

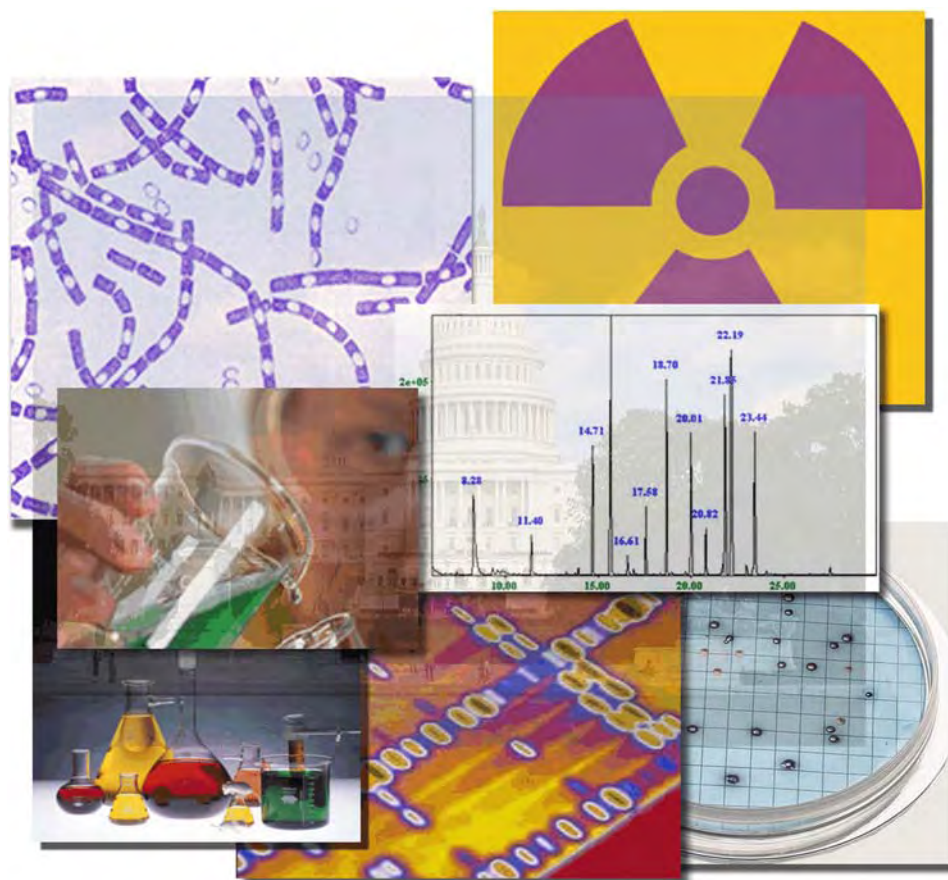


**United States  
Environmental Protection  
Agency**

# Standardized Analytical Methods for Use During Homeland Security Events

# Revision 2.0

*September 29, 2005*





# **Standardized Analytical Methods for Use During Homeland Security Events**

## **Revision 2.0**

September 29, 2005

Prepared by

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## Disclaimer

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## Use of This Document

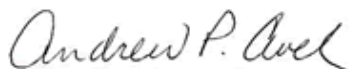
The information contained in this document represents the latest step in an ongoing National Homeland Security Research Center effort to provide standardized analytical methods for use by laboratories tasked with performing analyses in response to a homeland security event. Although at this time, many of the methods listed have not been tested for a particular analyte (e.g., analytes not explicitly identified in the method) or matrix, the methods are considered to contain the most appropriate currently available techniques. Unless a published method that is listed in this document states specific applicability to the analyte/matrix pair for which it has been selected, it should be assumed that method testing is needed. In these cases, adjustment may be required to accurately account for variations in environmental matrices, analyte characteristics, and target risk levels. Many of the target analytes listed in this document have only recently become an environmental concern, and EPA is actively pursuing development and validation of Standard Analytical Protocols (SAPs) based on the methods listed, including optimization of procedures for measuring target compounds. In those cases where method procedures are determined to be insufficient for a particular situation, EPA will provide guidance regarding appropriate actions. This will be an ongoing process as EPA will strive to establish a consistent level of validation for all listed analytes.

## Foreword

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a scientific base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Homeland Security Research Center (NHSRC) is the Agency's center for conducting research to facilitate protection and decontamination of structures and water infrastructure subject to chemical, biological, or radiological (CBR) terror attacks. NHSRC's research is designed to provide appropriate, effective, and validated technologies, methods, and guidance to understand the risk posed by CBR agents to enhance our ability to detect, contain, and clean up in the event of such attacks. NHSRC will also provide direct technical assistance to response personnel in the event of a CBR attack, as well as provide related interagency liaisons.

This publication has been produced as part of the Center's long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.



Andrew P. Avel, Acting Director  
National Homeland Security Research Center

## Abbreviations and Acronyms

AEM	<i>Applied and Environmental Microbiology</i>
AGI sampler	All Glass Impinger Sampler
AOAC	AOAC International (formerly the Association of Official Analytical Chemists)
ASTM	ASTM International (formerly the American Society for Testing and Materials)
BSL	Biosafety Level
°C	Degrees Celsius
Campy-BAC	<i>Campylobacter-Brucella</i> agar base with sheep blood and antibiotics
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CVAA	Cold Vapor Atomic Absorption
CVAFS	Cold Vapor Atomic Fluorescence Spectrometry
DAPI	4',6-diamidino-2-phenylindole
DHS	Department of Homeland Security
DIC	Differential Interference Contrast
DNA	Deoxyribonucleic Acid
DNPH	2,4-dinitrophenylhydrazine
DoD	Department of Defense
ED	Electron Diffraction
EDTA	Ethylenediaminetetraacetic acid
EDXA	Energy Dispersive X-ray Analysis
EEB	EHEC Enrichment Broth
EHEC	Enterohemorrhagic <i>Escherichia coli</i>
EIA	Enzyme Immunoassay
ELISA	Enzyme-Linked Immunosorbent Assay
EMMI	Environmental Monitoring Methods Index
EPA	U.S. Environmental Protection Agency
EQL	Estimated Quantitation Limit
FA	Fluorescence Assay
FBI	Federal Bureau of Investigation
FDA	Food and Drug Administration
FID	Flame Ionization Detector
FITC	Fluorescein isothiocyanate
FSIS	Food Safety and Inspection Service
GC	Gas Chromatograph or Gas Chromatography
GC/MS	Gas Chromatograph/Mass Spectrometer or Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption Spectrophotometer or Graphite Furnace Atomic Absorption Spectrophotometry
GITC	Guanidinium isothiocyanate
HAV	Hepatitis A Virus
HPLC	High Performance Liquid Chromatograph or High Performance Liquid Chromatography
HPLC-FL	High Performance Liquid Chromatograph - Fluorescence
HPLC-MS	High Performance Liquid Chromatograph - Mass Spectrometer
IC	Ion Chromatograph or Ion Chromatography
ICC	Integrated cell culture
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectrometry

ICR	Information Collection Rule
IMS	Immunomagnetic Separation
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. <a href="http://www.inchem.org/">http://www.inchem.org/</a>
IO	Inorganic
ISO	International Organization for Standardization
ISE	Ion Specific Electrode
K-D	Kuderna-Danish
LIA	Lysine Iron Agar
LRN	Laboratory Response Network
LSE	Liquid/Solid Extraction
MS	Mass Spectrometer or Mass Spectrometry or Matrix Spike
MSD	Matrix Spike Duplicate
MW	Molecular Weight
NA	Not Applicable
NEMI	National Environmental Methods Index
NERL-CI	National Exposure Risk Laboratory-Cincinnati
NHSRC	National Homeland Security Research Center
NIOSH	National Institute for Occupational Safety and Health
NOS	Not Otherwise Specified
NTIS	National Technical Information Service
ONPG	Ortho-nitrophenyl- $\beta$ -D-galactopyranoside
OSHA	Occupational Safety and Health Administration
OW	Office of Water
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated biphenyls
PCDDs	Polychlorinated dibenzo-p-dioxins
PCDFs	Polychlorinated dibenzofurans
PCR	Polymerase Chain Reaction
PFE	Pressurized Fluid Extraction
QC	Quality Control
RNA	Ribonucleic Acid
RP/HPLC	Reversed-Phase High Performance Liquid Chromatography
RT-PCR	Reverse Transcription-Polymerase Chain Reaction
SAED	Selected Area Electron Diffraction
SM	<i>Standard Methods for the Examination of Water and Wastewater</i>
SPE	Solid-Phase Extraction
SW	Solid Waste
TBD	To Be Determined
TCBS	Thiosulfate Citrate Bile Salts Sucrose
TC SMAC	Tellurite Cefixime Sorbitol MaConkey agar
TCLP	Toxicity Characteristic Leaching Procedure
TEM	Transmission Electron Microscope or Microscopy
TOXNET	National Library of Medicine, Toxicological Database
TRF	Time resolved fluorescence
TRU	Trans Uranic
TS	Thermospray
TSI	Triple Sugar Iron



USDA	U.S. Department of Agriculture
USGS	U.S. Geological Survey
UV	Ultraviolet
VEE	Venezuelan Equine Encephalitis
VOCs	Volatile Organic Compounds
VOA	Volatile Organic Analysis
XLD	Xylose lysine desoxycholate

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## Section 1.0: Introduction

In the aftermath of the terrorist attacks of September 11, 2001, and the anthrax attacks in the Fall of 2001, Federal and State personnel successfully carried out their mission to provide response, recovery, and remediation under trying circumstances, including an unprecedented demand on their capabilities to analyze environmental samples. In reviewing these incidents, the Environmental Protection Agency's (EPA) *9/11 Lessons Learned*<sup>1</sup> and its *Anthrax Lessons Learned*<sup>2</sup> reports identified several areas where the country could better prepare itself in the event of future terrorist incidents. One of the most important areas identified was the need to improve the nation's laboratory capacity and capability to respond to incidents requiring the analysis of large numbers of environmental samples in a short time.

