew@epa.gov	Sanjiv Shah - *Alternate* National Homeland Security Research Center U.S. EPA ORD - 8801RR 1200 Pennsylvania Avenue, NW Washington, DC 20460 (202) 564-9522 shah.sanjiv@epa.gov

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Section 5.0: Selected Chemical Methods

Appendix A provides a list of methods to be used in analyzing environmental samples for chemical contaminants during remediation activities that result from a homeland security event. Methods are listed for each analyte and for each sample type that potentially may need to be measured and analyzed when responding to an environmental emergency. Procedures from peer-reviewed journal articles are listed for those analyte-sample type combinations where methods are not available. Once standard procedures are available, the literature references will be replaced.

Please note: This section provides guidance for selecting chemical methods that have a high likelihood of assuring analytical consistency when laboratories are faced with a large scale environmental restoration crisis. Not all methods have been verified for the analyte/sample type combination listed in Appendix A. Please refer to the specified method to identify analyte/sample type combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 4.

Appendix A is sorted alphabetically by analyte and includes the following information:

- **Analyte(s).** The component, contaminant, or constituent of interest.
- **Chemical Abstracts Service Registration Number (CAS RN).** A unique identifier for chemical substances that provides an unambiguous way to identify a chemical or molecular structure when there are many possible systematic, generic, or trivial names.
- **Determinative technique.** An analytical instrument or technique used to determine the quantity and identification of compounds or components in a sample.
- **Method type.** Two method types (sample preparation and determinative) are used to complete sample analysis. In some cases, a single method contains information for both sample preparation and determinative procedures. In most instances, however, two separate methods may need to be used in conjunction.
- **Solid samples.** The recommended method/procedure to identify and measure the analyte of interest in solid phase samples.
- **Aqueous liquid samples.** The recommended method/procedure to identify and measure the analyte of interest in aqueous liquid phase samples.
- **Drinking water samples.** The recommended method/procedure to identify and measure the analyte of interest in drinking water samples.
- **Air samples.** The recommended method/procedure to identify and measure the analyte of interest in air samples.
- **Wipe samples.** The recommended method/procedure to identify and measure the analyte of interest in wipes used to collect a sample from a surface.

Following a homeland security event, it is assumed that only those areas with contamination greater than pre-existing/naturally prevalent levels commonly found in the environment would be subject to remediation. Dependent on site- and event-specific goals, investigation of background levels using methods listed in Appendix A is recommended.

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5.1 General Guidelines

This section provides a general overview of how to identify the appropriate chemical method(s) for a given analyte-sample type combination, as well as recommendations for QC procedures.

For additional information on the properties of the chemicals listed in Appendix A, TOXNET (http://toxnet.nlm.nih.gov/index.html), a cluster of databases on toxicology, hazardous chemicals, and related areas maintained by the National Library of Medicine, is an excellent resource. Additional resources include:

- SRC's PHYSPROP (http://srcinc.com/what-we-do/product.aspx?id=133) and CHEMFATE, part of the Environmental Fate Database supported by EPA (http://srcinc.com/what-we-do/product.aspx?id=132&terms=Environmental+Fate+and+Exposure).
- INCHEM at http://www.inchem.org/ contains both chemical and toxicity information.
- The Registry of Toxic Effects of Chemical Substances (RTECS) database can be accessed via the NIOSH Web site at http://www.cdc.gov/niosh/rtecs/default.html for toxicity information.
- EPA's Integrated Risk Information System (IRIS): http://www.epa.gov/iris/ contains toxicity information (searchable on TOXNET).
- *Forensic Science and Communications* published by the Laboratory Division of the FBI. http://www.fbi.gov/hq/lab/fsc/current/backissu.htm.
- Joint Research Centre/Institute for Health & Consumer Protection: http://ecb.jrc.it and http://ecb.jrc.it/testing-methods/ containing information regarding European Directive 67/548/EEC and Annex V.
- Agency of Toxic Substances & Disease Registry (ATSDR) Toxic Substances Portal, Toxicological Profiles: http://www.atsdr.cdc.gov/toxprofiles/index.asp.

Additional research on chemical contaminants is ongoing within EPA. Databases to manage this information are currently under development.

5.1.1 Standard Operating Procedures for Identifying Chemical Methods

To determine the appropriate method to be used on an environmental sample, locate the analyte of concern under the "Analyte(s)" column in Appendix A: Chemical Methods. After locating the analyte of concern, continue across the table to identify the appropriate determinative technique (e.g., high performance liquid chromatography [HPLC], gas chromatography – mass spectrometry [GC-MS]), then identify the appropriate sample preparation and determinative method(s) for the sample type of interest (solid, aqueous liquid, drinking water, air, or wipe). In some cases, two methods (sample preparation and determinative) are needed to complete sample analysis.

Sections 5.2.1 through 5.2.85 below provide summaries of the sample preparation and determinative methods listed in Appendix A. Once a method has been identified in Appendix A, **Table 5-1** can be used to locate the method summary.

Table 5-1. Chemical Methods and Corresponding Section Numbers

Analyte	CAS RN	Method	Section
Acephate	30560-19-1	538 (EPA OW)	5.2.10
		Chromatographia. 2006. 63(5/6): 233–237	5.2.82
		Journal of Chromatography A. 2007. 1154(1): 3–25	5.2.83

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Analyte	CAS RN	Method	Section
Acrylamide	79-06-1	3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8316 (EPA SW-846)	5.2.34
		PV2004 (OSHA)	5.2.68
Acrylonitrile	107-13-1	524.2	5.2.7
		3570 (EPA SW-846)	5.2.19
		5035A (EPA SW-846)	5.2.22
		8260C (EPA-SW846)	5.2.30
		8290A Appendix A (EPA SW-846)	5.2.32
		PV2004 (OSHA)	5.2.68
Aldicarb (Temik)	116-06-3	531.2 (EPA OW)	5.2.9
		3570 (EPA SW-846)	5.2.19
Aldicarb sulfone	1646-88-4	8290A Appendix A (EPA SW-846)	5.2.32
		8318A (EPA SW-846)	5.2.35
Aldicarb sulfoxide	1646-87-3	5601 (NIOSH)	5.2.51
		D7600-09 (ASTM)	5.2.75
Allyl alcohol	107-18-6	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		TO-15 (EPA ORD)	5.2.45
4-Aminopyridine	504-24-5	3535A (EPA SW-846)	5.2.16
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8330B (EPA SW-846)	5.2.37
Ammonia	7664-41-7	350.1 (EPA OW)	5.2.6
		6015 (NIOSH)	5.2.56
		4500-NH_3 B (SM)	5.2.77
		4500-NH_3 G (SM)	5.2.78
Ammonium metavanadate (analyze as total vanadium)	7803-55-6	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
Arsenic, Total	7440-38-2	6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
Arsenic trioxide (analyze as total arsenic)	1327-53-3	IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Arsine (analyze as total arsenic in non-air samples)	7784-42-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		6001 (NIOSH)	5.2.52
		9102 (NIOSH)	5.2.61
Asbestos	1332-21-4	D5755-03 (ASTM)	5.2.70
		D6480-05 (ASTM)	5.2.71
		10312:1995 (ISO)	5.2.76
Boron trifluoride	7637-07-2	ID216SG (OSHA)	5.2.67

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Analyte	CAS RN	Method	Section
Brodifacoum	56073-10-0	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
Bromadiolone	28772-56-7	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		D7600-09 (ASTM)	5.2.75
BZ [Quinuclidinyl benzilate]	6581-06-2	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
Calcium arsenate (analyze as total arsenic)	7778-44-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Carbofuran (Furadan)	1563-66-2	531.2 (EPA OW)	5.2.9
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8318A (EPA SW-846)	5.2.35
		5601 (NIOSH)	5.2.51
		D7600-09 (ASTM)	5.2.75
Carfentanil	59708-52-0	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36

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Analyte	CAS RN	Method	Section
Carbon disulfide	75-15-0	524.2 (EPA OW)	5.2.7
		5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		TO-15 (EPA ORD)	5.2.45
Chlorfenvinphos	470-90-6	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Chlorine	7782-50-5	4500-Cl G (SM)	5.2.79
		Analyst. 1999. 124: 1853–1857	5.2.80
2-Chloroethanol	107-07-3	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		2513 (NIOSH)	5.2.48
3-Chloro-1,2-propanediol	96-24-2	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Chloropicrin	76-06-2	551.1 (EPA OW)	5.2.12
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		PV2103 (OSHA)	5.2.69
Chlorosarin Chlorosoman	1445-76-7 7040-57-5	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Chlorpyrifos Chlorpyrifos oxon	2921-88-2 5598-15-2	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Crimidine	535-89-7	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
Cyanide, Amenable to chlorination	NA	RLAB Method 3135.2I	5.2.39
Cyanide, Total	57-12-5	335.4 (EPA OW)	5.2.5
		ISM01.2 CN (EPA CLP)	5.2.38
		6010 (NIOSH)	5.2.54
Cyanogen chloride	506-77-4	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		TO-15 (EPA ORD)	5.2.45
Cyclohexyl sarin (GF)	329-99-7	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
1,2-Dichloroethane	107-06-2	524.2 (EPA OW)	5.2.7
		5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		TO-15 (EPA ORD)	5.2.45

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Analyte	CAS RN	Method	Section
Dichlorvos	62-73-7	525.2 (EPA OW)	5.2.8
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Dicrotophos	141-66-2	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Diesel range organics	NA	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8015C (EPA SW-846)	5.2.29
		8290A Appendix A (EPA SW-846)	5.2.32
Diisopropyl methylphosphonate (DIMP)	1445-75-6	538 (EPA OW)	5.2.10
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7597-09 (ASTM)	5.2.72
Dimethylphosphite	868-85-9	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Dimethylphosphoramidic acid	33876-51-6	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
Diphacinone	82-66-6	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
Disulfoton	298-04-4	525.2 (EPA OW)	5.2.8
		3541 (EPA SW-846)	5.2.17
Disulfoton sulfone oxon	2496-91-5	3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
Disulfoton sulfoxide	2497-07-6	8270D (EPA SW-846)	5.2.31
Disulfoton sulfoxide oxon	2496-92-6	8290A Appendix A (EPA SW-846)	5.2.32
		5600 (NIOSH)	5.2.50
1,4-Dithiane	505-29-3	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
EA2192 [Diisopropylaminoethyl methyl-thiolophosphonate]	73207-98-4	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
Ethyl methylphosphonic acid (EMPA)	1832-53-7	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7597-09 (ASTM)	5.2.72
Ethyldichloroarsine (ED)	598-14-1	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		8270D (EPA SW-846)	5.2.31
		TO-15 (EPA ORD)	5.2.45
		9102 (NIOSH)	5.2.61

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Analyte	CAS RN	Method	Section
N-Ethyldiethanolamine (EDEA)	139-87-7	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7599-09 (ASTM)	5.2.74
Ethylene oxide	75-21-8	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		TO-15 (EPA ORD)	5.2.45
Fenamiphos	22224-92-6	525.2 (EPA OW)	5.2.8
		3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Fentanyl	437-38-7	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
Fluoride	16984-48-8	300.1, Rev 1.0 (EPA OW)	5.2.4
Fluoroacetamide	640-19-7	Journal of Chromatography B. 2008. 876, 103–108	5.2.84
Fluoroacetic acid and fluoroacetate salts	NA	300.1, Rev 1.0 (EPA OW)	5.2.4
		S301-1 (NIOSH)	5.2.62
		Analytical Letters. 1994. 27(14): 2703–2718	5.2.81
2-Fluoroethanol	371-62-0	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		2513 (NIOSH)	5.2.48
Formaldehyde	50-00-0	556.1 (EPA OW)	5.2.13
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8315A (EPA SW-846)	5.2.33
		2016 (NIOSH)	5.2.47

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Analyte	CAS RN	Method	Section
Gasoline range organics	NA	3570 (EPA SW-846)	5.2.19
		5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8015C (EPA SW-846)	5.2.29
		8290A Appendix A (EPA SW-846)	5.2.32
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	3535A (EPA SW-846)	5.2.16
		3570 (EPA SW-846)	5.2.19
Hexamethylenetriperoxidediamine (HMTD)	283-66-9	8290A Appendix A (EPA SW-846)	5.2.32
		8330B (EPA SW-846)	5.2.37
Hydrogen bromide Hydrogen chloride	10035-10-6 7647-01-0	7903 (NIOSH)	5.2.58
Hydrogen cyanide	74-90-8	6010 (NIOSH)	5.2.54
Hydrogen fluoride	7664-39-3	7903 (NIOSH)	5.2.58
Hydrogen sulfide	7783-06-4	6013 (NIOSH)	5.2.55
Isopropyl methylphosphonic acid (IMPA)	1832-54-8	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7597-09 (ASTM)	5.2.72
Kerosene	64742-81-0	3570 (EPA SW-846)	5.2.19
		5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8015C (EPA SW-846)	5.2.29
		8290A Appendix A (EPA SW-846)	5.2.32
Lead arsenate (analyze as total arsenic) Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic) Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic) Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic) Lewisite oxide	7645-25-2 541-25-3 40334-69-8 40334-70-1 1306-02-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Mercuric chloride (analyze as total mercury)	7487-94-7	245.1 (EPA OW)	5.2.3
		7473 (EPA SW-846)	5.2.27
		9102 (NIOSH)	5.2.61
Mercury, Total	7439-97-6	245.1 (EPA OW)	5.2.3
		7473 (EPA SW-846)	5.2.27
		IO-5 (EPA ORD)	5.2.43
		9102 (NIOSH)	5.2.61
Methamidophos	10265-92-6	538 (EPA OW)	5.2.10
		Chromatographia. 2006. 63(5/6): 233–237	5.2.82
		Journal of Chromatography A. 2007. 1154(1): 3–25	5.2.83

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Analyte	CAS RN	Method	Section
Methomyl	16752-77-5	531.2 (EPA OW)	5.2.9
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8318A (EPA SW-846)	5.2.35
		5601 (NIOSH)	5.2.51
		D7600-09 (ASTM)	5.2.75
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2	245.1 (EPA OW)	5.2.3
		7473 (EPA SW-846)	5.2.27
		IO-5 (EPA ORD)	5.2.43
		9102 (NIOSH)	5.2.61
Methyl acrylonitrile	126-98-7	524.2 (EPA OW)	5.2.7
		3570 (EPA SW-846)	5.2.19
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		8290A Appendix A (EPA SW-846)	5.2.32
		PV2004 (OSHA)	5.2.68
Methyl fluoroacetate (analyze as fluoroacetate ion)	453-18-9	300.1, Rev 1.0 (EPA OW)	5.2.4
		S301-1 (NIOSH)	5.2.62
		Analytical Letters. 1994. 27(14): 2703–2718	5.2.81
Methyl hydrazine	60-34-4	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		3510 (NIOSH)	5.2.49
Methyl isocyanate	624-83-9	OSHA 54	5.2.64
Methyl paraoxon Methyl parathion	950-35-6 298-00-0	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Methylamine	74-89-5	OSHA 40	5.2.63
N-Methyldiethanolamine (MDEA)	105-59-9	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7599-09 (ASTM)	5.2.74

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Analyte	CAS RN	Method	Section
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Methylphosphonic acid (MPA)	993-13-5	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7597-09 (ASTM)	5.2.72
Mevinphos	7786-34-7	525.2 (EPA OW)	5.2.8
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Monocrotophos	6923-22-4	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)-ethylamine]	538-07-8	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)-methylamine]	51-75-2	3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)-amine]	555-77-1	8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Mustard, sulfur / Mustard gas (HD)	505-60-2	3570 (EPA SW-846)	5.2.19
		3571 (EPA SW-846)	5.2.20
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
Nicotine compounds	54-11-5	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	3535A (EPA SW-846)	5.2.16
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8330B (EPA SW-846)	5.2.37
Osmium tetroxide (analyze as total osmium)	20816-12-0	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		9102 (NIOSH)	5.2.61
Oxamyl	23135-22-0	531.2 (EPA OW)	5.2.9
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8318A (EPA SW-846)	5.2.35
		5601 (NIOSH)	5.2.51
		D7600-09 (ASTM)	5.2.75
Paraquat	4685-14-7	549.2 (EPA OW)	5.2.11
Paraoxon	311-45-5	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
Parathion	56-38-2	3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Pentaerythritol tetranitrate (PETN)	78-11-5	3535A (EPA SW-846)	5.2.16
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8330B (EPA SW-846)	5.2.37
Phencyclidine	77-10-1	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
Phorate Phorate sulfone Phorate sulfone oxon Phorate sulfoxide Phorate sulfoxide oxon	298-02-2 2588-04-7 2588-06-9 2588-03-6 2588-05-8	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Phosgene	75-44-5	OSHA 61	5.2.65
Phosphamidon	13171-21-6	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Phosphine	7803-51-2	6002 (NIOSH)	5.2.53
Phosphorus trichloride	7719-12-2	6402 (NIOSH)	5.2.57
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7597-09 (ASTM)	5.2.72
Propylene oxide	75-56-9	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30
		1612 (NIOSH)	5.2.46
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Sarin (GB)	107-44-8	3570 (EPA SW-846)	5.2.19
		3571 (EPA SW-846)	5.2.20
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
Sodium arsenite (analyze as total arsenic)	7784-46-5	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Sodium azide (analyze as azide ion)	26628-22-8	300.1, Rev 1.0 (EPA OW)	5.2.4
		ID-211 (OSHA)	5.2.66
		Journal of Forensic Sciences. 1998. 43(1): 200–202	5.2.85
Soman (GD)	96-64-0	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Strychnine	57-24-9	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
Tabun (GA)	77-81-6	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Tetraethyl pyrophosphate	107-49-3	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44

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Analyte	CAS RN	Method	Section
Tetramethylenedisulfotetramine	80-12-6	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
Thallium sulfate (analyze as total thallium)	10031-59-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
Thiodiglycol (TDG)	111-48-8	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7598-09 (ASTM)	5.2.73
Thiofanox	39196-18-4	531.2 (EPA OW)	5.2.9
		3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		5601 (NIOSH)	5.2.51
1,4-Thioxane	15980-15-1	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
Titanium tetrachloride (analyze as total titanium)	7550-45-0	3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		9102 (NIOSH)	5.2.61

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Analyte	CAS RN	Method	Section
Triethanolamine (TEA)	102-71-6	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8290A Appendix A (EPA SW-846)	5.2.32
		8321B (EPA SW-846)	5.2.36
		TO-10A (EPA ORD)	5.2.44
		D7599-09 (ASTM)	5.2.74
Trimethyl phosphite	121-45-9	3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
		3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4	3535A (EPA SW-846)	5.2.16
		3570 (EPA SW-846)	5.2.19
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7	8290A Appendix A (EPA SW-846)	5.2.32
		8330B (EPA SW-846)	5.2.37
Vanadium pentoxide (analyze as total vanadium)	1314-62-1	200.7 (EPA OW)	5.2.1
		200.8 (EPA OW)	5.2.2
		3050B (EPA SW-846)	5.2.14
		6010C (EPA SW-846)	5.2.23
		6020A (EPA SW-846)	5.2.24
		IO-3.1 (EPA ORD)	5.2.40
		IO-3.4 (EPA ORD)	5.2.41
		IO-3.5 (EPA ORD)	5.2.42
		9102 (NIOSH)	5.2.61
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0	3520C (EPA SW-846)	5.2.15
		3535A (EPA SW-846)	5.2.16
		3541 (EPA SW-846)	5.2.17
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5	3545A (EPA SW-846)	5.2.18
		3570 (EPA SW-846)	5.2.19
VM [phosphonothioic acid, methyl-,S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5	8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9	3570 (EPA SW-846)	5.2.19
		3571 (EPA SW-846)	5.2.20
		8270D (EPA SW-846)	5.2.31
		8290A Appendix A (EPA SW-846)	5.2.32
		TO-10A (EPA ORD)	5.2.44
White phosphorus	12185-10-3	3570 (EPA SW-846)	5.2.19
		7580 (EPA SW-846)	5.2.28
		8290A Appendix A (EPA SW-846)	5.2.32
		7905 (NIOSH)	5.2.59

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Analyte	CAS RN	Method	Section
The following analytes should be prepared and/or analyzed by the following methods only if problems (e.g., insufficient recovery, interferences) occur when using the sample preparation/determinative techniques identified for these analytes in Appendix A.			
Allyl alcohol	107-18-6	TO-10A (EPA ORD)	5.2.44
BZ [Quinuclidinyl benzilate]	6581-06-2	8270D (EPA SW-846)	5.2.31
3-Chloro-1,2-propanediol	96-24-2	TO-15 (EPA ORD)	5.2.45
Chlorosarin Chlorosoman	1445-76-7 7040-57-5	TO-15 (EPA ORD)	5.2.45
Crimidine	535-89-7	8321B (EPA SW-846)	5.2.36
Diisopropyl methylphosphonate (DIMP)	1445-75-6	8270D (EPA SW-846)	5.2.31
		TO-15 (EPA ORD)	5.2.45
Dimethylphosphoramidic acid	33876-51-6	8270D (EPA SW-846)	5.2.31
EA2192 [Diisopropylaminoethyl methyl-thiolophosphonate] Ethyl methylphosphonic acid (EMPA)	73207-98-4 1832-53-7	8270D (EPA SW-846)	5.2.31
Hydrogen fluoride	7664-39-3	7906 (NIOSH)	5.2.60
Isopropyl methylphosphonic acid (IMPA)	1832-54-8	8270D (EPA SW-846)	5.2.31
Mercuric chloride (analyze as total mercury) Mercury, Total	7487-94-7 7439-97-6	7470A (EPA SW-846)	5.2.25
		7471B (EPA SW-846)	5.2.26
Methamidophos	10265-92-6	5600 (NIOSH)	5.2.50
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2	7470A (EPA SW-846)	5.2.25
		7471B (EPA SW-846)	5.2.26
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3	TO-15 (EPA ORD)	5.2.45
Methylphosphonic acid (MPA)	993-13-5	8270D (EPA SW-846)	5.2.31
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4	8270D (EPA SW-846)	5.2.31
Sarin (GB) Soman (GD)	107-44-8 96-64-0	TO-15 (EPA ORD)	5.2.45
1,4-Thioxane	15980-15-1	5030C (EPA SW-846)	5.2.21
		5035A (EPA SW-846)	5.2.22
		8260C (EPA SW-846)	5.2.30

Method summaries are listed in order of method selection hierarchy (see Figure 2-1), starting with EPA methods, followed by methods from other federal agencies, voluntary consensus standard bodies (VCSBs), and literature references. Methods are listed in numerical order under each publisher. Where available, a direct link to the full text of the method is provided in the method summary. For additional information on preparation procedures and methods available through consensus standards organizations, please use the contact information provided in **Table 5-2**.

Table 5-2. Sources of Chemical Methods

Name	Publisher	Reference
NEMI	EPA, USGS	http://www.nemi.gov
EPA OW Methods	EPA OW	http://www.epa.gov/safewater/methods/sourcalt.html
EPA SW-846 Methods	EPA OSWER	http://www.epa.gov/epaoswer/hazwaste/test/main.htm
EPA ORD Methods	EPA ORD	http://www.epa.gov/ttnamti1/

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Name	Publisher	Reference
EPA Air Toxics Methods	EPA OAR	http://www.epa.gov/ttn/amtic/airtox.html
OSHA Methods	OSHA	http://www.osha.gov/dts/sltc/methods/index.html
NIOSH Methods	NIOSH	http://www.cdc.gov/niosh/nmam/
Standard Methods for the Examination of Water and Wastewater (SM), 21st Edition, 2005*	American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF)	http://www.standardmethods.org
*Annual Book of ASTM Standards**	ASTM International	http://www.astm.org
GESTIS Substance Database	BGIA	http://www.dguv.de/bgia/en/gestis/stoffdb/index.jsp
ISO Methods*	ISO	http://www.iso.org
Official Methods of Analysis of AOAC International*	AOAC International	http://www.aoac.org
Analyst	Royal Society of Chemistry	http://www.rsc.org/Publishing/Journals/AN/
Analytical Letters*	Taylor & Francis	http://www.informaworld.com/smpp/title~content=t713597227
Journal of Chromatography A*	Elsevier Science Publishers	http://www.elsevier.com/
Journal of Forensic Sciences*	ASTM International	http://www.astm.org
Chromatographia	Vieweg+Teubner	http://www.chromatographia.de/
EPA Water Contamination Information Tool (WCIT)	EPA OW Water Security Division (WSD)	http://www.epa.gov/wcit
Analytical Chemistry	American Chemical Society (ACS)	pubs.acs.org/journal/anacham
Journal of Agricultural and Food Chemistry	ACS	pubs.acs.org/journal/jafcau

* Subscription and/or purchase required.

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5.1.2 General QC Guidelines for Chemical Methods

Having analytical data of appropriate quality requires that laboratories: (1) conduct the necessary QC activities to ensure that measurement systems are in control and operating correctly; (2) properly document results of the analyses; and (3) properly document measurement system evaluation of the analysis-specific QC, including corrective actions. In addition to the laboratories being capable of generating accurate and precise data during site remediation, they must be able to deliver results in a timely and efficient manner. Therefore, laboratories must be prepared with calibrated instruments, the proper standards, standard analytical procedures, standard operating procedures, and qualified and trained staff. Moreover, laboratories also must be capable of providing rapid turnaround of sample analyses and data reporting.

The level or amount of QC needed during sample analysis and reporting depends on the intended purpose of the data that are generated (e.g., the decision(s) to be made). The specific needs for data generation should be identified. QC requirements and data quality objectives should be derived based on those

needs, and should be applied consistently across laboratories when multiple laboratories are used. For almost all of the chemical warfare agents, most laboratories will not have access to analytical standards for calibration and QC. Use of these agents is strictly controlled by the DoD and access is limited. For information regarding laboratory analysis of samples containing CWAs or laboratory requirements to possess and use ultra-dilute agent standards, please contact Terry Smith, EPA's Office of Emergency Management, at (202) 564-2908.

A minimum set of analytical QC procedures should be planned, documented, and conducted for all chemical testing. Some method-specific QC requirements are described in many of the individual methods that are cited in this document and will be referenced in any SAPs developed to address specific analytes and sample types of concern. Individual methods, sampling and analysis protocols, or contractual statements of work should also be consulted to determine if any additional QC might be needed. Analytical QC requirements generally consist of analysis of laboratory control samples to document whether the analytical system is in control; matrix spikes to identify and quantify measurement system accuracy for the media of concern and, at the levels of concern, various blanks as a measure of freedom from contamination; as well as matrix spike duplicates or sample replicates to assess data precision.

In general, for measurement of chemical analytes, appropriate QC includes an initial demonstration of measurement system capability, as well as ongoing analysis of standards and other samples to ensure the continued reliability of the analytical results. Examples of appropriate QC include:

- Demonstration that the measurement system is operating properly:
  - Initial calibration; and
  - Method blanks.
- Demonstration of analytical method suitability for intended use:
  - Detection and quantitation limits;
  - Precision and recovery (verify measurement system has adequate accuracy); and
  - Analyte/matrix/level of concern-specific QC samples (verify that measurement system has adequate sensitivity at levels of concern).
- Demonstration of continued analytical method reliability:
  - Matrix spike/matrix spike duplicates (MS/MSDs) (recovery and precision);
  - QC samples (system accuracy and sensitivity at levels of concern);
  - Surrogate spikes (where appropriate);
  - Continuing calibration verification; and
  - Method blanks.

QC tests should be consistent with EPA's Good Laboratory Practice Standards (http://www.epa.gov/oecaerth/monitoring/programs/fifra/glp.html) and be run as frequently as necessary to ensure the reliability of analytical results. Additional guidance can be found at: www.epa.gov/quality/qatools.html; in Chapter 1 of EPA SW-846 Hazardous Test Methods (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/chap1.pdf); and in EPA's Drinking Water Laboratory Certification Manual (http://www.epa.gov/ogwdw000/methods/pdfs/manual_labcertification.pdf). As with the identification of needed QC samples, the frequency of QC sampling should be established based on an evaluation of data quality objectives. The type and frequency of QC tests can be refined over time.

Ensuring data quality also requires that laboratory results are properly assessed and documented. The results of the data quality assessment are transmitted to decision makers. This evaluation is as important as the data for ensuring informed and effective decisions. While some degree of data evaluation is necessary in order to be able to confirm data quality, 100% verification and/or validation is neither necessary nor conducive to efficient decision making in emergency situations. The level of such reviews should be determined based on the specific situation being assessed and on the corresponding data quality

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objectives. In every case, the levels of QC and data review necessary to support decision making should be determined as much in advance of data collection as possible.

Please note: The appropriate point of contact identified in Section 4 should be consulted regarding appropriate quality assurance (QA) and QC procedures prior to sample analysis. These contacts will consult with the EPA Environmental Response Laboratory Network (ERLN) coordinator responsible for laboratory activities during the specific event to ensure QA/QC procedures are performed consistently across laboratories. EPA program offices will be responsible for ensuring that the QA/QC practices are implemented.

5.1.3 Safety and Waste Management

It is imperative that safety precautions are used during collection, processing, and analysis of environmental samples. Laboratories should have a documented health and safety plan for handling samples that may contain the target CBR contaminants. Laboratory staff should be trained in, and need to implement, the safety procedures included in the plan. In addition, many of the methods summarized or cited in Section 5.2 contain some specific requirements, guidelines, or information regarding safety precautions that should be followed when handling or processing environmental samples and reagents.

These methods also provide information regarding waste management. Other resources that can be consulted for additional information include the following:

- CDC – Title 42 of the Code of Federal Regulations part 72 (42 CFR 72). Interstate Shipment of Etiologic Agents.
- CDC – 42 CFR part 73. Select Agents and Toxins.
- Department of Transportation (DOT) – 49 CFR part 172. Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements.
- EPA – 40 CFR part 260. Hazardous Waste Management System: General.
- EPA – 40 CFR part 270. EPA Administered Permit Programs: The Hazardous Waste Permit Program.
- OSHA – 29 CFR part 1910.1450. Occupational Exposure to Hazardous Chemicals in Laboratories.
- OSHA – 29 CFR part 1910.120. Hazardous Waste Operations and Emergency Response.

Please note that the Electronic Code of Federal Regulations (e-CFR) is available at http://ecfr.gpoaccess.gov/.

5.2 Method Summaries

Summaries for the analytical methods listed in Appendix A are provided in Sections 5.2.1 through 5.2.85. These sections contain summary information only, extracted from the selected methods. Each method summary contains a table identifying the contaminants in Appendix A to which the method applies, a brief description of the analytical method, and a link to, or source for, obtaining a full version of the method. The full version of the method should be consulted prior to sample analysis.

OUTDATED

5.2.1 EPA Method 200.7: Determination of Metals and Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Acid digestion
Determinative Technique: Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Method Developed for: Determination of metals in solution. This method is a consolidation of existing methods for water, wastewater, and solid wastes.
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples.
Detection and Quantitation: Method detection limits (MDLs) in aqueous samples have been found to be 0.008 mg/L for arsenic, 0.003 mg/L for vanadium, and 0.001 mg/L for thallium.

Description of Method: This method will determine metal-containing compounds only as the total metal (e.g., total arsenic) in aqueous samples. An aliquot of a well-mixed, homogeneous sample is accurately weighed or measured for sample processing. For total recoverable analysis of a sample containing undissolved material, analytes are first solubilized by gentle refluxing with nitric and hydrochloric acids. After cooling, the sample is made up to volume, mixed, and centrifuged or allowed to settle overnight prior to analysis. For determination of dissolved analytes in a filtered aqueous sample aliquot, or for the "direct analysis" total recoverable determination of analytes in drinking water where sample turbidity is < 1 nephelometric turbidity units (NTU), the sample is made ready for analysis by the addition of nitric acid, and then diluted to a predetermined volume and mixed before analysis. The prepared sample is analyzed using ICP-AES. Specific analytes targeted by Method 200.7 are listed in Section 1.1 of the method.

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques. Users should consult with the appropriate point of contact listed in Section 4.0 regarding use of graphite furnace atomic absorption spectrophotometry (GFAA) as a back-up or for additional confirmatory analyses.

Source: EPA. 1994. "Method 200.7: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 4.4.
http://www.epa.gov/sam/pdfs/EPA-200.7.pdf

OUTDATED

5.2.2 EPA Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Acid digestion
Determinative Technique: Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Method Developed for: Dissolved and total elements in ground water, surface water, drinking water, wastewater, sludges, and soils.
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples.
Detection and Quantitation: MDLs for arsenic in aqueous samples have been found to be 1.4 µg/L in scanning mode, and 0.4 µg/L in selected ion monitoring mode. The recommended calibration range is 10 to 200 µg/L.

Description of Method: This method will determine metal-containing compounds only as the total metal (e.g., total arsenic). An aliquot of a well-mixed, homogeneous sample is accurately weighed or measured for sample processing. For total recoverable analysis of a sample containing undissolved material, analytes are first solubilized by gentle refluxing with nitric and hydrochloric acids. After cooling, the sample is made up to volume, mixed, and centrifuged or allowed to settle overnight prior to analysis. For determination of dissolved analytes in a filtered aqueous sample aliquot, or for the "direct analysis" total recoverable determination of analytes in drinking water where sample turbidity is < 1 NTU, the sample is made ready for analysis by the addition of nitric acid, and then diluted to a predetermined volume and mixed before analysis. The prepared sample is analyzed using ICP-MS. Specific analytes targeted by Method 200.8 are listed in Section 1.1 of the method.

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques. Users should consult with the appropriate point of contact listed in Section 4.0 regarding use of graphite furnace atomic absorption spectrophotometry (GFAA) as a back-up or for additional confirmatory analyses.

Source: EPA. 1994. "Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry," Revision 5.4. http://www.epa.gov/sam/pdfs/EPA-200.8.pdf

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5.2.3 EPA Method 245.1: Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry (CVAA)

Analyte(s)	CAS RN
Mercuric chloride (analyze as total mercury)	7487-94-7
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Acid digestion
Determinative Technique: Cold vapor atomic absorption (CVAA)

Method Developed for: Mercury in surface waters. It may be applicable to saline waters, wastewaters, effluents, and domestic sewages providing potential interferences are not present.
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.
Detection and Quantitation: Applicable concentration range is 0.2 to 10.0 µg Hg/L. The detection limit for this method is 0.2 µg Hg/L.

Description of Method: This method will determine mercuric chloride and methoxyethylmercuric acetate as total mercury. If dissolved mercury is targeted, the sample is filtered prior to acidification. To detect total mercury (inorganic and organic mercury), the sample is treated with potassium permanganate and potassium persulfate to oxidize organic mercury compounds prior to analysis. Inorganic mercury is reduced to the elemental state (using stannous chloride) and aerated from solution. The mercury vapor passes through a cell positioned in the light path of a CVAA spectrophotometer. The concentration of mercury is measured using the CVAA spectrophotometer.

Source: EPA. 1994. "Method 245.1: Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry CVAA)." http://www.epa.gov/sam/pdfs/EPA-245.1.pdf

5.2.4 EPA Method 300.1, Revision 1.0: Determination of Inorganic Anions in Drinking Water by Ion Chromatography

Analyte(s)	CAS RN
Fluoride	16984-48-8
Fluoroacetic acid and fluoroacetate salts	NA
Methyl fluoroacetate	453-18-9
Sodium azide (analyze as azide ion)	26628-22-8

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: For fluoride, use direct injection. For fluoroacetic acid, fluoroacetate salts, and methyl fluoroacetate, use ultrasonic extraction by Analytical Letters, 1994, 27(14): 2703-2718 (solid and wipe samples), and water extraction by NIOSH Method S301-1 (air samples). For sodium azide, use water extraction, filtration, and acidification steps from the Journal of Forensic Science, 1998. 43(1):200-202 (solid samples), and filtration and acidification steps from this journal (aqueous liquid and drinking water samples).
Determinative Technique: Ion chromatography (IC) with conductivity detection

Method Developed for: Inorganic anions in reagent water, surface water, ground water, and finished drinking water

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Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples for fluoride, fluoroacetic acid, fluoroacetate salts, and methyl fluoroacetate. It also should be used for analysis of solid, air, and/or wipe samples for fluoroacetic acid, fluoroacetate salts, methyl fluoroacetate, and sodium azide when appropriate sample preparation techniques have been applied.
Detection and Quantitation: The detection limit for fluoride in reagent water is 0.009 mg/L. The MDL varies depending upon the nature of the sample and the specific instrumentation employed. The estimated calibration range should not extend over more than 2 orders of magnitude in concentration over the expected concentration range of the samples.

Description of Method: This method was developed for analysis of aqueous samples, and can be adapted for analysis of prepared solid and air samples when appropriate sample preparation techniques have been applied (see Appendix A). A small volume of an aqueous liquid sample (10 µL or 50 µL) is introduced into an ion chromatograph. The volume selected depends on the concentration of fluoroacetate ion in the sample. The anions of interest are separated and measured, using a system comprising a guard column, analytical column, suppressor device, and conductivity detector. The separator columns and guard columns, as well as eluent conditions, are identical. To achieve comparable detection limits, an ion chromatographic system must use suppressed conductivity detection, be properly maintained, and be capable of yielding a baseline with no more than 5 nS noise/drift per minute of monitored response over the background conductivity.

Special Considerations: For sodium azide, if analyses are problematic, refer to column manufacturer for alternate conditions.

Source: EPA. 1997. "Method 300.1: Determination of Inorganic Anions in Drinking Water by Ion Chromatography," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-300.1.pdf

5.2.5 EPA Method 335.4: Determination of Total Cyanide by Semi-Automated Colorimetry

Analyte(s)	CAS RN
Cyanide, Total	57-12-5

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Reflux-distillation
Determinative Technique: Visible spectrophotometry

Method Developed for: Cyanide in drinking, ground, surface, and saline waters, and domestic and industrial wastes
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.
Detection and Quantitation: The applicable range is 5 to 500 µg/L.

Description of Method: Cyanide is released from cyanide complexes as hydrocyanic acid by manual reflux-distillation, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reaction with chloramine-T, which subsequently reacts with pyridine and barbituric acid to give a red-colored complex.

Special Considerations: Some interferences include aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiocyanate, thiosulfate, and sulfide. These interferences can be eliminated or reduced by distillation.

Source: EPA. 1993. "Method 335.4: Determination of Total Cyanide by Semi-Automated Colorimetry," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-335.4.pdf

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5.2.6 EPA Method 350.1: Nitrogen, Ammonia (Colorimetric, Automated Phenate)

Analyte(s)	CAS RN
Ammonia	7664-41-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Distillation
Determinative Technique: Visible spectrophotometry

Method Developed for: Ammonia in drinking, ground, surface, and saline waters, and domestic and industrial wastes
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.
Detection and Quantitation: The working range for ammonia is 0.01 to 2.0 mg/L.

Description of Method: This method identifies and determines the concentration of ammonia in drinking water samples by spectrophotometry. Samples are buffered at a pH of 9.5 with borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds, and are distilled into a solution of boric acid. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside and measured spectrophotometrically.

Special Considerations: Reduced volume distillation techniques, such as midi-distillation or micro-distillation, can be used in place of traditional macro-distillation techniques.

Source: EPA. 1993. "Method 350.1: Nitrogen, Ammonia (Colorimetric, Automated Phenate)," Revision 2.0. http://www.epa.gov/sam/pdfs/EPA-350.1.pdf

5.2.7 EPA Method 524.2: Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography / Mass Spectrometry

Analyte(s)	CAS RN
Acrylonitrile	107-13-1
Carbon disulfide	75-15-0
1,2-Dichloroethane	107-06-2
Methyl acrylonitrile	126-98-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Purge-and-trap
Determinative Technique: GC-MS

Method Developed for: Purgeable volatile organic compounds (VOCs) in surface water, ground water, and drinking water in any stage of treatment
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples for carbon disulfide and 1,2-dichloroethane, and preparation and analysis of drinking and aqueous/liquid samples for acrylonitrile and methyl acrylonitrile.
Detection and Quantitation: Detection levels for acrylonitrile, carbon disulfide, 1,2-dichloroethane, and methyl acrylonitrile in reagent water have been found to be 0.22, 0.093, 0.02, and 0.11 µg/L, respectively. The applicable concentration range of this method is primarily column and matrix dependent, and is approximately 0.02 to 200 µg/L when a wide-bore thick-film capillary column is used. Narrow-bore thin-film columns may have a lower capacity, which limits the range to approximately 0.02 to 20 µg/L.

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Description of Method: VOCs and surrogates with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through the aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb the trapped sample components into a capillary gas chromatography (GC) column interfaced to a mass spectrometer (MS). The column is temperature programmed to facilitate the separation of the method analytes, which are then detected with the MS. Specific analytes targeted by Method 524.2 are listed in Section 1.1 of the method.

Special Considerations: The most recent version of this method (Method 524.3) requires instrumentation, such as cryogenic auto samplers, which are not currently in common use. If laboratory use of this equipment increases, Method 524.3 may be considered for SAM applications.

Source: EPA. 1992. "Method 524.2: Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry," Revision 4.0.
http://www.epa.gov/sam/pdfs/EPA-524.2.pdf

5.2.8 EPA Method 525.2: Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography / Mass Spectrometry

Analyte(s)	CAS RN
Dichlorvos	62-73-7
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6
Fenamiphos	22224-92-6
Mevinphos	7786-34-7

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Liquid-solid extraction (LSE) or solid-phase extraction (SPE)
Determinative Technique: GC-MS

Method Developed for: Organic compounds in finished drinking water, source water, or drinking water in any treatment stage
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and/or drinking water samples.
Detection and Quantitation: The applicable concentration range for most analytes is 0.1 to 10 µg/L.

Description of Method: Organic compounds, internal standards, and surrogates are extracted from a water sample by passing 1 L of sample through a cartridge or disk containing a solid matrix with chemically bonded C_{18} organic phase (LSE or SPE). The organic compounds are eluted from the LSE (SPE) cartridge or disk with small quantities of ethyl acetate followed by methylene chloride. The resulting extract is concentrated further by evaporation of some of the solvent. Sample components are separated, identified, and measured by injecting an aliquot of the concentrated extract into a high resolution fused silica capillary column of a GC-MS system. Specific analytes targeted by Method 525.2 are listed in Section 1.1 of the method.

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Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. SPE using C_{18} resin may not work for certain compounds having high water solubility. In these cases, other sample preparation techniques or different SPE resins may be required.

Source: EPA. 1995. "Method 525.2: Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry," Revision 2.0. http://www.epa.gov/sam/pdfs/EPA-525.2.pdf

5.2.9 EPA Method 531.2: Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization

Analyte(s)	CAS RN
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Carbofuran (Furadan)	1563-66-2
Methomyl	16752-77-5
Oxamyl	23135-22-0
Thiofanox	39196-18-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Direct injection
Determinative Technique: HPLC

Method Developed for: N-methylcarbamoyloximes and N-methylcarbamates in finished drinking water
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.
Detection and Quantitation: Detection limits range from 0.026 to 0.115 µg/L. The concentration range for target analytes in this method was evaluated between 0.2 µg/L and 10 µg/L.

Description of Method: An aliquot of sample is measured in a volumetric flask. Samples are preserved, spiked with appropriate surrogates and then filtered. Analytes are chromatographically separated by injecting a sample aliquot (up to 1000 µL) into a HPLC system equipped with a reverse phase (C_{18}) column. After elution from the column, the analytes are hydrolyzed in a post column reaction to form methylamine, which is in turn reacted to form a fluorescent isoindole that is detected by a fluorescence (FL) detector. Analytes also are quantitated using the external standard technique.

Source: EPA. 2001. "Method 531.2: Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-531.2.pdf

5.2.10 EPA Method 538: Determination of Selected Organic Contaminants in Drinking Water by Direct Aqueous Injection-Liquid Chromatography/Tandem Mass Spectrometry (DAI-LC/MS/MS)

Analyte(s)	CAS RN
Acephate	30560-19-1
Diisopropyl methylphosphonate (DIMP)	1445-75-6

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Analyte(s)	CAS RN
Methamidophos	10265-92-6

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Direct injection
Determinative Technique: Liquid Chromatography Tandem Mass Spectrometry (LC-MS-MS)

Method Developed for: Acephate, DIMP and methamidophos in drinking water samples
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.
Detection and Quantitation: The MDLs for acephate, DIMP, and methamidophos in reagent water were calculated to be 0.019, 0.014 and 0.017 µg/L, respectively. The Lowest Common Minimum Reporting Levels (LCMRLs) in reagent water were calculated to be 0.044, 0.022 and 0.032 µg/L, respectively

Description of Method: A 40-mL water sample is collected in a bottle containing sodium omadine and ammonium acetate. An aliquot of the sample is placed in an autosampler vial and internal standards are added. A 50-µL or larger injection is made into a liquid chromatograph (LC) equipped with a C18 column that is interfaced to an MS-MS operated in the electrospray ionization (ESI) mode. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC-MS-MS conditions. The concentration of each analyte is determined by internal standard calibration using procedural standards.

Source: EPA. 2009. "Method 538: Determination of Selected Organic Contaminants in Drinking Water by Direct Aqueous Injection-Liquid Chromatography/Tandem Mass Spectrometry (DAI-LC/MS/MS)," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-538.pdf

5.2.11 EPA Method 549.2: Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and High Performance Liquid Chromatography with Ultraviolet Detection

Analyte(s)	CAS RN
Paraquat	4685-14-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: LSE or SPE
Determinative Technique: HPLC-UV

Method Developed for: Diquat and paraquat in drinking water sources and finished drinking water
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples.
Detection and Quantitation: MDL for paraquat is 0.68 µg/L. The analytical range depends on the sample matrix and the instrumentation used.