In response, EPA formed the Homeland Security Laboratory Capacity Workgroup to identify and implement opportunities for near-term improvements and to develop recommendations for addressing longer-term, cross-cutting laboratory issues. The EPA Homeland Security Laboratory Capacity Workgroup consists of representatives from the Office of Research and Development, Office of Radiation and Indoor Air, Office of Water, Office of Solid Waste and Emergency Response, Office of Environmental Information, Office of Pesticide Programs, and several EPA Regional Offices.

A critical area identified by the workgroup was the need for a list of standardized analytical methods to be used by all laboratories when analyzing homeland security incident samples. Having standardized methods would reduce confusion, permit sharing of sample load between laboratories, improve data comparability, simplify the task of outsourcing analytical support to the commercial laboratory sector, and improve the follow-up activities of validating results, evaluating data and making decisions. To this end, workgroup members formed an Analytical Methods Subteam to address homeland security methods issues.

The Analytical Methods Subteam recognized that widely different needs for analytical methods could be required for various analytical activities that will be performed, including (1) constant monitoring and surveillance to determine if a terrorist event has occurred, (2) rapid screening for determining the presence of agents or contaminants of concern, (3) screening for identification of agents or contaminants used in an event, and (4) quantitation of the amount or levels of agents or contaminants identified for extent of contamination and the efficacy of decontamination determinations.

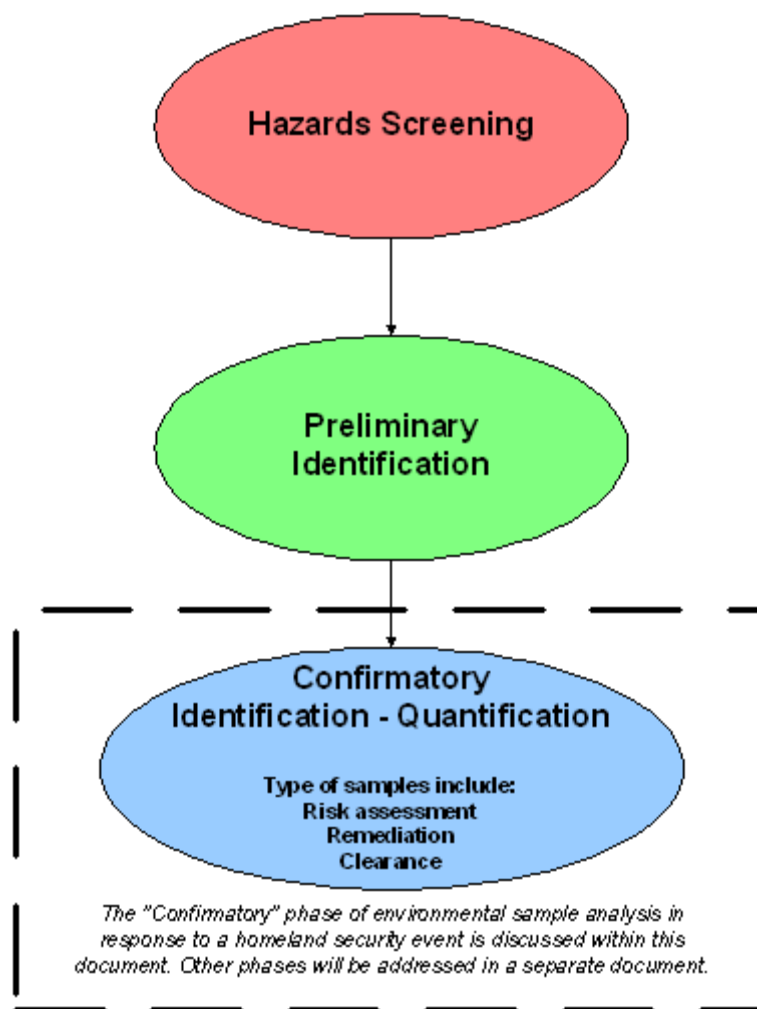
**Figure 1-1** represents the analytical decision tree for responding to homeland security incidents.

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<sup>1</sup>U.S. EPA internal report: Lessons Learned in the Aftermath of September 11, 2001 (February 2002).

<sup>2</sup>U.S. EPA internal report: Challenges Faced During the Environmental Protection Agency's Response to Anthrax and Recommendations for Enhancing Response Capabilities: A Lessons Learned Report (September 2002).

**Figure 1-1. Analytical Response Roadmap for Homeland Security Events**



During 2004, the Analytical Methods Subteam recognized that selection of standardized analytical methods is dependent on the intended application, and focused on selection of a single preferred confirmatory method per each individual analyte/matrix for use in assessing the extent of contamination and the efficacy of decontamination (e.g., identification and quantification of contaminants). 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-Chemical, Biological, and Radiological (NEMI-CBR)<sup>3</sup>
- Environmental Monitoring Method Index (EMMI)
- EPA Test Methods Index
- EPA Office of Solid Waste SW-846 Methods On-line
- EPA Microbiology Methods
- National Institute for Occupational Safety and Health (NIOSH) method index
- Occupational Safety and Health Administration (OSHA) method index
- AOAC International
- ASTM International
- International Organization for Standardization (ISO) methods
- Standard Methods for the Examination of Water and Wastewater
- PubMed Literature Database

In 2004, EPA's National Homeland Security Research Center brought together experts from across EPA and its sister agencies to develop a compendium of analytical methods to be used when responding to future incidents. Participants included representatives from U.S. EPA program offices, EPA regions, EPA national 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 both chemical and biological agents of concern in the types of environmental sample matrices that were anticipated for analysis in homeland security incidents. The primary objective of this effort was to identify appropriate SAM Analytical Methods Subteam consensus methods that represent a balance between existing determinative techniques and methodologies and providing consistent analytical results.

In September 2004, EPA published *Standardized Analytical Methods for Use During Homeland Security Events, Revision 1.0* (EPA/600/R-04/126), SAM. This document provided a list of analytical and sample preparation methods that were selected for measurement of 82 chemical analytes in aqueous/liquid, solid, oily solid, and air matrices, and 27 biological analytes in water, dust, and aerosol matrices.

During 2005, SAM was expanded to include several persistent chemical warfare agent degradation products and radioisotopes, a drinking water matrix, 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 radioisotopes and chemical warfare agent degradation products in all matrices, for measurement of chemical, biological, and radiochemical analytes in drinking water, and to determine the viability of biological organisms. These additional analytes and the corresponding methods selected are included in SAM Revision 2.0.

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<sup>3</sup>NEMI-CBR is being developed by the U.S. EPA Office of Water's Water Security Division to provide a central system for locating, evaluating, comparing, and retrieving analytical methods for rapid and effective analysis of environmental samples. NEMI-CBR displays and summarizes multiple screening and confirmatory methods for contaminants that may be associated with terrorist attacks, and is scheduled for release in December 2005.





## Section 2.0: Scope and Application

The purpose of this document is to identify and briefly describe the specific methodologies that EPA and its contracted laboratories will employ when called upon to analyze environmental samples in times of national emergency. The document also is intended as a tool that will be available to assist State and local laboratories in responding to homeland security events. The methodologies presented in this document should be used to:

- Evaluate the nature and extent of contamination
- Evaluate the effectiveness of decontamination

Methods are provided as corresponding to specific analyte/matrix pairs that are listed in Appendices A (chemical), B (biological), C (radiochemical), and D (biotoxin). Summaries of each method are provided in numerical order by developing agency, throughout Sections 4.2 (chemical), 5.2 (biological), 6.2 (radiochemical), and 7.2 (biotoxin).

The list of methods provided is limited to those methods that would be used to determine, to the extent possible within analytical limitations, the presence of chemical, biological, radiochemical, and biotoxin analytes of concern and to determine their concentrations in environmental media. The methods are not designed to be used for conducting an initial evaluation (triage or screening) of suspected material to determine if it poses an immediate danger or if it needs to be analyzed in specially designed, highly secure facilities. Methods for addressing these needs are and will be the subject of other efforts. It is hoped that this document will assist Federal, State and local governments in preparing for future emergencies.

It is important to note that, in some cases, the methods included in this document have not been verified for the analyte/matrix combination(s) for which they have been selected. The information contained in this document represents the latest step in an ongoing National Homeland Security Research Center effort to provide standardized analytical methods for use by laboratories tasked with performing analyses in response to a homeland security event. Although at this time, many of the methods listed have not been tested for a particular analyte (e.g., analytes not explicitly identified in the method) or matrix, the methods are considered to contain the most appropriate currently available techniques. Unless a published method that is listed in this document states specific applicability to the analyte/matrix pair for which it has been selected, it should be assumed that method testing is needed. In these cases, adjustment may be required to accurately account for variations in environmental matrices, analyte characteristics, and target risk levels. Many of the target analytes listed in this document have only recently become an environmental concern, and EPA is actively pursuing development and validation of Standard Analytical Protocols (SAPs) based on the methods listed, including optimization of procedures for measuring target compounds. In those cases where method procedures are determined to be insufficient for a particular situation, EPA will provide guidance 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 is developing and validating Standardized Analytical Protocols (SAPs) based on the methods that are listed in this document, where further development and verification are necessary. Once validation is complete, data regarding specific 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 periodically to address the needs of homeland security, reflect improvements in analytical methodology and new technologies, and incorporate changes in analytes based on needs. The Agency also anticipates that addendums may be generated to provide guidance 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 3.

Having data of known and documented quality is critical for public officials to accurately assess the activities that may be needed in responding to emergency situations. Quality control (QC) pertains to both sample collection and analysis. Data must be of sufficient quality to support decision making. Quality control, however, takes time and time is often critical in emergency response activities, where there will be tremendous pressure to conduct sampling and analytical operations quickly and efficiently. While reduced levels of QC might be tolerated during the rapid screening stage of emergency response, implementation of analytical methods for risk assessment and site release will require a higher and more appropriate level of QC. Many of the methods listed in this document include QC requirements for collecting and analyzing samples. These QC requirements may or may not be appropriate for addressing emergency response situations, and may be adjusted as necessary to maximize data and decision quality. Specific QC recommendations for analysis of samples for chemical, biological, radiochemical, and biotoxin analytes are provided in each corresponding section of this document (i.e., Sections 4.1.2, 5.1.2, 6.1.2, and 7.1.2, respectively).

Participants in the biological, chemical, and radiochemistry work groups, including representatives from the U.S. EPA, CDC, FDA, DHS, FBI, DoD, USDA, and USGS evaluated the suitability of existing methodologies and selected this set of methods for use by EPA laboratories and contract laboratories if called upon to respond to an emergency. The Agency 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 the Agency did not anticipate having to analyze for a particular analyte or analyte/matrix combination
- Preventing development and adoption of new and better measurement technologies

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

- Developing and specifying measurement quality objectives (i.e., 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) for all analyte/matrix combinations listed in this document
- Specifying minimum measurement system verification (e.g., ASTM Standard D6956-03) and documentation standards for homeland security analyses
- Working with other government agencies and the private sector to establish a laboratory accreditation system to ensure that laboratories selected to assist the Agency and its Federal, State, and local partners in responding to homeland security incidents have the requisite expertise and systems to perform this type of testing
- Continue working with multiple agencies and stakeholders to periodically update SAM and supporting documents

## Section 3.0: 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. 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. The appropriate point(s) of contact identified below should be consulted regarding any deviations.

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### Radiochemical Methods

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## Section 4.0: Chemical Methods

A list of analytical methods to be used in analyzing environmental samples for chemical contaminants during homeland security events is provided in Appendix A. Methods are listed for each analyte and for each sample matrix that potentially may need to be measured and analyzed when responding to an environmental emergency. Protocols from peer reviewed journal articles have been identified for analyte-matrix pairs where standardized methods are not available, and it should be noted that the limitations of these protocols differ from the limitations of the standardized methods identified. Once standard procedures are available, the literature references will be replaced.

The methods table in Appendix A is sorted alphabetically by analyte and includes the following information:

- **Analyte(s).** The compound or compound(s) of interest.
- **Chemical Abstract Survey 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.
- **Determinative method.** An analytical method or procedure used to determine the quantity and identification of compounds or components in a sample.
- **Determinative method identifier.** The unique identifier or number assigned to an analytical method by the method publisher.
- **Solid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in solid phase samples.
- **Oily solid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in oily phase samples.
- **Aqueous/Liquid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in aqueous and/or liquid phase samples.
- **Drinking water sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in drinking water samples.
- **Air sample preparation procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in air samples.

### 4.1 General Guidance

The guidance summarized in this section provides a general overview of how to identify the appropriate chemical method(s) for a given analyte-matrix combination as well as recommendations for quality control 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:

- Syracuse Research Corporation's Physprop and Chemfate, part of the Environmental Fate Database supported by EPA. <http://www.syrres.com/esc/databases.htm>
- 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/vz72d288.html#JWIDAW> for toxicity information.
- EPA's Integrated Risk Information System (IRIS): <http://www.epa.gov/iris/> contains toxicity information.
- The Forensic Science and Communications Journal published by the Laboratory Division of the Federal Bureau of Investigation. <http://www.fbi.gov/hq/lab/fsc/current/backissu.htm>.

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

#### 4.1.1 Standard Operating Procedures for Identifying Chemical Methods

Determine the appropriate method and sample preparation technique that is to be used on the environmental samples by locating the analyte of concern in Appendix A: Chemical Methods under the "Analyte" column. After locating the analyte of concern, continue across the table to identify the determinative technique and determinative method identifier for that particular compound. Determine the sample preparation technique by selecting the appropriate matrix column (Solid, Oily Solid, Aqueous/Liquid, Drinking Water, or Air) for that particular analyte.

Sections 4.2.1 through 4.2.73 below provide summaries of the determinative and sample preparation methods listed in Appendix A. Where available, a direct link to the full text of the selected analytical 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 1.

**Table 1. Sources of Chemical Methods**

Name	Publisher	Reference
National Environmental Methods Index (NEMI)	EPA, USGS	<a href="http://www.nemi.gov">http://www.nemi.gov</a>
U.S. EPA Office of Water (OW) Methods	EPA Office of Water	<a href="http://www.epa.gov/safewater/methods/sourcalt.html">http://www.epa.gov/safewater/methods/sourcalt.html</a>
U.S. EPA SW-846 Methods	EPA Office of Solid Waste	<a href="http://www.epa.gov/epaoswer/hazwaste/test/main.htm">http://www.epa.gov/epaoswer/hazwaste/test/main.htm</a>
U.S. EPA Office of Research and Development Methods	EPA Office of Research and Development	<a href="http://www.epa.gov/nerlcwww/ordmeth.htm">http://www.epa.gov/nerlcwww/ordmeth.htm</a>
U.S. EPA Air Toxics Methods	EPA Office of Air and Radiation	<a href="http://www.epa.gov/ttn/amtic/airtox.html">http://www.epa.gov/ttn/amtic/airtox.html</a>
Occupational Safety and Health Administration Methods	OSHA	<a href="http://www.osha-slc.gov/dts/sltc/methods/toc.html">http://www.osha-slc.gov/dts/sltc/methods/toc.html</a>
National Institutes for Occupational Safety and Health Methods	NIOSH	<a href="http://www.cdc.gov/niosh/nmam/">http://www.cdc.gov/niosh/nmam/</a>

Name	Publisher	Reference
Standard Methods for the Examination of Water and Wastewater, 20 <sup>th</sup> Edition, 1998*	American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF)	<a href="http://www.standardmethods.org">http://www.standardmethods.org</a>
Annual Book of ASTM Standards*	ASTM International	<a href="http://www.astm.org">http://www.astm.org</a>
International Organization for Standardization Methods*	ISO	<a href="http://www.iso.org">http://www.iso.org</a>
Official Methods of Analysis of AOAC International*	AOAC International	<a href="http://www.aoac.org">http://www.aoac.org</a>

\* Subscription and/or purchase required.

#### 4.1.2 General Quality Control (QC) Guidance for Chemical Methods

The level or amount of quality control (QC) needed during sample analysis and reporting depends on the intended purpose of the data that are generated (i.e., the decision(s) to be made). The specific decisions that will be made should be identified, and quality objectives (including QC requirements) should be derived based on those decisions. In establishing the appropriate level of QC, activities should be focused on specific elements needed to support decision making.

Having analytical data of appropriate quality 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, including corrective actions. In emergency response situations, however, speed and efficiency are also important. Laboratories must be prepared with calibrated instruments, the proper standards, method-specific standard analytical procedures, and qualified and trained technicians. Laboratories also must be capable of providing rapid turnaround of sample analyses and data reporting.

A minimum set of analytical QC procedures should be planned and conducted for all chemical testing. Method-specific QC requirements are described in many of the individual methods that are cited in this manual and will be referenced in any standardized analytical protocols developed to address specific analytes and matrices of concern. Individual methods, analysis protocols, or contractual statements of work also should be consulted to determine any additional QC that may 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, and matrix spike duplicates (MSD) 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 quality control includes:

- Demonstration that measurement system is operating properly
  - Initial calibration
  - Method blanks

- Demonstration of analytical method suitability for intended use
  - Detection and quantitation limits
  - Precision and recovery (verify measurement system has adequate accuracy)
  - 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 (recovery and precision)
  - QC samples (system accuracy and sensitivity at levels of concern)
  - Surrogate spikes (where appropriate)
  - Continuing calibration verification
  - Method blanks

QC tests should be run as frequently as necessary to ensure the reliability of analytical results. As with the identification of needed QC samples, frequency should be established based on an evaluation of data quality objectives. The type and frequency of QC can be focused over time. For example, as measurements become routine and the sources of analytical variability understood, the frequency of some types of QC samples (matrix spikes and matrix spike duplicates) or protocols (calibration checks, etc.) may be reduced without affecting analytical data quality.

Ensuring data quality also requires that laboratory results are properly evaluated and the results of the data quality evaluation are transmitted to decision makers. This evaluation is as important as the data in 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 response situations. The level of such reviews should be determined based on the specific situation being assessed and on the corresponding data quality 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 3 should be consulted regarding appropriate quality assurance and quality control (QA/QC) procedures prior to sample analysis. These contacts will consult with their respective QA/QC managers regarding QA/QC issues.

### 4.1.3 Safety and Waste Management

It is imperative that safety precautions are used during collection, processing, and analysis of environmental samples, particularly in emergency response situations that may include unknown hazards. Laboratories should have a documented health and safety plan for handling samples that may contain the target chemical, biological, or radiological 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 4.2 contain some specific requirements, guidance, 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:

- Occupational Health and Safety Administration's (OSHA) standard for Occupational Exposure to Hazardous Chemicals in Laboratories (29 CFR 1910.1450)
- OSHA regulations for hazardous waste operations and emergency response (29 CFR 1910)
- Environmental Protection Agency's standards regulating hazardous waste (40 CFR parts 260 - 270)



- U.S. Department of Transportation (DOT) regulations for transporting hazardous materials (49 CFR Part 172)
- U.S. Department of Health and Human Services, Centers for Disease Control and Prevention's requirements for possession, use, and transfer of select agents and toxins (42 CFR Part 1003)

## 4.2 Method Summaries

Method summaries for the analytical methods listed in Appendix A, including methods for sample preparation and determinative techniques, are provided in Sections 4.2.1 through 4.2.73. Information provided in these sections contains summary information only, extracted from the selected methods. The full version of the method needs to be consulted prior to sample analysis.