Description of Method: A 250-mL sample is extracted using a C_8 LSE cartridge or a C_8 disk that has been specially prepared for the reversed-phase, ion-pair mode. The LSE disk or cartridge is eluted with acidic aqueous solvent to yield the eluate/extract. An ion-pair reagent is added to the eluate/extract. The concentrations of paraquat in the eluate/extract are measured using a HPLC system equipped with an ultraviolet (UV) absorbance detector. A photodiode array detector is used to provide simultaneous detection and confirmation of the method analytes.

Source: EPA. 1997. "Method 549.2: Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and High Performance Liquid Chromatography with Ultraviolet Detection," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-549.2.pdf

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5.2.12 EPA Method 551.1: Determination of Chlorination Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides in Drinking Water by Liquid-Liquid Extraction and Gas Chromatography with Electron-Capture Detection

Analyte(s)	CAS RN
Chloropicrin	76-06-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent extraction
Determinative Technique: Gas chromatography-electron capture detector (GC-ECD)

Method Developed for: Chlorination disinfection byproducts, chlorinated solvents, and halogenated pesticides/herbicides in finished drinking water, drinking water during intermediate stages of treatment, and raw source water
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples.
Detection and Quantitation: The estimated detection limit (EDL) using MTBE and ammonium chloride-preserved reagent water on a 100% dimethylpolysiloxane (DB-1) column has been found to be 0.014 µg/L.

Description of Method: This is a GC-ECD method applicable to the determination of halogenated analytes in finished drinking water, drinking water during intermediate stages of treatment, and raw source water. A 50-mL sample aliquot is extracted with 3 mL of methyl *tert*-butyl ether (MTBE) or 5 mL of pentane. Two µL of the extract is then injected into a GC equipped with a fused silica capillary column and linearized ECD for separation and analysis. This liquid/liquid extraction technique efficiently extracts a wide boiling range of non-polar and polar organic components of the sample. Thus, confirmation is quite important, particularly at lower analyte concentrations. A confirmatory column is suggested for this purpose.

Special Considerations: The presence of chloropicrin should be confirmed by either a secondary GC column or by an MS.

Source: EPA. 1995. "Method 551.1: Determination of Chlorination Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides in Drinking Water by Liquid-Liquid Extraction and Gas Chromatography with Electron-Capture Detection," Revision 1.0.
http://www.epa.gov/sam/pdfs/EPA-551.1.pdf

5.2.13 EPA Method 556.1: Determination of Carbonyl Compounds in Drinking Water by Fast Gas Chromatography

Analyte(s)	CAS RN
Formaldehyde	50-00-0

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Liquid-liquid extraction with hexane
Determinative Technique: Fast gas chromatography with electron capture detection (FGC-ECD)

Method Developed for: Formaldehyde in drinking water samples
Method Selected for: SAM lists this method for preparation and analysis of drinking water samples.

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Detection and Quantitation: MDLs for formaldehyde in reagent water were calculated as 0.09 and 0.08 µg/L for primary and secondary columns, respectively. The applicable concentration range is approximately 5 to 40 µg/L.

Description of Method: A 20-mL volume of water sample is adjusted to pH 4 with potassium hydrogen phthalate (KHP) and the analytes are derivatized at 35ºC for 2 hours with 15 mg of O-(2,3,4,5,6-pentafluorobenzyl)-hydroxylamine (PFBHA) reagent. The oxime derivatives are extracted from the water with 4 mL of hexane. The extract is processed through an acidic wash step, and analyzed by FGC-ECD. The target analytes are identified and quantified by comparison to a procedural standard. Two chromatographic peaks will be observed for many of the target analytes. Both (E) and (Z) isomers are formed for carbonyl compounds that are asymmetrical, and that are not sterically hindered. The (E) and (Z) isomers may not be chromatographically resolved in a few cases. Compounds with two carbonyl groups, such as glyoxal and methyl glyoxal, can produce even more isomers. Chromatographic peaks used for analyte identification are provided in Section 17, Table 1 and Figure 1 of the method.

Special Considerations: All results should be confirmed on a second, dissimilar capillary GC column.

Source: EPA. 1999. "Method 556.1: Determination of Carbonyl Compounds in Drinking Water by Fast Gas Chromatography," Revision 1.0. http://www.epa.gov/sam/pdfs/EPA-556.1.pdf

5.2.14 EPA Method 3050B (SW-846): Acid Digestion of Sediments, Sludges, and Soils

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Titanium tetrachloride (analyze as total titanium)	7550-45-0
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Sample preparation
Sample Preparation Technique: Acid digestion
Determinative Technique: ICP-AES / ICP-MS
Determinative Method: EPA SW-846 Method 6010C or Method 6020A. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Metals in sediments, sludges, and soil samples
Method Selected for: SAM lists this method for preparation of solid samples.

Description of Method: This method is used to prepare samples for the determination of arsenic trioxide, arsine, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic; thallium sulfate as total thallium; titanium tetrachloride as titanium; osmium tetroxide as

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osmium; and ammonium metavanadate and vanadium pentoxide as total vanadium. A 1-g to 2-g sample is digested with nitric acid and hydrogen peroxide. Sample volumes are reduced, then brought up to a final volume of 100 mL. Samples are analyzed for total arsenic, total thallium, total titanium, or total vanadium by Method 6010C or 6020A (SW-846); use Method 6010C (SW-846) for total osmium; use Method 7010 (SW-846) for arsine.

Special Considerations: Concerns have been raised regarding the use of nitric acid when analyzing samples for osmium tetroxide; hydrochloric acid should be considered and evaluated as a possible alternative.

Source: EPA. 1996. "Method 3050B (SW-846): Acid Digestion of Sediments, Sludges, and Soils," Revision 2. http://www.epa.gov/sam/pdfs/EPA-3050b.pdf

5.2.15 EPA Method 3520C (SW-846): Continuous Liquid-Liquid Extraction

Analyte(s)	CAS RN
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
BZ [Quinuclidinyl benzilate]	6581-06-2
Carfentanil	59708-52-0
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Cyclohexyl sarin (GF)	329-99-7
Diesel range organics	NA
Diphacinone	82-66-6
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Methyl hydrazine	60-34-4
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Paraoxon	311-45-5
Parathion	56-38-2
Phosphamidon	13171-21-6
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Tetramethylenedisulfotetramine	80-12-6
Thiofanox	39196-18-4
Triethanolamine (TEA)	102-71-6
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5

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Analyte(s)	CAS RN
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5

Analysis Purpose: Sample preparation
Sample Preparation Technique: Continuous liquid-liquid extraction (CLLE)
Determinative Technique: Gas chromatography-flame ionization detector (GC-FID) / GC-MS / HPLC
Determinative Method: EPA SW-846 Method 8015C, Method 8270D, or Method 8321B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Organic compounds in aqueous samples
Method Selected for: SAM lists this method for preparation of aqueous liquid and/or drinking water samples. *Please note*: Drinking water samples for fenamiphos should be prepared and analyzed by EPA Method 525.2; drinking water samples for thiofanox should be prepared and analyzed by EPA Method 531.2; aqueous/liquid samples for bromadiolone should be analyzed using ASTM D7600-09; aqueous liquid samples for EDEA, MDEA, and TEA should be analyzed using ASTM D7599-09. All other drinking water and aqueous liquid samples should be prepared using this method (SW-846 Method 3520C).

Description of Method: This method is applicable to the isolation and concentration of water-insoluble and slightly soluble organics in preparation for a variety of chromatographic procedures. A measured volume of sample, usually 1 L, is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH and extracted with organic solvent for 18 to 24 hours. The extract is filtered through sodium sulfate to remove residual moisture, concentrated, and exchanged as necessary into a solvent compatible with the cleanup or determinative procedure used for analysis.

Special Considerations: Some of the target compounds will hydrolyze in water, with hydrolysis rates dependant on various factors such as sample pH and temperature. For more information on the preparation and analysis of thiofanox, see application note:
http://www.pickeringlabs.com/catalog/pdfs/MA112%20expanded%20Carbamates.pdf

Source: EPA. 1996. "Method 3520C (SW-846): Continuous Liquid-Liquid Extraction," Revision 3.
http://www.epa.gov/sam/pdfs/EPA-3520c.pdf

5.2.16 EPA Method 3535A (SW-846): Solid-Phase Extraction

Analyte(s)	CAS RN
4-Aminopyridine	504-24-5
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
BZ [Quinuclidinyl benzilate]	6581-06-2
Carfentanil	59708-52-0
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2

OUTDATED

Analyte(s)	CAS RN
Diesel range organics	NA
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid	33876-51-6
Diphacinone	82-66-6
1,4-Dithiane	505-29-3
EA2192 [Diisopropylaminoethyl methylthiolophosphonate]	73207-98-4
Ethyl methylphosphonic acid (EMPA)	1832-53-7
Ethyldichloroarsine (ED)	598-14-1
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
Hexamethylenetriperoxidediamine (HMTD)	283-66-9
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Nicotine compounds	54-11-5
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Paraoxon	311-45-5
Parathion	56-38-2
Pentaerythritol tetranitrate (PETN)	78-11-5
Phencyclidine	77-10-1
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[1]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[1]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
1,4-Thioxane	15980-15-1
Triethanolamine (TEA)	102-71-6

OUTDATED

Analyte(s)	CAS RN
Trimethyl phosphite	121-45-9
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation
Sample Preparation Technique: SPE
Determinative Technique: GC-FID / GC-MS / HPLC
Determinative Method: EPA SW-846 Method 8015C, Method 8270D, Method 8321B, or Method 8330B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Organic compounds in ground water, wastewater, and Toxicity Characteristic Leaching Procedure (TCLP, Method 1311) leachates
Method Selected for: SAM lists this method for preparation of aqueous liquid and/or drinking water samples. *Please note*: Drinking water samples for dichlorvos, fenamiphos, and mevinphos should be prepared and analyzed by EPA Method 525.2; drinking water samples for thiofanox should be prepared and analyzed by EPA Method 531.2; aqueous liquid samples for EMPA, IMPA, MPA, and PMPA should be prepared and analyzed using ASTM D7597-09; aqueous liquid samples for EDEA, MDEA and TEA should be prepared and analyzed using ASTM D7599-09; aqueous liquid samples for bromadiolone should be prepared and analyzed using ASTM 7600-09; aqueous liquid samples for thiodiglycol should be prepared and analyzed using ASTM 7598-09. All other drinking water samples and all aqueous liquid samples should be prepared using this method (SW-846 Method 3535A).

Description of Method: This method describes a procedure for isolating target organic analytes from aqueous and liquid samples using SPE media. Sample preparation procedures vary by analyte group. Following any necessary pH adjustment, a measured volume of sample is extracted by passing it through the SPE medium (disks or cartridges), which is held in an extraction device designed for vacuum filtration of the sample. Target analytes are eluted from the solid-phase media using an appropriate solvent which is collected in a receiving vessel. The resulting solvent extract is dried using sodium sulfate and concentrated, as needed.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. Tetramethylenedisulfotetramine may require SPE extraction using acetone or methyl ethylketone. Water samples that contain a high level of particulates or a large amount of humic products may not be extractable by SPE. Some of the target compounds will hydrolyze in water, with hydrolysis rates dependant on various factors such as sample pH and temperature.

Source: EPA. 1998. "Method 3535A (SW-846): Solid-Phase Extraction (SPE)," Revision 1. http://www.epa.gov/sam/pdfs/EPA-3535a.pdf

OUTDATED

5.2.17 EPA Method 3541 (SW-846): Automated Soxhlet Extraction

Analyte(s)	CAS RN
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
BZ [Quinuclidinyl benzilate]	6581-06-2
Carfentanil	59708-52-0
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Diesel range organics	NA
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid	33876-51-6
Diphacinone	82-66-6
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6
1,4-Dithiane	505-29-3
EA2192 [Diisopropylaminoethyl methylthiolophosphonate]	73207-98-4
Ethyl methylphosphonic acid (EMPA)	1832-53-7
Ethyldichloroarsine (ED)	598-14-1
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Nicotine compounds	54-11-5
Paraoxon	311-45-5
Parathion	56-38-2
Phencyclidine	77-10-1

OUTDATED

Analyte(s)	CAS RN
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[1]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[1]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
1,4-Thioxane	15980-15-1
Triethanolamine (TEA)	102-71-6
Trimethyl phosphite	121-45-9
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation
Sample Preparation Technique: Automated Soxhlet extraction
Determinative Technique: GC-FID / GC-MS / HPLC
Determinative Method: EPA SW-846 Method 8015C, Method 8270D, or Method 8321B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Organic compounds in soil, sediment, sludges, and waste solids
Method Selected for: SAM lists this method for preparation of solid samples.

Description of Method: Approximately 10 g of solid sample is mixed with an equal amount of anhydrous sodium sulfate and placed in an extraction thimble or between two plugs of glass wool. After adding the appropriate surrogate amount, the sample is extracted using an appropriate solvent in an automated Soxhlet extractor. The extract is dried with sodium sulfate to remove residual moisture, concentrated and exchanged, as necessary, into a solvent compatible with the cleanup or determinative procedure for analysis.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. Some of the target compounds will hydrolyze in water, with hydrolysis rates dependant on various factors such as sample pH and temperature.

Source: EPA. 1994. "Method 3541 (SW-846): Automated Soxhlet Extraction," Revision 0. http://www.epa.gov/sam/pdfs/EPA-3541.pdf

OUTDATED

5.2.18 EPA Method 3545A (SW-846): Pressurized Fluid Extraction (PFE)

Analyte(s)	CAS RN
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
BZ [Quinuclidinyl benzilate]	6581-06-2
Carfentanil	59708-52-0
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Diesel range organics	NA
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid	33876-51-6
Diphacinone	82-66-6
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6
1,4-Dithiane	505-29-3
EA2192 [Diisopropylaminoethyl methylthiolophosphonate]	73207-98-4
Ethyl methylphosphonic acid (EMPA)	1832-53-7
Ethyldichloroarsine (ED)	598-14-1
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Nicotine compounds	54-11-5
Paraoxon	311-45-5
Parathion	56-38-2
Phencyclidine	77-10-1

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Analyte(s)	CAS RN
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[1]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[1]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
1,4-Thioxane	15980-15-1
Triethanolamine (TEA)	102-71-6
Trimethyl phosphite	121-45-9
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation
Sample Preparation Technique: Pressurized Fluid Extraction (PFE)
Determinative Technique: GC-FID / GC-MS / HPLC
Determinative Method: EPA SW-846 Method 8015C, Method 8270D, or Method 8321B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Organic compounds in soils, clays, sediments, sludges, and waste solids
Method Selected for: SAM lists this method for preparation of solid samples.
Detection and Quantitation: This method has been validated for solid matrices containing 250 to 12,500 µg/kg of semivolatile organic compounds, 250 to 2500 µg/kg of organophosphorus pesticides, 5 to 250 µg/kg of organochlorine pesticides, 50 to 5000 µg/kg of chlorinated herbicides, and 1 to 2500 ng/kg of polychlorinated dibenzo-*p*-dioxins (PCDDs) / polychlorinated dibenzofurans (PCDFs).

Description of Method: Approximately 10 to 30 g of soil sample is prepared for extraction either by air drying the sample, or by mixing the sample with anhydrous sodium sulfate or pelletized diatomaceous earth. The sample is then ground and loaded into the extraction cell. The extraction cell containing the sample is heated to the extraction temperature, pressurized with the appropriate solvent system, and extracted for 5 minutes (or as recommended by the instrument manufacturer). The extract may be concentrated, if necessary, and exchanged into a solvent compatible with the cleanup or determinative step being employed.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. Sodium sulfate can cause clogging, and air-drying or pelletized diatomaceous earth may be preferred. Phencyclidine and VX require extraction with

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5% triethylamine in ethyl acetate. Some of the target compounds will hydrolyze in water, with hydrolysis rates dependant on various factors such as sample pH and temperature.

Source: EPA. 1998. "Method 3545A (SW-846): Pressurized Fluid Extraction (PFE)," Revision 1. http://www.epa.gov/sam/pdfs/EPA-3545a.pdf

5.2.19 EPA Method 3570 (SW-846): Microscale Solvent Extraction (MSE)

Analyte(s)	CAS RN
Acrylamide	79-06-1
Acrylonitrile	107-13-1
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
4-Aminopyridine	504-24-5
BZ [Quinuclidinyl benzilate]	6581-06-2
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
Carfentanil	59708-52-0
Carbofuran (Furadan)	1563-66-2
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Diesel range organics	NA
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid	33876-51-6
Diphacinone	82-66-6
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6
1,4-Dithiane	505-29-3
EA2192 [Diisopropylaminoethyl methylthiolophosphonate]	73207-98-4
Ethyl methylphosphonic acid (EMPA)	1832-53-7
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Formaldehyde	50-00-0
Gasoline range organics	NA
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
Hexamethylenetriperoxidediamine (HMTD)	283-66-9

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Analyte(s)	CAS RN
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Methomyl	16752-77-5
Methyl acrylonitrile	126-98-7
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Mustard, sulfur / Mustard gas (HD)	505-60-2
Nicotine compounds	54-11-5
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Oxamyl	23135-22-0
Paraoxon	311-45-5
Parathion	56-38-2
Pentaerythritol tetranitrate (PETN)	78-11-5
Phencyclidine	77-10-1
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[1]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[1]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Sarin (GB)	107-44-8
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
1,4-Thioxane	15980-15-1
Triethanolamine (TEA)	102-71-6
Trimethyl phosphite	121-45-9
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5

OUTDATED

Analyte(s)	CAS RN
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9
White phosphorus	12185-10-3

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation
Sample Preparation Technique: MSE
Determinative Technique: Gas chromatography – nitrogen-phosphorus detector (GC-NPD) / GC-FID / GC-MS / HPLC
Determinative Method: EPA SW-846 Methods 7580, 8015C, 8270D, 8315A, 8316, 8318A, 8321B, and 8330B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Extracting volatile, semivolatile, and nonvolatile organic compounds from solids such as soils, sludges, and wastes
Method Selected for: SAM lists this method for preparation of wipe samples.

Description of Method: Samples are prepared by shake extraction with an organic solvent in sealed extraction tubes. Careful manipulation of the sample, solvent, drying agent, and spiking solutions during the procedure minimizes loss of volatile compounds while maximizing extraction of volatile, semivolatile, and nonvolatile compounds. Sample extracts are collected, dried, and concentrated using a modification of the Kuderna-Danish concentration method or other appropriate concentration technique. By increasing the number of theoretical plates and reducing the distillation temperature, extracts are concentrated without loss of volatile constituents. Samples should be prepared one at a time to the point of solvent addition (i.e., do not pre-weigh a number of samples then add the solvent). Samples should be extracted as soon after collection as possible, and exposure to air before sample extraction is minimized as much as possible.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes.

Source: EPA. 2002. "Method 3570 (SW-846): Microscale Solvent Extraction (MSE)," Revision 0. http://www.epa.gov/sam/pdfs/EPA-3570.pdf

5.2.20 EPA Method 3571 (SW-846): Extraction of Solid and Aqueous Samples for Chemical Agents

Analyte(s)	CAS RN
Mustard, sulfur / Mustard gas (HD)	505-60-2
Sarin (GB)	107-44-8
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9

Analysis Purpose: Sample preparation
Sample Preparation Technique: MSE
Determinative Technique: GC-MS
Determinative Method: EPA SW-846 Method 8270D

OUTDATED

Method Developed for: HD, GB, and VX in concrete, charcoal, wood, water, brine, ash, coral, sand, and soil
Method Selected for: SAM lists this method for preparation of solid, aqueous liquid, and drinking water samples.

Description of Method: This method provides procedures for sample collection and extraction of the referenced compounds from solids and aqueous samples. A separate extract is required for each agent to be measured. Glacial acetic acid is added as a preservative to samples being assayed for GB and glacial acetic acid/sodium chloride is a preservative for samples assayed for HD. No preservative is added for VX. Samples are extracted with 10% isopropanol in dichloromethane by vortex mixing and filtered, if necessary. An optional water wash is included for VX that back-extracts the compound from heavy organics that could interfere with the assay. An optional column cleanup procedure is described to separate GB from heavy organics, if needed. Solvents are used to elute the extract first through the Carboprep90 column, then the silica column.

Source: EPA. 2007. "Method 3571 (SW-846): Extraction of Solid and Aqueous Samples for Chemical Agents," Revision 0. http://www.epa.gov/sam/pdfs/EPA-3571.pdf

5.2.21 EPA Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples

Analyte(s)	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
Ethylene oxide	75-21-8
2-Fluoroethanol	371-62-0
Gasoline range organics	NA
Kerosene	64742-81-0
Propylene oxide	75-56-9
The following analytes should be prepared by this method (and determined by the corresponding SW-846 Method 8260C) **only** if problems (e.g., insufficient recovery, interferences) occur when using the sample preparation/determinative techniques identified for these analytes in Appendix A.	
1,4-Thioxane	15980-15-1

Analysis Purpose: Sample preparation
Sample Preparation Technique: Purge-and-trap
Determinative Technique: GC-FID / GC-MS
Determinative Method: EPA SW-846 Method 8015C or Method 8260C. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: VOCs in aqueous and water miscible liquid samples
Method Selected for: SAM lists this method for preparation of aqueous liquid and/or drinking water samples. For carbon disulfide and 1,2-dichloroethane, EPA Method 524.2 (rather than Method 5030C) should be used for preparation of drinking water samples.

Description of Method: This method describes a purge-and-trap procedure for the analysis of VOCs in aqueous liquid samples and water miscible liquid samples. An inert gas is bubbled through a portion of the aqueous liquid sample at ambient temperature, and the volatile components are transferred from the aqueous liquid phase to the vapor phase. The vapor is swept through a sorbent column where the volatile

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components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a GC column.

Special Considerations: Heated purge may be required for poor-purging analytes.

Source: EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples, Revision 3. http://www.epa.gov/sam/pdfs/EPA-5030c.pdf

5.2.22 EPA Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

Analyte(s)	CAS RN
Acrylonitrile	107-13-1
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
Ethylene oxide	75-21-8
2-Fluoroethanol	371-62-0
Gasoline range organics	NA
Kerosene	64742-81-0
Methyl acrylonitrile	126-98-7
Propylene oxide	75-56-9
The following analytes should be prepared by this method (and determined by the corresponding SW-846 Method 8260C) **only** if problems (e.g., insufficient recovery, interferences) occur when using the sample preparation/determinative techniques identified for these analytes in Appendix A.	
1,4-Thioxane	15980-15-1

Analysis Purpose: Sample preparation
Sample Preparation Technique: Purge-and-trap
Determinative Technique: GC-FID / GC-MS
Determinative Method: EPA SW-846 Method 8015C or Method 8260C. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: VOCs in solid materials (e.g., soils, sediments, and solid waste) and oily wastes
Method Selected for: SAM lists this method for preparation of solid samples.

Description of Method: This method describes a closed-system purge-and-trap process for analysis of VOCs in solid samples containing low levels (0.5 to 200 µg/kg) of VOCs. The method also provides specific procedures for preparation of samples containing high levels (>200 µg/kg) of VOCs. For low-level VOCs, a 5-g sample is collected into a vial that is placed into an autosampler device. Reagent water, surrogates, and internal standards are added automatically, and the vial is heated to 40°C. The volatiles are purged into an appropriate trap using an inert gas combined with sample agitation. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a GC for analysis. For high-level VOCs, samples are either collected into a vial that contains a water-miscible organic solvent or a portion of sample is removed from the vial and dispersed in a water-miscible solvent. An aliquot of the solvent is added to reagent water, along with surrogates and internal standards, then purged and analyzed using an appropriate determinative method (e.g., Method 8015C or 8260C (SW-846)).

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Source: EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. http://www.epa.gov/sam/pdfs/EPA-5035a.pdf

5.2.23 EPA Method 6010C (SW-846): Inductively Coupled Plasma - Atomic Emission Spectrometry

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Titanium tetrachloride (analyze as total titanium)	7550-45-0
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Analyte determination and measurement
Determinative Technique: ICP-AES
Sample Preparation Method: EPA SW-846 Method 3050B (solid samples) and NIOSH Method 9102 (wipe samples)
Sample Preparation Technique: Acid digestion

Method Developed for: Trace elements in solution
Method Selected for: SAM lists this method for analysis of solid and wipe samples.
Detection and Quantitation: Detection limits vary with each analyte. Estimated instrument detection limits (IDLs) for arsenic and titanium are 30 µg/L and 5.0 µg/L, respectively. The upper end of the analytical range may be extended by sample dilution.

Description of Method: This method determines arsenic trioxide, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic; osmium tetroxide as osmium; thallium sulfate as thallium; titanium tetrachloride as titanium; and ammonium metavanadate and vanadium pentoxide as total vanadium. Soil samples (prepared using SW-846 Method 3050B) and wipe samples (prepared using NIOSH Method 9102) are analyzed by ICP-AES.

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques. Users should consult with the appropriate point of contact listed in Section 4.0 regarding use of GFAA as a back-up or for additional confirmatory analyses.

Source: EPA. 2007. "Method 6010C (SW-846): Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 3. http://www.epa.gov/sam/pdfs/EPA-6010c.pdf

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5.2.24 EPA Method 6020A (SW-846): Inductively Coupled Plasma - Mass Spectrometry

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Titanium tetrachloride (analyze as total titanium)	7550-45-0
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Analyte determination and measurement
Determinative Technique: ICP-MS
Sample Preparation Method: EPA SW-846 Method 3050B (solid samples) and NIOSH Method 9102 (wipe samples)
Sample Preparation Technique: Acid digestion

Method Developed for: Elements in water samples and in waste extracts or digests
Method Selected for: SAM lists this method for analysis of solid and wipe samples.
Detection and Quantitation: In relatively simple sample types, detection limits will generally be below 0.1 µg/L. Less sensitive elements, such as arsenic, may have detection limits of 1.0 µg/L or higher. The upper end of the analytical range may be extended by sample dilution.

Description of Method: This method will determine arsenic trioxide, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic. The method also will determine thallium sulfate as total thallium, titanium tetrachloride as titanium, and ammonium metavanadate and vanadium pentoxide as total vanadium. Soil samples (prepared using SW-846 Method 3050B) and wipe samples (prepared using NIOSH Method 9102) are analyzed by ICP-MS. IDLs, sensitivities, and linear ranges vary with sample type, instrumentation, and operation conditions.

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques. Users should consult with the appropriate point of contact listed in Section 4.0 regarding use of GFAA as a back-up or for additional confirmatory analyses.

Source: EPA. 1998. "Method 6020A (SW-846): Inductively Coupled Plasma-Mass Spectrometry," Revision 1. http://www.epa.gov/sam/pdfs/EPA-6020a.pdf

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5.2.25 EPA Method 7470A (SW-846): Mercury in Liquid Wastes (Manual Cold-Vapor Technique)

Analyte(s)	CAS RN
Mercuric chloride (analyze as total mercury)	7487-94-7
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Acid digestion (solid and aqueous liquid samples) and acid digestion by NIOSH Method 9102 (wipe samples)
Determinative Technique: CVAA

Method Developed for: Mercury in mobility-procedure extracts, aqueous wastes, and ground waters
Method Selected for: SAM lists this method for use if problems occur when using EPA SW-846 Method 7473 for these analytes during preparation and analysis of aqueous liquid samples. (See Footnote 12 of Appendix A.)
Detection and Quantitation: The detection limit for the method is 0.2 µg/L.

Description of Method: A 100-mL aqueous sample is digested with acids, permanganate solution, persulfate solution, and heat. The sample is cooled and reduced with hydroxylamine-sodium chloride solution. Just prior to analysis, the sample is treated with Sn(II), reducing the mercury to Hg(0). The reduced sample is sparged and the mercury vapor is analyzed by CVAA.

Special Considerations: Chloride and copper are potential interferences.

Source: EPA. 1994. "Method 7470A (SW-846): Mercury in Liquid Waste (Manual Cold-Vapor Technique)," Revision 1. http://www.epa.gov/sam/pdfs/EPA-7470a.pdf

5.2.26 EPA Method 7471B (SW-846): Mercury in Solid or Semisolid Wastes (Manual Cold-Vapor Technique)

Analyte(s)	CAS RN
Mercuric chloride (analyze as total mercury)	7487-94-7
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Acid digestion (solid and aqueous liquid samples) and acid digestion by NIOSH Method 9102 (wipe samples)
Determinative Technique: CVAA

Method Developed for: Total mercury in soils, sediments, bottom deposits, and sludge-type materials
Method Selected for: SAM lists this method for use if problems occur when using EPA SW-846 Method 7473 for these analytes during preparation and analysis of solid and wipe samples. (See Footnote 12 of Appendix A.)

Description of Method: A 0.5-g to 0.6-g sample is digested with aqua regia, permanganate solution, and heat. The sample is cooled and reduced with hydroxylamine-sodium chloride solution. Just prior to analysis, the sample is treated with Sn(II), reducing the mercury to Hg(0). The reduced sample is sparged and the mercury vapor is analyzed by CVAA.

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Special Considerations: Chloride and copper are potential interferences.

Source: EPA. 1998. "Method 7471B (SW-846): Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)," Revision 2. http://www.epa.gov/sam/pdfs/EPA-7471b.pdf

5.2.27 EPA Method 7473 (SW-846): Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry

Analyte(s)	CAS RN
Mercuric chloride (analyze as total mercury)	7487-94-7
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Thermal decomposition (solid and aqueous liquid samples) and acid digestion by NIOSH Method 9102 (wipe samples)
Determinative Technique: Visible spectrophotometry

Method Developed for: Total mercury in solids, aqueous samples, and digested solutions
Method Selected for: SAM lists this method for preparation and analysis of solid, aqueous liquid, and wipe samples.
Detection and Quantitation: The IDL is 0.01 ng total mercury. The typical working range for this method is 0.05 to 600 ng.

Description of Method: Controlled heating in an oxygenated decomposition furnace is used to liberate mercury from solid and aqueous samples. The sample is dried and then thermally and chemically decomposed within the furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace, where oxidation is completed and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

Special Considerations: If equipment is not available, use CVAA Methods 7471B (EPA SW-846) for solid samples and 7470A (EPA SW-846) for aqueous liquid samples.

Source: EPA. 1998. "Method 7473 (SW-846): Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry," Revision 0. http://www.epa.gov/sam/pdfs/EPA-7473.pdf

5.2.28 EPA Method 7580 (SW-846): White Phosphorus (P_4) by Solvent Extraction and Gas Chromatography

Analyte(s)	CAS RN
White phosphorus	12185-10-3

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Solvent extraction (solid, aqueous liquid, and drinking water samples) and MSE / solvent extraction by EPA SW-846 Method 3570/8290A Appendix A (wipe samples)

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Determinative Technique: GC-NPD

Method Developed for: White phosphorus in soil, sediment, and water
Method Selected for: SAM lists this method for preparation and analysis of solid, aqueous liquid, drinking water, and wipe samples.
Detection and Quantitation: MDLs for reagent water, well water, and pond water were calculated to be 0.008, 0.009, 0.008 µg/L, respectively. MDLs for sand, a sandy loam soil (Lebanon soil), and soil from the Rocky Mountain Arsenal (U.S. Army Environmental Center soil) were calculated to be 0.02, 0.43, 0.07 µg/kg, respectively. This procedure provides sensitivity on the order of 0.01 µg/L for water samples and 1 µg/kg for soil samples.

Description of Method: Method 7580 may be used to determine the concentration of white phosphorus in soil, sediment, and water samples using solvent extraction and GC. Water samples are extracted by one of two procedures, depending on the sensitivity required. For the more sensitive procedure, a 500-mL water sample is extracted with 50 mL of diethyl ether. The extract is concentrated by back extraction with reagent water, yielding a final extract volume of approximately 1.0 mL. A 1.0 µL aliquot of this extract is injected into a GC equipped with a nitrogen-phosphorus detector (NPD). Wet soil or sediment samples are analyzed by extracting a 40 g wet-weight aliquot of the sample with a mixture of 10.0 mL degassed reagent water and 10.0 mL isooctane. The extraction is performed in a glass jar on a platform shaker for 18 hours. A 1.0 µL aliquot of the extract is analyzed by GC-NPD.

Special Considerations: The presence of white phosphorus should be confirmed by either a secondary GC column or by an MS.

Source: EPA. 1996. "Method 7580 (SW-846): White Phosphorus (P_4) by Solvent Extraction and Gas Chromatography," Revision 0. http://www.epa.gov/sam/pdfs/EPA-7580.pdf

5.2.29 EPA Method 8015C (SW-846): Nonhalogenated Organics Using GC/FID

Analyte(s)	CAS RN
Diesel range organics	NA
Gasoline range organics	NA
Kerosene	64742-81-0

Analysis Purpose: Analyte determination and measurement
Determinative Technique: GC-FID
Sample Preparation Method: EPA SW-846 Method 3541/3545A or Method 5035A (solid samples), Method 3535A or 5030C (aqueous liquid and drinking water samples), and Method 3570/8290A Appendix A (wipe samples). Refer to Appendix A for which of these preparation methods should be used for a particular analyte/sample type combination.
Sample Preparation Technique: Automated Soxhlet extraction / PFE / purge-and-trap (solid samples), SPE / purge-and-trap (aqueous liquid and drinking water samples), and MSE / solvent extraction (wipe samples).

Method Developed for: Various nonhalogenated VOCs and semivolatile organic compounds in water samples
Method Selected for: SAM lists this method for analysis of solid, aqueous liquid, drinking water, and wipe samples.
Detection and Quantitation: The estimated MDLs vary with each analyte and range between 2 and 48 µg/L for aqueous liquid samples. The MDLs in other matrices have not been evaluated. The analytical range depends on the target analyte(s) and the instrument used.

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Description of Method: This method provides GC conditions for the detection of certain nonhalogenated volatile and semivolatile organic compounds. Depending on the analytes of interest, samples may be introduced into the GC by a variety of techniques including purge-and-trap, direct injection of aqueous liquid samples, and solvent extraction. An appropriate column and temperature program are used in the GC to separate the organic compounds. Detection is achieved by a flame ionization detector (FID). The method allows the use of packed or capillary columns for the analysis and confirmation of the non-halogenated individual analytes.

Special Considerations: The presence of the analytes listed in the table above should be confirmed by either a secondary GC column or by an MS.

Source: EPA. 2000. "Method 8015C (SW-846): Nonhalogenated Organics Using GC/FID," Revision 3. http://www.epa.gov/sam/pdfs/EPA-8015c.pdf

5.2.30 EPA Method 8260C (SW-846): Volatile Organic Compounds by Gas Chromatography-Mass Spectrometry (GC/MS)

Analyte(s)	CAS RN
Acrylonitrile	107-13-1
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
Ethylene oxide	75-21-8
2-Fluoroethanol	371-62-0
Methyl acrylonitrile	126-98-7
Propylene oxide	75-56-9
The following analytes should be determined by this method (and corresponding sample preparation methods) **only** if problems (e.g., insufficient recovery, interferences) occur when using the sample preparation/determinative techniques identified for these analytes in Appendix A.	
1,4-Thioxane	15980-15-1

Analysis Purpose: Analyte determination and measurement
Determinative Technique: GC-MS
Sample Preparation Method: EPA SW-846 Method 5035A (solid samples), Method 5030C (aqueous liquid and drinking water samples), and Method 3570/8290A Appendix A (wipe samples).
Sample Preparation Technique: Purge-and-trap (solid samples, aqueous liquid, and drinking water samples) and MSE / solvent extraction (wipe samples).

Method Developed for: Applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses (emulsified oil), tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.
Method Selected for: SAM lists this method for analysis of solid, aqueous liquid, drinking water, and/or wipe samples. For acrylonitrile, carbon disulfide, 1,2-dichloroethane, and methyl acrylonitrile only, EPA Method 524.2 (rather than 8260C) should be used for analysis of drinking water samples.
Detection and Quantitation: Using standard quadrupole instrumentation and the purge-and-trap, estimated quantitation limits are 5 ug/kg (wet weight) for soil/sediment samples and 5 ug/L for ground water. Somewhat lower limits may be achieved using an ion trap MS or other instrumentation of improved design. No matter which instrument is used, estimated quantitation limits (EQLs) will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample

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size is used to avoid saturation of the detector. The EQL for an individual analyte is dependent on the instrument as well as the choice of sample preparation/introduction method.

Description of Method: Volatile compounds are introduced into a GC by purge-and-trap or other procedures (see Section 1.2 in Method 8260C). The analytes can be introduced directly to a wide-bore capillary column or cryofocused on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. Alternatively, the effluent from the trap is sent to an injection port operating in the split mode for injection to a narrow-bore capillary column. The column is temperature-programmed to separate the analytes, which are then detected with a MS interfaced to the GC. Analytes eluted from the capillary column are introduced into the MS via a jet separator or a direct connection.

Source: EPA. 2006. "Method 8260C (SW-846): Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)," Revision 3. http://www.epa.gov/sam/pdfs/EPA-8260c.pdf

5.2.31 EPA Method 8270D (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC-MS)

Analyte(s)	CAS RN
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol[1]	96-24-2
Chloropicrin[2]	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine[3]	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Dimethylphosphite	868-85-9
Disulfoton	298-04-4
Disulfoton sulfone oxon[4]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[4]	2496-92-6
1,4-Dithiane	505-29-3
Ethyldichloroarsine (ED)	598-14-1
Fenamiphos	22224-92-6
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Mustard, sulfur / Mustard gas (HD)[5]	505-60-2
Nicotine compounds	54-11-5
Paraoxon	311-45-5
Parathion	56-38-2

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Analyte(s)	CAS RN
Phencyclidine	77-10-1
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[4]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[4]	2588-05-8
Phosphamidon	13171-21-6
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Sarin (GB)[5]	107-44-8
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine[2]	80-12-6
1,4-Thioxane[6]	15980-15-1
Trimethyl phosphite[2]	121-45-9
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate][5]	50782-69-9
The following analyte should be determined by this method **only** if liquid chromatography-mass spectrometry (LC-MS) [electrospray] procedures are not available to the laboratory. Sample preparation methods should remain the same.	
BZ [Quinuclidinyl benzilate][1]	6581-06-2
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphoramidic acid[1]	33876-51-6
EA2192 [Diisopropylaminoethyl methylthiolophosphonate][1]	73207-98-4
Ethyl methylphosphonic acid (EMPA)[1]	1832-53-7
Isopropyl methylphosphonic acid (IMPA)[1]	1832-54-8
Methylphosphonic acid (MPA)[1]	993-13-5
Pinacolyl methyl phosphonic acid (PMPA)[1]	616-52-4

[1] For this analyte, SW-846 Method 8270D must be modified to include a derivatization step.
[2] If problems occur with analyses, lower the injection temperature.
[3] If problems occur when using this method, it is recommended that SW-846 Method 8321B be used. Sample preparation methods should remain the same.
[4] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354
[5] For this analyte, refer to EPA SW-846 Method 8271 for GC-MS conditions.
[6] If problems occur when using this method, it is recommended that SW-846 Method 8260C and appropriate corresponding sample preparation procedures (i.e., Method 5035A for solid samples and Method 5030C for aqueous liquid and drinking water samples) be used.

Analysis Purpose: Analyte determination and measurement
Determinative Technique: GC-MS
Sample Preparation Method: EPA SW-846 Method 3541/3545A (solid samples), Method 3520C/3535A (aqueous liquid and drinking water samples), and Method 3570/8290A Appendix A or

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NIOSH 9102 (wipe samples). Refer to Appendix A for which of these preparation methods should be used for a particular analyte/sample type combination.
Sample Preparation Technique: Automated Soxhlet extraction / PFE (solid samples), CLLE / SPE (aqueous liquid and drinking water samples), and MSE / solvent extraction / acid digestion (wipe samples).

Method Developed for: Semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples
Method Selected for: SAM lists this method for analysis of solid, aqueous liquid, drinking water, and/or wipe samples. *Please note*: drinking water samples for dichlorvos, disulfoton, disulfoton sulfoxide, fenamiphos, and mevinphos should be prepared and analyzed by EPA Method 525.2; aqueous liquid and drinking water samples for chloropicrin should be prepared and analyzed by EPA Method 551.1; all other analyte/sample type combinations should be analyzed by this method (SW-846 8270D).
Detection and Quantitation: The EDLs vary with each analyte and range between 10 and 1000 µg/L for aqueous liquid samples and 660 and 3300 µg/kg for soil samples. The analytical range depends on the target analyte(s) and the instrument used.

Description of Method: Samples are prepared for analysis by GC-MS using the appropriate sample preparation and, if necessary, sample cleanup procedures. Semivolatile compounds are introduced into the GC-MS by injecting the sample extract into a GC with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a MS connected to the GC. Analytes eluted from the capillary column are introduced into the MS. For the determination of BZ, 3-chloro-1,2-propanediol, dimethylphosphoramidic acid, EA2192, EMPA, IMPA, MPA, and PMPA, a derivatization step is required prior to injection into the GC-MS. The phosphonic acids require derivatization with a trimethylsilyl agent and 3-chloro-1,2-propanediol requires derivatization with a heptafluorobutyryl agent.

Special Considerations: Refer to footnotes provided in analyte table above for special considerations that should be applied when measuring specific analytes. Procedures for derivatization are described in the following references:

Black et al. 1994. "Application of gas chromatography-mass spectrometry and gas chromatography-tandem mass spectrometry to the analysis of chemical warfare samples, found to contain residues of the nerve agent sarin, sulphur mustard and their degradation products." Journal of Chromatography A. 662(2): 301–321. http://www.sciencedirect.com/science/journal/00219673

Brereton, P., Kelly, J., Crews, C., Honour, S., and Wood, R. 2001. "Determination of 3-Chloro-1,2-Propanediol in Foods and Food Ingredients by Gas Chromatography with Mass Spectrometric Detection: Collaborative Study." Journal of AOAC International. 84(2): 455–465. http://www.atypon-link.com/AOAC/doi/abs/10.5555/jaoi.2001.84.2.455

Divinova, V., Svejkovska, B., Dolezal, M., and Velisek, J. 2004. "Determination of Free and Bound 3-Chloropropane-1,2-diol by Gas Chromatography with Mass Spectrometric Detection using Deuterated 3-Chloropropane-1,2-diol as Internal Standard." Czech Journal of Food Sciences. 22(5): 182–189. http://www.epa.gov/sam/pdfs/Czech_J_Food_Sci-22(5)_pg182-189.pdf

Retho, C., and Blanchard, F. 2005. "Determination of 3-chloropropane-1,2-diol as its 1,3-dioxolane derivative at the µg kg-1 level: Application to a wide range of foods." Food Additives & Contaminants: Part A Chemistry, Analysis, Control, Exposure & Risk Assessment. 22(12): 1189–1197. http://www.informaworld.com/smpp/content~db=all~content=a727751832

White et al. 1992. "Determination of 3-Quinuclidinyl Benzilate (QNB) and Its Major Methoabolites in Urine by Isotope Dilution Gas Chromatography/Mass Spectrometry." Journal of Analytical Toxicology. 16: 182–187. http://www.jatox.com/shop/shopexd.asp?id=4062

OUTDATED

Source: EPA. 1998. "Method 8270D (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)," Revision 4. http://www.epa.gov/sam/pdfs/EPA-8270d.pdf

5.2.32 EPA Method 8290A, Appendix A (SW-846): Procedure for the Collection, Handling, Analysis, and Reporting of Wipe Tests Performed within the Laboratory

Analyte(s)	CAS RN
Acrylamide	79-06-1
Acrylonitrile	107-13-1
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
4-Aminopyridine	504-24-5
BZ [Quinuclidinyl benzilate]	6581-06-2
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
Carfentanil	59708-52-0
Carbofuran (Furadan)	1563-66-2
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol	96-24-2
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Crimidine	535-89-7
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Diesel range organics	NA
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid	33876-51-6
Diphacinone	82-66-6
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6
1,4-Dithiane	505-29-3
EA2192 [Diisopropylaminoethyl methylthiolophosphonate]	73207-98-4
Ethyl methylphosphonic acid (EMPA)	1832-53-7
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Fentanyl	437-38-7
Formaldehyde	50-00-0
Gasoline range organics	NA
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
Hexamethylenetriperoxidediamine (HMTD)	283-66-9

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Analyte(s)	CAS RN
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Methomyl	16752-77-5
Methyl acrylonitrile	126-98-7
Methyl hydrazine	60-34-4
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Mustard, sulfur / Mustard gas (HD)	505-60-2
Nicotine compounds	54-11-5
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Oxamyl	23135-22-0
Paraoxon	311-45-5
Parathion	56-38-2
Pentaerythritol tetranitrate (PETN)	78-11-5
Phencyclidine	77-10-1
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[1]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[1]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Sarin (GB)	107-44-8
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
1,4-Thioxane	15980-15-1
Triethanolamine (TEA)	102-71-6
Trimethyl phosphite	121-45-9
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5

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Analyte(s)	CAS RN
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9
White phosphorus	12185-10-3

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation
Sample Preparation Technique: Solvent extraction
Determinative Technique: GC-NPD / GC-FID / GC-MS / HPLC
Determinative Method: EPA OW Method 300.1 Revision 1.0; EPA SW-846 Methods 7580, 8015C, 8270D, 8315A, 8316, 8318A, 8321B, and 8330B. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Evaluation of surface contamination by 2,3,7,8-substituted PCDD and PCDF congeners
Method Selected for: SAM lists this method for preparation of wipe samples.