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 the full version of the method or source for obtaining a full version of the method.

**Please note:** Not all methods have been verified for the analyte/matrix combination listed in Appendix A. Please refer to the specified method to identify analyte/matrix combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 3.

### 4.2.1 EPA CLP Method SOW ILM05.3 Cyanide: Analytical Methods for Total Cyanide Analysis

This method should be used for **preparation** and **analysis** of solid and aqueous/liquid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Cyanide	57-12-5

The method allows for either large volume (500-mL aqueous/liquid samples or 1-g to 5-g solid samples mixed with 500 mL of reagent water) or medium volume (50-mL aqueous/liquid samples, or 1-g solid samples mixed with 50 mL of reagent water) sample preparation. Aqueous/liquid samples are tested for sulfides and oxidizing agents prior to preparation. Sulfides are removed with cadmium carbonate or lead carbonate. Samples are treated with sulfuric acid and magnesium chloride and distilled into a sodium hydroxide solution. The solution is treated with color agents and the cyanide determined as an ion complex by visible spectrophotometry. The method quantitation limits are 10 µg/L or 2.5 mg/kg. Surfactants may interfere with the distillation procedure.

Source: <http://www.epa.gov/superfund/programs/clp/download/ilm/ilm53d.pdf>

#### 4.2.2 EPA Office of Air Quality Planning and Standards (OAQPS) Method 207-2: Analysis for Isocyanates by High Performance Liquid Chromatography (HPLC)

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Methyl isocyanate	624-83-9

Samples are withdrawn from an emission source at an isokinetic sampling rate and collected in a multicomponent sampling train that includes a heated probe, three impingers containing the derivatizing reagent in toluene, an empty impinger, an impinger containing charcoal, and an impinger containing silica gel. The impinger contents are concentrated to dryness under a vacuum, brought to volume with acetonitrile, and analyzed with a high pressure liquid chromatograph (HPLC). Known interferences come from a derivatizing agent, 1-(2-pyridyl)piperazine. The detection limit for methyl isocyanate is 228 ng/m<sup>3</sup>.

Source: <http://www.epa.gov/ttn/emc/proposed/m-207.pdf>

#### 4.2.3 EPA NERL Method 365.1, Revision 2: Determination of Phosphorus by Semi-Automated Colorimetry

This method should be used for **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Red Phosphorus	7723-14-0

This method measures all forms of phosphorus present in the sample, converting them to orthophosphate. The analyte is determined as a reduced antimony-phospho-molybdate complex. A 50-mL sample is digested with sulfuric acid and ammonium persulfate. The digestate is analyzed by automated spectrophotometry (colorimetry) in which the sample reacts with color agents. The range of the method is 0.01 to 1.0 mg P/L. Silica, arsenate, nitrite, and sulfide may cause interference.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4823](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4823)

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

This method should be used for **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic III compound	22569-72-8
Arsenic trichloride (analyze for Arsenic)	7784-34-1
Arsine	7784-42-1
Cadmium	7440-43-9
Metals, NOS *	NA

\* NOS = Not otherwise specified

This method will determine arsine, arsenic (III) compounds, and arsenic trichloride as 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 appropriate addition of nitric acid, and then diluted to a predetermined volume and mixed before analysis. The prepared sample is analyzed using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4665](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4665)

#### 4.2.5 EPA Method 245.2: Mercury (Automated Cold Vapor Technique)

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Mercury	7439-97-6

If dissolved mercury is desired, the sample is filtered. 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 tin sulfate or tin chloride) and aerated from solution. The mercury vapor passes through a cell positioned in the light path of a cold vapor atomic absorption (CVAA) spectrophotometer. The concentration of mercury is measured using the CVAA spectrophotometer. Applicable concentration range is 0.2 - 20.0 µg/L.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4822](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4822)

#### 4.2.6 EPA Method 252.2: Osmium (Atomic Absorption, Furnace Technique)

This method should be used for the **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Osmium tetroxide	20816-12-0

This method will determine osmium tetroxide as osmium. Samples are prepared according to Section 4.1 of EPA Method 200.0 (Metals: Atomic Absorption Methods) based on type of data desired (i.e., dissolved, suspended, total, or total recoverable). If only dissolved osmium is determined, the sample should be filtered before acidification with nitric acid. For total osmium, the sample is digested with nitric and hydrochloric acid and made up to volume. Samples are analyzed according to Section 9.3 “Furnace Procedure” of EPA Method 200.0, using a graphite furnace atomic absorption spectrometer. A representative aliquot of sample is placed in the graphite tube in the furnace, evaporated to dryness, chaffed, and atomized. Radiation from an excited element is passed through the vapor containing ground state atoms of the element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation. Optimal applicable concentration range is 50 - 500 µg/L.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=5299](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=5299)

#### 4.2.7 EPA Method 300.1: Determination of Inorganic Anions in Drinking Water by Ion Chromatography

This method should be used for the **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Fluoroacetate Salts	NA

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. The method detection limit varies depending upon the nature of the sample and the specific instrumentation employed. The estimated calibration range is approximately 2 orders of magnitude.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4674](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4674)

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

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Cyanide	57-12-5

Cyanide is released from cyanide complexes as hydrocyanic acid by means of a manual reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reactions with chloramine-T, which subsequently reacts with pyridine and barbituric acid to give a red-colored complex. Some interferences, such as aldehydes, nitrate-nitrite, oxidizing agents, thiocyanate, thiosulfate, and sulfide, are eliminated or reduced by distillation. The applicable range is 5 to 500 µg/L.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=5759](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=5759)

#### 4.2.9 EPA Method 350.3: Nitrogen, Ammonia (Potentiometric, Ion Selective Electrode)

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Ammonia	7664-41-7

Ammonia is determined potentiometrically using an ion selective ammonia electrode and a pH meter having an expanded millivolt scale or a specific ion meter. The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the internal solution, which is sensed by a pH electrode. The constant level of chloride in the internal solution is sensed by a chloride selective ion electrode which acts as the reference electrode. This method covers a range of 0.03 to 1400 mg NH<sub>3</sub>-N/L.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4871](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4871)

#### 4.2.10 EPA Method 508: Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Polychlorinated biphenyls (PCBs)	1336-36-3

A measured volume of sample is extracted with methylene chloride by shaking in a separatory funnel or mechanical tumbling in a bottle. The methylene chloride extract is isolated, dried and concentrated after solvent substitution with methyl tert-butyl ether. The concentration of pesticides in the extract are

measured using a capillary column gas chromatography (GC) system equipped with an electron capture detector (ECD). This method has been validated in a single laboratory. Resulting estimated detection limits (EDLs) and method detection limits (MDLs) differ depending on the congener.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4826](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4826)

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

This method should be used for **preparation** (purging) and **analysis** of drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Carbon disulfide	75-15-0
1,2-Dichloroethane	107-06-2
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

Volatile organic compounds 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.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4803](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4803)

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

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Dichlorvos	62-73-7
Fenamiphos	22224-92-6
Mevinphos	7786-34-7
Semivolatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

Organic compounds, internal standards, and surrogates are extracted from a water sample by passing 1 L of sample water through a cartridge or disk containing a solid matrix with chemically bonded C<sub>18</sub> organic phase (liquid-solid extraction, LSE). The organic compounds are eluted from the LSE 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 gas chromatography/mass spectrometry (GC/MS) system.

Source: [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4804](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4804)

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

This method should be used for **preparation** and **analysis** of drinking water samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Aldicarb (Temik)	116-06-3
Carbofuran (Furadan)	1563-66-2
Oxamyl	23135-22-0

An aliquot of sample is measured in a volumetric flask. Samples are preserved, spiked with appropriate surrogates and filtered. Analytes are chromatographically separated by injecting an aliquot (up to 1000 µL) into a high performance liquid chromatographic (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 methyl amine, which is in turn reacted to form a fluorescent isoindole that is detected by a fluorescence detector. Analytes also are quantitated using the external standard technique.

Source: [http://www.epa.gov/ogwdw000/methods/met531\\_2.pdf](http://www.epa.gov/ogwdw000/methods/met531_2.pdf)

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

This method should be used for **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Paraquat	4685-14-7

A 250-mL sample is extracted using a C-8 liquid/solid extraction (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 high performance liquid chromatography (HPLC) system equipped with a UV absorbance detector. A photodiode array detector is used to provide simultaneous detection and confirmation of the method analytes. The analytical range depends on the sample matrix and the instrumentation used.

Source: [http://www.epa.gov/nerlcwww/m\\_549\\_2.pdf](http://www.epa.gov/nerlcwww/m_549_2.pdf)

#### 4.2.15 EPA Method 3031 (SW-846): Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry

This method should be used for **preparation** of oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6010C or 6020A should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9
Metals, NOS *	NA

\* NOS = Not otherwise specified

This method will determine arsenic (III) compounds and arsenic trichloride as arsenic. The method also will determine osmium tetroxide as osmium. A 0.5-g sample of oil, oil sludge, tar, wax, paint, or paint sludge is mixed with potassium permanganate and sulfuric acid. The mixture is then treated with nitric and hydrochloric acids, filtered and diluted to volume. Excess manganese may be removed with ammonium hydroxide. Digestates are analyzed by Method 6020A or 6010C (SW-846).

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3031.pdf>

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

This method should be used for **preparation** of solid and/or oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6010C, 6020A, or 7010 should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic Trichloride	7784-34-1
Arsine	7784-42-1
Cadmium	7440-43-9
Metals, NOS *	NA
Osmium tetroxide	20816-12-0
Titanium tetrachloride	7550-45-0

\* NOS = Not otherwise specified

This method will determine arsine, arsenic (III) compounds, and arsenic trichloride as arsenic. The method also will determine titanium tetrachloride as titanium. A 1-g to 2-g sample is digested with nitric acid and hydrogen peroxide. Samples to be analyzed by Method 6010C (SW-846) for cadmium are also treated with hydrochloric acid. Sample volumes are reduced, then brought up to a final volume of 100 mL. Samples are analyzed for arsenic by Method 6020A (SW-846) and for cadmium by either Method 6010C or 6020A (SW-846).

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3050b.pdf>



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

This method should be used for **preparation** of aqueous/liquid and/or drinking water samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6020A, 8015C, 8082A, 8270D, or 8321B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Bromadiolone	28772-56-7
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
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
EA2192	73207-98-4
Ethylchloroarsine (ED)	598-14-1
Ethylmethyl phosphonate (EMPA)	1832-53-7
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2

<b>Contaminant</b>	<b>CAS RN</b>
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

This method describes a procedure for isolating organic compounds from aqueous/liquid samples. 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 (see Table 1 in Method 3520C), 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.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3520c.pdf>

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

This method should be used for **preparation** of aqueous/liquid and/or drinking water samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6020A, 8015C, 8082A, 8270D, or 8321B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Bromadiolone	28772-56-7
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
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
EA2192	73207-98-4
Ethylchloroarsine (ED)	598-14-1
Ethylmethyl phosphonate (EMPA)	1832-53-7
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2

Contaminant	CAS RN
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

This method describes a procedure for isolating target organic analytes from aqueous/liquid samples using solid-phase extraction (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 solid-phase extraction 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.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3535a.pdf>

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

This method should be used for **preparation** of solid and/or oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6020A, 8015C, 8082A, 8270D, or 8321B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Bromadiolone	28772-56-7
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
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
EA2192	73207-98-4
Ethylchloroarsine (ED)	598-14-1
Ethylmethyl phosphonate (EMPA)	1832-53-7
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2

Contaminant	CAS RN
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

Approximately 10 g of solid sample is mixed with an equal amount of anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and after adding the appropriate surrogate amount, 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.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3541.pdf>

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

This method should be used for **preparation** of solid and/or oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6020A, 8015C, 8082A, 8270D, or 8321B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Bromadiolone	28772-56-7
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
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
EA2192	73207-98-4
Ethylchloroarsine (ED)	598-14-1
Ethylmethyl phosphonate (EMPA)	1832-53-7
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2

Contaminant	CAS RN
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

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. (Note: Sodium sulfate can cause clogging, and air drying or diatomaceous earth may be preferred.) 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, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed. 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, 1 to 1400 µg/kg of PCBs, and 1 to 2500 ng/kg of PCDDs/PCDFs.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3545a.pdf>



#### 4.2.21 EPA Method 3580A (SW-846): Waste Dilution

This method should be used for **preparation** of oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 6020A, 8015C, 8082A, 8270D, or 8321B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Bromadiolone	28772-56-7
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
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
EA2192	73207-98-4
Ethylchloroarsine (ED)	598-14-1
Ethylmethyl phosphonate (EMPA)	1832-53-7
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Kerosene	64742-81-0
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Methylphosphonic acid (MPA)	993-13-5
Mevinphos	7786-34-7

Contaminant	CAS RN
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

This method describes a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. One gram of sample is weighed into a capped tube, and the sample is diluted to 10.0 mL with an appropriate solvent. The method is designed for wastes that may contain organic chemicals at a concentration greater than 20,000 mg/kg and that are soluble in the dilution solvent.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3580a.pdf>

#### 4.2.22 EPA Method 3585 (SW-846): Waste Dilution for Volatile Organics

This method should be used for **preparation** of oily solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 8015C or 8260B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
3-Chloro-1,2-propanediol	96-24-2
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
1,4-Dithiane	505-29-3
Ethylene oxide	75-21-8
Gasoline Range Organics	NA
Phosgene	75-44-5
Propylene oxide	75-56-9
1,4-Thioxane	15980-15-1
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

This method describes a solvent dilution of a non-aqueous waste sample prior to direct injection analysis. It is designed for use in conjunction with GC or GC/MS analysis of wastes that may contain organic chemicals at a concentration greater than 1 mg/kg and that are soluble in the dilution solvent. Highly contaminated or highly complex samples may be diluted prior to analysis for volatiles using direct injection. One gram of sample is weighed into a capped tube or volumetric flask. The sample is diluted to 2.0 to 10.0 mL with *n*-hexadecane or other appropriate solvent. Diluted samples are injected into the GC or GC/MS for analysis.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3585.pdf>

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

This method should be used for **preparation** of aqueous/liquid and/or drinking water samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 8015C or 8260B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
3-Chloro-1,2-propanediol	96-24-2

Contaminant	CAS RN
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
1,4-Dithiane	505-29-3
Ethylene oxide	75-21-8
Gasoline Range Organics	NA
Kerosene	64742-81-0
Propylene oxide	75-56-9
1,4-Thioxane	15980-15-1
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous/liquid samples and water miscible liquid samples. It also describes the analysis of high concentration soil and waste sample extracts prepared using Method 5035A (SW-846). The gas chromatographic determinative steps for this sample preparation technique can be found in the determinative method identified in Appendix A.

**Aqueous/Liquid Samples:** An inert gas is bubbled through a portion of the aqueous/liquid sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous/liquid phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

**High Concentration Extracts from Method 5035A (SW-846):** An aliquot of the extract prepared using Method 5035A is combined with organic-free reagent water in the purging chamber. It is then analyzed by purge-and-trap GC or GC/MS following the procedure used for the aqueous/liquid samples.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/5030c.pdf>

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

This method should be used for **preparation** of solid samples for the contaminants identified below and listed in Appendix A. Note: SW-846 Method 8015C or 8260B should be used for sample analysis (refer to Appendix A).

Contaminant	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
3-Chloro-1,2-propanediol	96-24-2
Cyanogen chloride	506-77-4

Contaminant	CAS RN
1,2-Dichloroethane	107-06-2
1,4-Dithiane	505-29-3
Ethylene oxide	75-21-8
Gasoline Range Organics	NA
Kerosene	64742-81-0
Phosgene	75-44-5
Propylene oxide	75-56-9
1,4-Thioxane	15980-15-1
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

This method describes a closed-system purge-and-trap process for analysis of volatile organic compounds (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 automatically added, and the vial 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 gas chromatograph 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., 8260B (SW-846)).

Source: [http://www.epa.gov/epaoswer/hazwaste/test/pdfs/5035a\\_r1.pdf](http://www.epa.gov/epaoswer/hazwaste/test/pdfs/5035a_r1.pdf)

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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9
Metals, NOS *	NA
Osmium tetroxide	20816-12-0
Titanium tetrachloride	7550-45-0

\* NOS = Not otherwise specified

This method determines arsenic (III) compounds and arsenic trichloride as arsenic, osmium tetroxide as osmium, and titanium tetrachloride as titanium. Any other metals are determined as the metal. Aqueous

samples (prepared using SW-846 Method 5050), soil samples (prepared using SW-846 Methods 3050B or 5050), oily solid samples (prepared using SW-846 Methods 3050B or 3031), and air filter/particle samples (prepared using Inorganic (IO) Method 3.4) are analyzed by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES). Detection limits vary with each analyte. The analytical range may be extended by sample dilution.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/6010c.pdf>

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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
Lewisite oxide	1306-02-1
Metals, NOS *	NA
Titanium tetrachloride	7550-45-0

\* NOS = Not otherwise specified

This method will determine arsenic (III) compounds, arsenic trichloride, Lewisite oxide, and CVAA as arsenic. The method also will determine titanium tetrachloride as titanium. Any other metals are determined as the metal. Aqueous samples (prepared using SW-846 Method 5050), soil samples (prepared using SW-846 Methods 3050B or 5050), oily solid samples (prepared using SW-846 Methods 3050B or 3031), and air filter/particle samples (prepared using IO Method 3.5) are analyzed by Inductively Coupled Plasma - Mass Spectrometry. Detection limits vary with each analyte. The analytical range may be extended by sample dilution.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/6020a.pdf>

#### 4.2.27 EPA Method 7010 (SW-846): Graphite Furnace Atomic Absorption Spectrophotometry

This method should be used for **analysis** of the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Arsine	7784-42-1

This method determines arsine as arsenic in environmental samples. Soil samples (prepared using SW-846 Method 3050B) are analyzed by Graphite Furnace Atomic Absorption Spectrophotometry (GFAA). A representative aliquot of the sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Detection limits vary with each matrix and instrument used. The analytical range may be extended by sample dilution.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/7010.pdf>

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

This method should be used for **preparation** and **analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Mercury	7439-97-6

A 100-mL aqueous or liquid waste 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 cold vapor atomic absorption. The detection limit for the method is less than 0.2 µg/L. Chloride and copper are potential interferences.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/7470a.pdf>

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

This method should be used for **preparation** and **analysis** of solid phase samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Mercury	7439-97-6

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 cold vapor atomic absorption. Chloride and copper are potential interferences.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/7471b.pdf>

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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Diesel Range Organics	NA
Gasoline Range Organics	NA
Kerosene	64742-81-0

This method provides gas chromatographic 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 gas chromatograph 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. The estimated method detection limits vary with each analyte and range between 2 and 48 µg/L for aqueous/liquid samples. The method detection limits in other matrices have not been evaluated for this method. The analytical range depends on the target analyte(s) and the instrument used.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8015c.pdf>

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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Allyl alcohol	107-18-6
Carbon disulfide	75-15-0
2-Chloroethanol	107-07-3
3-Chloro-1,2-propanediol	96-24-2
Cyanogen chloride	506-77-4
1,2-Dichloroethane	107-06-2
1,4-Dithiane	505-29-3
Ethylene oxide	75-21-8
Phosgene	75-44-5
Propylene oxide	75-56-9
1,4-Thioxane	15980-15-1
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