Description of Method: A surface area of 2 inches by 1 foot is wiped with glass fiber paper saturated with distilled-in-glass acetone. One wipe is used per designated area. Wipes are combined into a single composite sample in an extraction jar and solvent extracted using a wrist action shaker.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. The solvent systems described in this method extraction have been evaluated for PCDD and PCDF congeners only. Other analytes may require different solvent systems for optimal sample extraction.

Source: EPA. 2007. "Method 8290A, Appendix A (SW-846): Procedure for the Collection, Handling, Analysis, and Reporting of Wipe Tests Performed within the Laboratory," Revision 1. http://www.epa.gov/sam/pdfs/EPA-8290a.pdf

5.2.33 EPA Method 8315A (SW-846): Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)

Analyte(s)	CAS RN
Formaldehyde	50-00-0

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Solvent extraction (solid and aqueous liquid samples) and MSE / solvent extraction by EPA SW-846 Method 3570/8290A Appendix A (wipe samples)
Determinative Technique: HPLC

Method Developed for: Free carbonyl compounds in aqueous, soil, waste, and stack samples
Method Selected for: SAM lists this method for preparation and analysis of solid, aqueous liquid, and wipe samples.
Detection and Quantitation: The MDL for formaldehyde varies depending on sample conditions and instrumentation, but is approximately 6.2 µg/L for aqueous liquid samples.

Description of Method: A measured volume of aqueous liquid sample (approximately 100 mL), or an appropriate amount of solids extract (approximately 25 g), is buffered to pH 3 and derivatized with 2,4-

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dinitrophenylhydrazine (2,4-DNPH). Using the appropriate extraction technique, the derivatives are extracted using methylene chloride and the extracts are exchanged with acetonitrile prior to HPLC analysis. HPLC conditions are described permitting the separation and measurement of various carbonyl compounds in the extract by absorbance detection at 360 nm. If formaldehyde is the only analyte of interest, the aqueous liquid sample and/or solid sample extract should be buffered to pH 5.0 to minimize the formation of artifact formaldehyde.

Source: EPA. 1996. "Method 8315A (SW-846): Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)," Revision 1. http://www.epa.gov/sam/pdfs/EPA-8315a.pdf

5.2.34 EPA Method 8316 (SW-846): Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)

Analyte(s)	CAS RN
Acrylamide	79-06-1

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Direct injection (aqueous liquid and drinking water samples), water extraction (solid), and MSE / solvent extraction by EPA SW-846 Method 3570/8290A Appendix A (wipe samples)
Determinative Technique: HPLC

Method Developed for: Acrylamide, acrylonitrile, and acrolein in water samples
Method Selected for: SAM lists this method for preparation and/or analysis of solid, aqueous liquid, drinking water, and wipe samples.
Detection and Quantitation: Acrylamide has an MDL of 10 µg/L; acrylonitrile has an MDL of 20 µg/L.

Description of Method: Samples are analyzed by HPLC. A 200-µL aliquot is injected onto a C_{18} reverse-phase column, and compounds in the effluent are detected with a UV detector. Solid samples should be water extracted prior to injection. Aqueous liquid and drinking water samples can be directly injected.

Source: EPA. 1994. "Method 8316 (SW-846): Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)," Revision 0. http://www.epa.gov/sam/pdfs/EPA-8316.pdf

5.2.35 EPA Method 8318A (SW-846): *N*-Methylcarbamates by High Performance Liquid Chromatography (HPLC)

Analyte(s)	CAS RN
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Carbofuran (Furadan)	1563-66-2
Methomyl	16752-77-5
Oxamyl	23135-22-0

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Solvent extraction (solid samples), and MSE / solvent extraction by EPA SW-846 Method 3570/8290A Appendix A (wipe samples)
Determinative Technique: HPLC

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Method Developed for: *N*-methylcarbamates in soil, water, and waste matrices
Method Selected for: SAM lists this method for preparation and/or analysis of solid and wipe samples.
Detection and Quantitation: The estimated MDLs vary with each analyte and range from 1.7 to 9.4 µg/L for aqueous samples and 10 to 50 µg/kg for soil samples.

Description of Method: *N*-methylcarbamates are extracted from aqueous liquid samples with methylene chloride, and from soils, oily solid waste, and oils with acetonitrile. The extract solvent is exchanged to methanol/ethylene glycol, and the extract is cleaned using a C_{18} cartridge, filtered, and eluted on a C_{18} analytical column. After separation, the target analytes are hydrolyzed and derivatized post-column, then quantified fluorometrically. The sensitivity of the method usually depends on the level of interferences present, rather than on instrument conditions. Waste samples with a high level of extractable fluorescent compounds are expected to yield significantly higher detection limits.

Source: EPA. 2000. "Method 8318A (SW-846): N-Methylcarbamates by High Performance Liquid Chromatography (HPLC)," Revision 1. http://www.epa.gov/sam/pdfs/EPA-8318a.pdf

5.2.36 EPA Method 8321B (SW-846): Solvent-Extractable Nonvolatile Compounds by High Performance Liquid Chromatography-Thermospray-Mass Spectrometry (HPLC-TS-MS) or Ultraviolet (UV) Detection

Analyte(s)	CAS RN
Brodifacoum	56073-10-0
Bromadiolone	28772-56-7
BZ [Quinuclidinyl benzilate][1]	6581-06-2
Carfentanil	59708-52-0
Diisopropyl methylphosphonate (DIMP)[2]	1445-75-6
Dimethylphosphoramidic acid[1]	33876-51-6
Diphacinone	82-66-6
EA2192 [Diisopropylaminoethyl methylthiolophosphonate][1]	73207-98-4
Ethyl methylphosphonic acid (EMPA)[1]	1832-53-7
N-Ethyldiethanolamine (EDEA)	139-87-7
Fentanyl	437-38-7
Isopropyl methylphosphonic acid (IMPA)[1]	1832-54-8
N-Methyldiethanolamine (MDEA)	105-59-9
Methylphosphonic acid (MPA)[1]	993-13-5
Pinacolyl methyl phosphonic acid (PMPA)[1]	616-52-4
Thiodiglycol (TDG)	111-48-8
Thiofanox	39196-18-4
Triethanolamine (TEA)	102-71-6
The following analyte should be determined by this method **only** if problems (e.g., insufficient recovery, interferences) occur when using SW-846 Method 8270D. Sample preparation methods should remain the same as those listed in Appendix A.	
Crimidine[3]	535-89-7

[1] LC-MS (electrospray) procedures are preferred for these analytes; however, if this technique is not available to the laboratory, GC-MS procedures using derivatization based on SW-846 Method 8270D may be used. Sample preparation methods should remain the same. Both electrospray LC-MS and GC-MS derivatization procedures are currently under development.
[2] If problems occur with the analysis of DIMP using EPA SW-846 Method 8321B, use SW-846 Method 8270D.
[3] This analyte needs to be determined using a wavelength of 230 nm.

Analysis Purpose: Analyte determination and measurement

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Determinative Technique: HPLC
Sample Preparation Method: EPA SW-846 Method 3541/3545A (solid samples), 3520C/3535A (aqueous liquid and drinking water samples), and Method 3570/8290A Appendix A (wipe samples). For thiofanox, EPA Method 531.2 (rather than Method 3520C/3535A) should be used for preparation of drinking water samples. Refer to Appendix A for which of these preparation methods should be used for a particular analyte/sample type combination.
Sample Preparation Technique: Automated Soxhlet extraction / PFE (solid samples), CLLE / SPE (aqueous liquid and drinking water samples), and MSE / solvent extraction (wipe samples). For thiofanox, direct injection should be used for preparation of drinking water samples.

Method Developed for: Solvent-extractable nonvolatile compounds, including dyes, organophosphorus compounds, phenoxyacid herbicides, and carbamates in solid, and water samples
Method Selected for: SAM lists this method for analysis of solid, aqueous liquid, drinking water, and/or wipe samples. Aqueous liquid samples for DIMP, EMPA, IMPA, MPA, and PMPA should be analyzed using ASTM D7597-09; aqueous liquid samples for EDEA, MDEA, and TEA should be prepared and analyzed using ASTM D7599-09; aqueous liquid samples for bromadiolone should be prepared and analyzed using ASTM D7600-09; aqueous liquid samples for thiodiglycol should be prepared and analyzed using ASTM D7598-09. Drinking water samples for DIMP should be analyzed using EPA Method 538.

Description of Method: This method provides reversed-phase HPLC, thermospray (TSP) MS, and UV conditions for detection of the target analytes. Sample extracts can be analyzed by direct injection into the TSP or onto a LC-TSP interface. A gradient elution program is used to separate the compounds. Primary analysis may be performed by UV detection; however, positive results should be confirmed by TSP-MS. Quantitative analysis may be performed by either TSP-MS or UV detection, using either an external or internal standard approach. TSP-MS detection may be performed in either a negative ionization (discharge electrode) mode or a positive ionization mode, with a single quadrupole MS. The use of MS-MS techniques is an option. The analytical range and detection limits vary depending on the target analyte and instrument used.

Special Considerations: Refer to footnotes provided in analyte table above for special considerations that should be applied when measuring specific analytes.

Source: EPA. 1998. "Method 8321B (SW-846): Solvent-Extractable Nonvolatile Compounds by High Performance Liquid Chromatography-Thermospray-Mass Spectrometry (HPLC-TSP-MS) or Ultraviolet (UV) Detection," Revision 2. http://www.epa.gov/sam/pdfs/EPA-8321b.pdf

5.2.37 EPA Method 8330B (SW-846): Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)

Analyte(s)	CAS RN
4-Aminopyridine	504-24-5
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
Hexamethylenetriperoxidediamine (HMTD)	283-66-9
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Pentaerythritol tetranitrate (PETN)	78-11-5
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7

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Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Solvent extraction or direct injection (solid samples), SPE by EPA SW-846 Method 3535A (aqueous liquid and drinking water samples), and MSE / solvent extraction by EPA SW-846 Method 3570/8290A Appendix A (wipe samples)

Method Developed for: Trace analysis of explosives and propellant residues in water, soil, or sediment
Method Selected for: SAM lists this method for preparation and/or analysis of solid, aqueous liquid, drinking water, and wipe samples. Aqueous liquid and drinking water samples are prepared using Methods 3535A or 8330B prior to analysis.
Detection and Quantitation: The detection limits, ranges, and interferences depend on the target compound

Description of Method: This method is intended for the trace analysis of explosives and propellant residues by HPLC using a dual wavelength UV detector in a water, soil, or sediment matrix. All of the compounds listed in this method are either used in the manufacture of explosives or propellants, or they are the degradation products of compounds used for that purpose. Samples are prepared for analysis by high performance liquid chromatography – ultraviolet (HPLC-UV) using the appropriate sample preparation technique (solid phase extraction by 3535A or solvent extraction by 8330B) and, if necessary, sample cleanup procedures. Direct injection of diluted and filtered water samples can be used for water samples of higher concentration. Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered and chromatographed.

Source: EPA. 2006. "Method 8330B (SW-846): Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)," Revision 2. http://www.epa.gov/sam/pdfs/EPA-8330b.pdf

5.2.38 EPA CLP ISM01.2 Cyanide: Analytical Methods for Total Cyanide Analysis

Analyte(s)	CAS RN
Cyanide, Total	57-12-5

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Midi- or micro-distillation
Determinative Technique: Visible spectrophotometry

Method Developed for: Metals in water, sediment, sludge, and soil
Method Selected for: SAM lists this method for preparation and analysis of solid, aqueous liquid, and wipe samples.
Detection and Quantitation: The method quantitation limits are 10 µg/L for aqueous samples and 0.5 mg/kg for solid samples.

Description of Method: Cyanide as hydrocyanic acid is released from cyanide complexes by means of reflux-distillation, using either a midi- or micro-distillation process, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined spectrophotometrically. In the semiautomated spectrophotometric measurement, cyanide is converted to cyanogen chloride without hydrolyzing to the cyanate, by reaction with chloramine-T, at a pH less than 8. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent, and absorbance is read between 570 and 580 nanometers (nm). To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

Source: EPA Contract Laboratory Program (CLP). "ISM01.2: Exhibit D – Part D: Analytical Methods for Total Cyanide Analysis." http://www.epa.gov/sam/pdfs/EPA-ISM01.2.pdf

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5.2.39 EPA Region 7 RLAB Method 3135.2I: Cyanide, Total and Amenable in Aqueous and Solid Samples Automated Colorimetric with Manual Digestion

Analyte(s)	CAS RN
Cyanide, Amenable to chlorination	NA

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Acid digestion followed by distillation
Determinative Technique: Visible spectrophotometry

Method Developed for: Cyanide in drinking, ground, and surface waters, domestic and industrial waste waters, sediments and solid waste
Method Selected for: SAM lists this method for preparation and analysis of solid, aqueous liquid, drinking water, and wipe samples.
Detection and Quantitation: The applicable range is 0.003 to 0.500 mg/L cyanide in the distillate. This range can be expanded by sample dilution, either by using less sample for distillation or diluting the distillate.

Description of Method: This method detects inorganic cyanides that are present as either simple soluble salts or complex radicals. It may be used to determine values for both total cyanide and cyanide amenable to chlorination (also known as available cyanide). Cyanide in the sample released as hydrocyanic acid by refluxing the sample with strong acid. The hydrocyanic acid is distilled and collected in an absorber-scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated colorimetry. For determination of cyanide amenable to chlorination, a portion of the sample is chlorinated using sodium hypochlorite at a pH > 11 to decompose the cyanide. Cyanide levels are then determined in both the chlorinated sample portion of the sample and a portion of the sample that has not been chlorinated using the total cyanide method. Cyanides amenable to chlorination are then calculated by difference between unchlorinated and the chlorinated aliquots of the sample.

Special Considerations: Alternate cyanide analyzer equipment may be used, provided it is used according to the procedures described and the laboratory can demonstrate equivalent performance.

Source: EPA Region 7. 2008. "RLAB Method 3135.2I: Cyanide, Total and Amenable in Aqueous and Soil Samples Automated Colorimetric with Manual Digestion." http://www.epa.gov/sam/pdfs/EPA-3135.2I.pdf

5.2.40 IO [Inorganic] Compendium Method IO-3.1: Selection, Preparation, and Extraction of Filter Material

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5

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Analyte(s)	CAS RN
Thallium sulfate (analyze as total thallium)	10031-59-1
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Sample preparation
Sample Preparation Technique: Acid extraction
Determinative Technique: ICP-AES / ICP-MS
Determinative Method: EPA Method IO-3.4 or Method IO-3.5. Osmium tetroxide should be analyzed by Method IO-3.4.

Method Developed for: Particulate metals in air.
Method Selected for: SAM lists this method for preparation of air samples.
Description of Method: This method supports determination of arsenic trioxide, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic. Thallium sulfate is determined as total thallium and ammonium metavanadate and vanadium pentoxide are determined as total vanadium. A subsample (one-ninth of the overall filter) is obtained by cutting a strip from the filter used to collect the sample. The filter strip is extracted using a hydrochloric/nitric acid mix and microwave or hotplate heating. The extract is filtered, worked up to 20 mL, and analyzed using either Method IO-3.4 or Method IO-3.5.

Source: EPA. 1999. "IO Compendium Method IO-3.1: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Selection, Preparation and Extraction of Filter Material." http://www.epa.gov/sam/pdfs/EPA-IO-3.1.pdf

5.2.41 IO [Inorganic] Compendium Method IO-3.4: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma (ICP) Spectroscopy

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Analyte determination and measurement
Determinative Technique: ICP-AES
Sample Preparation Method: EPA Method IO-3.1
Sample Preparation Technique: Acid extraction

Method Developed for: Metals in ambient particulate matter
Method Selected for: SAM lists this method for analysis of air samples.

OUTDATED

Description of Method: This method determines arsenic trioxide, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic. Osmium tetroxide is determined as total osmium, thallium sulfate is determined as total thallium, and ammonium metavanadate and vanadium pentoxide are determined as total vanadium. Ambient air is sampled by high-volume filters using Method IO-2.1 (a sampling method) and the filters are extracted by Method IO-3.1. Detection limits, ranges, and interference corrections are dependent on the analyte and the instrument used.

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques. Concerns have been raised regarding the use of nitric acid when analyzing samples for osmium tetroxide; hydrochloric acid should be considered and evaluated as a possible alternative.

Source: EPA. 1999. "IO Compendium Method IO-3.4: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma (ICP) Spectroscopy." http://www.epa.gov/sam/pdfs/EPA-IO-3.4.pdf

EPA. 1999. "IO Compendium Method IO-2.1: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM_{10} Using High Volume (HV) Sampler." http://www.epa.gov/sam/pdfs/EPA-IO-2.1.pdf

5.2.42 IO [Inorganic] Compendium Method IO-3.5: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP-MS)

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Analyte determination and measurement
Determinative Technique: ICP-MS
Sample Preparation Method: EPA Method IO-3.1
Sample Preparation Technique: Acid extraction

Method Developed for: Metals in ambient particulate matter
Method Selected for: SAM lists this method for analysis of air samples.

Description of Method: This method determines arsenic trioxide, lewisite, lewisite degradation products, calcium and lead arsenate, and sodium arsenite as total arsenic. Thallium sulfate is determined as total thallium and ammonium metavanadate and vanadium pentoxide are determined as total vanadium. Ambient air is sampled by high-volume filters using Method IO-2.1 (a sampling method). The filters are extracted by Method IO-3.1. Detection limits, ranges, and interference corrections are dependent on the analyte and the instrument used.

OUTDATED

Special Considerations: Laboratory testing is currently underway for speciation of lewisite 1 using GC-MS techniques.

Source: EPA. 1999. "IO Compendium Method IO-3.5: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)." http://www.epa.gov/sam/pdfs/EPA-IO-3.5.pdf

EPA. 1999. "IO Compendium Method IO-2.1: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM_{10} Using High Volume (HV) Sampler." http://www.epa.gov/sam/pdfs/EPA-IO-2.1.pdf

5.2.43 IO [Inorganic] Compendium Method IO-5: Sampling and Analysis for Vapor and Particle Phase Mercury in Ambient Air Utilizing Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)

Analyte(s)	CAS RN
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Acid digestion for particulate mercury
Determinative Technique: Cold vapor atomic fluorescence spectrometry (CVAFS)

Method Developed for: Vapor and particle phase mercury in ambient air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limits are 30 pg/m^3 for particulate mercury and 45 pg/m^3 for vapor phase mercury. Detection limits, analytical range, and interferences are dependent on the instrument used.

Description of Method: Vapor phase mercury is collected using gold-coated glass bead traps at a flow rate of 0.3 L/min. The traps are directly desorbed onto a second (analytical) trap. The mercury desorbed from the analytical trap is determined by Atomic Fluorescence Spectrometry. Particulate mercury is sampled on glass-fiber filters at a flow rate of 30 L/min. The filters are extracted with nitric acid and microwave heating. The extract is oxidized with bromine chloride, then reduced with stannous chloride and purged from solution onto a gold-coated glass bead trap. This trap is desorbed onto a second trap, the second trap is desorbed, and the mercury is determined by CVAFS.

Special Considerations: There are no known positive interferences at 253.7 nm wavelength. Water vapor will cause a negative interference.

Source: EPA. 1999. "IO Compendium Method IO-5: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Sampling and Analysis for Vapor and Particle Phase Mercury in Ambient Air Utilizing Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)."
http://www.epa.gov/sam/pdfs/EPA-IO-5.pdf

OUTDATED

5.2.44 EPA Air Method, Toxic Organics - 10A (TO-10A): Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

Analyte(s)	CAS RN
BZ [Quinuclidinyl benzilate][1]	6581-06-2
Chlorfenvinphos	470-90-6
3-Chloro-1,2-propanediol[2]	96-24-2
Chlorosarin[2]	1445-76-7
Chlorosoman[2]	7040-57-5
Chlorpyrifos	2921-88-2
Chlorpyrifos oxon	5598-15-2
Cyclohexyl sarin (GF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Diisopropyl methylphosphonate (DIMP)[2]	1445-75-6
Dimethylphosphite	868-85-9
Dimethylphosphoramidic acid[1]	33876-51-6
EA2192 [Diisopropylaminoethyl methylthiolophosphonate][1]	73207-98-4
Ethyl methylphosphonic acid (EMPA)[1]	1832-53-7
N-Ethyldiethanolamine (EDEA)	139-87-7
Fenamiphos	22224-92-6
Isopropyl methylphosphonic acid (IMPA)[1]	1832-54-8
Methyl paraoxon	950-35-6
Methyl parathion	298-00-0
N-Methyldiethanolamine (MDEA)	105-59-9
1-Methylethyl ester ethylphosphonofluoridic acid (GE)[2]	1189-87-3
Methylphosphonic acid (MPA)[1]	993-13-5
Mevinphos	7786-34-7
Monocrotophos	6923-22-4
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1
Mustard, sulfur / Mustard gas (HD)	505-60-2
Paraoxon	311-45-5
Parathion	56-38-2
Phencyclidine	77-10-1
Phorate	298-02-2
Phorate sulfone	2588-04-7
Phorate sulfone oxon[3]	2588-06-9
Phorate sulfoxide	2588-03-6
Phorate sulfoxide oxon[3]	2588-05-8
Phosphamidon	13171-21-6
Pinacolyl methyl phosphonic acid (PMPA)[1]	616-52-4
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4
Sarin (GB)[2]	107-44-8
Soman (GD)[2]	96-64-0
Tabun (GA)	77-81-6

OUTDATED

Analyte(s)	CAS RN
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Triethanolamine (TEA)	102-71-6
Trimethyl phosphite	121-45-9
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9
The following analyte should be determined by this method **only** if problems (e.g., insufficient recovery, interferences) occur when using Method TO-15.	
Allyl alcohol	107-18-6

[1] For this analyte, HPLC is the preferred technique; however, if problems occur, Method TO-10A must be modified to include a derivatization step prior to analysis by GC-MS (see references listed under Special Considerations in Section 5.2.31).
[2] If problems occur when using this method, it is recommended that the canister Method TO-15 be used.
[3] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent extraction
Determinative Technique: GC-MS or HPLC

Method Developed for: Pesticides and polychlorinated biphenyls in ambient air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The limit of detection (LOD) will depend on the specific compounds measured, the concentration level, and the degree of specificity required. This method is applicable to multicomponent atmospheres, 0.001 to 50 µg/m^3 concentrations, and 4 to 24-hour sampling periods.

Description of Method: A low-volume (1 to 5 L/min) sample collection rate is used to collect vapors on a sorbent cartridge containing PUF in combination with another solid sorbent. Airborne particles also are collected, but the sampling efficiency is not known. Pesticides and other chemicals are extracted from the sorbent cartridge with 5% diethyl ether in hexane and determined by GC-MS. For common pesticides, HPLC coupled with a UV detector or electrochemical detector is preferable. If analyzed by GC-MS, BZ, dimethylphosphoramidic acid, EA2192, EMPA, IMPA, MPA, and PMPA require derivatization with a trimethylsilyl agent prior to injection into the GC.

Special Considerations: Refer to footnotes provided in analyte table above for special considerations that should be applied when measuring specific analytes. See Special Considerations for EPA SW-846 8270D in Section 5.2.31 for information regarding derivatization of compounds.

Source: EPA. 1999. "Air Method, Toxic Organics-10A (TO-10A): Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)." http://www.epa.gov/sam/pdfs/EPA-TO-10a.pdf

OUTDATED

5.2.45 EPA Air Method, Toxic Organics - 15 (TO-15): Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

Analyte(s)	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
Ethyldichloroarsine (ED)	598-14-1
Ethylene oxide	75-21-8
The following analytes should be determined by this method **only** if problems (e.g., insufficient recovery, interferences) occur when using Method TO-10A.	
3-Chloro-1,2-propanediol	96-24-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Diisopropyl methylphosphonate (DIMP)	1445-75-6
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3
Sarin (GB)	107-44-8
Soman (GD)	96-64-0

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Samples are collected using canisters.
Determinative Technique: GC-MS

Method Developed for: VOCs in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: This method applies to ambient concentrations of VOCs above 0.5 ppbv and typically requires VOC enrichment by concentrating up to 1 L of a sample volume; however, when using current technologies, quantifications of approximately 100 pptv have been achieved with 0.5-L sample volumes.

Description of Method: The atmosphere is sampled by introduction of air into a specially prepared stainless steel canister (electropolished or silica-coated). A sample of air is drawn through a sampling train comprising components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister. Grab samples also may be collected. After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to the laboratory for analysis. To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. Recovery of less volatile compounds may require heating the canister.

After the concentration and drying steps are completed, VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a cryo-focusing (ultra-low temperature) trap or small volume multisorbent trap. The sample is then released by thermal desorption and analyzed by GC-MS.

Special Considerations: If problems occur when using this method for determination of allyl alcohol, it is recommended that Method TO-10A be used. In cases where lower detection levels are needed, use procedures included in EPA Compendium Method TO-15: Reduction of Method Detection Limits to Meet Vapor Intrusion Monitoring Needs (http://www.epa.gov/ttnamti1/files/ambient/airtox/TO-15-Supplement.pdf).

OUTDATED

Source: EPA. 1999. "Air Method, Toxic Organics-15 (TO-15): Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)." http://www.epa.gov/sam/pdfs/EPA-TO-15.pdf

5.2.46 NIOSH Method 1612: Propylene Oxide

Analyte(s)	CAS RN
Propylene oxide	75-56-9

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Coconut shell charcoal solid sorbent tube
Determinative Technique: GC-FID

Method Developed for: Propylene oxide in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range is between 8 and 295 ppm for air samples of 5 L.

Description of Method: A sample tube containing coconut shell charcoal is used for sample collection with a flow rate of 0.01 to 0.2 L/min. One milliliter of carbon disulfide is added to the vial and allowed to sit for 30 minutes prior to analysis with occasional agitation. Analysis is performed on a GC-FID. No interferences have been found.

Special Considerations: The presence of propylene oxide should be confirmed by either a secondary GC column or by an MS.

Source: NIOSH. 1994. "Method 1612: Propylene Oxide," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-1612.pdf

5.2.47 NIOSH Method 2016: Formaldehyde

Analyte(s)	CAS RN
Formaldehyde	50-00-0

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent extraction
Determinative Technique: HPLC

Method Developed for: Formaldehyde in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit for formaldehyde is 0.07 µg/sample. The working range is 0.015 to 2.5 mg/m^3 (0.012 to 2.0 ppm) for a 15-L sample.

Description of Method: This method can be used for the determination of formaldehyde using HPLC with a UV detector. Air is sampled onto a cartridge containing silica gel coated with 2,4-DNPH, at a rate of 0.03 to 1.5 L/min. The cartridge is extracted with 10 mL of acetonitrile and analyzed by HPLC-UV at a wavelength of 360 nm. Ozone has been observed to consume the 2,4-DNPH reagent and to degrade the formaldehyde derivative. Ketones and other aldehydes can react with 2,4-DNPH; the derivatives produced, however, are separated chromatographically from the formaldehyde derivative.

OUTDATED

Source: NIOSH. 2003. "Method 2016: Formaldehyde," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-2016.pdf

5.2.48 NIOSH Method 2513: Ethylene Chlorohydrin

Analyte(s)	CAS RN
2-Chloroethanol	107-07-3
2-Fluoroethanol	371-62-0

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: GC-FID

Method Developed for: Ethylene chlorohydrin (2-chloroethanol) in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.5 to 15 ppm for a 20-L air sample.

Description of Method: Samples are drawn into a tube containing petroleum charcoal at a rate of 0.01 to 0.2 L/min and transferred into vials containing eluent (carbon disulfide, 2-propanol, and *n*-pentadiene as an internal standard). Vials must sit for 30 minutes prior to analysis by GC-FID. No interferences have been identified. Humidity may decrease the breakthrough volume during sample collection.

Special Considerations: The presence of 2-chloroethanol should be confirmed by either a secondary GC column or by an MS.

Source: NIOSH. 1994. "Method 2513: Ethylene Chlorohydrin," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-2513.pdf

5.2.49 NIOSH Method 3510: Monomethylhydrazine

Analyte(s)	CAS RN
Methyl hydrazine (monomethylhydrazine)	60-34-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Samples are collected into a bubbler containing hydrochloric acid.
Determinative Technique: Visible spectrophotometry

Method Developed for: Monomethylhydrazine in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.027 to 2.7 ppm for a 20-L air sample.

Description of Method: Samples are collected into a bubbler containing hydrochloric acid using a flow rate of 0.5 to 1.5 L/min. Samples are transferred to a 25-mL flask, mixed with phosphomolybdic acid solution, diluted to the mark with 0.1 M hydrochloric acid, and then transferred to a large test tube for spectrophotometric analysis. Positive interferences include other hydrazines, as well as stannous ion, ferrous ion, zinc, sulfur dioxide, and hydrogen sulfide. Negative interferences may occur by oxidation of the monomethylhydrazine by halogens, oxygen (especially in the presence of copper (I) ions) and hydrogen dioxide.

Source: NIOSH. 1994. "Method 3510: Monomethylhydrazine," Issue 1.
http://www.epa.gov/sam/pdfs/NIOSH-3510.pdf

OUTDATED

5.2.50 NIOSH Method 5600: Organophosphorus Pesticides

Analyte(s)	CAS RN
Disulfoton	298-04-4
Disulfoton sulfone oxon[1]	2496-91-5
Disulfoton sulfoxide	2497-07-6
Disulfoton sulfoxide oxon[1]	2496-92-6

[1] If problems occur when using this method for measurement of oxon compounds, analysts should consider use of procedures included in "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: Gas chromatography – flame photometric detector (GC-FPD)

Method Developed for: Organophosphorus pesticides in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit depends on the compound being measured. The working range for each analyte is provided in Table 5 of the method. These ranges cover from 0.1 to 2 times the OSHA Permissible Exposure Limits (PELs).

Description of Method: This method is used for the detection of organophosphorus pesticides using GC with a flame photometric detector (FPD). Samples are prepared by desorbing the XAD-2 resin with 2 mL of toluene/acetone (90/10 v/v) solution. The method also may be applicable to the determination of other organophosphorus compounds after evaluation for desorption efficiency, sample capacity, sample stability, and precision and accuracy. The working range for each analyte is provided in Table 5 of the method. These ranges cover from 0.1 to 2 times the OSHA PELs (see Table 5 of the method). The method also is applicable to Short Term Exposure Limit (STEL) measurements using 12-L samples.

Special Considerations: Refer to footnote provided in analyte table above for special considerations that should be applied when measuring specific analytes. Several organophosphates may co-elute with either target analytes or internal standards causing integration errors. These include other pesticides, and the following: tributyl phosphate, tris-(2-butoxy ethyl) phosphate, tricresyl phosphate, and triphenyl phosphate.

Source: NIOSH. 1994. "Method 5600: Organophosphorus Pesticides," Issue 1.
http://www.epa.gov/sam/pdfs/NIOSH-5600.pdf

5.2.51 NIOSH Method 5601: Organonitrogen Pesticides

Analyte(s)	CAS RN
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Carbofuran (Furadan)	1563-66-2
Methomyl	16752-77-5
Oxamyl	23135-22-0
Thiofanox	39196-18-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption

OUTDATED

Determinative Technique: HPLC

Method Developed for: Organonitrogen pesticides in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit for aldicarb is 1.2 µg per sample and 0.6 µg per sample for carbofuran, methomyl, and oxamyl. The working ranges for aldicarb, carbofuran, and oxamyl are listed in Table 2 of the method, and range from 0.5 to 10 times the OSHA PEL.

Description of Method: This method can be used for the determination of organonitrogen pesticides using HPLC with a UV detector. Samples are prepared by desorbing the XAD-2 resin with 2 mL of triethylamine-phosphate solution, rotating end-over-end for 45 minutes, and filtering. The method also may be applicable to the determination of other organonitrogen compounds and to a broad range of pesticides having UV chromophores, e.g., acetanilides, acid herbicides, organophosphates, phenols, pyrethroids, sulfonyl ureas, sulfonamides, triazines, and uracil pesticides. Because of the broad response of the UV detector at shorter wavelengths, there are many potential interferences. Those tested include solvents (chloroform and toluene), antioxidants (butylated hydroxytoluene [BHT]), plasticizers (dialkyl phthalates), nitrogen compounds (nicotine and caffeine), impurities in HPLC reagents (e.g., in triethylamine), other pesticides (2,4-Dichlorophenoxyacetic acid [2,4-D], atrazine, parathion, etc.), and pesticide hydrolysis products (1-naphthol). Confirmation techniques are recommended when analyte identity is uncertain.

Special Considerations: The presence of the analytes listed in the table above should be confirmed by either a secondary HPLC column or by an MS.

Source: NIOSH. 1998. "Method 5601: Organonitrogen Pesticides," Issue 1.
http://www.epa.gov/sam/pdfs/NIOSH-5601.pdf

5.2.52 NIOSH Method 6001: Arsine

Analyte(s)	CAS RN
Arsine (analyze as total arsenic in non-air samples)	7784-42-1

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Coconut shell charcoal solid sorbent tube
Determinative Technique: GFAA

Method Developed for: Arsine in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.001 to 0.2 mg/m^3 for a 10-L sample.

Description of Method: Arsine is determined as arsenic. A 0.1- to 10-L volume of air is drawn through a sorbent tube containing activated charcoal. The sorbent is extracted with a nitric acid solution, and arsenic is determined by GFAA.

Special Considerations: The method is subject to interferences from other arsenic compounds.

Source: NIOSH. 1994. "Method 6001: Arsine," Issue 2. http://www.epa.gov/sam/pdfs/NIOSH-6001.pdf

OUTDATED

5.2.53 NIOSH Method 6002: Phosphine

Analyte(s)	CAS RN
Phosphine	7803-51-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption with hot acidic permanganate solution
Determinative Technique: Visible spectrophotometry

Method Developed for: Phosphine in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.02 to 0.9 mg/m^3 for a 16-L sample.

Description of Method: In this method, phosphine is determined as phosphate. A volume of 1 to 16 L of air is drawn through a sorbent tube containing silica gel coated with mercuric cyanide. The sorbent is extracted with a potassium permanganate/sulfuric acid solution and washed with reagent water. Following treatment with the color agent and extraction into organic solvent, phosphate is determined by visible spectrometry.

Special Considerations: The method is subject to interferences from phosphorus trichloride, phosphorus pentachloride, and organic phosphorus compounds.

Source: NIOSH. 1994. "Method 6002: Phosphine," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-6002.pdf

5.2.54 NIOSH Method 6010: Hydrogen Cyanide

Analyte(s)	CAS RN
Cyanide, Total	57-12-5
Hydrogen cyanide	74-90-8

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: Visible spectrophotometry

Method Developed for: Hydrogen cyanide in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 3 to 260 mg/m^3 for a 3-L sample.

Description of Method: Hydrogen cyanide is determined as a cyanide ion complex by this method. A volume of 0.6 to 90 L of air is drawn through a soda lime sorbent tube. A glass-fiber filter is used to remove particulate cyanides prior to the sorbent tube. Cyanide is extracted from the sorbent with reagent water treated with sodium hydroxide. The extract is pH adjusted with hydrochloric acid, oxidized with N-chlorosuccinimide/succinimide, and treated with the coupling-color agent (barbituric acid/pyridine). The cyanide ion is determined by visible spectrophotometry using a wavelength of 580 nm.

Special Considerations: The method is subject to interference from high concentrations of hydrogen sulfide. Two liters is the minimum volume required to measure concentration of 5 ppm.

Source: NIOSH. 1994. "Method 6010: Hydrogen Cyanide," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-6010.pdf

OUTDATED

5.2.55 NIOSH Method 6013: Hydrogen Sulfide

Analyte(s)	CAS RN
Hydrogen sulfide	7783-06-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent extraction
Determinative Technique: IC with conductivity detection

Method Developed for: Hydrogen sulfide in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.9 to 20 mg/m^3 for a 20-L sample.

Description of Method: Hydrogen sulfide is determined as sulfate by this method. A volume of 15 to 40 L of air is drawn through charcoal sorbent. A prefilter is used to remove particulates. The sorbent portions are extracted with an ammonium hydroxide/hydrogen peroxide solution and the extract is analyzed for sulfate by IC.

Special Considerations: The method is subject to interference from sulfur dioxide.

Source: NIOSH. 1994. "Method 6013: Hydrogen Sulfide," Issue 1.
http://www.epa.gov/sam/pdfs/NIOSH-6013.pdf

5.2.56 NIOSH Method 6015: Ammonia

Analyte(s)	CAS RN
Ammonia	7664-41-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Water extraction
Determinative Technique: Visible spectrophotometry

Method Developed for: Ammonia in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 0.15 to 300 mg/m^3 for a 10-L sample. Twice the recommended sample volume should be collected in order to achieve an action level of 70 µg/m^3.

Description of Method: Ammonia is determined as indophenol blue by this method. A volume of 0.1 to 96 L of air is drawn through a sulfuric acid-treated silica gel sorbent. A prefilter is used to remove particulates. The sorbent is extracted with reagent water, the pH adjusted, and reagents are added to generate the indophenol blue compound in the presence of ammonium. The extract is analyzed by visible spectrophotometry. No interferences have been identified.

Source: NIOSH. 1994. "Method 6015: Ammonia," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-6015.pdf

OUTDATED

5.2.57 NIOSH Method 6402: Phosphorus Trichloride

Analyte(s)	CAS RN
Phosphorus trichloride	7719-12-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Add reagent to samples in bubbler solution and heat
Determinative Technique: Visible spectrophotometry

Method Developed for: Phosphorus trichloride in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of the method is 1.2 to 80 mg/m^3 for a 25-L sample.

Description of Method: In this method, phosphorus trichloride is determined as phosphate. A volume of 11 to 100 L of air is drawn through a bubbler containing reagent water. The resulting phosphorus acid solution is oxidized with bromine to phosphoric acid and color agent (sodium molybdate) and reducing agent (hydrazine sulfate) are added. The solution is analyzed for the resulting molybdenum blue complex by visible spectrophotometry. Phosphorus (V) compounds do not interfere. Sample solutions are stable to oxidation by air during sampling.

Source: NIOSH. 1994. "Method 6402: Phosphorus Trichloride," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-6402.pdf

5.2.58 NIOSH Method 7903: Acids, Inorganic

Analyte(s)	CAS RN
Hydrogen bromide	10035-10-6
Hydrogen chloride	7647-01-0
Hydrogen fluoride	7664-39-3

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: IC with conductivity detection

Method Developed for: Inorganic acids in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The working range of this method is 0.01 to 5 mg/m^3 for a 50-L sample.

Description of Method: Acids are analyzed as bromide, chloride, and fluoride. A volume of 3 to 100 L of air is drawn through a silica gel sorbent. The sorbent portions are extracted with a buffered carbonate/bicarbonate solution and the extract is analyzed by IC.

Special Considerations: Particulate salts of the acids are an interference (trapped on the glass wool filter plug in the sorbent tube). Chlorine and bromine are also interferences. Acetate, formate, and propionate interferences may be reduced by use of a weaker eluent. If problems occur when using this method for analysis of hydrogen fluoride, it is recommended that NIOSH Method 7906 be used.

Source: NIOSH. 1994. "Method 7903: Acids, Inorganic," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-7903.pdf

OUTDATED

5.2.59 NIOSH Method 7905: Phosphorus

Analyte(s)	CAS RN
White phosphorus	12185-10-3

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: GC solid sorbent tube and solvent extracted (desorbed)
Determinative Technique: GC-FPD

Method Developed for: Phosphorus in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The LOD for samples analyzed by GC-FPD is 0.005 µg per sample. The working range for samples analyzed by GC-FPD is 0.056 to 0.24 mg/m^3 for a 12-L sample.

Description of Method: This method identifies and determines the concentration of white phosphorus in air by using a GC-FPD. Five to 100 L of air is drawn through a GC solid sorbent tube, and the sorbent is extracted (desorbed) with xylene. The method is applicable to vapor-phase phosphorus only; if particulate phosphorus is expected, a filter could be used in the sampling train.

Special Considerations: The presence of white phosphorus should be confirmed by either a secondary GC column or by an MS.

Source: NIOSH. 1994. "Method 7905: Phosphorus," Issue 2.
http://www.epa.gov/sam/pdfs/NIOSH-7905.pdf

5.2.60 NIOSH Method 7906: Fluorides, Aerosol and Gas, by IC

Analyte(s)	CAS RN
Hydrogen fluoride	7664-39-3

Analysis Purpose: Sample preparation and analysis
Sample Preparation Technique: Water extraction
Determinative Technique: IC with conductivity detection

Method Developed for: Fluorides in aerosol and gas
Method Selected for: SAM lists this method for use if problems occur when using NIOSH Method 7903 for the analysis of hydrogen fluoride during preparation and analysis of air samples. (See Footnote 10 of Appendix A.)
Detection and Quantitation: The working range of the method is 0.04 to 8 mg/m^3 for 250-L samples.

Description of Method: Hydrogen fluoride is determined as fluoride ion by this method. A volume of 1 to 800 L of air is drawn through a 0.8-µm cellulose ester membrane (to trap particulate fluorides) and a cellulose pad treated with sodium carbonate (to trap gaseous fluoride). The pad is extracted with reagent water and the extract is analyzed for fluoride by IC.

Special Considerations: If other aerosols are present, gaseous fluoride may be slightly underestimated due to adsorption onto or reaction with particles, with concurrent overestimation of particulate/gaseous fluoride ratio.

Source: NIOSH. 1994. "Method 7906: Fluorides, Aerosol and Gas by IC," Issue 1.
http://www.epa.gov/sam/pdfs/NIOSH-7906.pdf

OUTDATED

5.2.61 NIOSH Method 9102: Elements on Wipes

Analyte(s)	CAS RN
Ammonium metavanadate (analyze as total vanadium)	7803-55-6
Arsenic, Total	7440-38-2
Arsenic trioxide (analyze as total arsenic)	1327-53-3
Arsine (analyze as total arsenic in non-air samples)	7784-42-1
Calcium arsenate (analyze as total arsenic)	7778-44-1
2-Chlorovinylarsonous acid (2-CVAA)	85090-33-1
Ethyldichloroarsine (ED)	598-14-1
Lead arsenate (analyze as total arsenic)	7645-25-2
Lewisite 1 (L-1) [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1
Lewisite oxide	1306-02-1
Mercuric chloride (analyze as total mercury)	7487-94-7
Mercury, Total	7439-97-6
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2
Osmium tetroxide (analyze as total osmium)	20816-12-0
Sodium arsenite (analyze as total arsenic)	7784-46-5
Thallium sulfate (analyze as total thallium)	10031-59-1
Titanium tetrachloride (analyze as total titanium)	7550-45-0
Vanadium pentoxide (analyze as total vanadium)	1314-62-1

Analysis Purpose: Sample preparation
Sample Preparation Technique: Acid digestion
Determinative Technique: ICP-AES / ICP-MS / Spectrophotometry
Determinative Method: EPA SW-846 Methods 6010C, 6020A, and 7473. Refer to Appendix A for which of these determinative methods should be used for a particular analyte.

Method Developed for: Measurement of metals on wipe surfaces using ICP-AES
Method Selected for: SAM lists this method for preparation of wipe samples.
Detection and Quantitation: The range for arsenic is 0.261 to105 µg/wipe; for thallium 0.136 to 50.0 µg/wipe; for vanadium 0.0333 to 25.0 µg/wipe.

Description of Method: Surface wipe samples are transferred to a clean beaker, followed by the addition of concentrated nitric and perchloric acids. The beaker contents are held at room temperature for 30 minutes, then heated at 150°C for 8 hours. Additional nitric acid is added until the wipe media is completely destroyed. The sample is then taken to near dryness and the residue dissolved and diluted before being analyzed.

Special Considerations: ICP-MS may also be used for the analysis of wipe samples; however, at this time, this technique has not been evaluated for wipes. Nitric and perchloric acids are strong oxidizers and extremely corrosive. Perform all perchloric acid digestions in a perchloric acid hood. When working with acids, use gloves and avoid inhalation or contact with skin or clothing.

Source: NIOSH. 2003. "Method 9102, Issue 1: Elements on Wipes."
http://www.epa.gov/sam/pdfs/NIOSH-9102.pdf

OUTDATED

5.2.62 NIOSH Method S301-1: Fluoroacetate Anion

Analyte(s)	CAS RN
Fluoroacetic acid and fluoroacetate salts	NA
Methyl fluoroacetate	453-18-9

Analysis Purpose: Sample preparation
Sample Preparation Technique: Water extraction
Determinative Technique: IC with electrolytic conductivity detection
Determinative Method: EPA Method 300.1 Rev 1.0

Method Developed for: Fluoroacetate anion in air
Method Selected for: SAM lists this method for preparation of air samples.
Detection and Quantitation: The detection limit is estimated to be 20 ng of sodium fluoroacetate per injection, corresponding to a 100-µL aliquot of a 0.2-µg/mL standard. The analytical range of this method is estimated to be 0.01 to 0.16 mg/m^3.

Description of Method: This method was developed specifically for sodium fluoroacetate, but also may be applicable to other fluoroacetate salts. The method determines fluoroacetate salts as fluoroacetate anion. A known volume of air (e.g., 480 L was used in validation of this method) is drawn through a cellulose ester membrane filter to collect sodium fluoroacetate. Sodium fluoroacetate is extracted from the filter with 5 mL of deionized water, and the resulting sample is analyzed by IC using electrolytic conductivity detection.

Special Considerations: When analyzing samples for methyl fluoroacetate (as fluoroacetate ion), addition of base is required to assist dissociation into fluoroacetate anion.

Source: NIOSH. 1977. "Method S301-1: Sodium Fluoroacetate."
http://www.epa.gov/sam/pdfs/NIOSH-S301-1.pdf

5.2.63 OSHA Method 40: Methylamine

Analyte(s)	CAS RN
Methylamine	74-89-5

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: HPLC

Method Developed for: Methylamine in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit of the overall procedure is 0.35 µg per sample (28 ppb or 35 µg/m^3). Quantitation limits of 28 ppb (35 µg/m^3) have been achieved. This is the smallest amount of methylamine that can be quantified within the requirements of a recovery of at least 75% and a precision (standard deviation of 1.96) of ± 25% or better.

Description of Method: This method is used for detection of methylamine using HPLC with a FL or visible (vis) detector. Samples are collected by drawing 10-L volumes of air at a rate of 0.2 L/min through standard size sampling tubes containing XAD-7 resin coated with 10% 7-chloro-4-nitrobenzo-2-oxa-1,3-diazole (NBD chloride) by weight. Samples are desorbed with 5% (w/v) NBD chloride in tetrahydrofuran (with a small amount of sodium bicarbonate present), heated in a hot water bath, and analyzed by high performance liquid chromatography – fluorescence (HPLC-FL) or high performance liquid chromatography – visible (HPLC-vis).

OUTDATED

Source: OSHA. 1982. "Method 40: Methylamine." Method originally obtained from www.osha.gov, but is provided here for reference. http://www.epa.gov/sam/pdfs/OSHA-Method40.pdf

5.2.64 OSHA Method 54: Methyl Isocyanate (MIC)

Analyte(s)	CAS RN
Methyl isocyanate	624-83-9

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: HPLC

Method Developed for: Methyl isocyanate in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.

Description of Method: This method determines the concentration of methyl isocyanate in air by using HPLC with a FL or UV detector. Samples are collected by drawing a known volume of air through XAD-7 tubes coated with 0.3 mg of 1-(2-pyridyl)piperazine (1-2PP). Samples are desorbed with acetonitrile and analyzed by HPLC using a FL or UV detector.

Source: OSHA. 1985. "Method 54: Methyl Isocyanate (MIC)." Method originally obtained from www.osha.gov, but is provided here for reference. http://www.epa.gov/sam/pdfs/OSHA-Method54.pdf

5.2.65 OSHA Method 61: Phosgene

Analyte(s)	CAS RN
Phosgene	75-44-5

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: GC-NPD

Method Developed for: Phosgene in air samples
Method Selected for: SAM lists this method for preparation and analysis of air samples.

Description of Method: This method determines the concentration of phosgene in air by using GC with an NPD. Air samples are collected by drawing known volumes of air through sampling tubes containing XAD-2 adsorbent that has been coated with 2-(hydroxymethyl)piperidine. The samples are desorbed with toluene and then analyzed by GC using an NPD.

Special Considerations: The presence of phosgene should be confirmed by either a secondary GC column or by MS

Source: OSHA. 1986. "Method 61: Phosgene." Method originally obtained from www.osha.gov, but is provided here for reference. http://www.epa.gov/sam/pdfs/OSHA-Method61.pdf

OUTDATED

5.2.66 OSHA Method ID-211: Sodium Azide and Hydrazoic Acid in Workplace Atmospheres

Analyte(s)	CAS RN
Sodium azide (analyze as azide ion)	26628-22-8

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Buffer desorption
Determinative Technique: IC-UV

Method Developed for: Sodium azide and hydrazoic acid in workplace atmospheres
Method Selected for: SAM lists this method for preparation and analysis of air and wipe samples.
Detection and Quantitation: The detection limit was found to be 0.001 ppm as hydrazoic acid (HN_3) or 0.003 mg/m^3 as sodium azide (NaN_3) for a 5-L air sample. The quantitation limit was found to be 0.004 ppm as HN_3 or 0.011 mg/m^3 as NaN_3 for a 5-L air sample.

Description of Method: This method describes sample collection and analysis of airborne azides [as NaN_3 and hydrazoic acid HN_3]. Particulate NaN_3 is collected on a PVC filter or in the glass wool plug of the sampling tube. Gaseous HN_3 is collected and converted to NaN_3 by the impregnated silica gel (ISG) sorbent within the sampling tube. The collected azide on either media is desorbed in a weak buffer solution, and the resultant anion (N_3^-) is analyzed by IC using a variable wavelength UV detector at 210 nm. A gravimetric conversion is used to calculate the amount of NaN_3 or HN_3 collected.

Source: OSHA. 1992. "Method ID-211: Sodium Azide and Hydrazoic Acid in Workplace Atmospheres." http://www.epa.gov/sam/pdfs/OSHA-ID-211.pdf

5.2.67 OSHA Method ID216SG: Boron Trifluoride (BF_3)

Analyte(s)	CAS RN
Boron trifluoride	7637-07-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Sample collected in bubbler (no sample preparation required)
Determinative Technique: Ion specific electrode (ISE)

Method Developed for: Boron trifluoride in air samples
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit is 10 µg in a 30-L sample.

Description of Method: Boron trifluoride is determined as fluoroborate. A volume of 30 to 480 L of air is drawn through a bubbler containing 0.1 M ammonium fluoride. The solution is diluted and analyzed with a fluoroborate ISE.

Source: OSHA. 1989. "Method ID216SG: Boron Trifluoride (BF_3)." Method originally obtained from www.osha.gov, but is provided here for reference. http://www.epa.gov/sam/pdfs/OSHA-ID216SG.pdf

OUTDATED

5.2.68 OSHA Method PV2004: Acrylamide

Analyte(s)	CAS RN
Acrylamide	79-06-1
Acrylonitrile	107-13-1
Methyl acrylonitrile	126-98-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: HPLC

Method Developed for: Acrylamide in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit was found to be 0.7 µg/mL (0.006 mg/m^3 for a 1-mL desorption volume or 0.029 mg/m^3 for a 5-mL desorption volume based on a 120-L air volume). Applicable working ranges for a 1-mL and 5-mL desorption volume are 0.017 - 1.5 mg/m^3 and 0.083 - 7.5 mg/m^3, respectively.

Description of Method: This method determines the concentration of acrylamide in air by using HPLC with a UV detector. Samples are collected by drawing known volumes of air through OSHA versatile sampler (OVS-7) tubes, each containing a glass fiber filter and two sections of XAD-7 adsorbent. Samples are desorbed with a solution of 5% methanol/95% water and analyzed by HPLC using a UV detector.

Source: OSHA. 1991. "Method PV2004: Acrylamide." http://www.epa.gov/sam/pdfs/OSHA-PV2004.pdf

5.2.69 OSHA Method PV2103: Chloropicrin

Analyte(s)	CAS RN
Chloropicrin	79-06-2

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent desorption
Determinative Technique: GC-ECD

Method Developed for: Chloropicrin in air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: The detection limit is 0.01 ng, with a 1-µL injection volume. This is the smallest amount that could be detected under normal operating conditions. The working range is 33.2 to 1330 µg/m^3.

Description of Method: This method determines the concentration of chloropicrin in air by GC-ECD. Samples are collected by drawing a known volume of air through two XAD-4 tubes in series. Samples are desorbed with ethyl acetate and analyzed by GC-ECD.

Special Considerations: The presence of chloropicrin should be confirmed by either a secondary GC column or by an MS. Chloropicrin is light sensitive, and samples should be protected from light.

Source: OSHA. 1991. "Method PV2103: Chloropicrin." http://www.epa.gov/sam/pdfs/OSHA-PV2103.pdf

OUTDATED

5.2.70 ASTM Method D5755-03: Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading

Analyte(s)	CAS RN
Asbestos	1332-21-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Direct transfer
Determinative Technique: Transmission electron microscopy (TEM)

Method Developed for: Asbestos in dust
Method Selected for: SAM lists this method for preparation and analysis of solid (e.g., soft surfaces-microvac) samples.

Description of Method: This method describes procedures to identify asbestos in dust and provide an estimate of the surface loading of asbestos reported as the number of asbestos structures per unit area of sampled surface. The sample is collected by vacuuming a known surface area with a standard 25- or 37-mm air sampling cassette using a plastic tube that is attached to the inlet orifice, which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane, and a section of the membrane is prepared and transferred to a TEM grid using a direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using select area electron diffraction (SAED) and energy dispersive X-ray analysis (EDXA) at a magnification of 15,000 to 20,000X.

Source: ASTM. 2003. "Method D5755-03: Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading." http://www.astm.org/Standards/D5755.htm

5.2.71 ASTM Method D6480-05: Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy

Analyte(s)	CAS RN
Asbestos	1332-21-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Direct transfer
Determinative Technique: TEM

Method Developed for: Asbestos in samples wiped from surfaces
Method Selected for: SAM lists this method for preparation and analysis of wipe (e.g., hard surfaces-wipes) samples.

Description of Method: This method describes a procedure to identify asbestos in samples wiped from surfaces and to provide an estimate of the concentration of asbestos reported as the number of asbestos structures per unit area of sampled surface. A sample is collected by wiping a surface of known area with a wipe material. The sample is transferred from the wipe material to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane filter, and a section of the membrane filter is prepared and transferred to a TEM grid, using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using electron diffraction and EDXA at a magnification from 15,000 to 20,000X.

OUTDATED

Source: ASTM. 2005. "Method D6480-05: Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy." http://www.astm.org/Standards/D6480.htm

5.2.72 ASTM Method D7597-09: Standard Test Method for Determination of Diisopropyl Methylphosphonate, Ethyl Hydrogen Dimethylamidophosphate, Ethyl Methylphosphonic Acid, Isopropyl Methylphosphonic Acid, Methylphosphonic Acid and Pinacolyl Methylphosphonic Acid in Water by Liquid Chromatography/Tandem Mass Spectrometry

Analyte(s)	CAS RN
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Ethyl methylphosphonic acid (EMPA)	1832-53-7
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Methylphosphonic acid (MPA)	993-13-5
Pinacolyl methyl phosphonic acid (PMPA)	616-52-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Filtered using a syringe-driven Millex HV polyvinylidene fluoride (PVDF) filter unit
Determinative Technique: LC-MS-MS

Method Developed for: Diisopropyl methylphosphonate, ethyl hydrogen dimethylamidophosphate, isopropyl methylphosphonic acid, methylphosphonic acid and pinacolyl methylphosphonic acid in surface water
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid samples.
Detection and Quantitation: The detection verification levels (DVLs) and reporting range for this method vary for each analyte and range from 0.25 to 20 µg/L and 5 to 1500 µg/L.

Description of Method: Target compounds are analyzed by direct injection without derivatization by LC-MS-MS. Samples are shipped to the laboratory at 0 to 6°C, spiked with surrogates, filtered using a syringe-driven filter unit and analyzed directly by LC-MS-MS within 1 day. The target compounds are identified by comparing the sample single reaction monitoring (SRM) transitions to the known standard SRM transitions. The retention time for the analytes of interest must also fall within the retention time of the standard by ± 5%. Target compounds are quantitated using the SRM transition of the target compounds and external standard calibration.

Source: ASTM. 2009. "Method D7597-09: Standard Test Method for Determination of Diisopropyl Methylphosphonate, Ethyl Hydrogen Dimethylamidophosphate, Ethyl Methylphosphonic Acid, Isopropyl Methylphosphonic Acid, Methylphosphonic Acid and Pinacolyl Methylphosphonic Acid in Water by Liquid Chromatography/Tandem Mass Spectrometry." http://www.astm.org/Standards/D7597.htm

5.2.73 ASTM Method D7598-09: Standard Test Method for Determination of Thiodiglycol in Water by Single Reaction Monitoring Liquid Chromatography/Tandem Mass Spectrometry

Analyte(s)	CAS RN
Thiodiglycol	111-48-8

Analysis Purpose: Sample preparation, and analyte determination and measurement

OUTDATED

Sample Preparation Technique: Filtered using a syringe-driven Millex HV polyvinyliden fluoride (PVDF) filter unit
Determinative Technique: LC-MS-MS

Method Developed for: Thiodiglycol in surface water samples
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid samples.
Detection and Quantitation: The DVL for thiodiglycol is 20 µg/L; the reporting range is 100 to 10000 µg/L.

Description of Method: Thiodiglycol is analyzed by direct injection without derivatization by LC-MS-MS. Samples are shipped to the laboratory at 0 to 6°C, spiked with surrogates, filtered using a syringe-driven filter unit and analyzed directly by LC-MS-MS within 7 days. The target compound is identified by comparing the sample primary SRM transition to the known standard SRM transition. The retention time must fall within the retention time of the standard by ± 5%. Thiodiglycol is quantitated using the primary SRM transition and external standard calibration.

Source: ASTM. 2009. "Method D7598-09: Standard Test Method for Determination of Thiodiglycol in Water by Single Reaction Monitoring Liquid Chromatography/Tandem Mass Spectrometry." http://www.astm.org/Standards/D7598.htm

5.2.74 ASTM Method D7599-09: Standard Test Method for Determination of Diethanolamine, Triethanolamine, *N*-Methyldiethanolamine and *N*-Ethyldiethanolamine in Water by Single Reaction Monitoring Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)

Analyte(s)	CAS RN
N-Ethyldiethanolamine (EDEA)	139-87-7
N-Methyldiethanolamine (MDEA)	105-59-9
Triethanolamine (TEA)	102-71-6

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Filtered using a syringe-driven Millex HV PVDF filter unit
Determinative Technique: LC-MS-MS

Method Developed for: Diethanolamine, triethanolamine, *n*-methyldiethanolamine and *n*-ethyldiethanolamine in surface water samples
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid samples.
Detection and Quantitation: The DVL and reporting range for EDEA and TEA are 5 µg/L and 25 to 500 µg/L, respectively. The DVL and reporting range for MDEA are 10 µg/L and 50 to 500 µg/L, respectively.

Description of Method: Target compounds are analyzed by direct injection without derivatization by LC-MS-MS. Samples are shipped to the laboratory at 0 to 6°C, spiked with surrogates, filtered using a syringe-driven filter unit and analyzed directly by LC-MS-MS within 7 days. Target compounds are identified by comparing sample SRM transitions to the known standard SRM transitions. The retention time for the analytes of interest must also fall within the retention time of the standard by ± 5%. Target compounds are quantitated using the SRM transition and external standard calibration.

Source: ASTM. 2009. "Method D7599-09: Standard Test Method for Determination of Diethanolamine, Triethanolamine, *N*-Methyldiethanolamine and *N*-Ethyldiethanolamine in Water by Single Reaction Monitoring Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)." http://www.astm.org/Standards/D7599.htm

OUTDATED

5.2.75 ASTM Method D7600-09: Standard Test Method for Determination of Aldicarb, Carbofuran, Oxamyl and Methomyl by Liquid Chromatography/Tandem Mass Spectrometry

Analyte(s)	CAS RN
Aldicarb (Temik)	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Bromadiolone	28772-56-7
Carbofuran (Furadan)	1563-66-2
Methomyl	16752-77-5
Oxamyl	23135-22-0

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Filtered using a syringe-driven Millex HV PVDF filter unit
Determinative Technique: LC-MS-MS

Method Developed for: Aldicarb, bromadiolone, carbofuran, oxamyl and methomyl in water
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid samples.
Detection and Quantitation: The DVL for aldicarb, carbofuran, methomyl, and oxamyl is 100 ng/L. The reporting range is 1 to 100 µg/L.

Description of Method: Target compounds are analyzed by direct injection without derivatization by LC-MS-MS. Samples are shipped to the laboratory at 0 to 6°C, spiked with surrogates, filtered using a syringe-driven filter unit, and analyzed directly by LC-MS-MS within 7 days. The target compounds are identified by comparing the sample primary and confirmatory multiple reaction monitoring (MRM) transitions to the known standard primary and confirmatory MRM transitions. The retention time for the analytes of interest must also fall within the retention time of the standard by ± 5%. Target compounds are quantitated using the primary SRM transition and external standard calibration.

Source: ASTM. 2009. "Method D7600-09: Standard Test Method for Determination of Aldicarb, Carbofuran, Oxamyl and Methomyl by Liquid Chromatography/Tandem Mass Spectrometry." http://www.astm.org/Standards/D7600.htm

5.2.76 ISO Method 10312:1995: Ambient Air - Determination of Asbestos Fibres - Direct-transfer Transmission Electron Microscopy Method

Analyte(s)	CAS RN
Asbestos	1332-21-4

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Direct transfer
Determinative Technique: TEM

Method Developed for: Asbestos in ambient air
Method Selected for: SAM lists this method for preparation and analysis of air samples.
Detection and Quantitation: In a 4000-L air sample with approximately 10 pg/m^3 (typical of clean or rural atmospheres), an analytical sensitivity of 0.5 structure/L can be obtained. This is equivalent to a detection limit of 1.8 structure/L when an area of 0.195 mm of the TEM specimen is examined. The range of concentrations that can be determined is 50 to 7,000 structures/mm^2 on the filter.

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Description of Method: This method determines the type(s) of asbestos fibers present, but cannot discriminate between individual fibers of the asbestos and non-asbestos analogues of the same amphibole mineral. The method is defined for polycarbonate capillan/pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn. The method is suitable for determination of asbestos in both exterior and building atmospheres.

Source: ISO. 2005. "Method 10312: 1995: Ambient Air - Determination of Asbestos Fibres - Direct Transfer Transmission Electron Microscopy Method."
http://www.iso.org/iso/iso_catalogue/catalogue_tc/catalogue_detail.htm?csnumber=18358

5.2.77 Standard Method 4500-NH_3 B: Nitrogen (Ammonia) Preliminary Distillation Step

Analyte(s)	CAS RN
Ammonia	7664-41-7

Analysis Purpose: Sample preparation
Sample Preparation Technique: Distillation
Determinative Technique: Visible spectrophotometry
Determinative Method: Standard Method 4500-NH_3 G

Method Developed for: Nitrogen (ammonia) in drinking waters, clean surface or groundwater, and good-quality nitrified wastewater effluent
Method Selected for: SAM lists this method for preparation of aqueous liquid samples.

Description of Method: A 0.5- to 1-L sample is dechlorinated, buffered, adjusted to pH 9.5, and distilled into a sulfuric acid solution. The distillate is brought up to volume, neutralized with sodium hydroxide, and analyzed by Method 4500-NH_3 G.

Source: APHA, AWWA, and WEF. 2005. "Method 4500-NH3 B: Nitrogen (Ammonia) Preliminary Distillation Step." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition.
http://www.standardmethods.org/

5.2.78 Standard Method 4500-NH_3 G: Nitrogen (Ammonia) Automated Phenate Method

Analyte(s)	CAS RN
Ammonia	7664-41-7

Analysis Purpose: Analyte determination and measurement
Determinative Technique: Visible spectrophotometry
Sample Preparation Method: Standard Method 4500-NH_3 B
Sample Preparation Technique: Distillation

Method Developed for: Nitrogen (ammonia) in drinking waters, clean surface or groundwater, and good-quality nitrified wastewater effluent
Method Selected for: SAM lists this method for analysis of aqueous liquid samples.
Detection and Quantitation: The range of the method is 0.02 to 2.0 mg/L.

Description of Method: Ammonia is determined as indophenol blue by this method. A portion of the neutralized sample distillate (from procedure 4500-NH_3 B) is run through a manifold. The ammonium in the distillate reacts with solutions of disodium ethylenediaminetetraacetic acid (EDTA), sodium phenate,

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sodium hypochlorite, and sodium nitroprusside. The resulting indophenol blue is detected by colorimetry in a flow cell. Photometric measurement is made between the wavelengths of 630 and 660 nm.

Source: APHA, AWWA, and WEF. 2005. "Method 4500-NH_3 G: Nitrogen (Ammonia) Automated Phenate Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

5.2.79 Standard Method 4500-Cl G: DPD Colorimetric Method

Analyte(s)	CAS RN
Chlorine	7782-50-5

Analysis Purpose: Sample preparation and/or analyte determination and measurement
Sample Preparation Technique: Water samples are buffered and colorimetric agent is added. Buffered water extraction by Analyst, 1999. 124: 1853–1857 are used for preparation of air samples.
Determinative Technique: Visible spectrophotometry

Method Developed for: Chlorine in water and wastewater
Method Selected for: SAM lists this method for preparation and analysis of aqueous liquid and drinking water samples. It also should be used for analysis of air samples when appropriate sample preparation techniques have been applied.
Detection and Quantitation: The method can detect 10 µg/L chlorine.

Description of Method: A portion of aqueous liquid sample is buffered and reacted with N,N-diethyl-*p*-phenylenediamine (DPD) color agent. The resulting free chlorine is determined by colorimetry. If total chlorine (including chloroamines and nitrogen trichloride) is to be determined, potassium iodide crystals are added. Results for chromate and manganese are blank corrected using thioacetamide solution.

Special Considerations: Organic contaminants and strong oxidizers may cause interference.

Source: APHA, AWWA, and WEF. 2005. "Method 4500-Cl G: DPD Colorimetric Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

5.2.80 Literature Reference for Chlorine (Analyst, 1999. 124(12): 1853–1857)

Analyte(s)	CAS RN
Chlorine	7782-50-5

Analysis Purpose: Sample preparation
Sample Preparation Technique: Buffered water extraction
Determinative Technique: Visible spectrophotometry
Determinative Method: Standard Method 4500-Cl G

Method Developed for: Active chlorine in air
Method Selected for: SAM lists this procedure for preparation of air samples.
Detection and Quantitation: Detection limit of 0.1 µg of chlorine; the collection efficiency was >90%; recovery of chlorine spikes from 0.05-g aliquots of the sorbent was not quantitative (~60%) but was reproducible.

Description of Method: A procedure is described for determination of total combined gas-phase active chlorine (i.e., Cl_2, hypochlorous acid [HOCl], and chloramines) and is based on a sulfonamide-functionalized silica gel sorbent. For determination of the collected chlorine, a modified version of the

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DPD colorimetric procedure is used, which yielded a detection limit of 0.1 µg of chlorine. At flow rates ranging from 31 to 294 mL/min, the collection efficiency was >90% based on breakthrough analysis. Recovery of chlorine spikes from 0.05-g aliquots of the sorbent was not quantitative (~60%) but was reproducible; the recovery is accounted for in samples by adding weighed amounts of sorbent to the standards.

Source: Johnson, B.J., Emerson, D.W., Song, L., Floyd, J., and Tadepalli, B. 1999. "Determination of active chlorine in air by bonded phase sorbent collection and spectrophotometric analysis." Analyst. 124(12): 1853–1857. www.epa.gov/sam/pdfs/Analyst124_pg1853-1857.pdf

5.2.81 Literature Reference for Fluoroacetate salts (Analytical Letters, 1994. 27(14): 2703–2718)

Analyte(s)	CAS RN
Fluoroacetic acid and fluoroacetate salts	NA
Methyl fluoroacetate	453-18-9

Analysis Purpose: Sample preparation
Sample Preparation Technique: Ultrasonic extraction
Determinative Technique: IC-ECD
Determinative Method: EPA Method 300.1, Revision 1.0

Method Developed for: Sodium fluoroacetate in soil
Method Selected for: SAM lists this procedure for preparation of solid and wipe samples.

Description of Method: Sodium fluoroacetate is determined at sub-microgram per gram concentrations in small (~1 g) soil samples. Samples are ultrasonically extracted with water, filtered, and analyzed by Method 300.1.

Source: Tomkins, B.A. 1994. "Screening-Procedure for Sodium Fluoroacetate (Compound 1080) at Sub-Microgram/Gram Concentrations in Soils." Analytical Letters. 27(14): 2703–2718. http://www.informaworld.com/smpp/content~content=a747219004~db=all~order=page

5.2.82 Literature Reference for Methamidophos (Chromatographia. 2006. 63(5/6): 233–237)

Analyte(s)	CAS RN
Acephate	30560-19-1
Methamidophos	10265-92-6

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: SPE
Determinative Technique: LC-MS-MS

Method Developed for: Pesticides (methamidophos) in water samples
Method Selected for: SAM lists this procedure for preparation and analysis of aqueous liquid samples.
Detection and Quantitation: The limit of detection for this limit is 30 µg/L.

Description of Method: A multi-residue analytical method is described for monitoring polar pesticides, such as acephate and methamidophos, in water with SPE (solid-phase extraction) and LC-MS-MS.

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Samples are analyzed using a C_{18} analytical column (150 mm x 3.2 mm I.D., 5µm particle size) coupled with a C_{18} guard cartridge system (4 mm x 3.0 mm I.D.).

Source: Liu, F., Bischoff, G., Pestemer, W., Xu, W., and Kofoet, A. 2006. "Multi-residue Analysis of Some Polar Pesticides in Water Samples with SPE and LC/MS/MS." Chromatographia. 63(5/6): 233–237. http://www.epa.gov/sam/pdfs/Chromatographia-63_pg233-237.pdf

5.2.83 Literature Reference for Methamidophos (Journal of Chromatography A, 2007. 1154: 3–25)

Analyte(s)	CAS RN
Acephate	30560-19-1
Methamidophos	10265-92-6

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Solvent extraction
Determinative Technique: LC-MS-MS

Method Developed for: Pesticides (methamidophos) in crops
Method Selected for: SAM lists this procedure for preparation and analysis of solid, air, and wipe samples.
Detection and Quantitation: The limit of detection for this method is 0.01 mg/kg.

Description of Method: A liquid chromatography–tandem quadrupole mass spectrometry (LC-MS-MS) multi-residue method for the simultaneous target analysis of a wide range of pesticides and metabolites in fruit, vegetables and cereals is described. Gradient elution has been used in conjunction with positive mode electrospray ionization tandem mass spectrometry to detect up to 171 pesticides and/or metabolites in different crop matrices using a single chromatographic run. Pesticide residues are extracted/partitioned from the samples with acetone/dichloromethane/light petroleum. Samples are analyzed by LC-MS-MS using a C_{18} analytical column (150 mm x 3.2 mm I.D., 5µm particle size) coupled with a C_{18} guard cartridge system (4 mm x 3.0 mm I.D.).

Special Considerations: The procedure has been developed for the analysis of various pesticides (methamidophos) in crops using LC-MS-MS; modifications will be needed for application to environmental samples such as soils, wipes, and air samples collected on sorbent/filters.

Source: Hiemstra, M., de Kok, A. 2007. "Comprehensive Multi-residue Method for the Target Analysis of Pesticides in Crops Using Liquid Chromatography-tandem Mass Spectrometry." Journal of Chromatography A. 1154(1): 3–25. http://www.sciencedirect.com/science/journal/00219673

5.2.84 Literature Reference for Fluoroacetamide (Journal of Chromatography B, 2008. 876(1): 103–108)

Analyte(s)	CAS RN
Fluoroacetamide	640-19-7

Analysis Purpose: Sample preparation, and analyte determination and measurement
Sample Preparation Technique: Water extraction
Determinative Technique: GC/MS

Method Developed for: Fluoroacetamide and tetramine in blood, urine and stomach contents

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Method Selected for: SAM lists this procedure for preparation and analysis of solid, aqueous liquid, drinking water, air, and wipe samples.
Detection and Quantitation: The detection limit of this method for fluoroacetamide is 0.01 µg/g.

Description of Method: Samples are extracted by microscale liquid-liquid extraction using acetonitrile, ENVI-CARB, and sodium chloride. Samples are analyzed by GC/MS using a 30-m DB-5MS capillary column (or equivalent) coupled with a 1.5 m Innowax capillary column (or equivalent) by a quartz capillary column connector. If analyzing for fluoroacetamide alone, only the Innowax capillary column is needed.

Special Considerations: The procedure has been developed for the analysis of fluoroacetamide and tetramine in blood, urine and stomach fluid samples; modifications will be needed for application to environmental samples.

Source: Xu, X., Song, G., Zhu, Y., Zhang, J., Zhao, Y., Shen, H., Cai, Z., Han, J., and Ren, Y. 2008. "Simultaneous Determination of two Accute Poisoning Rodenticides Tetramine and Fluoroacetamide with a Coupled Column in Poisoning Cases." Journal of Chromatography B. 876(1): 103–108. http://www.sciencedirect.com/science/journal/15700232

5.2.85 Literature Reference for Sodium Azide (Journal of Forensic Sciences, 1998. 43(1): 200–202)

Analyte(s)	CAS RN
Sodium azide (analyze as azide ion)	26628-22-8

Analysis Purpose: Sample preparation
Sample Preparation Technique: Water extraction, filtration, and/or acidification
Determinative Technique: IC with conductivity detection
Determinative Method: EPA Method 300.1, Revision 1.0

Method Developed for: Sodium azide in blood
Method Selected for: SAM lists this procedure for preparation of solid, aqueous liquid, and drinking water samples.
Detection and Quantitation: This method can routinely quantify to at least 100 µg/L, and the detection limit is estimated to be 30 µg/L.

Description of Method: Samples are analyzed by IC using suppressed conductivity detection. Water extraction and filtration steps should be used for the preparation of solid samples. Filtration steps should be used for preparation of aqueous liquid and drinking water samples.

Special Considerations: The procedure described above has been developed for the analysis of sodium azide in blood samples.

Source: Kruszyna, R., Smith, R.P., and Kruszyna, H. 1998. "Determining Sodium Azide Concentration in the Blood by Ion Chromatography." Journal of Forensic Sciences. 43(1): 200–202. http://www.astm.org/JOURNALS/FORENSIC/PAGES/2933.htm

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Section 6.0: Selected Radiochemical Methods

A list of analytical methods to be used in analyzing environmental samples for radiochemical contaminants during homeland security events is provided in Appendix B. Methods are listed for each isotope and for each sample type that potentially may need to be measured and analyzed when responding to an environmental emergency.

Please note: This section provides guidance for selecting radiochemical methods that have a high likelihood of assuring analytical consistency when laboratories are faced with a large scale environmental restoration crisis. Not all methods have been verified for the analyte/sample type combination listed in Appendix B. Please refer to the specified method to identify analyte/sample type combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 4.

Appendix B is sorted alphabetically by analyte and includes the following information:

- **Analyte(s).** The radionuclide(s) or contaminant(s) of interest.
- **CAS RN.** A unique identifier for chemical substances that provides an unambiguous way to identify a chemical or molecular structure when there are many possible systematic, generic, or trivial names. In this section (Section 6.0) and Appendix B, the CAS RNs correspond to the specific radionuclide identified.
- **Determinative technique.** An analytical instrument or technique used for qualitative and confirmatory determination of compounds or components in a sample.
- **Drinking water sample methods.** The recommended methods/procedures for sample preparation and analysis to measure the analyte of interest in drinking water samples. Methods have been identified for qualitative and confirmatory determination.
- **Aqueous and liquid phase sample methods.** The recommended methods/procedures for sample preparation and analysis to measure the analyte of interest in aqueous and/or non-aqueous liquid phase samples. Methods have been identified for qualitative and confirmatory determination.
- **Soil and sediment phase sample methods.** The recommended methods/procedures for sample preparation and analysis to measure the analyte of interest in soil and sediment samples. Methods have been identified for qualitative and confirmatory determination.
- **Surface wipe sample methods.** The recommended methods/procedures for sample preparation and analysis to measure the analyte of interest in surface wipe samples. Methods have been identified for qualitative and confirmatory determination.
- **Air filter sample methods.** The recommended methods/procedures for sample preparation and analysis to measure the analyte of interest in air filter samples. Methods have been identified for qualitative and confirmatory determination.
- **Qualitative determination method identifier.** A unique identifier or number assigned to an analytical method by the method publisher. The identified method is intended to determine the presence of a radionuclide. These methods are less precise than confirmatory methods, and are used when greater sample throughput and more rapid reporting of results is required.
- **Confirmatory method identifier.** A unique identifier or number assigned to an analytical method by the method publisher. The identified method is for measurement of the activity from a particular radionuclide per unit of mass, volume, or area sampled.

Following a homeland security event, it is assumed that only those areas with contamination greater than pre-existing/naturally prevalent levels commonly found in the environment would be subject to remediation. Dependent on site- and event-specific goals, investigation of background levels using methods listed in Appendix B is recommended.

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6.1 General Guidelines

The guidelines summarized in this section provide a general overview of how to identify the appropriate radiochemical method(s) for a given analyte-sample type combination, as well as recommendations for QC procedures.

For additional information on the properties of the radionuclides listed in Appendix B, TOXNET (http://toxnet.nlm.nih.gov/index.html), a cluster of databases on toxicology, hazardous chemicals, and related areas maintained by the National Library of Medicine, is an excellent resource. EPA's Radiation Protection (http://www.epa.gov/radiation/radionuclides/index.html) and the *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP) (http://www.epa.gov/radiation/marlap/manual.html) Web sites provide some additional information pertaining to radionuclides of interest and selection of radiochemical methods. Emergency response documents recently developed by EPA's Office of Radiation and Indoor Air (ORIA) may be found at http://www.epa.gov/narel/incident_guides.html.

6.1.1 Standard Operating Procedures for Identifying Radiochemical Methods

To determine the appropriate method to be used on an environmental sample, locate the analyte of concern in Appendix B: Radiochemical Methods under the "Analyte Class" or "Analyte(s)" column. After locating the analyte of concern, continue across the table to identify the appropriate determinative technique (e.g., alpha spectrometry), then identify the appropriate qualitative and/or confirmatory method for the sample type of interest (drinking water, aqueous and liquid phase, soil and sediment, surface wipes, and air filters) for the particular analyte.

Sections 6.2.1 through 6.2.36, below, provide summaries of the qualitative and confirmatory methods listed in Appendix B. Once a method has been identified in Appendix B, **Table 6-1** can be used to locate the method summary.

Table 6-1. Radiochemical Methods and Corresponding Section Numbers

Analyte / Analyte Class	CAS RN	Method	Section
Gross Alpha	NA	900.0 (EPA)	6.2.2
		FRMAC, Vol 2, pg. 33 (DOE)	6.2.21
Gross Beta	NA	AP1 (ORISE)	6.2.23
		7110 B (SM)	6.2.30
Gamma Select Mixed Fission Products*	NA	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.16
Americium-241	14596-10-2	Am-01-RC (HASL-300)	6.2.13
		Am-02-RC (HASL-300)	6.2.14
		Am-04-RC (HASL-300)	6.2.15
		Pu-12-RC (HASL-300)	6.2.18
		AP11 (ORISE)	6.2.26
		D3084-05 (ASTM)	6.2.28

* Please note that this category does not cover all fission products.

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Analyte / Analyte Class	CAS RN	Method	Section
Californium-252	13981-17-4	Am-01-RC (HASL-300)	6.2.13
		Am-04-RC (HASL-300)	6.2.15
		Pu-12-RC (HASL-300)	6.2.18
		AP11 (ORISE)	6.2.26
		D3084-05 (ASTM)	6.2.28
Cesium-137	10045-97-3	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.16
Cobalt-60	10198-40-0	7120 (SM)	6.2.31
Curium-244	13981-15-2	Am-01-RC (HASL-300)	6.2.13
		Am-04-RC (HASL-300)	6.2.15
		Pu-12-RC (HASL-300)	6.2.18
		AP11 (ORISE)	6.2.26
		D3084-05 (ASTM)	6.2.28
Europium-154	15585-10-1	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.16
		7120 (SM)	6.2.31
Iodine-125	14158-31-7	Procedure #9 (ORISE)	6.2.27
Iodine-131	10043-66-0	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.16
Iridium-192	14694-69-0	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.16
		7120 (SM)	6.2.31
Molybdenum-99	14119-15-4	901.1 (EPA)	6.2.3
		Ga-01-R (HASL-300)	6.2.15
Phosphorus-32	14596-37-3	R4-73-014 (EPA)	6.2.11
		RESL P-2 (DOE)	6.2.22
Plutonium-238	13981-16-3	EMSL-33 (EPA)	6.2.10
		AP11 (ORISE)	6.2.26
Plutonium-239	15117-48-3	D3084-05 (ASTM)	6.2.28
Polonium-210	13981-52-7	Method 111 (EPA)	6.2.1
		Po-02-RC (HASL-300)	6.2.17
Radium-226	13982-63-3	903.0 (EPA)	6.2.4
		903.1 (EPA)	6.2.5
		EMSL-19 (EPA)	6.2.9
		D3084-05 (ASTM)	6.2.28
		7500-Ra B (SM)	6.2.32
		7500-Ra C (SM)	6.2.33
Ruthenium-103	13968-53-1	901.1 (EPA)	6.2.3
Ruthenium-106	13967-48-1	Ga-01-R (HASL-300)	6.2.16
Selenium-75	14265-71-5	7120 (SM)	6.2.31

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Analyte / Analyte Class	CAS RN	Method	Section
Strontium-89	14158-27-1	905.0 (EPA)	6.2.6
		Stronium in Food and Bioenvironmental Samples	6.2.12
Strontium-90	10098-97-2	905.0 (EPA)	6.2.6
		Sr-03-RC (HASL-300)	6.2.19
		7500-Sr B (SM)	6.2.34
Technetium-99	14133-76-7	Tc-02-RC (HASL-300)	6.2.20
		AP5 (ORISE)	6.2.25
Tritium (Hydrogen-3)	10028-17-8	906.0 (EPA)	6.2.7
		AP2 (ORISE)	6.2.24
Uranium-234 Uranium-235 Uranium-238	13966-29-5 15117-96-1 7440-61-1	908.0 (EPA)	6.2.8
		EMSL-33 (EPA)	6.2.10
		AP11 (ORISE)	6.2.26
		D3084-05 (ASTM)	6.2.28
		D3972-02 (ASTM)	6.2.29
		7500-U B (SM)	6.2.35
		7500-U C (SM)	6.2.36

The method summaries are listed in order of method selection hierarchy (see Figure 2-1), starting with EPA methods, followed by methods from other federal agencies and VCSBs. Methods are listed in numerical order under each publisher. Where available, a direct link to the full text of the selected analytical method is provided in the method summary. For additional information regarding sample preparation and analysis procedures and on methods available through consensus standards organizations, please use the contact information provided in **Table 6-2**.

Table 6-2 Souces of Radiochemical Methods

Name	Publisher	Reference
NEMI	EPA, USGS	http://www.nemi.gov
CFR Promulgated Test Methods	EPA, Technical Transfer Network (TTN) Emission Measurement Center (EMC)	http://www.epa.gov/ttn/emc/promgate.html
Prescribed Procedures for Measurement of Radioactivity in Drinking Water (EPA-600 4-80-032, August 1980)	EPA, ORD, Environmental Monitoring and Support Laboratory (EMSL)	http://www.sld.state.nm.us/Documents/forewd.pdf Also available from National Technical Information Service (NTIS)*, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161, (703) 605-6000.
Radiochemical Analytical Procedures for Analysis of Environmental Samples, March 1979. EMSL-LV-0539-17	EPA, EMSL	Available NTIS*, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161, (703) 605-6000.

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Name	Publisher	Reference
EML Procedures Manual, Health and Safety Laboratory (HASL-300), 28th Edition, February, 1997	Department of Energy (DOE), Environmental Measurements Laboratory (EML) / Now DHS	http://www.eml.st.dhs.gov/publications/procman.cfm Also available from NTIS*, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161, (703) 605-6000.
Federal Radiological Monitoring and Assessment Center (FRMAC) Laboratory Manual	DOE, National Nuclear Security Administration (NNSA)	http://www.nv.doe.gov/nationalsecurity/homelandsecurity/frmac/manuals.aspx
Radiological and Environmental Sciences Laboratory (RESL) Analytical Chemistry Branch Procedures Manual	DOE, RESL	Available from NTIS, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161, (703) 605-6000.
Oak Ridge Institute for Science and Education (ORISE) Laboratory Procedures Manual	ORISE, Independent Environmental Assessment and Verification	http://orise.orau.gov/ieav/survey-projects/lab-manual.htm
Annual Book of ASTM Standards, Vol. 11.02*	ASTM International	http://www.astm.org
Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005*	APHA, AWWA, and WEF	http://www.standardmethods.org

* Subscription and/or purchase required.

6.1.2 General QC Guidelines for Radiochemical Methods

Having data of known and documented quality is critical so that public officials can accurately assess the activities that may be needed in remediating a site. Having such data requires that laboratories: (1) conduct the necessary QC to ensure that measurement systems are in control and operating correctly; (2) properly document results of the analyses; and (3) properly document measurement system evaluation of the analysis-specific QC. Ensuring data quality also requires that laboratory results are properly evaluated and the results of the data quality evaluation are transmitted to decision makers.

The level or amount of QC needed often depends on the intended purpose of the data that are generated. Various levels of QC may be required if the data are generated during contaminant presence/absence qualitative determinations versus confirmatory analyses. The specific needs for data generation should be identified. QC requirements and data quality objectives should be derived based on those needs, and should be applied consistently across laboratories when multiple laboratories are used. For example, during rapid sample screening analyses, minimal QC samples (e.g., blanks, duplicates) and documentation might be required to ensure data quality. Implementation of the analytical methods for evaluation of environmental samples during site assessment through site clearance, such as those identified in this document, might require increased QC.

Some method-specific QC requirements are described in many of the individual methods that are cited in this manual. QC requirements will be referenced in SAPs developed to address specific analytes and sample types of concern. Additional information regarding QC requirements specific to radiochemical methods is included in the MARLAP manual at: http://www.epa.gov/radiation/marlap/manual.html. Individual methods, sampling and analysis protocols, or contractual statements of work also should be consulted to determine any additional QC that may be needed.

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QC samples are required to assess the precision, bias, and reliability of sample results. All QC results are tracked on control charts and reviewed for acceptability and trends in analysis or instrument operation. QC parameters are measured as required per method at the prescribed frequency. QC of laboratory analyses using radiochemical methods includes ongoing analysis of QC samples and tracking QC parameters including, but not limited to the following:

- Method blanks;
- Calibration checks;
- Sample and sample duplicates;
- Laboratory control sample recoveries;
- MS/MSD recoveries; and
- Tracer and/or carrier yield.

Please note: The appropriate point of contact identified in Section 4 should be consulted regarding appropriate QA/QC procedures prior to sample analysis. These contacts will consult with the EPA coordinator responsible for laboratory activities during the specific event to ensure QA/QC procedures are performed consistently across laboratories. EPA program offices will be responsible for ensuring that the QA/QC practices are implemented.

6.1.3 Safety and Waste Management

It is imperative that safety precautions be used during collection, processing, and analysis of environmental samples. Laboratories should have a documented health and safety plan for handling samples that may contain target CBR contaminants, and laboratory staff should be trained in and implement the safety procedures included in the plan. In addition, many of the methods summarized or cited in Section 6.2 contain specific requirements, guidelines, or information regarding safety precautions that should be followed when handling or processing environmental samples and reagents. These methods may also provide information regarding waste management. Laboratories should consult with the responsible government agencies prior to disposal of waste materials. Other resources that can be consulted for additional information include the following:

- OSHA - 29 CFR part 1910.1450. Occupational Exposure to Hazardous Chemicals in Laboratories. http://www.access.gpo.gov/nara/cfr/waisidx_06/29cfr1910a_06.html
- EPA - 40 CFR part 260. Hazardous Waste Management System: General. http://www.access.gpo.gov/nara/cfr/waisidx_07/40cfr260_07.html
- EPA - 40 CFR part 270. EPA Administered Permit Programs: The Hazardous Waste Permit Program. http://www.access.gpo.gov/nara/cfr/waisidx_07/40cfr270_07.html
- NRC - 10 CFR part 20. Standards for Protection Against Radiation http://www.access.gpo.gov/nara/cfr/waisidx_00/10cfr20_00.html
- DOE. Order O 435.1: Radioactive Waste Management. July 1, 1999. Available at: www.directives.doe.gov/pdfs/doe/doetext/neword/435/o4351.html
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OUTDATED

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6.2 Method Summaries

Summaries for the analytical methods listed in Appendix B are provided in Sections 6.2.1 through 6.2.36. These summaries contain information that has been extracted from the selected methods. Each method summary contains a table identifying the contaminants in Appendix B to which the method applies, a brief description of the analytical method, and a link to the full version of the method or a source for obtaining a full version of the method. The full version of the method should be consulted prior to sample analysis.

6.2.1 EPA Method 111: Determination of Polonium-210 Emissions from Stationary Sources

Analyte(s)	CAS RN
Polonium-210	13981-52-7

Analysis Purpose: Qualitative and confirmatory determination
Determinative Technique: Alpha spectrometry

Method Developed for: Polonium-210 in particulate matter samples collected from stationary source exhaust stacks
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of surface wipes and air filters.

Description of Method: This method covers the determination of polonium-210 in particulate matter samples collected from stationary sources such as exhaust stacks. Polonium-210 in the sample is put in solution, deposited on a metal disc, and the radioactive disintegration rate measured. Polonium in acid solution spontaneously deposits on surface metals that are more electropositive than polonium. Po-209 tracers should be added to determine the chemical yield.

OUTDATED

Source: EPA EMC, prepared by the Office of Air Quality Planning and Standards (OAQPS). 2000. "Method 111: Determination of Polonium-210 Emissions from Stationary Sources." http://www.epa.gov/sam/pdfs/EPA-111.pdf

6.2.2 EPA Method 900.0: Gross Alpha and Gross Beta Radioactivity in Drinking Water

Analysis Purpose: Gross alpha and gross beta determination
Determinative Technique: Alpha/Beta counting

Method Developed for: Gross alpha and gross beta particle activities in drinking water
Method Selected for: SAM lists this method for gross alpha and gross beta determination in drinking water samples.