Volatile compounds are introduced into a gas chromatograph by purge-and-trap or other methods (see Sec. 1.2 in Method 8260B). 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 mass spectrometer (MS) interfaced to the gas chromatograph (GC). Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. The estimated quantitation limit (EQL) of Method 8260B for an individual analyte is dependent on the instrument as well as the choice of sample preparation/introduction method. Using standard quadrupole instrumentation and the purge-and-trap technique, estimated quantitation limits are 5 µg/kg (wet weight) for soil/sediment samples and 5 µg/L for ground water (see Table 3 in Method 8260B). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8260b.pdf>



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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
Cyclohexyl sarin (CF)	329-99-7
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Dimethylphosphite	868-85-9
Ethylchloroarsine (ED)	598-14-1
Fenamiphos	22224-92-6
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Methyl hydrazine	60-34-4
Methyl parathion	298-00-0
Mevinphos	7786-34-7
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Nicotine	54-11-5
Perfluoroisobutylene (PFIB)	382-21-8
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Sarin (GB)	107-44-8
Semivolatile Organic Compounds, NOS *	NA
Soman (GD)	96-64-0
Strychnine	57-24-9
Tabun (GA)	77-81-6

Contaminant	CAS RN
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Thiodiglycol (TDG)	111-48-8
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

Samples are prepared for analysis by gas chromatography/mass spectrometry (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 gas chromatograph (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 mass spectrometer (MS) connected to the gas chromatograph. Analytes eluted from the capillary column are introduced into the mass spectrometer. The estimated method detection limits 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.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8270d.pdf>

#### 4.2.33 EPA Method 8082A (SW-846): Polychlorinated Biphenyls (PCBs) by Gas Chromatography

This method should be used for **analysis** of samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Polychlorinated biphenyls (PCBs)	1336-36-3

This method is to be employed following sample preparation by the appropriate sample preparation procedure listed in Appendix A. Method 8082A is used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid, oily solid, aqueous, non-aqueous liquid, drinking water, and air samples. The method uses open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). The target compounds may be determined using either a single- or dual-column analysis system.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8082a.pdf>

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

This method should be used for **preparation** and **analysis** of solid, aqueous/liquid, and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Formaldehyde	50-00-0

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-dinitrophenylhydrazine (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. The method detection limit for formaldehyde varies depending on sample conditions and instrumentation but is approximately 6.2 µg/L for aqueous/liquid samples.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8315a.pdf>

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

This method should be used for **preparation** and **analysis** of solid, oily solid, and aqueous/liquid samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Aldicarb (Temik)	116-06-3
Carbofuran (Furadan)	1563-66-2
Oxamyl	23135-22-0

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 the instrumental conditions. Waste samples with a high level of extractable fluorescent compounds are expected to yield significantly higher detection limits. The estimated method detection limits vary with each analyte and range between 1.7 and 9.4 µg/L for aqueous/liquid samples and 10 and 50 µg/kg for soil samples.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8318a.pdf>

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

This method should be used for **analysis** of the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Bromadiolone	28772-56-7
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphoramidic acid	33876-51-6
EA2192	73207-98-4
Ethylmethyl phosphonate (EMPA)	1832-53-7
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Methylphosphonic acid (MPA)	993-13-5
Tetramethylenedisulfotetramine	80-12-6

This method provides reversed-phase high performance liquid chromatographic (RP/HPLC), thermospray (TS) mass spectrometric (MS), and ultraviolet (UV) conditions for detection of the target analytes. Sample extracts can be analyzed by direct injection into the thermospray or onto a liquid chromatographic-thermospray 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 TS/MS. Quantitative analysis may be performed by either TS/MS or UV detection, using either an external or internal standard approach. TS/MS detection may be performed in either a negative ionization (discharge electrode) mode or a positive ionization mode, with a single quadrupole mass spectrometer. The use of MS/MS techniques is an option. The analytical range and detection limits vary depending on the target analyte and instrument used.

Source: <http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8321b.pdf>

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

This method should be used for **preparation** and **analysis** of solid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Asbestos	1332-21-4

This method describes procedures to (a) identify asbestos in dust and (b) 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. 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:

[http://www.astm.org/cgi-bin/SoftCart.exe/STORE/filtrexx40.cgi?U+mystore+tavs3076+-L+D5755:03+/usr6/htdocs/astm.org/DATABASE.CART/REDLINE\\_PAGES/D5755.htm](http://www.astm.org/cgi-bin/SoftCart.exe/STORE/filtrexx40.cgi?U+mystore+tavs3076+-L+D5755:03+/usr6/htdocs/astm.org/DATABASE.CART/REDLINE_PAGES/D5755.htm)

#### **4.2.38 ASTM Method D6480-99: Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy**

This method should be used for **preparation** and **analysis** of solid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Asbestos	1332-21-4

This test 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. 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 (ED) and EDXA at a magnification from 15,000 to 20,000X.

Source:

[http://www.astm.org/cgi-bin/SoftCart.exe/STORE/filtrexx40.cgi?U+mystore+tavs3076+-L+D6480:99+/usr6/htdocs/astm.org/DATABASE.CART/REDLINE\\_PAGES/D6480.htm](http://www.astm.org/cgi-bin/SoftCart.exe/STORE/filtrexx40.cgi?U+mystore+tavs3076+-L+D6480:99+/usr6/htdocs/astm.org/DATABASE.CART/REDLINE_PAGES/D6480.htm)

#### **4.2.39 ISO Method - 10312: Ambient Air - Determination of Asbestos Fibres - Direct-transfer Transmission Electron Microscopy Method (TEM)**

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Asbestos	1332-21-4

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. The range of concentrations that can be determined is 50 structures/mm<sup>2</sup> to 7,000 structures/mm<sup>2</sup> on the filter. In a 4000-L air sample with approximately 10 pg/m<sup>3</sup> (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.

Source:

<http://www.iso.org/iso/en/CatalogueDetailPage.CatalogueDetail?CSNUMBER=18358&ICS1=13&ICS2=40&ICS3=20>

#### 4.2.40 ISO Method - 12884: Ambient Air – Determination of Total (Gas and Particle-phase) Polycyclic Aromatic Hydrocarbons – Collection on Sorbent-backed Filters with Gas Chromatographic/Mass Spectrometric Analysis

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Carbofuran (Furadan)	1563-66-2
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Dimethylphosphite	868-85-9
Fenamiphos	22224-92-6
Methyl parathion	298-00-0
Mevinphos	7786-34-7
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Semivolatile Organic Compounds, NOS *	NA
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3
Tetramethylenedisulfotetramine	80-12-6
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

This standard specifies sample collection, cleanup, and analysis procedures for determination of polycyclic aromatic hydrocarbons (PAHs) in air. It is designed to collect both gas-phase and particulate-phase compounds. It is a high-volume (100 to 250 L/min) method capable of detecting 0.05 ng/m<sup>3</sup> or less concentrations in 350-m<sup>3</sup> samples. An air sample is collected by pulling air at a maximum flow rate of 225 L/min through a fine-particle filter followed by a vapor trap containing polyurethane foam (PUF) or styrene/divinylbenzene polymer resin (XAD-2). The particle filter and sorbent cartridge are extracted

together in a Soxhlet extractor, and the sample extract is concentrated using a Kuderna-Danish concentrator (or other verified method), followed by further concentration under a nitrogen stream. An aliquot of sample is analyzed using GC/MS.

Source:

<http://www.iso.org/iso/en/CatalogueDetailPage.CatalogueDetail?CSNUMBER=1343&ICS1=13&ICS2=40&ICS3=20>

#### 4.2.41 ISO Method - 16000-3: Indoor Air – Part 3: Determination of Formaldehyde and Other Carbonyl Compounds – Active Sampling Method

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Formaldehyde	50-00-0

Samples are collected by passing air through a cartridge containing silica gel coated with acidified 2,4-dinitrophenylhydrazine (DNPH) reagent. The carbonyl group reacts with DNPH in the presence of an acid to form stable derivatives that are analyzed for parent aldehydes and ketones using high performance liquid chromatography (HPLC) with UV detection. Interferences are caused by certain isomeric aldehydes or ketones that may be unresolved by the HPLC. Interferences from organic compounds that have the same retention times and significant absorbance at 360 nm can be overcome by altering separation conditions (e.g., use of alternative columns or mobile gas phase compositions).