Description of Method: The method provides an indication of the presence of alpha and beta emitters, including the following SAM analytes:

- Americium-241 (CAS RN 14596-10-2) Alpha emitter
- Californium-252 (CAS RN 13981-17-4) Alpha emitter
- Cesium-137 (CAS RN 10045-97-3) Beta emitter
- Cobalt-60 (CAS RN 10198-40-0) Beta emitter
- Curium-244 (CAS RN 13981-15-2) Alpha emitter
- Europium-154 (CAS RN 15585-10-1) Beta emitter
- Iridium-192 (CAS RN 14694-69-0) Beta emitter
- Plutonium-238 (CAS RN 13981-16-3) Alpha emitter
- Plutonium-239 (CAS RN 15117-48-3) Alpha emitter
- Polonium-210 (CAS RN 13981-52-7) Alpha emitter
- Radium-226 (CAS RN 13982-63-3) Alpha emitter
- Ruthenium-103 (CAS RN 13968-53-1) Beta emitter
- Ruthenium-106 (CAS RN 13967-48-1) Beta emitter
- Strontium-90 (CAS RN 10098-97-2) Beta emitter
- Uranium-234 (CAS RN 13966-29-5) Alpha emitter
- Uranium-235 (CAS RN 15117-96-1) Alpha emitter
- Uranium-238 (CAS RN 7440-16-1) Alpha emitter

An aliquot of a preserved drinking water sample is evaporated to a small volume (3 to 5 mL) and transferred quantitatively to a tarred 2-inch planchet. The aliquot volume is determined based on a maximum total solids content of 100 mg. The sample aliquot is evaporated to dryness in the planchet to a constant weight, cooled, and counted using a gas proportional or scintillation counting system. The counting system is calibrated with thorium-230 for gross alpha, and with strontium-90 for gross beta analysis[3]. A traceable standards-based efficiency curve must be developed for each calibration nuclide (Th-230 and Sr-90) based on a range of total solids content in the 2-inch planchet from 0 to 100 mg (see method for specific recommendations and requirements for the use of cesium-137).

Special Considerations: Long counting time and increased sample size may be required to meet detection limits. Sensitivity is limited by the concentration of solids in the sample. The method provides an overall measure of alpha and beta activity, including activity for the radionuclides listed above, but does not permit the specific identification of any alpha or beta emitting radionuclides.

Source: EPA, EMSL. 1980. "Method 900.0: Gross Alpha and Gross Beta Radioactivity in Drinking Water." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-900.0.pdf

[3] EPA lists standards for use when analyzing drinking water in the table at 40 CFR 141.25 (footnote 11).

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6.2.3 EPA Method 901.1: Gamma Emitting Radionuclides in Drinking Water

Analyte(s)	CAS RN
Cesium-137	10045-97-3
Cobalt-60	10198-40-0
Europium-154	15585-10-1
Iodine-131	10043-66-0
Iridium-192	14694-69-0
Molybdenum-99	14119-15-4
Ruthenium-103	13968-53-1
Ruthenium-106	13967-48-1
Selenium-75	14265-71-5

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Gamma spectrometry

Method Developed for: Gamma emitting radionuclides in drinking water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of select gamma emitters in drinking water samples.

Description of Method: This method is applicable for analysis of water samples that contain radionuclides that emit gamma photons with energies ranging from approximately 60 to 2000 keV. The method uses gamma spectroscopy for measurement of gamma photons emitted from radionuclides without separating them from the sample matrix. A homogeneous aliquot of water is placed into a standard geometry (normally a Marinelli beaker) for gamma counting, typically using a high purity germanium (HP(Ge)) detector. Detectors such as Germanium (Lithium) (Ge(Li)) or thallium-activated sodium iodide (NaI(Tl)) also can be used. Sample aliquots are counted long enough to meet the required sensitivity of measurement. To reduce adsorbance of radionuclides on the walls of the counting container, the sample is acidified at collection time. Due to its lower resolution, significant interference can occur using the NaI(Tl) detector when counting a sample containing radionuclides that emit gamma photons of similar energies. When using this method, shielding is needed to reduce background interference. Detection limits are, in general, dependent on analyte radionuclide gamma-ray abundance, sample volume, geometry (physical shape), and counting time.

Source: EPA, EMSL. 1980. "Method 901.1: Gamma Emitting Radionuclides in Drinking Water." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-901.1.pdf

6.2.4 EPA Method 903.0: Alpha-Emitting Radium Isotopes in Drinking Water

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Qualitative determination
Determinative Technique: Alpha counting

Method Developed for: Total soluble alpha emitting isotopes of radium, namely radium-223, radium-224 and radium-226 in drinking water
Method Selected for: SAM lists this method for qualitative determination in drinking water samples.

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Description of Method: This method covers measurement of the total soluble alpha emitting isotopes of radium, namely radium-223, radium-224 and radium-226 in drinking water. The method does not give an accurate measurement of radium-226 content in the sample when other alpha emitters are present. If radium-223 and radium-224 are present, the results can be used to provide a gross determination of radium-226. When the total radium alpha activity of a drinking water sample is greater than 5 pCi/L, use of Method 903.1 (Radium-226 in Drinking Water) is preferred. Radium in the water sample is collected by coprecipitation with barium and lead sulfate, and purified by re-precipitation from EDTA solution. Citric acid is added to ensure that complete interchange occurs before the first precipitation step. The final barium sulfate precipitate is alpha counted to determine the total disintegration rate of the radium isotopes. By making a correction for the ingrowth of radon and its alpha emitting progeny for the elapsed time after separation, one can determine radium activity in the sample. Presence of significant natural barium in the sample can result in a falsely high yield. Based on a 1000-mL sample and 100-minute counting time, the minimum detectable level for this method is 0.5 pCi/L.

Source: EPA, EMSL. 1980. "Method 903.0: Alpha-Emitting Radium Isotopes in Drinking Water." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-903.0.pdf

6.2.5 EPA Method 903.1: Radium-226 in Drinking Water – Radon Emanation Technique

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha counting

Method Developed for: Radium-226 in drinking water
Method Selected for: SAM lists this method for confirmatory analysis of drinking water samples.

Description of Method: This method is specific for radium-226, and is based on the emanation and scintillation counting of radon-222, the immediate decay product of radium-226. Radium-226 is concentrated and separated from the water sample by coprecipitation on barium sulfate. The precipitate is dissolved in EDTA reagent, placed in a sealed bubbler and stored for ingrowth of radon-222. After ingrowth, the radon-222 gas is purged into a scintillation cell. When the short-lived radon-222 daughters are in equilibrium with the parent (after ~4h), the scintillation cell is counted for activity. The absolute measurement of radium-226 is effected by calibrating the scintillation cell system with a standard solution of the nuclide. There are no radioactive interferences in this method. Based on a 1000-mL sample and 100-minute counting time, the minimum detectable level for this method is 0.5 pCi/L.

Source: EPA, EMSL. 1980. "Method 903.1: Radium-226 in Drinking Water – Radon Emanation Technique." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-903.1.pdf

6.2.6 EPA Method 905.0: Radioactive Strontium in Drinking Water

Analyte(s)	CAS RN
Strontium-89	14158-27-1
Strontium-90	10098-97-2

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Beta counting

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Method Developed for: Strontium-89, strontium-90, and total strontium in drinking water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of aqueous/liquid and drinking water samples for strontium-89 and analysis of drinking water samples for strontium-90.

Description of Method: Stable strontium carrier is added to the water sample. Both strontium-89 and strontium-90 are precipitated from the solution as insoluble carbonates. Interferences from calcium and from some radionuclides are removed by one or more precipitations of the strontium carrier as strontium nitrate. Barium and radium are removed by precipitation as chromates. The yttrium-90 decay product of strontium-90 is removed by a hydroxide precipitation step. The separated strontium-89 and strontium-90 are precipitated as carbonates, weighed for determination of the chemical recovery, and counted for beta particle activity. The counting result, ascertained immediately after separation, represents the total strontium activity (strontium-89 and strontium-90) plus an insignificant fraction of the yttrium-90 that has grown into the separated strontium-90. The yttrium-90 decay product is allowed to in-grow for approximately two weeks and then is separated with stable yttrium carrier as hydroxide and finally precipitated as the oxalate, weighed for chemical recovery, and mounted for beta counting. The strontium-90 concentration is determined from the yttrium-90 activity; strontium-89 concentration is determined from the difference.

Source: EPA, EMSL. 1980. "Method 905.0: Radioactive Strontium in Drinking Water, *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-905.0.pdf

6.2.7 EPA Method 906.0: Tritium in Drinking Water

Analyte(s)	CAS RN
Tritium (Hydrogen-3)	10028-17-8

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Liquid scintillation

Method Developed for: Tritium (as T_2O or HTO) in drinking water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of drinking water and aqueous/liquid phase samples.

Description of Method: An unpreserved 100-mL aliquot of a drinking water sample is distilled after adjusting pH with a small amount of sodium hydroxide and adding potassium permanganate. The alkaline treatment prevents other radionuclides, such as radioiodine and radiocarbon, from distilling with the tritium. The permanganate treatment oxidizes trace organics that may be present in the sample and prevents their appearance in the distillate. To determine the concentration of tritium, the middle fraction of the distillate is used, because the early and late fractions are more apt to contain materials interfering with the liquid scintillation counting process. A portion of this collected fraction is added to a liquid scintillator cocktail, and the solution is mixed, dark adapted and counted for beta particle activity. The efficiency of the system can be determined by the use of prepared tritiated water standards having the same density and color as the sample.

Source: EPA, EMSL. 1980. "Method 906.0: Tritium in Drinking Water." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-906.0.pdf

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6.2.8 EPA Method 908.0: Uranium in Drinking Water – Radiochemical Method

Analyte(s)	CAS RN
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Qualitative determination
Determinative Technique: Alpha counting

Method Developed for: Total uranium alpha particle activity in drinking water
Method Selected for: SAM lists this method for qualitative determination in drinking water samples.

Description of Method: This method measures total uranium alpha activity of a sample, without doing an isotopic uranium analysis. The sample is acidified with hydrochloric acid and boiled to eliminate carbonate and bicarbonate ions. Uranium is coprecipitated with ferric hydroxide and separated from the sample. The uranium is then separated from other radionuclides that were carried down with the ferric hydroxide by dissolving the hydroxide precipitate in hydrochloric acid, putting the solution through an anion exchange column, washing the column with hydrochloric acid, and finally eluting the uranium with hydrochloric acid. The uranium eluate is evaporated and the uranium chemical form is converted to nitrate. The residue is transferred to a stainless steel planchet, dried, flamed, and counted for alpha particle activity. Since uranium is a naturally occurring radionuclide, reagents must be checked for uranium contamination by analyzing a complete reagent blank by the same procedure as used for the samples. Based on a 1000- mL sample and 100-minute counting time in a single laboratory study, the minimum detectable level for this method is 1.0 pCi/L.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: EPA, EMSL. 1980. "Method 908.0: Uranium in Drinking Water – Radiochemical Method." *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA/600/4/80/032. http://www.epa.gov/sam/pdfs/EPA-908.0.pdf

6.2.9 EPA Method EMSL-19: Determination of Radium-226 and Radium-228 in Water, Soil, Air and Biological Tissue

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha counting

Method Developed for: Radium-226 and radium-228 in water, soil, air, biological tissues, and biological fluids
Method Selected for: SAM lists this method for confirmatory analysis of soil/sediment, surface wipe, and air filter samples.

Description of Method: Following acid digestion and filtration of soil, sediment, surface wipe, or air filter samples, radium is precipitated with barium sulfate. Barium-radium-sulfate is dissolved in a pentasodium diethylenetriamine-pentaacetate (DTPA) solution and transferred to an emanation tube. The radon is allowed to come to equilibrium for approximately 30 days. Radium-226 decays by alpha

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emission to radon-222. Radon-222 is separated and collected from the liquid by a de-emanation technique. The radon is counted by alpha scintillation 4.5 hours after de-emanation, at which time the short-lived progeny have reached 97% of equilibrium. An applicable measurement range has not been determined; however, samples that contain 0.1 pCi of Radium-226 have been analyzed.

Source: EPA, EMSL. 1979. "EMSL-19: Determination of Radium-226 and Radium-228 in Water, Soil, Air and Biological Tissue." *Radiochemical Analytical Procedures for Analysis of Environmental Samples*. http://www.epa.gov/sam/pdfs/EPA-EMSL-19.pdf

6.2.10 EPA Method EMSL-33: Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue

Analyte(s)	CAS RN
Plutonium-238	13981-16-3
Plutonium-239	15117-48-3
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Isotopic plutonium, uranium, and thorium, together or individually, in soil, water, air filters, urine, or ashed residues of vegetation, animal tissues, and bone
Method Selected for: SAM lists this method for confirmatory analysis of drinking water, aqueous/liquid, soil/sediment, surface wipe, and/or air filter samples.

Description of Method: This method is appropriate for the analysis of isotopic plutonium, uranium, and thorium, together or individually, by alpha spectrometry. Plutonium-236, uranium-232, and thorium-234 tracer standards are added for the determination of chemical yields. Samples are decomposed by nitric-hydrofluoric acid digestion or ignition to assure that all of the plutonium is dissolved and chemically separated from the sample by coprecipitation with sodium and ammonium hydroxide, anion exchange, and electrodeposition. The residues are dissolved in dilute nitric acid and successive sodium and ammonium hydroxide precipitations are performed in the presence of boric acid to remove fluoride and soluble salts. The hydroxide precipitate is dissolved, the solution is pH-adjusted with hydrochloric acid, and plutonium and uranium are adsorbed on an anion exchange column, separating them from thorium. Plutonium is eluted with hydrobromic acid. The actinides are electrodeposited on stainless steel discs from an ammonium sulfate solution and subsequently counted by alpha spectrometry. This method is designed to detect environmental levels of activity as low as 0.02 pCi per sample. To avoid possible cross-contamination, sample aliquot activities should be limited to 25 pCi or less.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: EPA, EMSL. 1979. "EMSL-33: Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue." *Radiochemical Analytical Procedures for Analysis of Environmental Sample*. http://www.epa.gov/sam/pdfs/EPA-EMSL-33.pdf

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6.2.11 EPA Method R4-73-014: Radioactive Phosphorus

Analyte(s)	CAS RN
Phosphorus-32	14596-37-3

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Cerenkov counting with Liquid Scintillation counter

Method Developed for: Phosporus-32 in nuclear reactor solutions
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of water samples.

Description of Method: 200 mL or less of a water sample is acidified with nitric acid and carriers of phosphorus (standardized), cobalt, zirconium, silver, and manganese are added. Hydroxides are precipitated by the addition of hydrogen peroxide and potassium hydroxide, and the hot solution is filtered through filter paper. Carriers of cobalt and zirconium are added to the filtrate, and the hydroxides are precipitated by the addition of hydrogen peroxide and potassium hydroxide. The solution is filtered and the hydroxides are discarded. The filtrate is acidified with hydrochloric acid, and phosphorous is precipitated as magnesium ammonium phosphate by the addition of a magnesium mixture and ammonium hydroxide. The magnesium ammonium phosphate is collected on a tared filter, dried, and weighed to determine the chemical yield. The precipitate is mounted and beta counted with a gas-flow proportional counter.

Source: EPA, EMSL. 1980. "Method R4-73-14: Radioactive Phosphorus." *Prescribed Procedures for Radiochemical Analysis of Nuclear Reactor Solutions.*

6.2.12 EPA Method: Determination of Radiostrontium in Food and Bioenvironmental Samples

Analyte(s)	CAS RN
Strontium-89	14158-27-1

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Low background alpha/beta counter

Method Developed for: Strontium-89 and strontium-90 in food, vegetation, and tissue samples
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of soil, sediment, wipes, and air filters.

Description of Method: This method is use for the determination of strontium-89 and strontium-90 in various bio-environmental samples. Ten grams or less of the sample is placed in a nickel crucible. Barium and strontium (standardized) carriers are added to the sample. Sodium hydroxide pellets and anhydrous sodium carbonate are added and mixed, and the sample is fused as a carbonate. The strontium-calcium carbonates are dissolved in hydrochloric acid, complexed with di-sodium EDTA and passed through a cation column where the strontium is absorbed, and the complexed calcium passes through. The strontium is eluted from the column and precipitated as a carbonate. The strontium carbonate is weighed and mounted on a planchet for beta counting with a low background gas-flow alpha beta counter. The chemical yield is determined gravimetrically, using calculations provided in the method.

Special Considerations: This method was developed for analysis of food, vegetation, and tissue. Additional laboratory development and testing is necessary for application to soil, sediment, air filters, and wipes.

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Source: EPA, National Environmental Research Center. 1975. "Determination of Radiostrontium in Food and Bioenvironmental Samples." *Handbook of Radiochemical Methods*, EPA-680/4-75-001.

6.2.13 EML HASL-300 Method Am-01-RC: Americium in Soil

Analyte(s)	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Curium-244	13981-15-2

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Americium in soil
Method Selected for: SAM lists this method for confirmatory analysis of soil/sediment samples.

Description of Method: This method uses alpha spectrometry for determination of americium-241 in soil, and also can be applied for determination of californium. Americium is leached from soil with nitric acid and hydrochloric acid. Americium-243 is added as a tracer to determine chemical yield. The soil is processed through the plutonium separation steps using ion exchange resin according to Method Pu-11-RC. Americium is collected with a calcium oxalate precipitation and finally isolated and purified by ion exchange. Californium-252 and curium-244 are eluted with americium as americium is stripped off the column. After source preparation by microprecipitation, americium-241, californium-252, and curium-244 are determined by alpha spectrometry analysis. The counting period chosen depends on the sensitivity required of the measurement and the degree of uncertainty in the result that is acceptable. The lower limit of detection (LLD) for americium-241 is 0.5 mBq when counted for 1000 minutes. In cases where less than 100 g of sample is available, use of Pu-12-RC is recommended.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Am-01-RC: Americium in Soil." *EML Procedures Manual*, HASL-300, 28th Edition.
http://www.epa.gov/sam/pdfs/EML-Am-01-RC.pdf

6.2.14 EML HASL-300 Method Am-02-RC: Americium-241 in Soil-Gamma Spectrometry

Analyte(s)	CAS RN
Americium-241	14596-10-2

Analysis Purpose: Qualitative determination
Determinative Technique: Gamma spectrometry

Method Developed for: Americium-241 in large volume soil samples
Method Selected for: SAM lists this method for qualitative determination in soil/sediment samples.

Description of Method: This method uses gamma spectrometry for determination of americium-241 in soil. Americium-241 decays with the emission of a gamma ray at 59.5 keV with a decay frequency (abundance or yield) of 35.9%. The sample is placed into an appropriately sized standard geometry (normally a Marinelli beaker) after drying and grinding the sample for homogenization. Gamma-ray

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attenuation corrections are required if the calibration source and the sample are in a different matrix or are of different densities. The LLD for 600 to 800 g of soil in a Marinelli beaker is 0.74 mBq for a 1000-minute count.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Am-02-RC: Americium-241 in Soil-Gamma Spectrometry." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Am-02-RC.pdf

6.2.15 EML HASL-300 Method Am-04-RC: Americium in QAP Water and Air Filters - Eichrom's TRU Resin

Analyte(s)	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Curium-244	13981-15-2

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Americium (but not lanthanides) in water and air filters
Method Selected for: SAM lists this method for confirmatory analysis of drinking water, aqueous/liquid samples, surface wipes, and air filters.

Description of Method: This method is specific to measurement of americium isotopes in samples that do not contain lanthanides, but also can be used for measurement of californium and curium. The method uses microprecipitation and determination by alpha spectrometry. Americium-243 is added to the sample to determine chemical yield. The sample is processed through separation steps using ion exchange resins. The eluate from the ion exchange column containing americium (and all other ions, except plutonium) is evaporated, redissolved, and loaded onto a Transuranic (TRU) Resin extraction column. Americium (and curium and californium, if present) is separated and purified on the column and finally stripped with dilute nitric acid stripping solution. Microprecipitation is used to prepare for alpha spectrometry. The method involves sample preparation steps from EML HASL-300 Method Pu-10-RC for water samples. The LLD for total americium is 0.3 mBq when counted for 1000 minutes.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Am-04-RC: Americium in QAP Water and Air Filters - Eichrom's TRU Resin." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Am-04-RC.pdf

6.2.16 EML HASL-300 Method Ga-01-R: Gamma Radioassay

Analyte(s)	CAS RN
Cesium-137	10045-97-3
Cobalt-60	10198-40-0
Europium-154	15585-10-1
Iodine-131	10043-66-0
Iridium-192	14694-69-0

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Analyte(s)	CAS RN
Molybdenum-99	14119-15-4
Ruthenium-103	13968-53-1
Ruthenium-106	13967-48-1
Selenium-75	14265-71-5

Analysis Purpose: Qualitative and confirmatory analysis or gross gamma determination
Determinative Technique: Gamma spectrometry

Method Developed for: Gamma-ray emitting radionuclides in a variety of environmental matrices
Method Selected for: SAM lists this method for qualitative and/or confirmatory analysis of select gamma emitters in aqueous/liquid, soil/sediment, surface wipes, and/or air filter samples.

Description of Method: This method uses gamma spectrometry for the measurement of gamma photons emitted from radionuclides without separating them from the sample matrix. Samples are placed into a standard geometry for gamma counting, typically using an HP(Ge) detector. Detectors such as Ge(Li) or NaI(Tl) also can be used. The sample is placed into a standard geometry for gamma counting. Soil samples and sludge are placed into an appropriately sized Marinelli beaker after drying and grinding the sample for homogenization. Air filters and surface wipes can be counted directly or pressed into a planchet and counted. Samples are counted long enough to meet the required sensitivity of measurement. For typical counting systems and sample types, activity levels of approximately 40 Bq are measured, and sensitivities as low as 0.002 Bq can be achieved for many nuclides. Because of electronic limitations, count rates higher than 2000 counts per second (cps) should be avoided. High activity samples may be diluted, reduced in size, or moved away from the detector (a limited distance) to reduce the count rate and allow for analysis. The method is applicable for analysis of samples that contain radionuclides emitting gamma photons with energies above approximately 20 keV for germanium (Ge) (both HP(Ge) and GeLi) detectors and above 50 keV for NaI(Tl) detectors.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Ga-01-R: Gamma Radioassay." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Ga-01-R.pdf

6.2.17 EML HASL-300 Method Po-02-RC: Polonium in Water, Vegetation, Soil, and Air Filters

Analyte(s)	CAS RN
Polonium-210	1-3981-52-7

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Polonium in water, vegetation, soil, and air filters
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of drinking water, aqueous/liquid, and soil/sediment samples.

Description of Method: This method uses alpha spectrometry for determination of polonium in water, vegetation, soil, and air filter samples. Polonium equilibrated with Po-208 or Po-209 tracer is isolated from most other elements by coprecipitation with lead sulfide. The sulfide precipitate is dissolved in weak hydrochloric acid solution. Polonium is quantitatively deposited on a nickel disc, and the plated disc is counted on an alpha spectrometer to measure chemical yield and activity of the sample. The

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solution from the deposition may be retained and analyzed for Pb-210. When counted for 1000 minutes, the LLD for polonium is 1.0 mBq for water and 1.3 mBq for vegetation, soil and filters.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Po-02-RC: Polonium in Water, Vegetation, Soil, and Air Filters." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Po-02-RC.pdf

6.2.18 EML HASL-300 Method Pu-12-RC: Plutonium and/or Americium in Soil or Sediments

Analyte(s)	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Curium-244	13981-15-2

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Plutonium and americium in soil
Method Selected for: This method is listed in SAM for use when small soil and sediment sample sizes (≤100 g) will be analyzed.

Description of Method: A sample of soil of up to 100 g in size is equilibrated with Am-243 tracer. Contaminant isotopes are leached with nitric and hydrochloric acid. Plutonium is removed by ion exchange. The eluent from the plutonium separation is saved for determination of americium, curium, and californium. Americium, curium, and californium are collected with a calcium oxalate coprecipitation, isolated and purified by extraction chromatography. Microprecipitation is used to prepare the sample for analysis by alpha spectrometry of americium, curium, and californium. The LLD for americium is 0.5 mBq when counted for 1000 minutes.

Special Considerations: In cases where only small sample sizes (≤100 g) will be analyzed, this method is recommended for confirmatory analysis. If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Pu-12-RC: Plutonium and/or Americium in Soil or Sediments." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Pu-12-RC.pdf

6.2.19 EML HASL-300 Method Sr-03-RC: Strontium-90 in Environmental Samples

Analyte(s)	CAS RN
Strontium-90	10098-97-2

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Beta counting

Method Developed for: Strontium-90 in vegetation, water, air filters and soil
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of soil/sediment, surface wipe, and air filter samples.

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Description of Method: Strontium is separated from calcium, other fission products, and natural radioactive elements. Fuming nitric acid separations remove the calcium and most other interfering ions. Radium, lead and barium are removed with barium chromate. Traces of other fission products are scavenged with iron hydroxide. After strontium-90 and yttrium-90 equilibrium has been attained, yttrium-90 is precipitated as the hydroxide and converted to oxalate for counting on a low-background gas proportional beta counter. Chemical yield is determined with strontium-85 tracer by counting in a gamma well detector.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Sr-03-RC: Strontium-90 in Environmental Samples." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Sr-03-RC.pdf

6.2.20 EML HASL-300 Method Tc-02-RC: Technetium-99 in Water – TEVA® Resin

Analyte(s)	CAS RN
Technetium-99	14133-76-7

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Liquid scintillation

Method Developed for: Technetium-99 (Tc-99) in water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of drinking water and aqueous/liquid phase samples.

Description of Method: The sample containing Tc-99 is mixed with Technetium-95m (Tc-95m) added as a gamma-emitting tracer. The two isotopes of technetium are brought to an isotopic equilibrium and separated from other elements by ferrous and ferric hydroxide coprecipitation. The precipitate is dissolved with dilute nitric acid and passed through a commercially available resin column (TEVA® Resin) which is highly specific for technetium in the pertechnetate form. The resin is washed with dilute nitric acid to remove possible interferences and then it is extruded directly into a suitable liquid scintillation cocktail. The sample is typically counted for 1 hour to simultaneously determine Tc-99 activity and the Tc-95m radiochemical yield. Quench/efficiency calibration curves need to be established for the liquid scintillation spectrometer for both Tc-95m and Tc-99.

Source: EML, DOE (EML is currently part of the DHS). 1997. "HASL-300 Method Tc-02-RC: Technetium-99 in Water – TEVA® Resin." *EML Procedures Manual*, HASL-300, 28th Edition. http://www.epa.gov/sam/pdfs/EML-Tc-02-RC.pdf

6.2.21 DOE FRMAC Method Volume 2, Page 33: Gross Alpha and Beta in Air

Analysis Purpose: Gross alpha and gross beta determination
Determinative Technique: Alpha/Beta counting

Method Developed for: Gross alpha and beta in air
Method Selected for: SAM lists this method for gross alpha and gross beta determination in air filters, and for direct counting of surface wipes.

Description of Method: A thin-window gas-flow proportional counter is used for counting gross alpha and beta radioactivity. The method supplies an approximation of the alpha and beta activity present in the air or the removable surface activity dependent on the sample type. The method provides an indication of the presence of alpha and beta emitters, including the following SAM analytes:

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- Americium-241 (CAS RN 14596-10-2) Alpha emitter
- Californium-252 (CAS RN 13981-17-4) Alpha emitter
- Cesium-137 (CAS RN 10045-97-3) Beta emitter
- Cobalt-60 (CAS RN 10198-40-0) Beta emitter
- Curium-244 (CAS RN 13981-15-2) Alpha emitter
- Europium-154 (CAS RN 15585-10-1) Beta emitter
- Iridium-192 (CAS RN 14694-69-0) Beta emitter
- Plutonium-238 (CAS RN 13981-16-3) Alpha emitter
- Plutonium-239 (CAS RN 15117-48-3) Alpha emitter
- Polonium-210 (CAS RN 13981-52-7) Alpha emitter
- Radium-226 (CAS RN 13982-63-3) Alpha emitter
- Ruthenium-103 (CAS RN 13968-53-1) Beta emitter
- Ruthenium-106 (CAS RN 13967-48-1) Beta emitter
- Strontium-90 (CAS RN 10098-97-2) Beta emitter
- Uranium-234 (CAS RN 13966-29-5) Alpha emitter
- Uranium-235 (CAS RN 15117-96-1) Alpha emitter
- Uranium-238 (CAS RN 7440-16-1) Alpha emitter

For this application, the procedure requires the use of thorium-230 for alpha counting efficiency and cesium-137 for beta counting efficiency in the calibration of the detector. An air filter or swipe sample is placed onto a planchet then counted for alpha and beta radioactivity. Activity is reported in activity units per volume of air sampled, as units of activity per surface area sampled, or as total units of activity in cases where sample collection information is not available.

Source: FRMAC. 1998. "Gross Alpha and Beta in Air." *FRMAC Monitoring and Analysis Manual – Sample Preparation and Analysis* - Volume 2, DOE/NV/11718-181 Vol. 2, UC-707, p. 33. http://www.epa.gov/sam/pdfs/FRMAC-Vol2-pg33.pdf

6.2.22 DOE RESL Method P-2: ^{32}P Fish, Vegetation, Dry Ash, Ion Exchange

Analyte(s)	CAS RN
Phosphorus-32	14596-37-3

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Cerenkov counting with Liquid Scintillation

Method Developed for: Phosphorus-32 in fish and vegetation
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of soil, sediment, wipes, and air filters.

Description of Method: Samples up to 500 g are dry ashed at 550ºC and dissolved in two portions of nitric acid. The sample is evaporated to half volume and transferred to a perchloric acid hood. Concentrated nitric acid and concentrated perchloric acid are added, and the sample is evaporated to dryness. The residue is dissolved in hydrochloric acid and filtered through a glass fiber filter. Fe-55 is removed by precipitation with cupferron. The solution containing phosphate is purified by passing it through anion and cation columns to remove possible contaminants. The purified phosphate is precipitated as magnesium ammonium phosphate, filtered onto a glass fiber filter, and dried. The magnesium ammonium phosphate is dissolved in nitric acid and transferred to a counting vial. P-32 is assayed by counting the Cerenkov radiation with a liquid scintillation counter.

Special Considerations: Laboratories using this method must have a designated perchloric acid fume hood. This method was developed for analysis of fish and vegetation. Additional development and

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testing is necessary for application to soil, sediment, wipes, and air filters. Phosphorus and iron carrier must be added to matrices that do not contain mg quantities of both elements.

Source: RESL, DOE. 1977. "Method P-2: ^{32}P Fish, Vegetation, Dry Ash, Ion Exchange." *RESL Analytical Chemistry Branch Procedures Manual*, IDO-12096.

6.2.23 ORISE Method AP1: Gross Alpha and Beta for Various Matrices

Analysis Purpose: Gross alpha and gross beta determination
Determinative Technique: Alpha/Beta counting

Method Developed for: Gross alpha and beta in water, soil, vegetation, and other solids
Method Selected for: SAM lists this method for gross alpha and gross beta determination in soil/sediment samples.

Description of Method: This method provides an indication of the presence of alpha and beta emitters, including the following SAM analytes:

- Americium-241 (CAS RN 14596-10-2) Alpha emitter
- Californium-252 (CAS RN 13981-17-4) Alpha emitter
- Cesium-137 (CAS RN 10045-97-3) Beta emitter
- Cobalt-60 (CAS RN 10198-40-0) Beta emitter
- Curium-244 (CAS RN 13981-15-2) Alpha emitter
- Europium-154 (CAS RN 15585-10-1) Beta emitter
- Iridium-192 (CAS RN 14694-69-0) Beta emitter
- Plutonium-238 (CAS RN 13981-16-3) Alpha emitter
- Plutonium-239 (CAS RN 15117-48-3) Alpha emitter
- Polonium-210 (CAS RN 13981-52-7) Alpha emitter
- Radium-226 (CAS RN 13982-63-3) Alpha emitter
- Ruthenium-103 (CAS RN 13968-53-1) Beta emitter
- Ruthenium-106 (CAS RN 13967-48-1) Beta emitter
- Strontium-90 (CAS RN 10098-97-2) Beta emitter
- Uranium-234 (CAS RN 13966-29-5) Alpha emitter
- Uranium-235 (CAS RN 15117-96-1) Alpha emitter
- Uranium-238 (CAS RN 7440-16-1) Alpha emitter

This procedure provides screening measurements to indicate whether specific chemical analyses are required for water, soil, vegetation, and other solids. Liquid samples are acidified, concentrated, dried in a planchet, and counted in a low-background proportional counter. Solid samples are dried and processed to provide homogeneity, and a known quantity is transferred to a planchet and counted in a low-background proportional counter.

Special Considerations: Volatile radionuclides will not be accurately determined using this procedure.

Source: ORISE, Oak Ridge Associated Universities (ORAU). 2001. "Method AP1: Gross Alpha and Beta for Various Matrices." *Laboratory Procedures Manual for the Environmental Survey and Site Assessment Program*. http://www.epa.gov/sam/pdfs/ORISE-AP1.pdf

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6.2.24 ORISE Method AP2: Determination of Tritium

Analyte(s)	CAS RN
Tritium (Hydrogen-3)	10028-17-8

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Liquid scintillation

Method Developed for: Tritium in soil, sediment, animal tissue, vegetation, smears, and water samples
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of soil/sediment and surface wipe samples.

Description of Method: The tritium in aqueous and solid samples is distilled using an Allihn condenser. For solid samples, an appropriate volume of laboratory reagent water is added to facilitate distillation. Certain solid samples may be refluxed to ensure distribution of any tritium that may be in the sample. The sample may be spiked with a standard tritium solution to evaluate quenching and counting efficiency. After the sample has been distilled, an aliquot of the distillate is added to a scintillation cocktail and the sample is counted using a liquid scintillation analyzer.

Special Considerations: Other volatile radionuclides such as iodine and carbon isotopes may interfere and may require that the sample be made alkaline using solid sodium hydroxide before distillation. Organic impurities may interfere and may require the addition of an oxidizing agent to the sample as well as spiking the samples with a standard tritium solution. The addition of a standard tritium solution to each sample allows for counting efficiencies to be calculated for each individual sample.

Source: ORISE, ORAU. 2001. "Method AP2: Determination of Tritium." *Laboratory Procedures Manual for the Environmental Survey and Site Assessment Program*.
http://www.epa.gov/sam/pdfs/ORISE-AP2.pdf

6.2.25 ORISE Method AP5: Determination of Technetium-99

Analyte(s)	CAS RN
Technetium-99	14133-76-7

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Liquid scintillation

Method Developed for: Technetium-99 in sediment, soil, smears, and water at environmental levels
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of soil/sediment, surface wipe, and air filter samples.

Description of Method: Solid samples are leached with dilute nitric acid. The leachates are passed through a commercially available resin column (TEVA® resin) which is highly specific for technetium in the pertechnetate form. The technetium is absorbed onto the extraction resin. The resin is added to a scintillation vial containing an appropriate cocktail and counted using a liquid scintillation analyzer. Most interfering beta emitting radionuclides (including C-14, P-32, S-35, Sr-90, Y-90, and Th-234) are effectively removed using TEVA® resin under the conditions in this procedure.

Special Considerations: Tritium may follow technetium due to the absorption of some tritium-labeled compounds by the resin. Possible tritium interferences are eliminated by setting the technetium counting window above the maximum energy of tritium beta particles.

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Source: ORISE, ORAU. 2001. "Method AP5: Determination of Technetium-99." *Laboratory Procedures Manual for the Environmental Survey and Site Assessment Program*. http://www.epa.gov/sam/pdfs/ORISE-AP5.pdf

6.2.26 ORISE Method AP11: Sequential Determination of the Actinides in Environmental Samples Using Total Sample Dissolution and Extraction Chromatography

Analyte(s)	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Curium-244	13981-15-2
Plutonium-238	13981-16-3
Plutonium-239	15117-48-3
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Americium, curium, plutonium, neptunium, thorium, and/or uranium in water and solid samples
Method Selected for: SAM recommends this method for confirmatory analysis when a sample exists in a refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem. In the event of refractory radioactive material, SAM recommends this method for both qualitative determination and confirmatory analysis of drinking water, aqueous/liquid, soil/sediment, surface wipes, and air filter samples.

Description of Method: Solid and unfiltered aqueous samples are dissolved completely by a combination of potassium hydrogen fluoride and pyrosulfate fusions. Filtered aqueous samples are evaporated to dryness followed by a pyrosulfate fusion. The fusion cake is dissolved and, for analyses requiring uranium only, two barium sulfate precipitations are performed, and the uranium is separated using EDTA. For all other analyses, one barium sulfate precipitation is performed and all alpha emitters are coprecipitated on barium sulfate. The barium sulfate is dissolved and the actinides are separated by extraction chromatography. An optional section is presented for the separation of americium from the lanthanides. All actinides are coprecipitated on cerium fluoride and counted with an alpha spectrometer system.

Source: ORISE, ORAU. 2001. "Method AP11: Sequential Determination of the Actinides in Environmental Samples Using Total Sample Dissolution and Extraction Chromatography." *Laboratory Procedures Manual for the Environmental Survey and Site Assessment Program*. http://www.epa.gov/sam/pdfs/ORISE-AP11.pdf

6.2.27 ORISE Method Procedure #9: Determination of I-125 in Environmental Samples

Analyte(s)	CAS RN
Iodine-125	14158-31-7

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Gamma spectrometry

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Method Developed for: Iodine-125 in environmental samples, such as soil, sediment, vegetation, water, milk, filters (air or water), etc.
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of drinking water, aqueous/liquid, soil/sediment, surface wipe, and air filter samples.

Description of Method: In this method a direct comparison is made between the sample and a source prepared from a National Institute of Standards and Technology (NIST) traceable standard. If it is known, either by the sample preparation procedure or by a qualitative analysis on some device (high resolution intrinsic planar detector) that I-125 is the only radionuclide contributing to the observed peak, then this method can be used as a rapid quantitative method.

The sample is prepared by matrix specific techniques and the final sample is placed in a 16 millimeter culture tube and counted in a 3" x 3" thin window sodium iodide (NaI) well detector attached to a pulse height analyzer. I-125 gamma counting rate is determined in the 25 to 35 keV energy range by pulse height analysis. NIST traceable liquid standards are also counted in the same geometric configuration as the samples to determine I-125 counting efficiency.

Special Considerations: Due to the low photon energy of I-125, the Compton scattering and x-ray photons from other radionuclides may cause significant interferences in this procedure.

Source: ORISE, ORAU. 1995. "Procedure #9: Determination of I-125 in Environmental Samples." *Laboratory Procedures Manual for the Environmental Survey and Site Assessment Program.* http://www.epa.gov/sam/pdfs/ORISE-Procedure9-1995.pdf

6.2.28 ASTM Method D3084-05: Standard Practice for Alpha Spectrometry in Water

Analyte(s)	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Curium-244	13981-15-2
Plutonium-238	13981-16-3
Plutonium-239	15117-48-3
Radium-226	13982-63-3
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Qualitative determination
Determinative Technique: Alpha spectrometry

Method Developed for: Alpha particle spectra in water
Method Selected for: SAM lists this method for qualitative determination in drinking water, aqueous/liquid, soil and sediment, surface wipes, and/or air filter samples.

Description of Method: This standard practice covers the process that is required to obtain well-resolved alpha spectra from water samples and discusses the associated problems. This practice is typically preceded with specific chemical separations and mounting techniques that are included in referenced methods. A chemical procedure is required to isolate and purify the radionuclides (see ASTM Methods D3865, *Standard Test Method for Plutonium in Water* and D3972, *Standard Test Method for Isotopic Uranium in Water by Radiochemistry*), and a radioactive tracer is added to determine yield. A source is prepared by employing electrodeposition, microprecipitation, or evaporation (depositing the solution onto a stainless steel or platinum disc). Electrodeposition and microprecipitation are preferred.

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The source's radioactivity is then measured in an alpha spectrometer according to manufacturer's operating instructions. The counting period chosen depends on the sensitivity required of the measurement and the degree of uncertainty in the result that is acceptable.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11 for sample preparation instead of the methods referenced in ASTM Method D3084.

Source: ASTM. 2005. "Method D3084-05: Standard Practice for Alpha Spectrometry in Water." *Annual Book of ASTM Standards*, Vol. 11.02. http://www.astm.org/Standards/D3084.htm

6.2.29 ASTM Method D3972-02: Standard Test Method for Isotopic Uranium in Water by Radiochemistry

Analyte(s)	CAS RN
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Confirmatory analysis
Determinative Technique: Alpha spectrometry

Method Developed for: Alpha-particle-emitting isotopes of uranium in water
Method Selected for: SAM lists this method for confirmatory analysis of drinking water samples.

Description of Method: Uranium is chemically separated from a water sample by coprecipitation with ferrous hydroxide followed by anion exchange, and electrodeposition. When suspended matter is present, an acid dissolution step is added to ensure that all of the uranium dissolves. The sample is acidified, and uranium-232 is added as an isotopic tracer to determine chemical yield. Uranium is coprecipitated from the sample with ferrous hydroxide. This precipitate is dissolved in concentrated hydrochloric acid, or is subjected to acid dissolution with concentrated nitric and hydrofluoric acids, if the hydrochloric acid fails to dissolve the precipitate. Uranium is separated from other radionuclides by adsorption on anion exchange resin, followed by elution with hydrochloric acid. The uranium is finally electrodeposited onto a stainless steel disc and counted using alpha spectrometry.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: ASTM. 2002. "Method D3972-02: Standard Test Method for Isotopic Uranium in Water by Radiochemistry." *Annual Book of ASTM Standards*, Vol. 11.02.
http://www.astm.org/DATABASE.CART/HISTORICAL/D3972-02.htm

6.2.30 Standard Method 7110 B: Gross Alpha and Gross Beta Radioactivity (Total, Suspended, and Dissolved)

Analysis Purpose: Gross alpha and gross beta determination
Determinative Technique: Alpha/Beta counting

Method Developed for: Gross alpha and gross beta activity in water
Method Selected for: SAM lists this method for gross alpha and gross beta determination in aqueous/liquid samples.

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Description of Method: This method allows for measurement of gross alpha and gross beta radiation in water samples. The method provides an indication of the presence of alpha and beta emitters, including the following SAM analytes:

- Americium-241 (CAS RN 14596-10-2) Alpha emitter
- Californium-252 (CAS RN 13981-17-4) Alpha emitter
- Cesium-137 (CAS RN 10045-97-3) Beta emitter
- Cobalt-60 (CAS RN 10198-40-0) Beta emitter
- Curium-244 (CAS RN 13981-15-2) Alpha emitter
- Europium-154 (CAS RN 15585-10-1) Beta emitter
- Iridium-192 (CAS RN 14694-69-0) Beta emitter
- Plutonium-238 (CAS RN 13981-16-3) Alpha emitter
- Plutonium-239 (CAS RN 15117-48-3) Alpha emitter
- Polonium-210 (CAS RN 13981-52-7) Alpha emitter
- Radium-226 (CAS RN 13982-63-3) Alpha emitter
- Ruthenium-103 (CAS RN 13968-53-1) Beta emitter
- Ruthenium-106 (CAS RN 13967-48-1) Beta emitter
- Strontium-90 (CAS RN 10098-97-2) Beta emitter
- Uranium-234 (CAS RN 13966-29-5) Alpha emitter
- Uranium-235 (CAS RN 15117-96-1) Alpha emitter
- Uranium-238 (CAS RN 7440-16-1) Alpha emitter

This method recommends using a thin-window gas-flow proportional counter for counting gross alpha and beta radioactivity. An internal proportional or Geiger counter may also be used. An aliquot of sample is evaporated to a small volume and transferred to a tared counting pan. The sample residue is dried to constant weight, cooled, and reweighed to determine dry residue weight, then counted for alpha and beta radioactivity.

Special Considerations: Ground water samples containing elevated levels of dissolved solids will require use of smaller sample volumes.

Source: APHA, AWWA, and WEF. 2005. "Method 7110 B: Gross Alpha and Gross Beta Radioactivity (Total, Suspended, and Dissolved)." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

6.2.31 Standard Method 7120: Gamma-Emitting Radionuclides

Analyte(s)	CAS RN
Cesium-137	10045-97-3
Cobalt-60	10198-40-0
Europium-154	15585-10-1
Iridium-192	14694-69-0
Ruthenium-103	13968-53-1
Ruthenium-106	13967-48-1
Selenium-75	14265-71-5

Analysis Purpose: Qualitative and confirmatory determination
Determinative Technique: Gamma spectrometry

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Method Developed for: Gamma emitting radionuclides in water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of select gamma emitters in aqueous/liquid samples.

Description of Method: The method uses gamma spectroscopy using either Ge detectors or NaI(Tl) crystals for the measurement of gamma photons emitted from radionuclides present in water. The method can be used for qualitative and confirmatory determinations with Ge detectors or semi-qualitative and semi-quantitative determinations (using NaI(Tl) detectors). Exact confirmation using NaI is possible for single nuclides or when the gamma emissions are limited to a few well-separated energies. A homogeneous water sample is placed into a standard geometry (normally a Marinelli beaker) for gamma counting. Sample portions are counted long enough to meet the required sensitivity of measurement. A standard containing a mixture of gamma energies from approximately 100 to 2000 keV is used for energy calibration.

Source: APHA, AWWA, and WEF. 2005. "Method 7120: Gamma-Emitting Radionuclides." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

6.2.32 Standard Method 7500-Ra B: Radium: Precipitation Method

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Qualitative determination
Determinative Technique: Alpha counting

Method Developed for: Alpha-emitting isotopes of radium in water
Method Selected for: SAM lists this method for qualitative determination in aqueous/liquid samples.

Description of Method: This method is for determination of all alpha-emitting radium isotopes by alpha decay analysis. Lead and barium carriers are added to the sample containing alkaline citrate, then sulfuric acid is added to precipitate radium, barium, and lead as sulfates. The precipitate is purified by washing with nitric acid, dissolving in alkaline EDTA, and re-precipitating as radium-barium sulfate after pH adjustment to 4.5. This slightly acidic EDTA keeps other naturally occurring alpha-emitters and the lead carrier in solution. Radium-223, -224, and -226 are identified by the rate of ingrowth of their daughter products in barium sulfate precipitate. The results are corrected by the rate of ingrowth of daughter products to determine radium activity. This method involves alpha counting by a gas-flow internal proportional counter, scintillation counter, or thin end-window gas-flow proportional counter.

Source: APHA, AWWA, and WEF. 2005. "Method 7500-Ra B: Radium: Precipitation Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

6.2.33 Standard Method 7500-Ra C: Radium: Emanation Method

Analyte(s)	CAS RN
Radium-226	13982-63-3

Analysis Purpose: Confirmatory determination
Determinative Technique: Alpha counting

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Method Developed for: Soluble, suspended, and total radium-226 in water
Method Selected for: SAM lists this method for confirmatory analysis of aqueous/liquid samples.

Description of Method: Radium in water is concentrated and separated from sample solids by coprecipitation with a relatively large amount of barium as the sulfate. The precipitate is treated to remove silicates, if present, and to decompose insoluble radium compounds, fumed with phosphoric acid to remove sulfite, and dissolved in hydrochloric acid. The completely dissolved radium is placed in a bubbler, which is then closed and stored for a period of several days to 4 weeks for ingrowth of radon. The bubbler is connected to an evacuation system and the radon gas is removed from the liquid by aeration and helium, dried with a desiccant, and collected in a counting cell. Four hours after radon collection, the cell is counted. The activity of the radon is equal to the radium concentration. The minimum detectable concentration depends on counter characteristics, background-counting rate of scintillation cell, cell efficiency, length of counting period, and contamination of apparatus and environment by radium-226. Without reagent purification, the overall reagent blank (excluding background) should be between 0.03 and 0.05 pCi radium-226, which may be considered the minimum detectable amount under routine conditions.

Source: APHA, AWWA, and WEF. 2005. "Method 7500-Ra C: Radium: Emanation Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

6.2.34 Standard Method 7500-Sr B: Total Radioactive Strontium and Strontium-90: Precipitation Method

Analyte(s)	CAS RN
Strontium-90	10098-97-2

Analysis Purpose: Qualitative and confirmatory analysis
Determinative Technique: Beta counting

Method Developed for: Strontium-90 or total radioactive strontium in drinking water or filtered raw water
Method Selected for: SAM lists this method for qualitative and confirmatory analysis of aqueous/liquid samples.

Description of Method: A known amount of inactive strontium ions, in the form of strontium nitrate, is added as a "carrier." The carrier, alkaline earths, and rare earths are precipitated as the carbonate to concentrate the radiostrontium. The carrier, along with the radionuclides of strontium, is separated from other radioactive elements and inactive sample solids by precipitation as strontium nitrate using fuming nitric acid solution. The carrier and radionuclides of strontium are precipitated as strontium carbonate, which is dried, weighed to determine recovery of carrier, and measured for radioactivity. The activity of the final precipitate is due to radioactive strontium only, because all other radioactive elements have been removed. Because it is impossible to separate the isotopes of strontium-89 and strontium-90 by any chemical procedure, the amount of strontium-90 is determined by separating and measuring the activity of yttrium-90, its decay product. This method involves beta counting by a gas-flow internal proportional counter or thin end-window low-background proportional counter. A correction is applied to compensate for loss of carriers and activity during the various purification steps.

Source: APHA, AWWA, and WEF. 2005. "Method 7500-Sr B: Total Radioactive Strontium and Strontium-90: Precipitation Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

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6.2.35 Standard Method 7500-U B: Uranium: Radiochemical Method

Analyte(s)	CAS RN
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Qualitative determination
Determinative Technique: Alpha counting

Method Developed for: Total uranium alpha activity in water
Method Selected for: SAM lists this method for qualitative determination in aqueous/liquid samples.

Description of Method: The sample is acidified with hydrochloric or nitric acid and boiled to eliminate carbonate and bicarbonate ions. Uranium is coprecipitated with ferric hydroxide and subsequently separated. The ferric hydroxide is dissolved, passed through an anion-exchange column, and washed with acid, and the uranium is eluted with dilute hydrochloric acid. The acid eluate is evaporated to near dryness, the residual salt is converted to nitrate, and the alpha activity is counted by a gas-flow proportional counter or alpha scintillation counter.

Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: APHA, AWWA, and WEF. 2005. "Method 7500-U B: Uranium: Radiochemical Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition.
http://www.standardmethods.org/

6.2.36 Standard Method 7500-U C: Uranium: Isotopic Method

Analyte(s)	CAS RN
Uranium-234	13966-29-5
Uranium-235	15117-96-1
Uranium-238	7440-61-1

Analysis Purpose: Confirmatory determination
Determinative Technique: Alpha spectrometry

Method Developed for: Isotopic content of the uranium alpha activity; determining the differences among naturally occurring, depleted, and enriched uranium in water
Method Selected for: SAM lists this method for confirmatory analysis of aqueous/liquid samples.

Description of Method: This method is a radiochemical procedure for determination of the isotopic content of uranium alpha activity. The sample is acidified with hydrochloric or nitric acid and uranium-232 is added as an isotopic tracer. Uranium is coprecipitated with ferric hydroxide and subsequently separated from the sample. The ferric hydroxide precipitate is dissolved and the solution passed through an anion-exchange column. The uranium is eluted with dilute hydrochloric acid. The acid eluate is evaporated to near dryness, and the residual salt is converted to nitrate and electrodeposited onto a stainless steel disc and counted by alpha spectrometry.

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Special Considerations: If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11.

Source: APHA, AWWA, and WEF. 2005. "Method 7500-U C: Uranium: Isotopic Method." *Standard Methods for the Examination of Water and Wastewater*. 21st Edition. http://www.standardmethods.org/

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Section 7.0: Selected Pathogen Methods

Per decision of the NHSRC SAM Pathogens Committee, "Section 7.0: Selected Pathogen Methods" has been temporarily withdrawn from the SAM Revision 6.0. Section 7.0 is currently undergoing a significant restructuring to better address the complexity of environmental samples in a more user-friendly format. End-users, expert scientists, and federal agencies are contributing to the new design templates.

During this transition period, the following personnel can be contacted for any emergency technical support need:

- EPA's Office of Emergency Management, Homeland Security Laboratory Research Center, manages the ERLN. The pathogens contact for ERLN is: Michele Burgess (burgess.michele@epa.gov, 202-564-8006).
- NHSRC SAM Pathogens Contact: Sanjiv Shah, Lead (shah.sanjiv@epa.gov, 202-564-9522)

Users may also refer to the SAM Version 5.0, Pathogen Methods section. SAM Revision 5.0 can be accessed at http://www.epa.gov/sam/. The SAM 5.0 Pathogens section is available in a searchable format at http://www.epa.gov/sam/searchpath.htm

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Section 8.0: Selected Biotoxin Methods

A list of methods or procedures to be used in analyzing environmental samples for biotoxin contaminants is provided in Appendix D. These methods should be used to support remediation activities (site assessment through clearance) following a homeland security event. Procedures have been compiled for each biotoxin that may need to be identified and/or quantified following a contamination incident. Analytical procedures are not currently available for all the analyte-sample type combinations included in this document. Future research needs include identification of additional methods as well as development and validation of the procedures listed. Appendix D is sorted alphabetically by analyte, within each of two analyte types (i.e., protein and small molecule).

> ***Please note:*** This section provides guidance for selecting biotoxin methods that have a high likelihood of assuring analytical consistency when laboratories are faced with a large-scale environmental restoration crisis. Not all methods have been verified for the analyte/sample type combination listed in Appendix D. Please refer to the specified method to identify analyte/sample type combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 4.

Appendix D provides the following information:

- **Analyte(s).** The compound or compound(s) of interest.
- **CAS RN / Description.** A unique identifier for substances that provides an unambiguous way to identify a toxin or toxin isoform when there are many possible systematic, generic, or trivial names and/or a brief statement describing the toxin.
- **Analysis type.** Tests are either for presumptive identification, confirmatory identification, or biological activity determination; tests types are described below.
- **Analytical Technique.** An analytical instrument or technique used to determine the quantity and identification of compounds or components in a sample.
- **Analytical Method.** The recommended method or procedure, and the corresponding publisher.
- **Aerosol (filter/cassette or liquid impinger).** The recommended method/procedure to measure the analyte of interest in air sample collection media such as filter cassettes and liquid impingers.
- **Solid (soil, powder).** The recommended method/procedure to measure the analyte of interest in solid samples such as soil and powders.
- **Particulate (swabs, wipes, filters).** The recommended method/procedure to measure the analyte of interest in particulate sample collection media such as swabs, wipes and dust-collecting socks used with vacuum collection.
- **Liquid/water.** The recommended method/procedure to measure the analyte of interest in liquid and water samples.
- **Drinking water.** The recommended method/procedure to measure the analyte of interest in drinking water samples.

Following a homeland security event, it is assumed that only those areas with contamination greater than pre-existing, naturally prevalent levels commonly found in the environment would be subject to remediation. Dependent on site- and event-specific goals, investigation of background levels using methods listed in Appendix D is recommended.

The "analysis type" listed for each biotoxin method in Appendix D is intended to address: (1) the level of certainty of results and (2) the remediation phase (e.g., site mapping, assessment, clearance). Presumptive methods are intended to provide results consistent with a reasonable level of certainty and would generally be used during remediation where a large number of samples may need to be processed (e.g., requiring high throughput). Immunoassays are common presumptive methods, may be adapted for large-

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scale sample processing, and are listed for many of the biotoxin analytes. Confirmatory methods are intended to provide results with a high level of certainty. Confirmatory methods should be considered for remediation when: (1) presumptive analysis indicates the presence of the biotoxin, (2) a smaller subset of samples requires processing, or (3) as required for a tiered approach (e.g., algorithm) to remediation. Several techniques are listed in Appendix D as confirmatory; these are generally more time consuming and expensive. The use of these terms in this document is not intended to redefine or supersede the Laboratory Response Network's (LRN) use of these terms. The terms presumptive and confirmatory as used by the LRN are described in Section 8.1.4. Methods that address biological activity are intended to provide a high level of certainty in corroborating other assay results. Biotoxins may be detectable but inactive (either before or after remediation); therefore these assays may also provide information about potential impact on human safety. Biological activity may be determined directly using *in vivo* (e.g., mouse bioassay) or *in vitro* (e.g., enzymatic activity) methods or inferred using indirect methods (e.g., HPLC). However, biological availability (i.e., biotoxin accessibility to site of action) and activity are both required to elicit toxicity and some *in vitro* methods may not address both parameters. Confirmatory procedures listed for the small molecule biotoxins involve a determination of intact compound structure (an indication of biological activity); therefore, only presumptive and confirmatory methods are listed for these biotoxins. Tiered approaches for specific contaminants will be described in a future revision of SAM.

Numerous analytical techniques using a variety of instrumentation (e.g., high performance liquid chromatography – mass spectrometer [HPLC-MS], HPLC-FL, immunoassay [enzyme-linked immunosorbent assay (ELISA)], immunoassay [lateral flow device (LFD)], etc.) have been cited in Appendix D. It is expected that a reduced number of these analytical techniques and instruments will be necessary after method verification and validation. In addition, it is recognized that new reports detailing advances in biotoxin analysis appear in the literature frequently. Accordingly, the individual techniques and methods listed in Appendix D are to be regarded as a starting point; after thoughtful consideration of current technologies at the time of remediation and consultation with the authority in charge of the remediation activity, these techniques and methods can be modified as necessary for analysis of a particular sample.

The presence of disinfectants (e.g., chlorine) and/or preservatives added during water sample collection to slow degradation (e.g., pH adjustors, de-chlorinating agents) could possibly affect analytical results. When present, the impact of these agents on method performance should be evaluated, if not previously determined. EPA's NHSRC is working on a sample collection document that is intended as a companion to SAM. This sample collection document will provide information regarding sampling container/media, preservation, holding time, sample size, and shipping and is intended to complement the laboratory analytical methods that are the focus of the SAM document.

8.1 General Guidelines

This section provides a general overview of how to identify the appropriate method(s) for a given biotoxin as well as recommendations for QC procedures.

For additional information on the properties of the biotoxins listed in Appendix D, TOXNET (http://toxnet.nlm.nih.gov/index.html), a cluster of databases on toxicology, hazardous chemicals, and related areas maintained by the National Library of Medicine, is an excellent resource.

Additional resources include:

- *Defense Against Toxin Weapons*, published by the U.S. Army Medical Research Institute of Infectious Diseases (http://www.usamriid.army.mil/education/defensetox/toxdefbook.pdf) contains information regarding sample collection, toxin analysis and identification, as well as decontamination and water treatment.
- Select Agent Rules and Regulations found at the National Select Agent Registry (http://www.selectagents.gov/)

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- The CDC has additional information regarding select agent toxins at the following Web site: http://www.cdc.gov/od/sap/sap/toxinamt.htm
- SRC's PHYSPROP and Chemfate, part of the Environmental Fate Database supported by EPA. See http://srcinc.com/what-we-do/product.aspx?id=133.
- INCHEM at http://www.inchem.org/ contains both chemical and toxicity information.
- The RTECS database can be accessed via the NIOSH Web site at http://www.cdc.gov/niosh/rtecs/default.html for toxicity information.
- The Forensic Science and Communications Journal published by the Laboratory Division of the FBI. See http://www.fbi.gov/hq/lab/fsc/current/backissu.htm.

Additional research on biotoxin contaminants is ongoing within EPA.

8.1.1 Standard Operating Procedures for Identifying Biotoxin Methods

To determine the appropriate method that is to be used on an environmental sample, locate the biotoxin of concern in Appendix D: Selected Biotoxin Methods under the "Analyte(s)" column. After locating the biotoxin, continue across the table and identify the appropriate analysis type. After an analysis type has been chosen, find the analytical technique (e.g., immunoassay) and analytical method applicable to the sample type of interest (aerosol, solid, particulate, liquid, or drinking water) corresponding to that particular analyte.

Once a method has been identified in Appendix D, the corresponding method summary can be found in Sections 8.2.1 through 8.3.12. Method summaries are listed first by alphabetical order within each biotoxin subcategory (i.e., protein and small molecule) and then in order of method selection hierarchy (see Figure 2-1), starting with EPA methods, followed by methods from other federal agencies, VCSBs, and journal articles. Where available, a direct link to the full text of the method is provided with the method summary. For additional information on sample preparation procedures and methods available through consensus standards organizations, please use the contact information provided in **Table 8-1**.

Table 8-1. Sources of Biotoxin Methods

Name	Publisher	Reference
FDA, Bacteriological Analytical Manual Online	FDA	http://www.cfsan.fda.gov/~ebam/bam-toc.html
Official Methods of Analysis of AOAC International*	AOAC International	http://www.aoac.org
NEMI	EPA, USGS	http://www.nemi.gov/
Pharmacology & Toxicology*	Blackwell Synergy	http://www.blackwell-synergy.com/loi/pto
Analytical Biochemistry*	Science Direct	http://www.sciencedirect.com/
Biochemical Journal*	Portland Press Ltd.	http://www.biochemj.org/
Journal of Medicinal Chemistry*	American Chemical Society	http://www.acs.org/
Journal of Food Protection*	International Association for Food Protection	http://www.foodprotection.org/
Journal of Chromatography B*	Elsevier Science Publishers	http://www.elsevier.com/
Biomedical Chromatography*	John Wiley And Sons Ltd	http://www.wiley.com/

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Name	Publisher	Reference
Environmental Health Perspectives*	National Institute of Environmental Health Sciences	http://www.niehs.nih.gov/
Toxicon*	Elsevier Science Publishers	http://www.elsevier.com/
Federation of European Microbiological Societies (FEMS) Microbiology Letters*	Wiley-Blackwell	http://www.wiley.com/
International Journal of Food Microbiology*	Elsevier Science Publishers	http://www.elsevier.com/
Rapid Communications in Mass Spectrometry *	John Wiley And Sons Ltd.	http://www.wiley.com/
Journal of AOAC International*	AOAC International	http://www.aoac.org
Analyst*	Royal Society of Chemistry	http://www.rsc.org/
Journal of Pharmaceutical and Biomedical Analysis*	Elsevier Science Publishers	http://www.elsevier.com/
Journal of Clinical Microbiology	American Society for Microbiology (ASM)	http://www.asm.org/
Journal of Clinical Laboratory Analysis*	John Wiley And Sons Ltd.	http://www.wiley.com/
Journal of Analytical Toxicology*	S. Tinsley Preston	http://www.jatox.com/
Lateral Flow Immunoassay Kits	Environmental Technology Verification (ETV) Program	http://www.epa.gov/etv/
Journal of Agricultural and Food Chemistry*	ACS Publications	http://pubs.acs.org/
Applied and Environmental Microbiology (AEM)*	ASM	http://aem.asm.org/
Journal of Chemical Health and Safety*	Elsevier Science Publishers	http://www.elsevier.com/

* Subscription and/or purchase required.

8.1.2 General QC Guidelines for Biotoxin Methods

Having data of known and documented quality is critical so that public officials can accurately assess the activities that may be needed in remediating a site during and following emergency situations. Having such data requires that laboratories: (1) conduct the necessary QC to ensure that measurement systems are in control and operating properly; (2) properly document results of the analyses; and (3) properly document measurement system evaluation of the analysis-specific QC. Ensuring data quality also requires that laboratory results are properly evaluated and the results of the data quality evaluation are transmitted to decision makers.

The level or amount of QC needed often depends on the intended purpose of the data that are generated. Various levels of QC may be required if the data are generated during presence/absence determinations versus confirmatory analyses. The specific needs for data generation should be identified. QC requirements and data quality objectives should be derived based on those needs and should be applied consistently across laboratories when multiple laboratories are used. For example, during rapid sample screening, minimal QC samples (e.g., blanks, replicates) and documentation might be required to ensure data quality. Sample analyses for environmental evaluation during site assessment through site clearance, such as those identified in this document, might require increased QC (e.g., demonstrations of method sensitivity, precision, and accuracy).

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While method-specific QC requirements may be included in many of the procedures that are cited in this document, and will be referenced in any SAPs developed to address specific analytes and sample types of concern, the following describes a minimum set of QC samples and procedures that should be conducted for all analyses. Individual methods, sampling and analysis protocols, or contractual statements of work also should be consulted to determine any additional QC that may be needed. QC tests should be run as frequently as necessary to ensure the reliability of analytical results. In general, sufficient QC includes an initial demonstration of measurement system capability as well as ongoing assessments to ensure the continued reliability of the analytical results.

Examples of sufficient QC for the presumptive tests listed in Appendix D include:

- Method blanks;
- Positive test samples / negative test samples;
- Calibration check samples;
- Use of test kits and reagents prior to expiration; and
- Accurate temperature controls.

Examples of sufficient QC for the confirmatory tests listed in Appendix D include:

- Demonstration that the measurement system is operating properly
  - Initial calibration
  - Method blanks

- Demonstration of measurement system suitability for intended use
  - Precision and recovery (verify measurement system has adequate accuracy)
  - Analyte/sample type/level of concern-specific QC samples (verify that measurement system has adequate sensitivity at levels of concern)

- Demonstration of continued measurement system reliability
  - MS/MSDs (recovery and precision)
  - QC samples (system accuracy and sensitivity at levels of concern)
  - Continuing calibration verification
  - Method blanks

Please note: The appropriate point of contact identified in Section 4 should be consulted regarding appropriate QA/QC procedures prior to sample analysis. These contacts will consult with the EPA ERLN coordinator responsible for laboratory activities during the specific event to ensure QA/QC procedures are performed consistently across laboratories. EPA program offices will be responsible for ensuring that the QA/QC practices are implemented.

8.1.3 Safety and Waste Management

It is imperative that safety precautions be used during collection, processing, and analysis of environmental samples. Laboratories should have a documented health and safety plan for handling samples that may contain target CBR contaminants, and laboratory staff should be trained in and implement the safety procedures included in the plan. In addition, many of the methods summarized or cited in Section 8.2 contain some specific requirements, guidelines, or information regarding safety precautions that should be followed when handling or processing environmental samples and reagents. These methods also provide information regarding waste management. Other resources that can be consulted for additional information include the following:

- American Biological Safety Association, Risk Group Classifications for Infectious Agents, available at http://www.absa.org/riskgroups/index.html.
- *Biosafety in Microbiological and Biomedical Laboratories* (BMBL), 5th Edition, found at http://www.cdc.gov/OD/ohs/biosfty/bmbl5/bmbl5toc.htm.

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- Biological Safety: Principles and Practices, 4th Ed. ASMPress (http://estore.asm.org/).
- CDC – 42 CFR part 72. Interstate Shipment of Etiologic Agents.
- CDC – 42 CFR part 73. Select Agents and Toxins.
- DOT – 49 CFR part 172. Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements.
- EPA – 40 CFR part 260. Hazardous Waste Management System: General.
- EPA – 40 CFR part 270. EPA Administered Permit Programs: The Hazardous Waste Permit Program.
- OSHA – 29 CFR part 1910.1450. Occupational Exposure to Hazardous Chemicals in Laboratories.
- OSHA – 29 CFR part 1910.120. Hazardous Waste Operations and Emergency Response.
- USDA – 9 CFR part 121. Possession, Use, and Transfer of Select Agents and Toxins.

Please note that the e-CFR is available at http://ecfr.gpoaccess.gov/.

8.1.4 Laboratory Response Network (LRN)

The LRN was created in accordance with Presidential Decision Directive 39, which established terrorism preparedness responsibilities for federal agencies. The LRN is primarily a national network of local, state, federal, military, food, agricultural, veterinary, and environmental laboratories; however, additional LRN laboratories are located in strategic international locations. The CDC provides technical and scientific support to member laboratories as well as secure access to standardized procedures and reagents for rapid (within 4 to 6 hours) presumptive detection of biothreat agents and emerging infectious disease agents. These rapid presumptive assays are part of agent-specific algorithms of assays which lead to a confirmed result. The algorithm for a confirmed result is often a combination of one or more presumptive positive results from a rapid assay in combination with a positive result from one of the "gold standard" methods, such as culture. The standardized procedures, reagents, and agent-specific algorithms are considered to be sensitive and are available only to LRN member laboratories. Thus, these procedures are not available to the general public and are not discussed in this document.

Additional information on select agents and regulations may be obtained at the National Select Agent Registry at: http://www.selectagents.gov/.

For additional information on the LRN, including selection of a laboratory capable of receiving and processing the specified sample type/analyte, please use the contact information provided below or visit http://www.bt.cdc.gov/lrn/.

Centers for Disease Control and Prevention
Laboratory Response Branch
Division of Bioterrorism Preparedness and Response (DBPR)
National Center for Prevention, Detection, and Control of Infectious Diseases (NCPDCID)
Coordinating Center for Infectious Diseases (CCID)
Centers for Disease Control and Prevention (CDC)
1600 Clifton Road NE, Mailstop C-18
Atlanta, GA 30333
Telephone: (404) 639-2790
E-mail: lrn@cdc.gov

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Local public health laboratories, private, and commercial laboratories with questions about the LRN should contact their state public health laboratory director or the Association of Public Health Laboratories (APHL) (contact information provided below).

Association of Public Health Laboratories
8515 Georgia Avenue, Suite 700
Silver Spring, MD 20910
Telephone: (240) 485-2745
Fax: (240) 485-2700
Web site: www.aphl.org
E-mail: info@aphl.org

8.2 Method Summaries for Protein Biotoxins

Summaries of the analytical methods for protein biotoxins listed in Appendix D are provided in Sections 8.2.1 through 8.2.5. These sections contain summary information only, extracted from the selected methods. The full version of the method should be consulted prior to sample analysis.

Each summary contains a brief description of the method, intended method application, performance data (if available), and a link to or source for obtaining a full version of the method.

8.2.1 Abrin

Abrin – CAS RN: 1393-62-0.
Description: Glycoprotein consisting of a deadenylase (25–32 kDa A chain) and lectin (35 kDa B chain); an agglutinin (A2B2) may be present in crude preparations.
Abrine – CAS RN: 526-31-8
Description: Small molecule, indole alkaloid marker for abrin.

Method	Analytical Technique	Section
Journal of Food Protection. 2008. 71(9): 1868–1874	Immunoassay	8.2.1.1
Journal of Agricultural and Food Chemistry. 2008. 56(23): 11139–11143	LC-MS-MS	8.2.1.2
Pharmacology & Toxicology. 2001. 88(5): 255–260	Ribosome inactivation assay	8.2.1.3
Analytical Biochemistry. 2008. 378: 87–89	Enzyme activity	8.2.1.4

8.2.1.1 Literature Reference for Abrin (Journal of Food Protection. 2008. 71(9): 1868–1874)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay
Method Developed for: Abrin in food
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for using mouse monoclonal antibodies (mAbs) and rabbit-derived polyclonal antibodies prepared against a mixture of abrin isozymes for three separate ELISA and electrochemiluminescence (ECL)-based assays in food products. The three assays vary by use of antibody combination (e.g, assay configuration): (1) polyclonal (capture)/polyclonal (detection) ELISA, (2) polyclonal/monoclonal ELISA, and (3) polyclonal/monoclonal ECL assay. The LODs, with purified Abrin C and various abrin extracts in buffer, are between 0.1 and 0.5 ng/mL for all three assays. The LOD for abrin spiked into food

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products ranged from 0.1 to 0.5 ng/mL, using the ECL assay. The LOD for abrin spiked into food products for the ELISA assays ranged between 0.5 and 10 ng/mL depending on the antibody combination. In all cases, the LODs were less than the concentration at which abrin may pose a health concern.

Special Considerations: Crude preparations of abrin may also contain agglutinins that are unique to rosary peas and that can cross-react in the immunoassays. Addition of non-fat milk powder to the sample buffer may eliminate false-positive results (Dayan-Kenigsberg, J., Bertocchi, A., and Garber, E.A. 2008. "Rapid Detection of Ricin in Cosmetics and Elimination of Artifacts Associated with Wheat Lectin." Journal of Immunological Methods. 336(2): 251–254). http://www.sciencedirect.com/science/journal/00221759

Source: Garber, E.A., Walker, J.L., and O'Brien, T.W. 2008. "Detection of Abrin in Foods Using Enzyme-Linked Immunosorbent Assay and Electrochemiluminescence Technologies." Journal of Food Protection. 71(9): 1868–1874. http://www.ingentaconnect.com/content/iafp/jfp/2008/00000071/00000009/art00015

8.2.1.2 Literature Reference for Abrin by Abrine Detection (Journal of Agricultural and Food Chemistry. 2008. 56(23): 11139-11143)

Analysis Purpose: Complementary presumptive for abrin
Analytical Technique: LC-MS-MS

Method Developed for: Abrine in beverages
Method Selected for: SAM lists these procedures for complementary presumptive analysis of abrin by abrine detection in aerosol, solid, particulate, liquid, and water samples. Abrine, an alkaloid present in equal concentrations with abrin in rosary peas (*Abrus precatorius* L.), is found in crude preparations of abrin and may be an indicator of abrin contamination. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for sample extraction by SPE or liquid-liquid extraction, followed by tandem mass spectrometry. The method was verified in beverages (bottled water, cola, juice drink, 1% low fat milk, bottled tea) spiked with abrine at either 0.5µg/mL or 0.05µg/mL. These samples were prepared for LC-MS-MS by either an optimized SPE procedure or a liquid-liquid extraction procedure. For SPE, optimal abrine recoveries were achieved with sample pH adjusted to 2 – 6 with formic acid, inclusion of a water/methanol (95/5, v/v) washing step prior to elution, and use of a Strata-X SPE cartridge. Liquid-liquid extraction was with an equal volume (2 mL) of acetonitrile/water (75/25, v/v). Differences in recovery between the two extraction methods were determined using the two-sided Student's *t* test, assuming equal variance. At 0.5 µg/mL, recovery of abrine by SPE was significantly higher ($P < 0.01$) for water and juice drink as compared to liquid-liquid extraction, but no significant differences were observed for cola and tea. At 0.05 µg/mL, the differences in recovery of abrine in water, tea, cola, and juice drink were highly statistically different ($P < 0.001$), with better recoveries for the optimized SPE procedure. The method had a MDL of 0.025 µg/mL and limit of quantitation (LOQ) of 0.05 µg/mL. Storage stability was also tested for abine at 10 µg/mL in a water/methanol stock solution (90/10, v/v) at three temperatures (0°C, 4°C, and 23°C). Aliquots were analyzed in triplicate at 0, 1, 7, and 21 days after sample preparation. There was no statistically significant difference between abrine standards stored at the three temperatures at 21 days and no loss of abrine concentration.

Special Considerations: The biotoxin methods points of contact listed in Section 4.0 of SAM should be consulted for additional information regarding water and drinking water analyses.

Source: Owens, J. and Koester, C. 2008. "Quantitation of Abrine, an Indole Alkaloid Marker of the Toxic Glycoproteins Abrin, by Liquid Chromatography/Tandem Mass Spectrometry When

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Spiked into Various Beverages." Journal of Agriculture and Food Chemistry. 56(23): 11139–11143. http://pubs.acs.org/doi/pdf/10.1021/jf802471y

8.2.1.3 Literature Reference for Abrin and Ricin (Analytical Biochemistry. 2008. 378(1): 87-89)

Analysis Purpose: Biological activity
Analytical Technique: Enzyme activity

Method Developed for: Jequirity seed (abrin) and castor bean (ricin) extracts in buffer
Method Selected for: SAM lists these procedures for biological activity analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: This *in vitro* assay is a ribonucleic acid (RNA) N-glycosidase enzyme activity assay for the detection of purified abrin and ricin toxins (Types I and II) or in jequirity seed (abrin) and castor bean (ricin) extracts. Synthetic biotinylated RNA substrates with varied loop sequences are cleaved by either the ricin or abrin toxin and the RNA products are hybridized to ruthenylated-oligodeoxynucleotides to generate an ECL signal. Assays require incubation for 2 hours at 48°C. Commercially available ECL-based reagents and RNase inactivators are used. Control experiments for the jequirity seed experiments and the distinct GdAA/GdAGA ratio for the castor bean assay demonstrate lack of non-specific cleavage for the assay. The undiluted castor bean extract contained 22.0 ± 0.7 mg/mL total protein and 4.1 ± 0.3 mg/mL ricin equivalents as determined by standard protein determination and by ECL immunoassay assays respectively. The undiluted jequirity seed extract was similarly assayed, with a resultant 21.6 ± 0.6 mg/mL total protein and 3.7 ± 0.3 µg/mL equivalents of toxin. Dilutions were performed to determine effective signal-to-background ratio and the linear range for calculation of toxin activity. Resultant calculations for ricin activity equivalents in the undiluted castor bean extract were equivalent to those obtained with the ECL immunoassays: 4.4 ± 0.2 mg/mL activity equivalents. In contrast, the undiluted jequirity seed extract contained a calculated level of 740 ± 50 µg/mL activity equivalents, which greatly exceeded the immunoassay-based value.

Special Considerations: This enzyme activity assay does not test for cell binding; cell culture assays are being developed to test for cell binding but are not currently available. The only readily available assay to test for both the cell binding and enzymatic activity of the intact (whole) toxin is the mouse bioassay.

Source: Keener, W.K., Rivera, V.R., Cho, C.R., Hale, M.L., Garber, E.A.E., and Poli, M.A. 2008. "Identification of the RNA N-glycosidase Activity of Ricin in Castor bean extracts by an Electrochemiluminescence-based Assay." Analytical Biochemistry. 378(1): 87–89. http://www.sciencedirect.com/science/journal/00032697

8.2.1.4 Literature Reference for Abrin, Shiga Toxin, and Shiga-like Toxins (Pharmacology Toxicology. 2001. 88(5): 255–260)

Analysis Purpose: Confirmatory for abrin; biological activity for shiga and shiga-like toxins
Analytical Technique: Ribosome inactivation assay

Method Developed for: Abrin in phosphate buffered saline (PBS)
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

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Description of Method: Procedures are described for measuring the biological activity of ribosome-inactivating proteins using a microtiter plate format for detection of abrin in PBS. Nuclease-treated rabbit reticulocyte lysate containing luciferase messenger ribonucleic acid (mRNA) is used to measure toxin activity via inhibition of protein synthesis. The relative biological activity is determined by comparing luminescence levels in treated samples versus those of untreated controls. The amount of luciferase translated, as measured by luminescence, is inversely proportional to the toxin concentration. Linear dose response curves are generated for abrin, with a 50% inhibition of translation at 0.5 nM. Coupling this procedure, or a modification of this procedure, with an immunoassay will provide more information regarding the specificity and toxicity of the target biotoxin.

Special Considerations: For abrin, as well as shiga and shiga-like toxins, this assay does not test for cell binding; cell culture assays are being developed to test for cell binding but are not currently available. The only readily available assay to test for both the cell binding and enzymatic activity of the intact (whole) toxin is the mouse bioassay.

Source: Hale, M.L. 2001. "Microtiter-based Assay for Evaluating the Biological Activity of Ribosome-inactivation Proteins." Pharmacology Toxicology. 88(5): 255–260. http://www3.interscience.wiley.com/journal/120703798/abstract

8.2.2 Botulinum Neurotoxins (Serotypes A, B, E, F)

Botulinum neurotoxins – Description: Protein composed of ~100 kDa heavy chain and ~50 kDa light chain; can be complexed with hemagglutinin and non-hemagglutinin components for total MW of ~900 kDa.
SNAP-25 – Description: Synaptosomal-associated protein 25; 25 kDa membrane-associated protein cleaved by botulinum neurotoxin Serotypes A, C, and E
VAMP 2 – Description: Vesicle-associated membrane protein 2 (also known as synaptobreven 2); cleaved by botulinum neurotoxin Serotypes B, D, F, and G

Method	Analytical Technique	Section
LRN	Immunoassay, Immunoassay (ELISA) and Mouse bioassay	8.1.4
FDA, Bacteriological Analytical Manual Online, January 2001, Chapter 17, *Clostridium botulinum*	Immunoassay (ELISA) and Mouse bioassay	8.2.2.1
Journal of Chemical Health and Safety. 2008. 15(6): 14–19	Endopep-MS	8.2.2.2
Lateral Flow Immunoassay Kits	Immunoassay	8.2.2.3

8.2.2.1 FDA, Bacteriological Analytical Manual Online, Chapter 17, 2001: Botulinum Neurotoxins

Analysis Purpose: Confirmatory and biological activity
Analytical Technique: Immunoassay (ELISA) and mouse bioassay

Method Developed for: Botulinum neurotoxins (Serotypes A, B, E, F) in food
Method Selected for: SAM lists this procedure for confirmation and biological activity assessment in aerosol samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: An amplified-enzyme-linked immunosorbent assay (amp-ELISA) and a digoxigenin-labeled enzyme-linked immunosorbent assay (DIG-ELISA) are described for the detection of Types A, B, E, and F botulinum neurotoxins in food products. The amp-ELISA method uses goat anti-A or E, rabbit anti-B, or horse anti-F serum to capture the toxins in a 96-well plate, and a corresponding biotinylated goat antitoxin to detect the toxin. Visualization is

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with streptavidin-alkaline phosphatase. The DIG-ELISA method is a modification of the amp-ELISA method, with digoxigenin-labeled antitoxin IgG's substituted for the streptavidin-alkaline phosphatase. Toxin can be detected at approximately 10 minimum lethal doses (MLD)/mL (0.12 to 0.25 ng/mL). High concentration samples (greater than 10,000 MLD/mL) may give a positive absorbance for more than one toxin type. Further dilution of the sample will remove cross-reactivity.

The mouse bioassay detects biologically active toxin using a three part approach: toxin screening; toxin titer; and finally, toxin neutralization using monovalent antitoxin sera. Samples are prepared by centrifugation for suspended solids under refrigeration, or solids are extracted with an equal volume of pH 6.2 gel-phosphate buffer and then centrifuged. Toxins from nonproteolytic strains of *C. botulinum* may need trypsin activation to be detected. Serial dilutions of untreated and trypsin-treated sample fluids are injected in separate pairs of mice intraperitoneally (i.p.). Mice are also injected with heated, untreated, undiluted sample. Death of mice, along with symptoms of botulism, confirms presence of botulinum toxin. After calculation of an MLD, dilute monovalent antitoxin sera types A, B, E, and F are injected into mice 30 minutes to 1 hour before challenging them with the i.p. injection of each dilution that gave the highest MLD from the toxic preparation.

Special Considerations: Immunoassays with botulinum toxins may produce variable results with uncomplexed forms of toxin.

Source: FDA, Center for Food Safety and Applied Nutrition (CFSAN). 2001. "Chapter 17 – *Clostridium botulinum*." *Bacteriological Analytical Manual Online.*
http://www.epa.gov/sam/pdfs/FDA-BAM-Chap17.pdf

8.2.2.2 Literature Reference for Botulinum Neurotoxins by SNAP-25 and VAMP 2 Cleavage Product Detection (Journal of Chemical Health and Safety. 2008. 15(6): 14–19)

Analysis Purpose: Complementary presumptive for botulinum neurtotoxins
Analytical Technique: LC-MS

Method Developed for: Botulinum neurotoxins Serotypes A, B, E, and F in clinical samples (stool, serum)
Method Selected for: SAM lists these procedures for complementary presumptive analysis of botulinum neurotoxins by SNAP-25 and VAMP 2 cleavage product detection in aerosol samples. SNAP-25 and VAMP 2 function as substrates for botulinum neurotoxins and may be an indicator of botulinum neurotoxin contamination. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for antibody-based sample extraction, followed by synthetic peptide cleavage and high resolution matrix-assisted laser-desorbtion ionization (MALDI) time of flight MS. The method is verified for stool and serum clinical samples obtained from an exposed individual. Botulinum neurotoxin Serotypes A, B, D, and E are obtained from Metabiologics (Madison, WI) and used as positive controls. Rabbit polyclonal antibodies specific for Serotypes A, B, E, and F are also obtained from Metabiologics and are coupled to Dynabeads® Protein G beads. Twenty microliters of beads are added to 100 µL of stool sample, along with a cocktail of protease inhibitors. The mixture is incubated for two hours at 37°C, washed in buffer, followed by a water wash. Five hundred microliters of serum sample is added to 100 µL of beads and similarly incubated and washed. Protease inhibitors are not required for serum samples. After antibody isolation, the bead-extracted sample is incubated in a reaction buffer with synthetic peptide substrates specific for Serotypes A, B, E, and F. Samples are incubated at 37°C for four hours. A 2-µL aliquot of the reaction mixture supernatant is mixed

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with 18 µL of a matrix solution and 0.5 µL of the resultant mixture is placed on a 192-spot MALDI plate. Mass spectra are collected from 650 to 4500 *m/z* in the positive ion reflector mode on either an Applied Biosystems™ 4700 Proteomics Analyzer or an Applied Biosystems™ 4800 TOF/TOF. Cleavage product peaks specific for Serotypes A, B, E, and F can be for observed for the positive controls and positive stool and serum samples. Negative controls do not show these peaks.

Special Considerations: Additional detector platforms are available such as described in "Development of an *In Vitro* Activity Assay as an Alternative to the Mouse Bioassay for Clostridium botulinum Neurotoxin Type A," 2008. Applied and Environmental Microbiology. 74(14): 4309–4313. (http://www.epa.gov/sam/pdfs/AEM-74(14)-pgs4309-4313.pdf). FRET based assays are also available as commercial products (http://www.biosentinelpharma.com/products.php).

Source: Barr, J.R., Kalb, S.R., Moura, H., and Pirkle, J.L. 2008. "Biological Monitoring of Exposure to Botulinum Neurotoxins." Journal of Chemical Health and Safety. 15(6): 14–19. http://www.sciencedirect.com/science/journal/18715532

8.2.2.3 EPA Environmental Technology Verification (ETV) Program Reports – Lateral Flow Immunoassay Kits

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Botulinum neurotoxins (Types A, B) and ricin in buffer or water samples
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Test strips are self-contained, qualitative assays for screening environmental samples for the presence of botulinum toxin and ricin. After the sample is collected, it is transferred onto the test strip where dye-labeled antibodies detect trace amounts of the contaminant, as indicated by the presence of two bands in the test result window. After 15 minutes, the results are read visually. Botulinum neurotoxin Type A can be detected at 5 mg/L and Type B at 4 mg/L, 33% of the time. Ricin toxin can be detected at 20 mg/L, with no cross-reactivity to certain substances (i.e., lectin from soybeans).

An alternative lateral flow immunochromatographic device also can be used. This device uses two antibodies in combination to specifically detect target antigen in solution. When a sufficient amount of target antigen is present, the colloidal gold label accumulates in the sample window on a test strip, forming a visible reddish-brown colored line. The presence of two bands indicates a positive reading. Botulinum neurotoxin Type A can be detected at 0.01 mg/L and Type B at 0.5 mg/L, with no false negatives detected when interferents are present. Ricin toxin can be detected at 0.035 mg/L, with 88% accuracy.

Lateral flow immunoassay kits have been evaluated by the EPA ETV Program using BADD™ and BioThreat Alert® test strips for the detection of botulinum neurotoxins Types A and B and ricin. Reports and information associated with these evaluations are available at: http://www.epa.gov/sam/pdfs/ETV-BADD091904.pdf (BADD™ test strips) and http://www.epa.gov/sam/pdfs/ETV-BioThreat092104.pdf (BioThreat Alert® test strips).

Special Considerations: Immunoassays with botulinum toxins may produce variable results with uncomplexed form of toxin. Addition of non-fat milk powder to the sample buffer may eliminate false-positive results (Dayan-Kenigsberg, Bertocchi, J.A., and Garber, E.A.E. 2008. "Rapid

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Detection of Ricin in Cosmetics and Elimination of Artifacts Associated with Wheat Lectin." Journal of Immunological Methods. 336(2): 251–254). http://www.sciencedirect.com/science/journal/00221759

Source: ETV. 2006. http://www.epa.gov/etv/

8.2.3 Ricin (Ricinine)

Ricin – CAS RN: 9009-86-3.
Description: 60 kDa glycoprotein composed of two subunits (~32 kDa A chain and ~34 kDa B chain); an agglutinin of MW 120 kDa may be present in crude preparations.
Ricinine – CAS RN: 5254-40-3.
Description: Small molecule, alkaloid marker for ricin.

Method	Analytical Technique	Section
LRN	Immunoassay	8.1.4
Analytical Biochemistry. 2008. 378: 87–89	Enzyme activity	8.2.1.3
Lateral Flow Immunoassay Kits	Immunoassay	8.2.2.2
Journal of AOAC International. 2008. 91(2): 376–382	Immunoassay	8.2.3.1
Journal of Analytical Toxicology. 2005. 29: 149–155	LC-MS	8.2.3.2

8.2.3.1 Literature Reference for Ricin (Journal of AOAC International. 2008. 91(2): 376–382)

Analysis Purpose: Confirmatory
Analytical Technique: Immunoassay

Method Developed for: Ricin for food products
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: This immunoassay is for the detection of various concentrations of purified ricin in food products (e.g., juice, dairy products, vegetables, bakery products, condiments). The immunoassay uses ECL detection in a 96-well plate format with a monoclonal capture antibody against ricin (19A-2C6) and either a polyclonal or monoclonal detector antibody. The samples and detector antibodies can be added sequentially or in combination during the capture step. Using the polyclonal antibody, ricin was detected at concentrations as low as 0.04 ng/mL. Simultaneous addition of sample and detector antibody allowed for a shortened procedure with only a single 20 minute incubation with no false negatives caused by "hook" effects at high concentrations of ricin. Quantitation can be performed either with the sequential procedure or with the simultaneous procedure if it is know that the ricin concentration is not in the "hook" region. The simultaneous procedure should not be used when a sample contains constituents that may react with the ruthenium tag. Polyclonal/monoclonal antibodies are commercially available as an ELISA test kit.

Special Considerations: Crude preparations of ricin may also contain agglutinins that are unique to castor beans and that can cross-react in the immunoassays.