Source:

<http://www.iso.org/iso/en/CatalogueDetailPage.CatalogueDetail?CSNUMBER=29049&ICS1=13&ICS2=40&ICS3=20>

#### 4.2.42 NIOSH Method 1402: Alcohols III

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Allyl alcohol	107-18-6

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 eluent is added to a sampling vial, which is crimped and then allowed to sit for 30 minutes with occasional agitation. High humidity reduces the sampling capacity. Volatile compounds can be replaced on the sorbent by less volatile substances. The working range is dependant on the size of the compound. For a 10-L sample, compounds with a lower molecular weight have a working range from 45 to 140 mg/m<sup>3</sup> and compounds with a higher molecular weight have a working range between 175 to 680 mg/m<sup>3</sup>.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/1402.pdf>

#### 4.2.43 NIOSH Method 1612: Propylene Oxide

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Propylene oxide	75-56-9

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 (CS<sub>2</sub>) is added to the vial and allowed to sit for 30 minutes prior to analysis with occasional agitation. No interferences have been found in this method. The working range is between 8 to 295 ppm for air samples of 5 L.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/1612.pdf>

#### 4.2.44 NIOSH Method 2010: Amines, Aliphatic

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Methylamine	74-89-5

A silica gel sorbent tube is used for sample collection with a flow rate of 0.01 to 1 L/min. An aliquot of 0.1 M H<sub>2</sub>SO<sub>4</sub> in aqueous methanol is added to sample vials, and the vials are agitated in a sonic water bath. Samples are neutralized using 0.3 M KOH prior to analysis by GC. Methylamine can be collected on a sampling tube containing XAD-7 resin coated with 10% NBD chloride (7-chloro-4-nitrobenzo-2-oxa-1,3-diazole) by weight. A stable derivative is formed on the coated resin. The derivative is extracted with 5% (w/v) NBD chloride in tetrahydrofuran (THF) and analyzed by high-performance liquid chromatography (<http://www.osha-slc.gov/dts/sltc/methods/organic/org040/org040.html>). A methanol peak can interfere with low-level analysis, and high humidity can cause reduced capacity of sample adsorption. The working range is between 8 to 183 ppm for diethylamine and 4 to 71 ppm for dimethylamine in air samples of 20 L.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/2010.pdf>

#### 4.2.45 NIOSH Method 2513: Ethylene Chlorohydrin

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
2-Chloroethanol	107-07-3

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. No interferences have been identified. Humidity may decrease the breakthrough volume during sample collection. The working range of the method is 0.5 to 15 ppm for a 20-L air sample.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/2513.pdf>



#### 4.2.46 NIOSH Method 3510: Monomethylhydrazine

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A:

Contaminant	CAS RN
Methyl hydrazine	60-34-4

Samples are collected into a bubbler containing HCl using a flow rate of 0.5 to 1.5 L/min. Samples are then mixed with phosphomolybdic acid solution and transferred to a large test tube for analysis. Positive interferences that have been noted include 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. The working range of the method is 0.027 to 2.7 ppm for a 20-L air sample.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/3510.pdf>

#### 4.2.47 NIOSH Method 6001: Arsine

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Arsine	7784-42-1

In this method, arsine is determined as arsenic. A volume of 0.1 to 10 L of air is drawn through a sorbent tube containing activated charcoal. The sorbent is extracted with a nitric acid solution. The arsine is determined by graphite furnace atomic absorption. The working range of the method is 0.001 to 0.2 mg/m<sup>3</sup> for a 10-L sample. The method is subject to interferences from other arsenic compounds.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6001.pdf>

#### 4.2.48 NIOSH Method 6002: Phosphine

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Phosphine	7803-51-2

In this method, phosphine is determined as phosphate. One to 16 liters of air are drawn through a sorbent tube containing silica gel coated with Hg(CN)<sub>2</sub>. 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, the phosphate is determined by visible spectrometry. The working range of the method is 0.02 to 0.9 mg/m<sup>3</sup> for a 16-L sample. The method is subject to interferences from phosphorus trichloride, phosphorus pentachloride, and organic phosphorus compounds.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6002.pdf>

#### 4.2.49 NIOSH Method 6004: Sulfur Dioxide

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Sulfur dioxide	7446-09-5

In this method, sulfur dioxide is determined as sulfite plus sulfate. A volume of 40 to 200 liters of air is drawn through a sodium carbonate-treated filter that is preceded by a 0.8  $\mu\text{m}$  filter to remove particulates and sulfuric acid. The treated filter is extracted with a carbonate/bicarbonate solution and the extract analyzed by ion chromatography for sulfite and sulfate. The sulfur dioxide is present as sulfite on the filter; however, because sulfite oxidizes to sulfate, both ions must be determined and the results summed. The working range of the method is 0.5 to 20  $\text{mg}/\text{m}^3$  for a 100-L sample. The method is subject to interference from sulfur trioxide in dry conditions.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6004.pdf>

#### 4.2.50 NIOSH Method 6010: Hydrogen Cyanide

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Hydrogen cyanide	74-90-8

Hydrogen cyanide is determined as a cyanide ion complex by this method. A volume of 2 to 90 liters 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. The extract is pH adjusted and treated with the coupling-color agent. The cyanide ion is determined by visible spectrophotometry. The working range of the method is 3 to 260  $\text{mg}/\text{m}^3$  for a 3-L sample. The method is subject to interference from high concentrations of hydrogen sulfide.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6010.pdf>

#### 4.2.51 NIOSH Method 6011: Bromine and Chlorine

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Chlorine	7782-50-5

In this method, chlorine is determined as chloride. A volume of 2 to 90 liters of air is drawn through a silver membrane filter. A prefilter is used to remove particulate chlorides. The filter is extracted with sodium hyposulfate solution, and the extract analyzed for chloride by ion chromatography. The working range of the method is 0.02 to 1.5  $\text{mg}/\text{m}^3$  for a 90-L sample. The method is subject to positive interference by HCl and negative interference by hydrogen sulfide.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6011.pdf>

#### 4.2.52 NIOSH Method 6013: Hydrogen Sulfide

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Hydrogen sulfide	7783-06-4

Hydrogen sulfide is determined as sulfate by this method. A volume of 1.2 to 40 liters 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 ion chromatography. The working range of the method is 0.9 to 20 mg/m<sup>3</sup> for a 20-L sample. The method is subject to interference from sulfur dioxide.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6013.pdf>

#### 4.2.53 NIOSH Method 6015: Ammonia

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Ammonia	7664-41-7

Ammonia is determined as indophenol blue by this method. A volume of 0.1 to 96 liters 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. The working range of the method is 0.15 to 300 mg/m<sup>3</sup> for a 10-L sample. No interferences have been identified.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6015.pdf>

#### 4.2.54 NIOSH Method 6402: Phosphorus Trichloride

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Phosphorus trichloride	7719-12-2

In this method, phosphorus trichloride is determined as phosphate. A volume of 11 to 100 liters of air is drawn through a bubbler containing reagent water. The resulting H<sub>3</sub>PO<sub>3</sub> solution is oxidized to H<sub>3</sub>PO<sub>4</sub> and color agents are added. The solution is analyzed by visible spectrophotometry. The working range of the method is 1.2 to 80 mg/m<sup>3</sup> for a 25-L sample. Phosphorus (V) compounds do not interfere. The sample solutions are stable to oxidation by air during sampling.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/6402.pdf>

#### 4.2.55 NIOSH Method 7903: Acids, Inorganic

This method should be used for **preparation** and **analysis** of air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Hydrogen bromide	10035-10-6
Hydrogen chloride	7647-01-0
Hydrogen fluoride	7664-39-3

Acids are analyzed as bromide, chloride, and fluoride, respectively, by this method. A volume of 3 to 100 liters 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 ion chromatography. The working range of this method is 0.01 to 5 mg/m<sup>3</sup> for a 50-L sample. 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.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/7903.pdf>

#### 4.2.56 NIOSH Method 7904: Cyanides, Aerosol and Gas

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Cyanide	57-12-5

In this method, cyanide(s) are determined as cyanide ion. A volume of 10 to 180 liters of air is drawn through a 0.8-µm PVC membrane filter and a bubbler containing 0.1N KOH solution. The filter collects aerosols of cyanide solutions and the bubbler collects HCN. The filters are extracted with the KOH solution. Sulfide must be removed from the solutions prior to analysis. Analyze the solutions by cyanide ion specific electrode (ISE). The working range of the method is 0.5 to 15 mg/m<sup>3</sup> for a 90-L sample. Sulfide, chloride, iodide, bromide, cadmium, zinc, silver, nickel, cuprous iron, and mercury are interferences.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/7904.pdf>

#### 4.2.57 NIOSH Method 7906: Fluorides, Aerosol and Gas

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Hydrogen fluoride	7664-39-3

Hydrogen fluoride is determined as fluoride ion by this method. A volume of 1 to 800 liters of air is drawn through a 0.8- $\mu$ m 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 ion chromatography. The working range of the method is 0.04 to 8 mg/m<sup>3</sup> for 250-L samples. If other aerosols are present, gaseous fluoride may be slightly underestimated owing to adsorption onto or reaction with particles; with concurrent overestimation of particulate/gaseous fluoride ratio.

Source: <http://www.cdc.gov/niosh/nmam/pdfs/7906.pdf>

#### 4.2.58 NIOSH Method S301-1: Fluoroacetate Anion

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Fluoroacetate salts	NA

This method was developed specifically for sodium fluoroacetate, but also may be applicable to other fluoroacetate salts. 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 ion chromatography using electrolytic conductivity detection. The analytical range of this method is estimated to be 0.01 to 0.16 mg/m<sup>3</sup>. The detection limit of the analytical method is estimated to be 20 ng of sodium fluoroacetate per injection, corresponding to a 100- $\mu$ L aliquot of a 0.2- $\mu$ g/mL standard.

Source: <http://www.cdc.gov/niosh/pdfs/s301.pdf>

#### 4.2.59 OSHA Method ID-188: Ammonia in Workplace Atmospheres - Solid Sorbent

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Ammonia	7664-41-7

In this method, ammonia is determined as ammonium ion. A volume of 7.5 to 24 liters of air is drawn through a sulfuric acid-treated carbon bead sorbent. The sorbent is extracted with reagent water and the extract analyzed for ammonium by ion chromatography. The detection limit for the method is 0.60 ppm for 24-L samples and the quantitation limit is 1.5 ppm for 24-L samples. Volatile amines, monethanolamine, isopropanolamine, and propanolamine may be interferences. Particulate ammonium salts can be a positive interference (trapped on the glass wool filter plug in the sorbent tube).

Source: <http://www.osha-slc.gov/dts/sltc/methods/inorganic/id188/id188.html>

#### 4.2.60 OSHA Method ID-216SG: Boron Trifluoride (BF<sub>3</sub>)

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Boron trifluoride	7637-07-2

Boron trifluoride is determined as fluoroborate by this method. A volume of 30 to 480 liters of air is drawn through a bubbler containing 0.1-M ammonium fluoride. The solution is diluted and analyzed with a fluoroborate ion specific electrode (ISE). The detection limit is 10 µg in a 30-L sample.