Source: Garber, E.A.E., and O'Brien, T. W. 2008. "Detection of Ricin in Food Using Electrochemiluminescence-Based Technology." Journal of AOAC International. 91(2): 376–382. http://www.atypon-link.com/AOAC/doi/abs/10.5555/jaoi.91.2.376

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8.2.3.2 Literature Reference for Ricin by Ricinine Detection (Journal of Analytical Toxicology. 2005. 29(3): 149–155)

Analysis Purpose: Complementary presumptive for ricin
Analytical Technique: LC-MS

Method Developed for: Ricinine in human and rat urine samples
Method Selected for: SAM lists these procedures for complementary presumptive analysis of ricin by ricinine detection in aerosol, solid, particulate, liquid, and water samples. Ricinine, an alkaloid component of castor beans, is found in crude preparations of ricin, and may be an indicator of ricin contamination. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for sample extraction by SPE, isocratic HPLC, followed by ESI tandem mass spectrometry. For MS analyses, protonated molecular ions are selected in the multiple reaction monitoring mode and quantified by isotope dilution with $^{13}C_6$-labeled ricinine as the internal reference. Urine pools enriched with ricinine at two concentrations were used as quality controls for validation of the method in urine samples. The calculated limit of detection was 0.04 ng/mL. In addition to the validation with urine samples, testing was performed on a single human urine sample (forensic), a crude ricin preparation, and urine samples from an animal ricinine exposure study. For the human urine sample, the concentration of ricinine was measured to be 4.24 ng/mL. After a series of simple extraction and filtration steps to provide a crude castor bean preparation, the final ricinine level was 502 ng/mL. For the animal exposure study, rats were injected with ricinine at 1, 5, and 10 mg/kg, with mean 24-hour urine concentrations of 1010, 6364, and 17,152 ng/mL, respectively. Mean 48-hour urine concentrations were 40, 324, and 610 mg/mL. Stability of ricinine in human urine was also tested, with ricinine found to be stable in human urine samples when heated at 90°C for 1 hour and when stored at 25°C and 5°C for 3 weeks.

Special Considerations: The following updated literature reference adds the analyte abrine for detection of select agent abrin: Rudolph C. Johnson, Yingtao Zhou, Ram Jain, Sharon W. Lemire, Shannon Fox, Pat Sabourin, and John R. Barr. 2009. "Quantification of L-Abrine in Human and Rat Urine: A Biomarker for the Toxin Abrin." Journal of Analytical Toxicology, 33, (2), 77−84.

Source: Johnson, R.C., Lemire, S.W., Woolfitt, Ospina, M., Preston, K.P, Olson, C.T., and Barr, J.R. 2005. "Quantification of Ricinine in Rat and Human Urine: A Biomarker for Ricin Exposure." Journal of Analytical Toxicology. 29(3): 149–155.
http://www.jatox.com/abstracts/2005/April/149-johnson.html

8.2.4 Shiga and Shiga-like Toxins (Stx, Stx-1, Stx-2)

CAS RN: 75757-64-1 (Stx).
Description: Protein composed of one ~32 kDa A chain and five 7.7 kDa B chains.

Method	Analytical Technique	Section
Pharmacology & Toxicology. 2001. 88(5): 255–260	Ribosome inactivation assay	8.2.1.4
FDA, Bacteriological Analytical Manual Online, January 2001, Appendix 1, Rapid Methods for Detecting Foodborne Pathogens	Immunoassay (ELISA)	8.2.4.1
Journal of Clinical Microbiology. 2007. 45(10): 3377–3380	Optical immunoassay	8.2.4.2

8.2.4.1 FDA, Bacteriological Analytical Manual Online, Appendix 1, 2001: Rapid Methods for Detecting Foodborne Pathogens

Analysis Purpose: Confirmatory

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Analytical Technique: Immunoassay (ELISA)

Method Developed for: Shiga and shiga-like toxins in food
Method Selected for: SAM lists this manual for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental samples.

Description of Method: Shiga toxin (Stx) is produced by *Shigella dysenteriae* and Shiga-like toxins (Shiga toxin Types 1 [Stx-1] and 2 [Stx-2]) are produced by various Shiga-toxigenic *E. coli* (STEC). An ELISA is described for the detection of these toxins. Diluted samples are added to microwells coated with an anti-Shiga toxin capture antibody. After incubation at room temperature, a wash is performed to remove unbound material. A second anti-Shiga toxin antibody is added for detection and incubation continued at room temperature. A wash is performed to remove unbound antibody. Enzyme conjugated anti-IgG visualization antibody, directed against the species from which the second anti-Shiga toxin antibody was derived, is added and the plate incubated then rinsed. Substrate is added, and after incubation to develop the color, stop solution is added. The results are interpreted spectrophotometrically.

Source: FDA, CFSAN. 2001. "Rapid Methods for Detecting Foodborne Pathogens." *Bacteriological Analytical Manual Online.* http://www.epa.gov/sam/pdfs/FDA-BAM-Appendix1.pdf

8.2.4.2 Literature Reference for Shiga and Shiga-like Toxins (Journal of Clinical Microbiology. 2007. 45(10): 3377–3380)

Analysis Purpose: Presumptive
Analytical Technique: Optical immunoassay

Method Developed for: Shiga toxin in foods
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for a rapid optical immunoassay for the detection of Stx-1 and Stx-2 using a commercially available kit. Fecal samples (742 specimens) are assayed for Shiga toxins with and without enrichment of the specimens in broth. Duplicate assays are applied using either the rapid optical immunoassay or a commercially available ELISA kit. Samples producing positive results by immunoassay are confirmed by Vero cell cytotoxicity assay. Sensitivities of 96.8% are achieved for direct stool sample assays.

Special Considerations: At the time of publication, the manufacturer no longer supports this assay. The CDC lists possible alternative kits for identification of Shiga toxin, in Table 4 of the following Web site: http://www.cdc.gov/mmwr/preview/mmwrhtml/rr5812a1.htm#tab4.

Source: Teel, L.D., Daly, J.A., Jerris, R.C., Maul, D., Svanas, G., O'Brien, A.D., and Park, C.H. 2007. "Rapid Detection of Shiga Toxin-Producing *Escherichia coli* by Optical Immunoassay." Journal of Clinical Microbiology. 45(10): 3377–3380. www.epa.gov/sam/pdfs/JCM-45(10)-pgs3377-3380.pdf

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8.2.5 Staphylococcal Enterotoxins (SEA, SEB, SEC)

CAS RNs: 37337-57-8 (SEA), 39424-53-8 (SEB), 39424-54-9 (SEC)
Description: Monomeric protein of ~ 28 kDa (SEB), monomeric proteins of ~ 27–27.5 kDa (SEA and SEC)

Method	Analytical Technique	Section
LRN	Immunoassay	8.1.4
AOAC Official Method 993.06	Immunoassay	8.2.5.1
Applied and Environmental Microbiology. 1997. 63(6): 2361–2365	T-cell proliferation assay	8.2.5.2

8.2.5.1 AOAC Official Method 993.06: Staphylococcal Enterotoxins in Selected Foods

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Staphylococcal enterotoxins in selected foods
Method Selected for: SAM lists this method for presumptive analysis of staphylococcal enterotoxins Type B (SEB) in aerosol samples, and Types A (SEA) and C (SEC) in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: This method is an enzyme immunoassay (EIA) using a mixture of high-affinity capture antibodies for identification of toxin(s) in food samples. Samples are prepared by dilution in Tris buffer, centrifugation, and filtration of the supernatant through a syringe, with adjustment to a final pH of 7.0 to 8.0. Samples are incubated in 96-well plates with the mixture of antibodies conjugated to horseradish peroxidase (HRP), and visualized with a peroxidase substrate. Assay results are determined visually or using a microtiter plate reader. Test is considered positive for staphylococcal enterotoxins if absorbance is >0.200 and is considered negative if absorbance is ≤0.200. Specific toxin serotypes are not differentiated. This method detects from 1.3 to 3.3 ng/mL staphylococcal enterotoxin in extracts prepared from food containing 4 to 10 ng/mL staphylococcal enterotoxin.

Source: AOAC International. 1994. "Method 991.06: Staphylococcal Enterotoxins in Selected Foods." *Official Methods of Analysis of AOAC International*. 16th Edition, 4th Revision; Vol. I. http://www.aoac.org/

8.2.5.2 Literature Reference for Staphylococcal Enterotoxins Types A, B, and C (Applied and Environmental Microbiology. 1997. 63(6): 2361–2365)

Analysis Purpose: Biological activity
Analytical Technique: T-cell proliferation assay

Method Developed for: Staphylococcal enterotoxin Type A (SEA) in selected foods
Method Selected for: SAM lists this method for biological activity assessment of staphylococcal enterotoxins Type B in aerosol samples, and Types A and C in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: This method is a T-cell proliferation assay using lymphocytes in a 96-well plate format for identification of staphyloccal enterotoxin(s) in food samples. Lyphocytes are prepared from heparinized Lewis rat blood or human blood using Ficoll-Paque™. Cells are

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aliquoted at 0.5×10^5 to 1.0×10^5 cell per well in 100 µL culture medium into a U-bottomed 96-well tissue culture plate. Food samples (potato salad, canned mushrooms, hot dogs, dry milk) are homogenized in PBS (1:1, wt/wt), centrifuged, the supernatants diluted 1:10 in PBS, and added directly to sample wells containing lymphocytes. Varying concentrations of SEA can be used as a standard curve. The treated samples are added to the lymphocytes and incubated for two to five days at 37°C. On the last day either 1 µCi of [methyl-^{3}H] thymidine or 20 µL of Alamar blue is added to the well. After 24 hours, supernatant is either harvested onto glass fiber filters and the beta-radioactivity counted or the color reaction of the Alamar blue treated wells is read on a plate reader at 570 nm. Both human and rat lymphocytes produce strong T-cell proliferation in response to SEA. The radioactive assay shows a significant level of proliferation ($P < 0.05$) as compared to control medium at levels as low as 0.1 pg SEA per well. The Alamar blue assay detects SEA at 1 ng per well. Diluted food samples without SEA do not induce T-cell proliferation.

Special Considerations: This method was developed for SEA in selected foods and has not been tested with SEB and SEC or in other sample types. However, because the T-cell proliferation assay is not antigen specific, the method may be appropriate for SEB and SEC, both of which have superantigen T-cell proliferation activity. This assay cannot identify the specific superantigen nor can it assess emetic activity; additional testing to determine specificity and assess toxin activity should be performed.

Source: Rasooly, L., Rose, N.R., Shah, D.B., and Rasooly, A. 1997. "In Vitro Assay of *Staphylococcus aureus* Enterotoxin A Activity in Food." Applied and Environmental Microbiology. 63(6): 2361–2365. www.epa.gov/sam/pdfs/AEM-63(6)-pgs2361-2365.pdf

8.3 Method Summaries for Small Molecule Biotoxins

Summaries of the analytical methods for small molecule biotoxins listed in Appendix D are provided in Sections 8.3.1 through 8.3.12. These sections contain summary information only, extracted from the selected methods. The full version of the method should be consulted prior to sample analysis. Each summary contains a brief description of the method, intended method application, performance data (if available), and a link to or source for obtaining a full version of the method.

8.3.1 Aflatoxin (Type B1)

CAS RN: 27261-02-5

Method	Analytical Technique	Section
AOAC Official Method 991.31	Immunoassay and HPLC-FL	8.3.1.1

8.3.1.1 AOAC Official Method 991.31: Aflatoxins in Corn, Raw Peanuts, and Peanut Butter

Analysis Purpose: Presumptive and confirmatory
Analytical Technique: Immunoassay and HPLC-FL

Method Developed for: Aflatoxins (Type B1) in corn, raw peanuts, and peanut butter
Method Selected for: SAM lists this method for presumptive and confirmatory analyses in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: This method is for the detection of aflatoxins in agricultural products. The sample is extracted with methanol-water (7 + 3), filtered, diluted with water, and applied to

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an affinity column containing mAbs specific for aflatoxins B1, B2 (CAS RN 22040-96-6), G1 (CAS RN 1385-95-1), and G2 (CAS RN 7241-98-7). Antibody-bound aflatoxins are removed from the column with methanol. For detection and quantitation of total aflatoxins, fluorescence measurement after reaction with bromine solution is performed. For individual aflatoxins, fluorescence detection and postcolumn iodine derivatization are performed and quantitation is by LC. Method performance was characterized using various commodities (e.g., corn) at aflatoxin levels over a range of 10 to 30 ng/g. This method was originally designed for the analysis of aflatoxins (B_1, B_2, G_1, and G_2) in samples where cleanup was necessary to remove food components, such as fats and proteins; the cleanup procedure may not be necessary for analysis of water samples.

Special Considerations: AOAC Official Method 994.08: Aflatoxin in Corn, Almonds, Brazil Nuts, Peanuts, and Pistachio Nuts, (AOAC International. 1998. *Official Methods of Analysis of AOAC International*, 16th Edition, 4th Revision, Vol. II. http://www.aoac.org/) may be used as a complementary HPLC-FL method in order to provide more flexibility for analyses.

Source: AOAC International. 1994. "Method 991.31: Aflatoxins in Corn, Raw Peanuts, and Peanut Butter." *Official Methods of Analysis of AOAC International*. 16th Edition, 4th Revision; Vol. II. http://www.aoac.org/

8.3.2 α-Amanitin

CAS RN: 23109-05-9

Method	Analytical Technique	Section
Journal of Chromatography B. 1991. 563(2): 299–311	HPLC amperometric detection	8.3.2.1
Journal of Food Protection. 2005. 68(6): 1294–1301	Immunoassay	8.3.2.2

8.3.2.1 Literature Reference for α-Amanitin (Journal of Chromatography B. 1991. 563(2): 299–311)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC with amperometric detection

Method Developed for: α-Amanitin in plasma
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for the selective determination in human plasma of α-amanitin using HPLC with amperometric detection. After extraction of plasma with disposable C_{18} silica cartridges, the extracts are separated by isocratic reversed-phase chromatography using a macroporous polystyrene-divinylbenzene column and a mobile phase of 0.05 M phosphate buffer-acetonitrile (91:9) at pH 9.5. Amperometric detection is performed by applying an oxidation potential as low as +350 mV (vs. Ag/AgCl) to a glassy carbon electrode, in a thin-layer flow-cell. The linear range for alpha-amanitin is 3 to 200 ng/mL, and the relative LOD in plasma is 2 ng/mL at a signal-to-noise ratio of 2. The intra-assay precision has been evaluated at levels of 10 and 200 ng/mL.

Source: Tagliaro, F., Schiavon, G., Bontempelli, G., Carli, G., and Marigo, M. 1991. "Improved High-performance Liquid Chromatographic Determination with Amperometric Detection of Alpha-amanitin in Human Plasma Based on its Voltammetric Study." Journal of Chromatography B. 563(2): 299–311. http://www.ncbi.nlm.nih.gov/pubmed/2055993

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8.3.2.2 Literature Reference for α-Amanitin, T-2 Mycotoxin (Journal of Food Protection. 2005. 68(6): 1294–1301)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: α-Amanitin, ricin, and T-2 mycotoxin in food and beverages
Method Selected for: SAM lists these procedures for presumptive analysis of α-amanitin and T-2 toxin in aerosol, solid, particulate, liquid, and water samples and for confirmatory analysis of ricin in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Commercially available ELISAs are described and assessed for detection of ricin, amanitin, and T-2 toxin at levels below those described as a health concern in food samples. Solid food samples are prepared by washing the sample with sodium phosphate buffer followed by dilution with phosphate-buffered saline. Liquid beverage samples are prepared by dilution in sodium phosphate buffer. Amanitin samples are similarly prepared using water instead of buffer, and T-2 toxin samples are similarly prepared using 35% methanol in water instead of buffer. The prepared samples are used with commercially obtained ELISA kits according to the manufacturer's directions, except for the incorporation of an eight-point calibration curve and reading the plates at both 405 and 650 nm after 26 minutes of incubation at 37°C. This assay detects ricin in food products at 0.01 µg/mL with acceptable background levels. Amanitin can be detected in food products at 1 µg/g with the ELISA. Background responses occurred, but at less than the equivalent of 0.5 ppm for amanitin. The ELISA kit will successfully detect T-2 toxin at targeted levels of 0.2 µg/g. The ELISA kit successfully detects T-2 toxin at targeted levels of 0.2 µg/g; the immunoassay for T-2 toxin, however, shows significant background responses and varies up to 0.1 ppm.

Source: Garber, E.A., Eppley, R.M., Stack, M.E., McLaughlin, M.A., and Park, D.L. 2005. "Feasibility of Immunodiagnostic Devices for the Detection of Ricin, Amanitin, and T-2 Toxin in Food." Journal of Food Protection. 68(6): 1294–1301.
http://www.ingentaconnect.com/content/iafp/jfp/2005/00000068/00000006/art00027

8.3.3 Anatoxin-a

CAS RN: 64285-06-9

Method	Analytical Technique	Section
Biomedical Chromatography. 1996. 10: 46–47	HPLC-FL (precolumn derivatization)	8.3.3.1

8.3.3.1 Literature Reference for Anatoxin-a (Biomedical Chromatography. 1996. 10(1): 46–47)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC-FL (precolumn derivatization)

Method Developed for: Anatoxin-a in potable water
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Procedures are described for HPLC analysis with fluorimetric detection of anatoxin-a in water samples after derivatization with 7-fluoro-4-nitro-2,1,3-benzoxadiazole (NBD-F). Samples are extracted at pH 7 with SPE using a weak cation exchanger. The toxin is eluted with methanol containing 0.2% trifluoroacetic acid (TFA). Samples are evaporated,

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reconstituted with acetonitrile, and re-evaporated prior to derivatization. This procedure detects anatoxin-a at concentrations of 0.1 µg/L with a good linear calibration.

Source: James, K.J., and Sherlock, I.R. 1996. "Determination of the Cyanobacterial Neurotoxin, Anatoxin-a, by Derivatisation Using 7-Fluoro-4-Nitro-2,1,3-Benzoxadiazole (NBD-F) and HPLC Analysis with Fluorimetric Detection." Biomedical Chromatography. 10(1): 46–47. http://www3.interscience.wiley.com/journal/18562/abstract

8.3.4 Brevetoxins (B form)

CAS RN: 79580-28-2

Method	Analytical Technique	Section
Environmental Health Perspectives. 2002. 110(2): 179–185	Immunoassay	8.3.4.1
Toxicon. 2004. 43(4): 455–465	HPLC-MS-MS	8.3.4.2

8.3.4.1 Literature Reference for Brevetoxins (Environmental Health Perspectives. 2002. 110(2): 179–185)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Brevetoxins in shellfish
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for a competitive ELISA used to detect brevetoxins in shellfish. The assay uses goat anti-brevetoxin antibodies in combination with a three-step signal amplification process: (1) secondary biotinylated antibodies; (2) streptavidin-HRP conjugate; and (3) chromogenic enzyme substrate. Sample preparation for liquids is dilution in PBS. Sample preparation for solids (oysters) is homogenization in PBS, or extraction in acetone. The working range for the assay is 0.2 to 2.0 ng/mL for diluted and undiluted liquid samples, and 0.2 to 2.0 ng/mL for solid samples, corresponding to 0.8 to 8.0 µg brevetoxins/100.0 g shellfish. The method has been compared to the mouse bioassay and is equivalent in sensitivity.

Source: Naar, J., Bourdelais, A., Tomas, C., Kubanek, J., Whitney, P.L., Flewelling, L., Steidinger, K., Lancaster, J., and Badan, D.G. 2002. "A Competitive ELISA to Detect Brevetoxins from *Karenia brevis* (Formerly *Gymnodinium breve*) in Seawater, Shellfish, and Mammalian Body Fluid." Environmental Health Perspectives. 110(2): 179–185. http://www.epa.gov/sam/pdfs/EHP-110(2)-pgs179-185.pdf

8.3.4.2 Literature Reference for Brevetoxins (Toxicon. 2004. 43(4): 455–465)

Analysis Purpose: Confirmatory
Analytical Technique: High performance liquid chromatography tandem mass spectrometers (HPLC-MS-MS)

Method Developed for: Brevetoxins in shellfish
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

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Description of Method: Shellfish sample homogenates are extracted with acetone, and centrifuged. The supernatants are combined, evaporated, and re-solubilized in 80% methanol. Following a wash with 95% n-hexane, the methanolic layer is evaporated, and the residue re-solubilized in 25% methanol and applied to a C_{18} SPE column. Analytes are eluted with 100% methanol, evaporated, and re-solubilized in methanol for analysis. Analysis of prepared samples is performed using HPLC-MS-MS with a mobile phase of water and acetonitrile with acetic acid. Analytes are detected by an MS with ESI interface. Brevetoxins are extensively metabolized, with many sub-forms. This method describes multiple liquid chromatography/electrospray ionization mass spectrometry (LC-ESI-MS) profiles for metabolites of brevetoxins from oysters.

Source: Wang, Z., Plakas, S.M., El Said, K.R., Jester, E.L., Granade, H.R., and Dickey, R.W. 2004. "LC/MS Analysis of Brevetoxin Metabolites in the Eastern Oyster (*Crassostrea virginica*)." Toxicon. 43(4): 455–465. http://cat.inist.fr/?aModele=afficheN&cpsidt=15668117

8.3.5 α-Conotoxin

CAS RN: 156467-85-5

Method	Analytical Technique	Section
Biochemical Journal. 1997. 328: 245–250	Immunoassay	8.3.5.1
Journal of Medicinal Chemistry. 2004. 47(5): 1234–1241	HPLC-MS	8.3.5.2

8.3.5.1 Literature Reference for α-Conotoxin (Biochemical Journal. 1997. 328(1): 245–250)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Purified α-Conotoxin GI in phosphate buffer
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: A biologically active fluorescein derivative of *Conus geographus* α-conotoxin (FGI) is used in solution-phase-binding assays with two purified *Torpedo californica* monoclonal antibodies (mAbs) to detect the toxin in laboratory samples. For competitive ligand-displacement spin-column assays, FGI was premixed with various dilutions of unlabelled ligands and then incubated with the two mAbs (5A1 and 8D2) at room temperature. Fluorescence is measured in ratio mode using cuvettes with excitation and emission monochromators set at gamma = 490 nm and gamma = 525 nm, respectively. The binding of FGI to the mAbs had apparent dissociation constants of 10 to 100 nM. The binding specificity and epitopes recognized by the two mAbs against α-conotoxin GI are also characterized. Competitive displacement assays showed that both mAbs specifically bound α-conotoxin GI with high avidity. Cross-reactivity with α-conotoxins M1 and S1 was not observed for either mAb in a direct ELISA. With spin-column assay, however, 5A1, but not 8D2, cross-reacted at a low level (100 – 300-fold less avid) with these α-conotoxins. An antibody/α-conotoxin GI molar ratio of 1:1 afforded complete protection in mouse lethal assays.

Source: Ashcom, J.D., and Stiles, B.G. 1997. "Characterization of α-Conotoxin Interactions with the Nicotinic Acetylcholine Receptor and Monoclonal Antibodies." Biochemical Journal. 328(1): 245–250. http://www.epa.gov/sam/pdfs/BJ-328-pgs245-250.pdf

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8.3.5.2 Literature Reference for α-Conotoxin (Journal of Medicinal Chemistry. 2004. 47(5): 1234–1241)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC-MS

Method Developed for: *Conus anemone* venom (α-Conotoxins AnIA, AnIB, and AnIC) in buffer
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are discussed for the detection of peptides within the α-conotoxin molecular mass range using an HPLC-MS. A crude extract of *Conus anemone* venom sample is made using 30% acetonitrile/water acidified with 0.1% TFA, with the insoluble portion of the sample removed by centrifugation. A portion of the sample extract is fractionated by size-exclusion chromatography in order to prepare a sample containing small peptides in the range of 1000 to 2500 Da. Chromatography conditions are elution with 30% acetonitrile / 0.048% TFA at a flow rate of 0.5 mL/minute, with detection at 214 nm. Three sulfated α-conotoxins (AnIA, AnIB, and AnIC) can be identified by LC-MS that are within the molecular mass range of other α-conotoxins (i.e., 1400–2200 Da). Peptides can be quantified by reversed-phase HPLC using an external reference standard for each peptide.

Source: Loughnan, M.L., Nicke, A., Jones, A., Adams, D.J., Alewood, P.F., and Lewis, R.J. 2004. "Chemical and Functional Identification and Characterization of Novel Sulfated Alpha-conotoxins from the Cone Snail *Conus anemone*." Journal of Medicinal Chemistry. 47(5): 1234–1241. http://pubs.acs.org/cgi-bin/abstract.cgi/jmcmar/2004/47/i05/abs/jm031010o.html

8.3.6 Cylindrospermopsin

CAS RN: 143545-90-8

Method	Analytical Technique	Section
FEMS Microbiology Letters. 2002. 216: 159-164	HPLC-PDA	8.3.6.1
ELISA Kits for Cylindrospermopsin	Immunoassay	8.3.6.2

8.3.6.1 Literature Reference for Cylindrospermopsin (FEMS Microbiology Letters. 2002. 216(2): 159–164)

Analysis Purpose: Confirmatory
Analytical Technique: High performance liquid chromatography – Photodiode array detector (HPLC-PDA)

Method Developed for: Cylindrospermopsin in eutrophic waters
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Cylindrospermopsin is detected using HPLC with photodiode array detector (PDA) in environmental waters. The suggested solvent range for cylindrospermopsin is below 50% methanol and 30% acetonitrile. Complex samples (culture medium) are purified on a C_{18} column with a linear gradient of 1 to 12% (v/v) methanol/water over 24 minutes at 40°C, with monitoring at 262 nm. The use of C_{18} columns for environmental waters is suggested for removal of the large number of organic compounds that may be present. This method detects and recovers cylindrospermopsin from spiked environmental water samples at 1 µg/L.

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Source: Metcalf, J.S., Beattie, K.A., Saker, M.L., and Codd, G.A. 2002. "Effects of Organic Solvents on the High Performance Liquid Chromatographic Analysis of the Cyanobacterial Toxin Cylindrospermopsin and Its Recovery from Environmental Eutrophic Waters by Solid Phase Extraction." FEMS Microbiology Letters. 216(2): 159–164.
http://cat.inist.fr/?aModele=afficheN&cpsidt=14002569

8.3.6.2 ELISA Kits for Cylindrospermopsin

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Cylindrospermopsin in ground water, surface water, and well water
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Cylindrospermopsin is detected using a colorimetric immunoassay (competitive ELISA) procedure. A sample (0.05 mL), enzyme conjugate (cylindrospermopsin-HRP), and an antibody solution containing rabbit anti-cylindrospermopsin antibodies are added to plate wells containing immobilized sheep anti-rabbit antibodies. Both the cylindrospermopsin (if present) in the sample and cylindrospermopsin-HRP conjugate compete in solution to bind to the rabbit anti-cylindrospermopsin antibodies in proportion to their respective concentrations. The anti-cylindrospermopsin antibody-target complexes are then bound to the immobilized sheep anti-rabbit antibodies on the plate. After incubation, the unbound molecules are washed and decanted. A specific substrate is then added which is converted from a colorless to a blue solution by the HRP enzyme conjugate solution. The reaction is terminated with the addition of a dilute acid. The concentration of cylindrospermopsin in the sample is determined photometrically by comparing sample absorbance to the absorbance of the calibrators (standards) at a specific wavelength (450 nm). The applicable concentration range is 0.4–2.0 µg/L, with a minimum detection level of 0.4 µg/L.

Source: NEMI. 2006.
http://infotrek.er.usgs.gov/pls/apex/f?p=119:38:7526698938332159::::P38_METHOD_ID:9507

8.3.7 Diacetoxyscirpenol (DAS)

CAS RN: 2270-40-8

Method	Analytical Technique	Section
International Journal of Food Microbiology. 1988. 6(1): 9–17	Immunoassay	8.3.7.1
Rapid Communications in Mass Spectrometry. 2006. 20(9): 1422–1428	LC/APCI-MS	8.3.7.2

8.3.7.1 Literature Reference for Diacetoxyscirpenol (DAS) (International Journal of Food Microbiology. 1988. 6(1): 9–17)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: DAS in food
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

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Description of Method: An ELISA is used for the detection of DAS in food samples. Antibodies against DAS are obtained after immunization of rabbits with DAS-hemiglutarate-human serum albumin (DAS-HG-HSA), and a DAS-hemisuccinate-HRP conjugate (DAS-HS-HRP) is prepared by an ester method for use as enzyme-labeled toxin in the competitive assay. The detection limit for DAS using this assay is approximately 10 pg/mL. This assay will cross-react related toxins. The relative cross-reactivities of the assay are 597.5, 5.2, 100.0, 2.5, and 1.5% for 3 alpha-acetyl-DAS, DAS, T-2 toxin, neosolaniol, and 15-acetoxyscirpenol, respectively.

Source: Klaffer, U., Martlbauer, E., and Terplan, G. 1988. "Development of a Sensitive Enzyme-linked Immunosorbent Assay for the Detection of Diacetoxyscirpenol." International Journal of Food Microbiology. 6(1): 9–17.
http://www.sciencedirect.com/science/journal/01681605

8.3.7.2 Literature Reference for Diacetoxyscirpenol (DAS) and T-2 Mycotoxin (Rapid Communications in Mass Spectrometry. 2006. 20(9): 1422–1428)

Analysis Purpose: Confirmatory
Analytical Technique: Liquid chromatography/atmospheric pressure chemical ionization mass spectrometry (LC/APCI-MS)

Method Developed for: DAS and T-2 mycotoxin in food
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: A LC/APCI-MS procedure based on time-of-flight mass spectrometry (TOFMS), with a real-time reference mass correction, is used for simultaneous determination of *Fusarium* mycotoxins (to include DAS and T-2 mycotoxin) in foodstuffs. Mycotoxin samples are extracted with acetonitrile/water (85:15) and centrifuged, and the supernatant is applied to a column for cleanup. Prepared samples are separated by liquid chromatography with an aqueous mobile phase of ammonium acetate and methanol detection is provided in exact mass chromatograms with a mass window of 0.03 Th. The limits of detection range from 0.1 to 6.1 ng/g in analyzed foodstuffs.

Source: Tanaka, H., Takino, M., Sugita-Konishi, Y., and Tanaka, T. 2006. "Development of Liquid Chromatography/Time-of-flight Mass Spectrometric Method for the Simultaneous Determination of Trichothecenes, Zearalenone, and Aflatoxins in Foodstuffs." Rapid Communications in Mass Spectrometry. 20(9): 1422–1428.
http://cat.inist.fr/?aModele=afficheN&cpsidt=17697070

8.3.8 Microcystins (Principal isoforms: LA, LR, LW, RR, YR)

CAS RNs: 96180-79-9 (LA), 101043-37-2 (LR), 157622-02-1 (LW), 111755-37-4 (RR), 101064-48-6 (YR)

Method	Analytical Technique	Section
Journal of AOAC International. 2001. 84(4): 1035–1044	Immunoassay/Phosphatase assay	8.3.8.1
Analyst. 1994. 119(7): 1525–1530	HPLC-PDA	8.3.8.2

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8.3.8.1 Literature Reference for Microcystins (Journal of AOAC International. 2001. 84(4): 1035–1044)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay/Phosphatase assay

Method Developed for: Microcystins-LA, -LR, -LW, -RR, -YR in algae products
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: ELISA and protein phosphatase inhibition assays are used to detect microcystins in blue-green algae products. Solid samples are prepared by homogenization in methanol (75% in water), with centrifugation to remove solids. Immunoassays are performed on the prepared samples using a commercially available ELISA test kit as described by the manufacturer. Samples are quantitated by comparison with a microcystins-LR standard curve. Quantitation with the colorimetric protein phosphatase inhibition assay is based on a comparison with a microcystin-LR standard curve. ELISA and phosphatase assay results agree over a concentration range of 0.5 to 35 µg/g. Neither assay is specific for a particular isoform.

Source: Lawrence, J.F., Niedzwiadek, B., Menard, C., Lau, B.P., Lewis, D., Kuper-Goodman, T., Carbone, S., and Holmes, C. 2001. "Comparison of Liquid Chromatography/Mass Spectrometry, ELISA, and Phosphatase Assay for the Determination of Microcystins in Blue-green Algae Products." Journal of AOAC International. 84(4): 1035–1044.
http://cat.inist.fr/?aModele=afficheN&cpsidt=1135453

8.3.8.2 Literature Reference for Microcystins (Analyst. 1994. 119(7): 1525–1530)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC-PDA

Method Developed for: Microcystins-LA, -LR, -LW, -RR, -YR in raw and treated waters
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Procedures are discussed to test the presence of microcystin-LR, -LY, -LW, -LF (CAS RN 154037-70-4), and -RR in treated and untreated water samples. Cyanobacterial cells are separated from the water by filtration through 110-mm glass fiber grade C (GF/C) discs. The cellular components collected on the discs are extracted three times with methanol; the collected extraction fluids are combined and dried. The residue is resuspended in methanol and analyzed by HPLC-PDA. The liquid portion of the filtered water sample is subjected to trace enrichment using a C_{18} SPE cartridge, followed by identification and determination by HPLC-PDA. This procedure can detect microcystin concentrations as low as 250 ng/L and is the basis of the World Health Organization (WHO) method for the detection of microcystins.

Source: Lawton, L.A., Edwards, C., and Codd, G.A. 1994. "Extraction and High-performance Liquid Chromatographic Method for the Determination of Microcystins in Raw and Untreated Waters." Analyst. 119(7): 1525–1530.
http://www.rsc.org/Publishing/Journals/AN/article.asp?doi=AN9941901525

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8.3.9 Picrotoxin

CAS RN: 124-87-8

Method	Analytical Technique	Section
Journal of Pharmaceutical and Biomedical Analysis. 1989. 7(3): 369–375	HPLC	8.3.9.1

8.3.9.1 Literature Reference for Picrotoxin (Journal of Pharmaceutical & Biomedical Analysis. 1989. 7(3): 369–375)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC

Method Developed for: Picrotoxin in serum
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for quantification of the two components of picrotoxin (picrotin [CAS RN 21416-53-5] and picrotoxinin [CAS RN 17617-45-7]) in serum samples. Serum samples are prepared by washing with *n*-hexane, followed by extraction with chloroform. The chloroform is evaporated and the sample is reconstituted in acetonitrile-1 mM ammonium acetate buffer (pH 6.4) 34:66 (v/v) for assay by reversed-phase HPLC. The effluent is monitored at 200 nm, and quantification is based on peak-height ratio of analyte to the internal standard. A linear response is obtained for both analytes (picrotin and picrotoxinin) in the range 0.2 to 20.0 µg/mL.

Source: Soto-Otero, R., Mendez-Alvarez, E., Sierra-Paredes, G., Galan-Valiente, J., Aguilar-Veiga, E., and Sierra-Marcuno, G. 1989. "Simultaneous Determination of the Two Components of Picrotoxin in Serum by Reversed-phase High-performance Liquid Chromatography with Application to a Pharmacokinetic Study in Rats." Journal of Pharmaceutical & Biomedical Analysis. 7(3): 369–375. http://www.sciencedirect.com/science/journal/07317085

8.3.10 Saxitoxins (Principal isoforms: STX, NEOSTX, GTX, dcGTX, dcSTX)

CAS RNs: 35523-89-8 (STX), 64296-20-4 (NEOSTX), 77462-64-7 (GTX), 58911-04-9 (dcSTX)

Method	Analytical Technique	Section
Journal of AOAC International. 1995. 78: 528–532	HPLC-FL (post column derivatization)	8.3.10.1
ELISA Kits for Saxitoxin	Immunoassay	8.3.10.2

8.3.10.1 Literature Reference for Saxitoxin (Journal of AOAC International. 1995. 78(2): 528–532)

Analysis Purpose: Confirmatory
Analytical Technique: HPLC-FL (post column derivatization)

Method Developed for: Saxitoxins (STX, NEOSTX, GTX, dcGTX, dcSTX) in shellfish
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described to detect multiple analogues of saxitoxin in shellfish using ion-interaction chromatography on a silica-based reversed-phase (C_8) column with

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postcolumn periodate oxidation and FL detection. Toxin groups of different net charges are determined separately by isocratic elution using either sodium 1-heptanesulfonate in ammonium phosphate (GTX-1, GTX-6, dcGTX2, dcGTX3) or sodium 1-heplanesulfonate in ammonium phosphate and acetonitrile (STX [CAS RN 35523-89-8], neoSTX [CAS RN 64296-20-4], dcSTX [CAS RN 58911-04-9]). For biological sample types, a cleanup procedure using a C_{18} SPE cartridge is effective in preventing false peaks. High sensitivity with detection limits ranging from 20 to 110 fmol are achieved as a result of reduced band broadening and optimized reaction conditions. This method, when applied to low-toxicity shellfish, gives higher values than the standard mouse bioassay.

Source: Oshima, Y. 1995. "Postcolumn Derivatization Liquid Chromatographic Method for Paralytic Shellfish Toxins." Journal of AOAC International. 78(2): 528–532.
http://cat.inist.fr/?aModele=afficheN&cpsidt=3469391

8.3.10.2 ELISA Kits for Saxitoxins

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: STX in water and solid samples (e.g., shellfish)
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types other than water.

Description of Method: Saxitoxin is detected using a colorimetric immunoassay (competitive ELISA) procedure. A sample (0.05 mL), enzyme conjugate (saxitoxin-HRP), and an antibody solution containing rabbit anti-saxitoxin antibodies are added to plate wells containing immobilized sheep anti-rabbit antibodies. Both the saxitoxin (if present) in the sample and saxitoxin-HRP conjugate compete in solution to bind to the rabbit anti-saxitoxin antibodies in proportion to their respective concentrations. The anti-saxitoxin antibody-target complexes are then bound to the immobilized sheep anti-rabbit antibodies on the plate. After incubation, the unbound molecules are washed and decanted. A specific substrate is then added which is converted from a colorless to a blue solution by the HRP enzyme conjugate solution. The reaction is terminated with the addition of a dilute acid. The concentration of saxitoxin in the sample is determined photometrically by comparing sample absorbance to the absorbance of the calibrators (standards) at a specific wavelength (450 nm). The applicable concentration range is 0.015–0.4 ng/mL, with a minimum detection level of 0.015 ng/mL.

Special Considerations: This kit is not intended for other types of saxitoxin. Cross-reactivity is observed with the following saxitoxin types: dcSTX (29%), GTX 2, 3, and 5B (23%), sulfo GTX 1 and 2 (2.0%, dcGTX 2 and 3 (1.4%), NEOSTX (1.3%), dcNEOSTX (0.6%), GTX 1 and 4 (<0.2%). High concentrations (e.g., above 0.1 ng/mL for toxins with >20% cross-reactivity) of these other types of saxitoxin may produce false positive responses.

Source: NEMI. 2006.
http://infotrek.er.usgs.gov/pls/apex/f?p=119:38:8989971104293493::::P38_METHOD_ID:9512

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8.3.11 T-2 Mycotoxin

CAS RN: 21259-20-1

Method	Analytical Technique	Section
Journal of Food Protection. 2005. 68(6): 1294–1301	Immunoassay	8.3.2.2
Rapid Communications in Mass Spectrometry. 2006. 20(9): 1422–1428	LC/APCI-MS	8.3.7.2

See Sections 8.3.2.2 and 8.3.7.2 for information on immunoassay and LC/APCI-MS procedures for T-2 Mycotoxin.

8.3.12 Tetrodotoxin

CAS RN: 9014-39-5

Method	Analytical Technique	Section
Analytical Biochemistry. 2001. 290: 10-17	LC/ESI-MS	8.3.12.1
Journal of Clinical Laboratory Analysis. 1992. 6: 65–72	Immunoassay	8.3.12.2

8.3.12.1 Literature Reference for Tetrodotoxin (Analytical Biochemistry. 2001. 290(1): 10–17)

Analysis Purpose: Confirmatory
Analytical Technique: LC/ESI-MS

Method Developed for: Tetrodotoxin (TTX) from puffer fish and newt tissues
Method Selected for: SAM lists these procedures for confirmatory analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

Description of Method: Procedures are described for LC/ESI-MS analysis of TTXs in tissue samples from puffer fish and newts by a combination of chromatography on a reversed-phase column with long carbon chains (C30) and with the mobile phase containing an ion pair reagent (ammonium heptafluorobutyrate). The relationship between the amount of applied standard TTX and its peak area on the mass chromatogram (m/z 320) shows good linearity over a range of 50 to 1000 pmol. The detection limit of TTX in the SIM mode is estimated to be 0.7 pmol, with a signal to noise ratio of 2:1.

Source: Shoji, Y., Yotsu-Yamashita, M., Miyazawa, T., and Yasumoto, T. 2001. "Electrospray Ionization Mass Spectrometry of Tetrodotoxin and its Analogs: Liquid Chromatography/Mass Spectrometry, Tandem Mass Spectrometry, and Liquid Chromatography/Tandem Mass Spectrometry." Analytical Biochemistry. 290(1): 10–17.
http://www.sciencedirect.com/science/journal/00032697

8.3.12.2 Literature Reference for Tetrodotoxin (Journal of Clinical Laboratory Analysis. 1992. 6(2): 65–72)

Analysis Purpose: Presumptive
Analytical Technique: Immunoassay

Method Developed for: Tetrodotoxin in buffer
Method Selected for: SAM lists these procedures for presumptive analysis in aerosol, solid, particulate, liquid, and water samples. Further research is needed to develop and standardize the procedures for environmental sample types.

OUTDATED

Description of Method: Procedures are described for a competitive inhibition enzyme immunoassay (CIEIA) for tetrodotoxin in biological samples. An anti-TTX mAb, designated T20G10, is directly labeled with alkaline phosphatase for use in the assay. Sensitivities of 6 to 7 ng/mL (IC 50) and 2 to 3 ng/mL (IC 20) are achieved.

Source: Raybould, T.J., Bignami, G.S., Inouye, L.K., Simpson, S.B., Byrnes, J.B., Grothaus, P.G., and Vann, D.C. 1992. "A Monoclonal Antibody-based Immunoassay for Detecting Tetrodotoxin in Biological Samples." Journal of Clinical Laboratory Analysis. 6(2): 65–72. http://www3.interscience.wiley.com/journal/112131435/abstract

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Section 9.0: Conclusions

Methods listed in Appendix A (chemical methods), Appendix B (radiochemical methods), and Appendix D (biotoxin methods) are recommended for use in assessment of the extent of contamination and the effectiveness of decontamination following a homeland security event. For pathogen methods, please refer to the points of contact listed in the Appendix C text box, or Appendix C in SAM 5.0.

The primary objective of this document is not necessarily to identify the "best" analytical methods, but rather to identify appropriate methods that represent a balance between providing existing, documented, determinative techniques and providing consistent and valid analytical results. The method selected for each analyte/sample type combination was deemed the most general, appropriate, and broadly applicable of available methods. This is a living document, which can be used as a guide by EPA and EPA-contracted laboratories tasked with analysis of environmental samples following a homeland security event. Recommended methods are subject to change based on procedure testing and advances in technology.

Any questions concerning the information in this document should be directed to the appropriate point(s) of contact listed in Section 4.

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Appendix A: Selected Chemical Methods

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Appendix A: Selected Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Acephate	30560-19-1	LC-MS-MS	Sample Prep	Adapted from Journal of Chromatography A, 1154(1): 3-25	Adapted from Chromatographia, 63(5/6): 233-237	538 (EPA OW)	Adapted from Journal of Chromatography A, 1154(1): 3-25	Adapted from Journal of Chromatography A, 1154(1): 3-25
			Determinative					
Acrylamide	79-06-1	HPLC	Sample Prep	Water extraction	8316 (EPA SW-846)	8316 (EPA SW-846)	PV2004 (OSHA)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8316 (EPA SW-846)				8316 (EPA SW-846)
Acrylonitrile	107-13-1	HPLC	Sample Prep	5035A (EPA SW-846)	524.2 (EPA OW)	524.2 (EPA OW)	PV2004 (OSHA)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8260C (EPA SW-846)				8260C (EPA SW-846)
Aldicarb (Temik)	116-06-3	HPLC	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Aldicarb sulfone	1646-88-4	HPLC	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Aldicarb sulfoxide	1646-87-3	HPLC	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Allyl alcohol	107-18-6	GC-MS	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	TO-15[21] (EPA ORD)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
4-Aminopyridine	504-24-5	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Ammonia	7664-41-7	Visible spectrophotometry	Sample Prep	Not of concern	4500- NH_3 B (SM)	350.1 (EPA OW)	6015 (NIOSH)	Not of concern
			Determinative		4500- NH_3 G (SM)			
Ammonium metavanadate (analyze as total vanadium)	7803-55-6	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Arsenic, Total	7440-38-2	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Arsenic trioxide (analyze as total arsenic)	1327-53-3	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Arsine (analyze as total arsenic in non-air samples)	7784-42-1	GFAA / ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	6001 (NIOSH)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)				6010C/6020A (EPA SW-846)
Asbestos	1332-21-4	TEM	Sample Prep	D5755-03 (soft surfaces-microvac) (ASTM)	Not of concern	Not of concern	10312:1995 (ISO)	D6480-05 (hard surfaces-wipes) (ASTM)
			Determinative					
Boron trifluoride	7637-07-2	ISE	Sample Prep	Not of concern	Not of concern	Not of concern	ID216SG (OSHA)	Not of concern
			Determinative					
Brodifacoum	56073-10-0	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)	8321B (EPA SW-846)	8321B (EPA SW-846)		8321B (EPA SW-846)
Bromadiolone	28772-56-7	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7600-09 (ASTM)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)		8321B (EPA SW-846)		8321B (EPA SW-846)
BZ [Quinuclidinyl benzilate]	6581-06-2	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Calcium arsenate (analyze as total arsenic)	7778-44-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Carbofuran (Furadan)	1563-66-2	HPLC / LC-MS-MS	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Carbon disulfide	75-15-0	GC-MS	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	524.2 (EPA OW)	TO-15 (EPA ORD)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)			
Carfentanil	59708-52-0	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)	8321B (EPA SW-846)	8321B (EPA SW-846)		8321B (EPA SW-846)
Chlorfenvinphos	470-90-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Chlorine	7782-50-5	Visible spectrophotometry	Sample Prep	Not of concern	4500-Cl G (SM)	4500-Cl G (SM)	Adapted from Analyst, 124(12): 1853-1857	Not of concern
			Determinative				4500-Cl G (SM)	
2-Chloroethanol	107-07-3	GC-MS / GC-FID	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	2513 (NIOSH)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
3-Chloro-1,2-propanediol	96-24-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[4] (EPA SW-846)	8270D[4] (EPA SW-846)	8270D[4] (EPA SW-846)		8270D[4] (EPA SW-846)
Chloropicrin	76-06-2	GC-MS / GC-ECD	Sample Prep	3541/'3545A (EPA SW-846)	551.1 (EPA OW)	551.1 (EPA OW)	PV2103 (OSHA)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[6] (EPA SW-846)				8270D[6] (EPA SW-846)
Chlorosarin	1445-76-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Chlorosoman	7040-57-5	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
2-Chlorovinylarsonous acid (2-CVAA) (degradation product of Lewisite)	85090-33-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Chlorpyrifos	2921-88-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Chlorpyrifos oxon	5598-15-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Crimidine	535-89-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[7] (EPA SW-846)	8270D[7] (EPA SW-846)	8270D[7] (EPA SW-846)		8270D[7] (EPA SW-846)
Cyanide, Amenable to chlorination	NA	Visible spectrophotometry	Sample Prep	3135.2I (EPA RLAB)	3135.2I (EPA RLAB)	3135.2I (EPA RLAB)	Not of concern	3135.2I (EPA RLAB)
			Determinative					
Cyanide, Total	57-12-5	Visible spectrophotometry	Sample Prep	ISM01.2 CN (EPA CLP)	ISM01.2 CN (EPA CLP)	335.4 (EPA OW)	6010 (NIOSH)	ISM01.2 CN (EPA CLP)
			Determinative					

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Cyanogen chloride	506-77-4	GC-MS	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	TO-15 (EPA ORD)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
Cyclohexyl sarin (GF)	329-99-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
1,2-Dichloroethane (degradation product of HD)	107-06-2	GC-MS	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	524.2 (EPA OW)	TO-15 (EPA ORD)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)			
Dichlorvos	62-73-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	525.2 (EPA OW)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)			8270D (EPA SW-846)
Dicrotophos	141-66-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Diesel range organics	NA	GC-FID	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8015C (EPA SW-846)	8015C (EPA SW-846)	8015C (EPA SW-846)		8015C (EPA SW-846)
Diisopropyl methylphosphonate (DIMP) (degradation product of GB)	1445-75-6	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7597-09 (ASTM)	538 (EPA OW)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[8] (EPA SW-846)				8321B[8] (EPA SW-846)
Dimethylphosphite	868-85-9	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Dimethylphosphoramidic acid (degradation product of GA)	33876-51-6	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Diphacinone	82-66-6	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)	8321B (EPA SW-846)	8321B (EPA SW-846)		8321B (EPA SW-846)
Disulfoton	298-04-4	GC-MS / GC-FPD	Sample Prep	3541/3545A (EPA SW-846)	525.2 (EPA OW)	525.2 (EPA OW)	5600 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)				8270D (EPA SW-846)
Disulfoton sulfone oxon[9]	2496-91-5	GC-MS / GC-FPD	Sample Prep	3541/3545A (EPA SW-846)	525.2 (EPA OW)	525.2 (EPA OW)	5600 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)				8270D (EPA SW-846)

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Disulfoton sulfoxide	2497-07-6	GC-MS / GC-FPD	Sample Prep	3541/3545A (EPA SW-846)	525.2 (EPA OW)	525.2 (EPA OW)	5600 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)				8270D (EPA SW-846)
Disulfoton sulfoxide oxon[9]	2496-92-6	GC-MS / GC-FPD	Sample Prep	3541/3545A (EPA SW-846)	525.2 (EPA OW)	525.2 (EPA OW)	5600 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)				8270D (EPA SW-846)
1,4-Dithiane (degradation product of HD)	505-29-3	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
EA2192 [Diisopropylaminoethyl methylthiolophosphonate] (hydrolysis product of VX)	73207-98-4	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Ethyl methylphosphonic acid (EMPA) (degradation product of VX)	1832-53-7	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7597-09 (ASTM)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Ethyldichloroarsine (ED)	598-14-1	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-15 (EPA ORD)	9102 (NIOSH)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
N-Ethyldiethanolamine (EDEA) (degradation product of HN-1)	139-87-7	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7599-09 (ASTM)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)		8321B (EPA SW-846)		8321B (EPA SW-846)
Ethylene oxide	75-21-8	GC-MS	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	TO-15 (EPA ORD)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
Fenamiphos	22224-92-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	525.2 (EPA OW)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)			8270D (EPA SW-846)
Fentanyl	437-38-7	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)	8321B (EPA SW-846)	8321B (EPA SW-846)		8321B (EPA SW-846)
Fluoride	16984-48-8	IC-conductivity detection	Sample Prep	Not of concern	300.1, Rev 1.0 (EPA OW)	300.1, Rev 1.0 (EPA OW)	Not of concern	Not of concern
			Determinative					
Fluoroacetamide	640-19-7	GC-MS	Sample Prep	Adapted from Journal of Chromatography B, 876(1): 103-108	Adapted from Journal of Chromatography B, 876(1): 103-108	Adapted from Journal of Chromatography B, 876(1): 103-108	Adapted from Journal of Chromatography B, 876(1): 103-108	Adapted from Journal of Chromatography B, 876(1): 103-108
			Determinative					

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Fluoroacetic acid and fluoroacetate salts (analyze as fluoroacetate ion)	NA	IC	Sample Prep	Adapted from Analytical Letters, 27(14): 2703-2718	300.1, Rev 1.0 (EPA OW)	300.1, Rev 1.0 (EPA OW)	S301-1 (NIOSH)	Adapted from Analytical Letters, 27)14): 2703-2718
			Determinative	300.1, Rev 1.0 (EPA OW)			300.1, Rev 1.0 (EPA OW)	300.1, Rev 1.0 (EPA OW)
2-Fluoroethanol	371-62-0	GC-MS / GC-FID	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	2513 (NIOSH)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
Formaldehyde	50-00-0	FGC-ECD / HPLC	Sample Prep	8315A (EPA SW-846)	8315A (EPA SW-846)	556.1 (EPA OW)	2016 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8315A (EPA SW-846)
Gasoline range organics	NA	GC-FID	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8015C (EPA SW-846)	8015C (EPA SW-846)	8015C (EPA SW-846)		8015C (EPA SW-846)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Hexamethylenetriperoxidediamine (HMTD)	283-66-9	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Hydrogen bromide	10035-10-6	IC-conductivity detection	Sample Prep	Not of concern	Not of concern	Not of concern	7903 (NIOSH)	Not of concern
			Determinative					
Hydrogen chloride	7647-01-0	IC-conductivity detection	Sample Prep	Not of concern	Not of concern	Not of concern	7903 (NIOSH)	Not of concern
			Determinative					
Hydrogen cyanide	74-90-8	Visible spectrophotometry	Sample Prep	Not of concern	Not of concern	Not of concern	6010 (NIOSH)	Not of concern
			Determinative					
Hydrogen fluoride	7664-39-3	IC-conductivity detection	Sample Prep	Not of concern	Not of concern	Not of concern	7903[10] (NIOSH)	Not of concern
			Determinative					
Hydrogen sulfide	7783-06-4	IC-conductivity detection	Sample Prep	Not of concern	Not of concern	Not of concern	6013 (NIOSH)	Not of concern
			Determinative					

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Isopropyl methylphosphonic acid (IMPA) (degradation product of GB)	1832-54-8	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7597-09 (ASTM)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Kerosene	64742-81-0	GC-FID	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8015C (EPA SW-846)	8015C (EPA SW-846)	8015C (EPA SW-846)		8015C (EPA SW-846)
Lead arsenate (analyze as total arsenic)	7645-25-2	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Lewisite 1 (L-1)[11] [2-chlorovinyldichloroarsine] (analyze as total arsenic)	541-25-3	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Lewisite 2 (L-2) [bis(2-chlorovinyl)chloroarsine] (analyze as total arsenic)	40334-69-8	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Lewisite 3 (L-3) [tris(2-chlorovinyl)arsine] (analyze as total arsenic)	40334-70-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Lewisite oxide (degradation product of Lewisite)	1306-02-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Mercuric chloride (analyze as total mercury)	7487-94-7	Visible spectrophotometry / CVAA / CVAFS	Sample Prep	7473[12] (EPA SW-846)	7473[12] (EPA SW-846)	245.1 (EPA OW)	Not of concern	9102 (NIOSH)
			Determinative					7473[12] (EPA SW-846)
Mercury, Total	7439-97-6	Visible spectrophotometry / CVAA / CVAFS	Sample Prep	7473[12] (EPA SW-846)	7473[12] (EPA SW-846)	245.1 (EPA OW)	IO-5 (EPA ORD)	9102 (NIOSH)
			Determinative					7473[12] (EPA SW-846)
Methamidophos	10265-92-6	LC-MS-MS	Sample Prep	Adapted from Journal of Chromatography A, 1154(1): 3-25	Adapted from Chromatographia, 63(5/6): 233-237	538 (EPA OW)	Adapted from Journal of Chromatography A, 1154(1): 3-25	Adapted from Journal of Chromatography A, 1154(1): 3-25
			Determinative					
Methomyl	16752-77-5	HPLC / LC-MS-MS	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Methoxyethylmercuric acetate (analyze as total mercury)	151-38-2	Visible spectrophotometry / CVAA / CVAFS	Sample Prep	7473[12] (EPA SW-846)	7473[12] (EPA SW-846)	245.1 (EPA OW)	IO-5 (EPA ORD)	9102 (NIOSH)
			Determinative					7473[12] (EPA SW-846)

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Methyl acrylonitrile	126-98-7	HPLC	Sample Prep	5035A (EPA SW-846)	524.2 (EPA OW)	524.2 (EPA OW)	PV2004 (OSHA)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8260C (EPA SW-846)				8260C (EPA SW-846)
Methyl fluoroacetate (analyze as fluoroacetate ion)	453-18-9	IC	Sample Prep	Adapted from Analytical Letters, 27(14): 2703-2718	300.1, Rev 1.0 (EPA OW)	300.1, Rev 1.0 (EPA OW)	S301-1 (NIOSH)	Adapted from Analytical Letters 27(14): 2703-2718
			Determinative	300.1, Rev 1.0 (EPA OW)			300.1, Rev 1.0 (EPA OW)	300.1, Rev 1.0 (EPA OW)
Methyl hydrazine	60-34-4	GC-MS / visible spectrophotometry	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	3510 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Methyl isocyanate	624-83-9	HPLC	Sample Prep	Not of concern	Not of concern	Not of concern	OSHA 54	Not of concern
			Determinative					
Methyl paraoxon	950-35-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Methyl parathion	298-00-0	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Methylamine	74-89-5	HPLC	Sample Prep	Not of concern	Not of concern	Not of concern	OSHA 40	Not of concern
			Determinative					
N-Methyldiethanolamine (MDEA) (degradation product of HN-2)	105-59-9	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7599-09 (ASTM)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)		8321B (EPA SW-846)		8321B (EPA SW-846)
1-Methylethyl ester ethylphosphonofluoridic acid (GE)	1189-87-3	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Methylphosphonic acid (MPA) (degradation product of VX, GB, & GD)	993-13-5	HPLC	Sample Prep	3541/3545A (EPA SW-846)	D7597-09 (ASTM)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)
Mevinphos	7786-34-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	525.2 (EPA OW)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)			8270D (EPA SW-846)

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Monocrotophos	6923-22-4	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Mustard, nitrogen (HN-1) [bis(2-chloroethyl)ethylamine]	538-07-8	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Mustard, nitrogen (HN-2) [2,2'-dichloro-N-methyldiethylamine N,N-bis(2-chloroethyl)methylamine]	51-75-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Mustard, nitrogen (HN-3) [tris(2-chloroethyl)amine]	555-77-1	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Mustard, sulfur / Mustard gas (HD)	505-60-2	GC-MS	Sample Prep	3571 (EPA SW-846)	3571 (EPA SW-846)	3571 (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)		8270D[13] (EPA SW-846)
Nicotine compounds (analyze as nicotine)	54-11-5	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Osmium tetroxide (analyze as total osmium)	20816-12-0	ICP-AES	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C (EPA SW-846)			IO-3.4 (EPA ORD)	6010C (EPA SW-846)
Oxamyl	23135-22-0	HPLC / LC-MS-MS	Sample Prep	8318A (EPA SW-846)	D7600-09 (ASTM)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					8318A (EPA SW-846)
Paraquat	4685-14-7	HPLC-UV	Sample Prep	Not of concern	549.2 (EPA OW)	549.2 (EPA OW)	Not of concern	Not of concern
			Determinative					
Paraoxon	311-45-5	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Parathion	56-38-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Pentaerythritol tetranitrate (PETN)	78-11-5	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Phencyclidine	77-10-1	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phorate	298-02-2	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phorate sulfone	2588-04-7	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phorate sulfone oxon[9]	2588-06-9	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phorate sulfoxide	2588-03-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phorate sulfoxide oxon[9]	2588-05-8	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phosgene	75-44-5	GC-NPD	Sample Prep	Not of concern	Not of concern	Not of concern	OSHA 61	Not of concern
			Determinative					
Phosphamidon	13171-21-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Phosphine	7803-51-2	Visible spectrophotometry	Sample Prep	Not of concern	Not of concern	Not of concern	6002 (NIOSH)	Not of concern
			Determinative					
Phosphorus trichloride	7719-12-2	Visible spectrophotometry	Sample Prep	Not of concern	Not of concern	Not of concern	6402 (NIOSH)	Not of concern
			Determinative					
Pinacolyl methyl phosphonic acid (PMPA) (degradation product of GD)	616-52-4	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7597-09 (ASTM)	3535A (EPA SW-846)	TO-10A[3] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)		8321B[2] (EPA SW-846)

OUTDATED

Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Propylene oxide	75-56-9	GC-MS / GC-FID	Sample Prep	5035A (EPA SW-846)	5030C (EPA SW-846)	5030C (EPA SW-846)	1612 (NIOSH)	Not of concern
			Determinative	8260C (EPA SW-846)	8260C (EPA SW-846)	8260C (EPA SW-846)		
R 33 (VR) [methylphosphonothioic acid, S-[2-(diethylamino)ethyl] O-2-methylpropyl ester]	159939-87-4	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Sarin (GB)	107-44-8	GC-MS	Sample Prep	3571 (EPA SW-846)	3571 (EPA SW-846)	3571 (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)		8270D[13] (EPA SW-846)
Sodium arsenite (analyze as total arsenic)	7784-46-5	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
Sodium azide (analyze as azide ion)	26628-22-8	IC-UV	Sample Prep	Adapted from J. of Forensic Sciences, 43(1): 200-202[14]	Adapted from J. of Forensic Sciences, 43(1): 200-202[14]	Adapted from J. of Forensic Sciences, 43(1): 200-202[14]	ID-211 (OSHA)	ID-211 (OSHA)
			Determinative	300.1, Rev 1.0[15] (EPA OW)	300.1, Rev 1.0[15] (EPA OW)	300.1, Rev 1.0[15] (EPA OW)		
Soman (GD)	96-64-0	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A[5] (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Strychnine	57-24-9	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Tabun (GA)	77-81-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Tetraethyl pyrophosphate	107-49-3	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
Tetramethylenedisulfotetramine	80-12-6	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[6] (EPA SW-846)	8270D[6] (EPA SW-846)	8270D[6] (EPA SW-846)		8270D[6] (EPA SW-846)
Thallium sulfate (analyze as total thallium)	10031-59-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6020A/6010C (EPA SW-846)
Thiodiglycol (TDG) (degradation product of HD)	111-48-8	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7598-09 (ASTM)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)		8321B (EPA SW-846)		8321B (EPA SW-846)

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
Thiofanox	39196-18-4	HPLC	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	531.2 (EPA OW)	5601 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)	8321B (EPA SW-846)			8321B (EPA SW-846)
1,4-Thioxane (degradation product of HD)	15980-15-1	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[16] (EPA SW-846)	8270D[16] (EPA SW-846)	8270D[16] (EPA SW-846)		8270D[16] (EPA SW-846)
Titanium tetrachloride (analyze as total titanium)	7550-45-0	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	Not of concern	Not of concern	Not of concern	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)				6010C/6020A (EPA SW-846)
Triethanolamine (TEA) (degradation product of HN-3)	102-71-6	HPLC / LC-MS-MS	Sample Prep	3541/3545A (EPA SW-846)	D7599-09 (ASTM)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8321B (EPA SW-846)		8321B (EPA SW-846)		8321B (EPA SW-846)
Trimethyl phosphite	121-45-9	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3535A (EPA SW-846)	3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[6] (EPA SW-846)	8270D[6] (EPA SW-846)	8270D[6] (EPA SW-846)		8270D[6] (EPA SW-846)
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7	HPLC	Sample Prep	8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	3535A/8330B (EPA SW-846)	Not of concern	3570/8290A Appendix A (EPA SW-846)
			Determinative		8330B (EPA SW-846)	8330B (EPA SW-846)		8330B (EPA SW-846)
Vanadium pentoxide (analyze as total vanadium)	1314-62-1	ICP-AES / ICP-MS	Sample Prep	3050B (EPA SW-846)	200.7/200.8 (EPA OW)	200.7/200.8 (EPA OW)	IO-3.1 (EPA ORD)	9102 (NIOSH)
			Determinative	6010C/6020A (EPA SW-846)			IO-3.4/IO-3.5 (EPA ORD)	6010C/6020A (EPA SW-846)
VE [phosphonothioic acid, ethyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21738-25-0	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
VG [phosphonothioic acid, S-(2-(diethylamino)ethyl) O,O-diethyl ester]	78-53-5	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
VM [phosphonothioic acid, methyl-, S-(2-(diethylamino)ethyl) O-ethyl ester]	21770-86-5	GC-MS	Sample Prep	3541/3545A (EPA SW-846)	3520C/3535A (EPA SW-846)	3520C/3535A (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D (EPA SW-846)	8270D (EPA SW-846)	8270D (EPA SW-846)		8270D (EPA SW-846)
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl-phosphonothiolate]	50782-69-9	GC-MS	Sample Prep	3571 (EPA SW-846)	3571 (EPA SW-846)	3571 (EPA SW-846)	TO-10A (EPA ORD)	3570/8290A Appendix A (EPA SW-846)
			Determinative	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)	8270D[13] (EPA SW-846)		8270D[13] (EPA SW-846)

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Analyte(s)	CAS RN	Determinative Technique	Method Type	Solid Samples	Aqueous Liquid Samples	Drinking Water Samples	Air Samples	Wipes
White phosphorus	12185-10-3	GC-NPD / GC-FPD	Sample Prep	7580 (EPA SW-846)	7580 (EPA SW-846)	7580 (EPA SW-846)	7905 (NIOSH)	3570/8290A Appendix A (EPA SW-846)
			Determinative					7580 (EPA SW-846)

Footnotes

[1] If problems occur when using this method, it is recommended that TO-10A be used.

[2] LC-MS (electrospray) procedures are preferred for these analytes; however, if this technique is not available to the laboratory, GC-MS procedures using derivatization based on SW-846 Method 8270D may be used. Sample preparation methods should remain the same. Both electrospray LC-MS and GC-MS derivatization procedures are currently under development.

[3] For this analyte, HPLC is the preferred technique; however, if problems occur, Method TO-10A must be modified to include a derivatization step prior to analysis by GC-MS.

[4] For this analyte, SW-846 Method 8270D must be modified to include a derivatization step.

[5] If problems occur when using this method, it is recommended that the canister Method TO-15 be used.

[6] If problems occur with analyses, lower the injection temperature.

[7] If problems occur when using this method, it is recommended that SW-846 Method 8321B be used as the determinative method. Sample preparation methods should remain the same.

[8] If problems occur with the analysis of DIMP using EPA SW-846 Method 8321B, use SW-846 Method 8270D.

[9] If problems occur during measurement of oxon compounds, analysts should consider use of procedures included in Kamal, A. et al., "Oxidation of selected organophosphate pesticides during chlorination of simulated drinking water." Water Research. 2009. 43(2): 522–534. http://www.sciencedirect.com/science/journal/00431354

[10] If problems occur when using this method, it is recommended that NIOSH Method 7906 be used.

[11] Laboratory testing is currently under way for speciation of Lewisite 1 using GC-MS techniques.

[12] If equipment is not available, use CVAA Methods 7471B (EPA SW-846) for solid samples and 7470A (EPA SW-846) for aqueous liquid samples.

[13] For this analyte, refer to EPA SW-846 Method 8271 for GC-MS conditions.

[14] Water extraction, filtration, and acidification steps from the Journal of Forensic Science. 1998. 43(1): 200-202 should be used for the preparation of solid samples. Filtration and acidification steps from this journal should be used for preparation of aqueous liquid and drinking water samples.

[15] If analyses are problematic, refer to column manufacturer for alternate conditions

[16] If problems occur when using this method, it is recommended that SW-846 Method 8260C and appropriate corresponding sample preparation procedures (i.e., 5035A for solid samples, and 5030C for aqueous liquid and drinking water samples) be used.

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Appendix B: Selected Radiochemical Methods

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Appendix B: Selected Radiochemical Methods

Analyte Class	Determinative Technique	Drinking Water Samples	Aqueous and Liquid Phase Samples	Soil and Sediment Samples	Surface Wipes	Air Filters
Gross Alpha	Alpha/Beta counting	900.0 (EPA)	7110 B (SM)	AP1 (ORISE)	FRMAC, Vol 2, pg. 33 (DOE)	FRMAC, Vol 2, pg. 33 (DOE)
Gross Beta	Alpha/Beta counting	900.0 (EPA)	7110 B (SM)	AP1 (ORISE)	FRMAC, Vol 2, pg. 33 (DOE)	FRMAC, Vol 2, pg. 33 (DOE)
Gamma	Gamma spectrometry	901.1 (EPA)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Select Mixed Fission Products[1]	Gamma spectrometry	901.1 (EPA)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)

Analyte(s)	CAS RN	Determinative Technique	Drinking Water Samples		Aqueous and Liquid Phase Samples		Soil and Sediment Samples		Surface Wipes		Air Filters	
			Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory
Americium-241[3]	14596-10-2	Alpha/Gamma spectrometry	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	Am-02-RC (HASL-300)	Am-01-RC[4] (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)
Californium-252[3]	13981-17-4	Alpha spectrometry	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-01-RC[4] (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)
Cesium-137	10045-97-3	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Cobalt-60	10198-40-0	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Curium-244[3]	13981-15-2	Alpha spectrometry	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-01-RC[4] (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)	D3084-05 (ASTM)	Am-04-RC (HASL-300)
Europium-154	15585-10-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Iodine-125	14158-31-7	Gamma spectrometry	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9 (ORISE)	Procedure #9[5] (ORISE)	Procedure #9[5] (ORISE)
Iodine-131	10043-66-0	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R[5] (HASL-300)	Ga-01-R[5] (HASL-300)
Iridium-192	14694-69-0	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)

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Analyte(s)	CAS RN	Determinative Technique	Drinking Water Samples		Aqueous and Liquid Phase Samples		Soil and Sediment Samples		Surface Wipes		Air Filters	
			Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory
Molybdenum-99	14119-15-4	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Phosphorus-32	14596-37-3	Liquid scintillation	R4-73-014 (EPA)	R4-73-014 (EPA)	R4-73-014 (EPA)	R4-73-014 (EPA)	RESL P-2 (DOE)	RESL P-2 (DOE)	RESL P-2 (DOE)	RESL P-2 (DOE)	RESL P-2 (DOE)	RESL P-2 (DOE)
Plutonium-238[3]	13981-16-3	Alpha spectrometry	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)
Plutonium-239[3]	15117-48-3	Alpha spectrometry	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)
Polonium-210	13981-52-7	Alpha spectrometry	Po-02-RC (HASL-300)	Po-02-RC (HASL-300)	Po-02-RC (HASL-300)	Po-02-RC (HASL-300)	Po-02-RC (HASL-300)	Po-02-RC (HASL-300)	Method 111 (EPA)	Method 111 (EPA)	Method 111 (EPA)	Method 111 (EPA)
Radium-226	13982-63-3	Alpha counting / spectrometry	903.0 (EPA)	903.1 (EPA)	7500-Ra B (SM)	7500-Ra C (SM)	D3084-05 (ASTM)	EMSL-19 (EPA)	D3084-05 (ASTM)	EMSL-19 (EPA)	D3084-05 (ASTM)	EMSL-19 (EPA)
Ruthenium-103	13968-53-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Ruthenium-106	13967-48-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Selenium-75	14265-71-5	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)	Ga-01-R (HASL-300)
Strontium-89	14158-27-1	Beta counting	905.0 (EPA)	905.0 (EPA)	905.0 (EPA)	905.0 (EPA)	Stronium in Food and Bioenvironmental Samples (EPA)		Stronium in Food and Bioenvironmental Samples (EPA)		Stronium in Food and Bioenvironmental Samples (EPA)	
Strontium-90	10098-97-2	Beta counting	905.0 (EPA)	905.0 (EPA)	7500-Sr B (SM)	7500-Sr B (SM)	Sr-03-RC (HASL-300)	Sr-03-RC (HASL-300)	Sr-03-RC (HASL-300)	Sr-03-RC (HASL-300)	Sr-03-RC (HASL-300)	Sr-03-RC (HASL-300)
Technetium-99	14133-76-7	Liquid scintillation	Tc-02-RC (HASL-300)	Tc-02-RC (HASL-300)	Tc-02-RC (HASL-300)	Tc-02-RC (HASL-300)	AP5 (ORISE)	AP5 (ORISE)	AP5 (ORISE)	AP5 (ORISE)	AP5 (ORISE)	AP5 (ORISE)
Tritium (Hydrogen-3)	10028-17-8	Liquid scintillation	906.0 (EPA)	906.0 (EPA)	906.0 (EPA)	906.0 (EPA)	AP2 (ORISE)	AP2 (ORISE)	AP2 (ORISE)	AP2 (ORISE)	Not applicable[6]	Not applicable[6]
Uranium-234[3]	13966-29-5	Alpha counting / spectrometry	908.0[7] (EPA)	D3972-02 (ASTM)	7500-U B[7] (SM)	7500-U C (SM)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)

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Analyte(s)	CAS RN	Determinative Technique	Drinking Water Samples		Aqueous and Liquid Phase Samples		Soil and Sediment Samples		Surface Wipes		Air Filters	
			Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory	Qualitative Determination[2]	Confirmatory
Uranium-235[3]	15117-96-1	Alpha counting / spectrometry	908.0[7] (EPA)	D3972-02 (ASTM)	7500-U B[7] (SM)	7500-U C (SM)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)
Uranium-238[3]	7440-61-1	Alpha counting / spectrometry	908.0[7] (EPA)	D3972-02 (ASTM)	7500-U B[7] (SM)	7500-U C (SM)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)	D3084-05 (ASTM)	EMSL-33 (EPA)

Footnotes

[1] Please note that this category does not cover all fission products. In addition to the specific radionuclides listed in this appendix, gamma-ray spectometry with a high resoution HP(Ge) detector will identify and quantify fission products with gamma rays in the energy range of 30 keV to 2000 keV. The sensitivity will be dependent on the detector efficiency and the gamma-ray emission probabilities (branching ratio) for the specific radionuclide.

[2] In those cases where the same method is listed for qualitative determination and confirmatory analysis, qualitative determination can be performed by application of the method over a shorter count time than that used for confirmatory analysis.

[3] If it is suspected that the sample exists in refractory form (i.e., non-digestible or dissolvable material after normal digestion methods) or if there is a matrix interference problem, use ORISE Method AP11 for qualitative determination or confirmatory analysis of alpha radioactivity.

[4] In cases where only small sample volumes (≤100 g) are available, use HASL-300 Method Pu-12-RC.

[5] This procedure should be used only for filters specifically designed for iodine.

[6] Because tritium is not sampled using traditional air filters, this matrix is not applicable.

[7] This method was developed for measurement of total uranium and does not distinguish between uranium isotopes.

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Appendix C: Selected Pathogen Methods

Per decision of the NHSRC SAM Pathogens Committee, "Section 7.0: Selected Pathogen Methods" has been temporarily withdrawn from the SAM Revision 6.0. Section 7.0 is currently undergoing a significant restructuring to better address the complexity of environmental samples in a more user-friendly format. End-users, expert scientists, and federal agencies are contributing to the new design templates.

During this transition period, the following personnel can be contacted for any emergency technical support need:

- EPA's Office of Emergency Management, Homeland Security Laboratory Research Center, manages the ERLN. The pathogens contact for ERLN is: Michele Burgess (burgess.michele@epa.gov, 202-564-8006).
- NHSRC SAM Pathogens Contact: Sanjiv Shah, Lead (shah.sanjiv@epa.gov, 202-564-9522)

Users may also refer to the SAM Version 5.0, Pathogen Methods section. SAM Revision 5.0 can be accessed at http://www.epa.gov/sam/. The SAM 5.0 Pathogens section is available in a searchable format at http://www.epa.gov/sam/searchpath.htm

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Appendix D: Selected Biotoxin Methods

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Appendix D: Selected Biotoxin Methods

Note: The presence of disinfectants (e.g., chlorine) and/or preservatives added during water sample collection to slow degradation (e.g., pH adjustors, de-chlorinating agents) could possibly affect analytical results. When present, the impact of these agents on method performance should be evaluated if not previously determined.

Analyte(s)	CAS RN / Description	Analysis Type[1]	Analytical Technique	Aerosol (filter/cassette, liquid impinger)	Solid (soil, powder)	Particulate (swabs, wipes, dust socks)	Liquid Water	Drinking Water
Protein								
Abrin	1393-62-0 (abrin) / Glycoprotein consisting of a deadenylase (25–32 kDa A chain) and lectin (35 kDa B chain); an agglutinin (A2B2) may be present in crude preparations 526-31-8 (abrine) / small molecule, abrin marker	Presumptive	Immunoassay[2]	Adapted from Journal of Food Protection 71(9): 1868-1874	Adapted from Journal of Food Protection 71(9): 1868-1874	Adapted from Journal of Food Protection 71(9): 1868-1874	Adapted from Journal of Food Protection 71(9): 1868-1874	Adapted from Journal of Food Protection 71(9): 1868-1874
		Complementary Presumptive (abrine)	LC-MS-MS	Adapted from Journal of Agricultural and Food Chemistry 56(23): 11139–11143	Adapted from Journal of Agricultural and Food Chemistry 56(23): 11139–11143	Adapted from Journal of Agricultural and Food Chemistry 56(23): 11139–11143	Adapted from Journal of Agricultural and Food Chemistry 56(23): 11139–11143	Adapted from Journal of Agricultural and Food Chemistry 56(23): 11139–11143
		Confirmatory	Ribosome inactivation assay	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260
		Biological Activity	Enzyme activity[3]	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89
Botulinum neurotoxins (Serotoypes A, B, E, F)	Protein composed of ~100 kDa heavy chain and ~50 kDa light chain; can be complexed with hemagglutinin and non-hemagglutinin components for total MW of ~900 kDa SNAP-25, VAMP 2 / botulinum neurotoxin markers	Presumptive	Immunoassay[4]	Adapted from EPA Environmental Technology Verification report	**LRN** If analysis for this agent is required in solid, particulate, or liquid samples, contact the LRN at (404) 639-2790 for information of the closest LRN laboratory capable of receiving and processing the sample. The terms presumptive and confirmatory as used for LRN methods are described in Section 8.1.4.			
		Complementary Presumptive (SNAP25, VAMP 2)	LC-MS	Adapted from Journal of Chemical Health and Safety 15(6): 14–19				
		Confirmatory	Immunoassay[4] (ELISA)	Adapted from FDA Bacteriological Analytical Manual, Chapter 17				
		Biological Activity	Mouse Bioassay	Adapted from FDA Bacteriological Analytical Manual, Chapter 17				

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Analyte(s)	CAS RN / Description	Analysis Type[1]	Analytical Technique	Aerosol (filter/cassette, liquid impinger)	Solid (soil, powder)	Particulate (swabs, wipes, dust socks)	Liquid Water	Drinking Water
Ricin	9009-86-3 (ricin) / 60 kDa glycoprotein composed of two subunits (~32 kDa A chain and ~34 kDa B chain); an agglutinin of MW 120 kDa may be present in crude preparations 5254-40-3 (ricinine) / small molecule, ricin marker	Presumptive	Immunoassay[2]	Adapted from EPA Environmental Technology Verification report	**LRN** If analysis for this agent is required in solid, particulate, or liquid samples, contact the LRN at (404) 639-2790 for information of the closest LRN laboratory capable of receiving and processing the sample. The terms presumptive and confirmatory as used for LRN methods are described in Section 8.1.4.			
		Complementary Presumptive (ricinine)	LC-MS	Adapted from Journal of Analytical Toxicology 29(3): 149-155	Adapted from Journal of Analytical Toxicology 29(3): 149-155	Adapted from Journal of Analytical Toxicology 29(3): 149-155	Adapted from Journal of Analytical Toxicology 29(3): 149-155	Adapted from Journal of Analytical Toxicology 29(3): 149-155
		Confirmatory	Immunoassay	Adapted from Journal of AOAC International 91(2): 376-382	Adapted from Journal of AOAC International 91(2): 376-382	Adapted from Journal of AOAC International 91(2): 376-382	Adapted from Journal of AOAC International 91(2): 376-382	Adapted from Journal of AOAC International 91(2): 376-382
		Biological Activity	Enzyme activity[3]	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89	Adapted from Analytical Biochemistry 378(1): 87-89
Shiga and Shiga-like Toxins (Stx, Stx-1, Stx-2)	75757-64-1 (Stx) / Protein composed of one ~32 kDa A chain and five 7.7 kDa B chains	Presumptive	Optical immunoassay	Adapted from Journal of Clinical Microbiology 45(10): 3377–3380	Adapted from Journal of Clinical Microbiology 45(10): 3377–3380	Adapted from Journal of Clinical Microbiology 45(10): 3377–3380	Adapted from Journal of Clinical Microbiology 45(10): 3377–3380	Adapted from Journal of Clinical Microbiology 45(10): 3377–3380
		Confirmatory	Immunoassay (ELISA)	Adapted from FDA Bacteriological Analytical Manual, Appendix 1	Adapted from FDA Bacteriological Analytical Manual, Appendix 1	Adapted from FDA Bacteriological Analytical Manual, Appendix 1	Adapted from FDA Bacteriological Analytical Manual, Appendix 1	Adapted from FDA Bacteriological Analytical Manual, Appendix 1
		Biological Activity	Ribosome inactivation assay[3]	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260	Adapted from Pharmacology & Toxicology 88(5): 255-260
Staphylococcal enterotoxins (SEB)	39424-53-8 (SEB) / Monomeric protein of ~ 28 kDa	Presumptive	Immunoassay	Adapted from 993.06 (AOAC)	**LRN** If analysis for this agent is required in solid, particulate, or liquid samples, contact the LRN at (404) 639-2790 for information of the closest LRN laboratory capable of receiving and processing the sample. The terms presumptive and confirmatory as used for LRN methods are described in Section 8.1.4.			
		Confirmatory	TBD	TBD	TBD	TBD	TBD	TBD
		Biological Activity	T-cell proliferation assay	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365

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Analyte(s)	CAS RN / Description	Analysis Type[1]	Analytical Technique	Aerosol (filter/cassette, liquid impinger)	Solid (soil, powder)	Particulate (swabs, wipes, dust socks)	Liquid Water	Drinking Water
Staphylococcal enterotoxins (SEA, SEC)	37337-57-8 (SEA) 39424-54-9 (SEC) / Monomeric proteins of ~ 27–27.5 kDa	Presumptive	Immunoassay	Adapted from 993.06 (AOAC)	Adapted from 993.06 (AOAC)	Adapted from 993.06 (AOAC)	Adapted from 993.06 (AOAC)	Adapted from 993.06 (AOAC)
		Confirmatory	TBD	TBD	TBD	TBD	TBD	TBD
		Biological Activity	T-cell proliferation assay	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365	Adapted from Applied and Environmental Microbiology 63(6): 2361–2365
Small Molecule								
Aflatoxin (Type B1)	27261-02-5	Presumptive	Immunoassay	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)
		Confirmatory	HPLC-FL	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)	Adapted from 991.31 (AOAC)
α-Amanitin	23109-05-9	Presumptive	Immunoassay	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301
		Confirmatory	HPLC amperometric detection	Adapted from Journal of Chromatography 563(2): 299-311	Adapted from Journal of Chromatography 563(2): 299-311	Adapted from Journal of Chromatography 563(2): 299-311	Adapted from Journal of Chromatography 563(2): 299-311	Adapted from Journal of Chromatography 563(2): 299-311
Anatoxin-a	64285-06-9	Presumptive	TBD	TBD	TBD	TBD	TBD	TBD
		Confirmatory	HPLC-FL (precolumn derivatization)	Adapted from Biomedical Chromatography B 10(1): 46-47	Adapted from Biomedical Chromatography B 10(1): 46-47	Adapted from Biomedical Chromatography B 10(1): 46-47	Adapted from Biomedical Chromatography B 10(1): 46-47	Adapted from Biomedical Chromatography B 10(1): 46-47
Brevetoxins (B form)	79580-28-2	Presumptive	Immunoassay	Adapted from Environmental Health Perspectives 110(2): 179-185	Adapted from Environmental Health Perspectives 110(2): 179-185	Adapted from Environmental Health Perspectives 110(2): 179-185	Adapted from Environmental Health Perspectives 110(2): 179-185	Adapted from Environmental Health Perspectives 110(2): 179-185
		Confirmatory	HPLC-MS-MS	Adapted from Toxicon 43(4): 455-465	Adapted from Toxicon 43(4): 455-465	Adapted from Toxicon 43(4): 455-465	Adapted from Toxicon 43(4): 455-465	Adapted from Toxicon 43(4): 455-465
α-Conotoxin	156467-85-5	Presumptive	Immunoassay	Adapted from Biochemical Journal 328(1): 245-250	Adapted from Biochemical Journal 328(1): 245-250	Adapted from Biochemical Journal 328(1): 245-250	Adapted from Biochemical Journal 328(1): 245-250	Adapted from Biochemical Journal 328(1): 245-250
		Confirmatory	HPLC-MS	Adapted from Journal of Medicinal Chemistry 47(5): 1234-1241	Adapted from Journal of Medicinal Chemistry 47(5): 1234-1241	Adapted from Journal of Medicinal Chemistry 47(5): 1234-1241	Adapted from Journal of Medicinal Chemistry 47(5): 1234-1241	Adapted from Journal of Medicinal Chemistry 47(5): 1234-1241
Cylindrospermopsin	143545-90-8	Presumptive	Immunoassay	Adapted from ELISA kits for Cylindrospermopsin	Adapted from ELISA kits for Cylindrospermopsin	Adapted from ELISA kits for Cylindrospermopsin	Adapted from ELISA kits for Cylindrospermopsin	Adapted from ELISA kits for Cylindrospermopsin
		Confirmatory	HPLC-PDA	Adapted from FEMS Microbiology Letters 216(2): 159-164	Adapted from FEMS Microbiology Letters 216(2): 159-164	Adapted from FEMS Microbiology Letters 216(2): 159-164	Adapted from FEMS Microbiology Letters 216(2): 159-164	Adapted from FEMS Microbiology Letters 216(2): 159-164

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Analyte(s)	CAS RN / Description	Analysis Type[1]	Analytical Technique	Aerosol (filter/cassette, liquid impinger)	Solid (soil, powder)	Particulate (swabs, wipes, dust socks)	Liquid Water	Drinking Water
Diacetoxyscirpenol (DAS)	2270-40-8	Presumptive	Immunoassay	Adapted from International Journal of Food Microbiology 6(1): 9-17	Adapted from International Journal of Food Microbiology 6(1): 9-17	Adapted from International Journal of Food Microbiology 6(1): 9-17	Adapted from International Journal of Food Microbiology 6(1): 9-17	Adapted from International Journal of Food Microbiology 6(1): 9-17
		Confirmatory	LC/APCI-MS	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428
Microcystins Principal isoforms: LA, LR, LW, RR, YR	96180-79-9 (LA) 101043-37-2 (LR) 157622-02-1 (LW) 111755-37-4 (RR) 101064-48-6 (YR)	Presumptive	Immunoassay/ Phosphatase assay	Adapted from Journal of AOAC International 84(4): 1035-1044	Adapted from Journal of AOAC International 84(4): 1035-1044	Adapted from Journal of AOAC International 84(4): 1035-1044	Adapted from Journal of AOAC International 84(4): 1035-1044	Adapted from Journal of AOAC International 84(4): 1035-1044
		Confirmatory	HPLC-PDA	Adapted from Analyst 119(7): 1525-1530	Adapted from Analyst 119(7): 1525-1530	Adapted from Analyst 119(7): 1525-1530	Adapted from Analyst 119(7): 1525-1530	Adapted from Analyst 119(7): 1525-1530
Picrotoxin	124-87-8	Presumptive	Immunoassay	TBD	TBD	TBD	TBD	TBD
		Confirmatory	HPLC	Adapted from Journal of Pharmaceutical and Biomedical Analysis 7(3): 369-375	Adapted from Journal of Pharmaceutical and Biomedical Analysis 7(3): 369-375	Adapted from Journal of Pharmaceutical and Biomedical Analysis 7(3): 369-375	Adapted from Journal of Pharmaceutical and Biomedical Analysis 7(3): 369-375	Adapted from Journal of Pharmaceutical and Biomedical Analysis 7(3): 369-375
Saxitoxins Principal isoforms: Saxitoxin (STX) Neosaxitoxin (NEOSTX) Gonyautoxin (GTX) Decarbamoylgonyautoxin (dcGTX) Decarbamoylsaxitoxin (dcSTX)	35523-89-8 (STX) 64296-20-4 (NEOSTX) 77462-64-7 (GTX) None given (dcGTX) 58911-04-9 (dcSTX)	Presumptive	Immunoassay	Adapted from ELISA kits for Saxitoxins	Adapted from ELISA kits for Saxitoxins	Adapted from ELISA kits for Saxitoxins	Adapted from ELISA kits for Saxitoxins	Adapted from ELISA kits for Saxitoxins
		Confirmatory	HPLC-FL (post column derivatization)	Adapted from Journal of AOAC International 78(2): 528-532	Adapted from Journal of AOAC International 78(2): 528-532	Adapted from Journal of AOAC International 78(2): 528-532	Adapted from Journal of AOAC International 78(2): 528-532	Adapted from Journal of AOAC International 78(2): 528-532
T-2 Mycotoxin	21259-20-1	Presumptive	Immunoassay	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301	Adapted from Journal of Food Protection 68(6): 1294-1301
		Confirmatory	LC/APCI-MS	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428	Adapted from Rapid Communications in Mass Spectrometry 20(9): 1422-1428
Tetrodotoxin	9014-39-5	Presumptive	Immunoassay	Adapted from Journal of Clinical Laboratory Analysis 6(2): 65-72	Adapted from Journal of Clinical Laboratory Analysis 6(2): 65-72	Adapted from Journal of Clinical Laboratory Analysis 6(2): 65-72	Adapted from Journal of Clinical Laboratory Analysis 6(2): 65-72	Adapted from Journal of Clinical Laboratory Analysis 6(2): 65-72
		Confirmatory	LC/ESI-MS	Adapted from Analytical Biochemistry 290(1): 10-17	Adapted from Analytical Biochemistry 290(1): 10-17	Adapted from Analytical Biochemistry 290(1): 10-17	Adapted from Analytical Biochemistry 290(1): 10-17	Adapted from Analytical Biochemistry 290(1): 10-17

[1] Descriptions for presumptive, confirmatory, and biological activity assays are provided in Section 8.0.

[2] Crude preparations of ricin and abrin may also contain agglutinins that are unique to castor beans and rosary peas, respectively, and that can cross-react in the immunoassays.

[3] This assay does not test for cell binding; cell culture assays are being developed to test for cell binding but are not currently available. The only readily available assay to test for both the cell binding and enzymatic activity of the intact (whole) toxin is a mouse bioassay.

[4] Immunoassays may produce variable results with uncomplexed form of toxin.

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Attachment 1:
SAM Supporting Documents

The documents and tools listed in this attachment have been developed by EPA to provide information regarding collection, screening, rapid analysis, and disposal of samples that may be needed during environmental restoration following a homeland security event. The information included in the documents is intended to be complementary to information provided in the analytical methods listed in SAM. As additional documents containing similar complementary information become available, they will be added to the list contained in this Attachment.

- Searchable SAM Web site at: www.epa.gov/sam/
- "Guidelines for Development of Sample Collection Plans for Radiochemical Analytes in Environmental Matrices Following Homeland Security Events," EPA/600/R-08/128, February 2009. http://www.epa.gov/nhsrc/pubs/600r08128.pdf
- "Sample Collection Procedures for Radiochemistry Analytes in Environmental Matrices," EPA/600/S-07/001, December 2006. http://www.epa.gov/nhsrc/pubs/600s07001.pdf
- "Sample Collection Information Document – Companion to SAM Revision 5.0," EPA/600/R-09/074, June 2010. http://www.epa.gov/nhsrc/pubs/600r09074.pdf
- "Field Screening Equipment Information Document – Companion to SAM Revision 5.0," EPA/600/R-10/091, September 2010
- "Rapid Screening and Preliminary Identification Techniques and Methods – Companion to SAM Revision 5.0," EPA/600/R-10/090, September 2010
- "Laboratory Environmental Sample Disposal Information Document – Companion to SAM Revision 5.0," EPA/600/R-10/092, September 2010

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