Source: <http://www.osha-slc.gov/dts/sltc/methods/partial/id216sg/id216sg.html>

#### 4.2.61 Standard Method 4110 B: Ion Chromatography with Chemical Suppression of Eluent Conductivity

This method should be used for **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Hydrogen bromide	10035-10-6
Hydrogen chloride	7647-01-0

The contaminants are determined as bromide and chloride respectively by this method. Aqueous/liquid samples are pre-filtered and injected onto the ion chromatograph. The anions are identified on the basis of retention time and quantified by measurement of peak area or height. The method can detect bromide and chloride at 0.1 mg/L. Lower values can be achieved using a higher scale setting and an electronic integrator. Other salts of the anions are a positive interference. Low molecular weight organic acids may interfere with chloride.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

#### 4.2.62 Standard Method 4500-NH<sub>3</sub> B: Preliminary Distillation Step

This method should be used for **preparation** of aqueous/liquid samples for the contaminant identified below and listed in Appendix A. Note: Method 4500-NH<sub>3</sub> G should be used for sample analysis.

Contaminant	CAS RN
Ammonia	7664-41-7

A 0.5-L to 1-L water sample is dechlorinated, buffered, adjusted to pH 9.5, and distilled into a sulfuric acid solution. The distillate is brought up to volume and neutralized with sodium hydroxide. The distillate is analyzed by Method 4500-NH<sub>3</sub> G.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

#### 4.2.63 Standard Method 4500-NH<sub>3</sub> G: Automated Phenate Method

This method should be used for **analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Ammonia	7664-41-7

Ammonia is determined as indophenol blue by this method. A portion of the neutralized distillate from procedure 4500-NH<sub>3</sub> B is run through the manifold described in the method. The ammonium in the distillate reacts with solutions of disodium EDTA, sodium phenate, sodium hypochlorite, and sodium nitroprusside. The resulting indophenol blue is detected by colorimetry in a flow cell. The range of the method is 0.02 to 2.0 mg/L.

*Source:* American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

#### 4.2.64 Standard Method 4500-Cl G: DPD Colorimetric Method

This method should be used for **preparation** and **analysis** of aqueous/liquid and drinking water samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Chlorine	7782-50-5

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 chloramines and nitrogen trichloride) is to be determined, KI crystals are added. Results for chromate and manganese are blank corrected using thioacetamide solution. The method can detect 10 µg/L chlorine. Organic contaminants and strong oxidizers may cause interference.

*Source:* American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

#### 4.2.65 IO Compendium Method IO-3.1: Selection, Preparation, and Extraction of Filter Material

This method should be used for **preparation** and **analysis** of air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9

This method will determine arsenic (III) compounds and arsenic trichloride as arsenic. 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 hydrochloric/nitric acid mix and microwave or hotplate heating. The extract is filtered and worked up to 20 mL. The extract is analyzed by compendium methods IO-3.4 or IO-3.5.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-3-1.pdf>

#### 4.2.66 IO Compendium Method IO-3.4: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma (ICP) Spectroscopy

This method should be used for **preparation** and **analysis** of air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9
Osmium tetroxide	20816-12-0

All analytes are determined as the metal by this method. Ambient air is sampled by high-volume filters using compendium method IO-2.1 (a sampling method). The filters are extracted by compendium method IO-3.1 and the extracts analyzed by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES). Detection limits, ranges, and interference corrections are dependent on the analyte and the instrument used.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-3-4.pdf>  
<http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-2-1.pdf>



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

This method should be used for **preparation** and **analysis** of air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Arsenic (III) compounds	22569-72-8
Arsenic trichloride	7784-34-1
Cadmium	7440-43-9

All analytes are determined as the metal by this method. Ambient air is sampled by high-volume filters using compendium method IO-2.1 (a sampling method). The filters are extracted by compendium method IO-3.1 and the extracts analyzed by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). Detection limits, ranges, and interference corrections are dependent on the analyte and the instrument used.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-3-5.pdf>  
<http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-2-1.pdf>

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

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Mercury	7439-97-6

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 BrCl, 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 Atomic Fluorescence Spectrometry. The detection limits are 30 pg/m<sup>3</sup> for particulate mercury and 45 pg/m<sup>3</sup> for vapor mercury. Detection limits, analytical range, and interferences are dependent on the instrument used. There are no known positive interferences at 253.7 nm wavelength. Water vapor will cause a negative interference.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-5.pdf>

#### 4.2.69 EPA Air Method, Toxic Organics - 6 (TO-6): Method for the Determination of Phosgene in Ambient Air Using High Performance Liquid Chromatography

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Phosgene	75-44-5

This method can be used to detect phosgene in air at the 0.1 ppbv level. Ambient air is drawn through a midjet impinger containing 10 mL of 2/98 aniline/toluene (by volume). Phosgene readily reacts with aniline to form carbanilide (1,3-diphenylurea), which is stable indefinitely. After sampling, the impinger contents are transferred to a screw capped vial having a Teflon-lined cap and returned to the laboratory for analysis. The solution is heated to dryness and the residue is dissolved in acetonitrile. Carbanilide is determined in the acetonitrile solution using reverse-phase HPLC with an ultraviolet (UV) absorbance detector operating at 254 nm. Precision for phosgene spiked into a clean air stream is  $\pm 15$  to 20% relative standard deviation. Recovery is quantitative within that precision, down to less than 3 ppbv.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-6.pdf>

#### 4.2.70 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)

This method should be used for **sampling** and **analysis** of ambient air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Carbofuran (Furadan)	1563-66-2
Dichlorvos	62-73-7
Dicrotophos	141-66-2
Dimethylphosphite	868-85-9
Fenamiphos	22224-92-6
Methyl parathion	298-00-0
Mevinphos	7786-34-7
Phencyclidine	77-10-1
Phenol	108-95-2
Phorate	298-02-2
Polychlorinated biphenyls (PCBs)	1336-36-3
Semivolatile Organic Compounds, NOS *	NA
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1
Tetraethyl pyrophosphate	107-49-3

Contaminant	CAS RN
Tetramethylenedisulfotetramine	80-12-6
Trimethyl phosphite	121-45-9
VE	21738-25-0
VG	78-53-5
VM	21770-86-5
VX [O-ethyl-S-(2-diisopropylaminoethyl)methyl phosphonothiolate]	50782-69-9

\* NOS = Not otherwise specified

A low-volume (1 to 5 L/minute) sample is used to collect vapors on a sorbent cartridge containing PUF or PUF in combination with another solid sorbent. Airborne particles may also be 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 gas chromatography coupled with an electron capture detector (ECD). Nitrogen-phosphorous detector (NPD), flame photometric detector (FPD), Hall electrolytic conductivity detector (HECD), or mass spectrometer (MS) also may be used. This method is applicable to multicomponent atmospheres, 0.001 to 50 µg/m<sup>3</sup> concentrations, and 4 to 24-hour sampling periods. The limit of detection will depend on the specific compounds measured, the concentration level, and the degree of specificity required.

Source: <http://www.epa.gov/ttnamti1/files/ambient/airtox/to-10ar.pdf>

#### 4.2.71 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)

This method should be used for **preparation** and **analysis** of air samples for the contaminants identified below and listed in Appendix A.

Contaminant	CAS RN
Carbon disulfide	75-15-0
3-Chloro-1,2-propanediol	96-24-2
Chloropicrin	76-06-2
Chlorosarin	1445-76-7
Chlorosoman	7040-57-5
2-Chlorovinylarsonous acid (CVAA)	85090-33-1
Cyanogen chloride	506-77-4
Cyclohexyl sarin (GF)	329-99-7
1,2-Dichloroethane	107-06-2
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethylphosphoramidic acid	33876-51-6

<b>Contaminant</b>	<b>CAS RN</b>
1,4-Dithiane	505-29-3
EA2192	73207-98-4
Ethyldichloroarsine (ED)	598-14-1
Ethylene oxide	75-21-8
Ethylmethyl phosphonate (EMPA)	1832-53-7
GE (1-methylethyl ester ethyl-phosphonofluoridic acid)	1189-87-3
Isopropyl methylphosphonic acid (IMPA)	1832-54-8
Lewisite 1 (L-1) [2-chlorovinylchloroarsine]	541-25-3
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1
Lewisite oxide	1306-02-1
Methylphosphonic acid (MPA)	993-13-5
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2
Mustard, sulfur (HD) / Mustard gas (H)	505-60-2
Oxamyl	23135-22-0
Perfluoroisobutylene (PFIB)	382-21-8
Phosgene	75-44-5
Sarin (GB)	107-44-8
Soman (GD)	96-64-0
Tabun (GA)	77-81-6
Thiodiglycol (TDG)	111-48-8
1,4-Thioxane	15980-15-1
Volatile Organic Compounds, NOS *	NA

\* NOS = Not otherwise specified

The atmosphere is sampled by introduction of air into a specially prepared stainless steel canister (specially 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. After the concentration and drying steps are completed, the 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. 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.

Source: <http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-15r.pdf>

#### 4.2.72 Journal of Analytical Atomic Spectrometry, 2000, 15, pp. 277-279: Boron Trichloride Analysis

This method should be used for **preparation** and **analysis** of air samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Boron trichloride	10294-34-5

An analytical procedure is described for the determination of boron trichloride by ICP-AES. A modified sampling and gas introduction system allows on-line monitoring of the investigated gas. The air sample is introduced into an aqueous mannitol solution, and the boron trichloride hydrolyzed to boric acid. Since this method is designed for measuring boron in trichlorosilane, the detection limit was found to be 0.63 µg of boron/g dichlorosilane.

Source: Eschwey, M., Pulvermacher E., Benninghoff, C., and Telgheder, U., "On-line monitoring of boron in dichlorosilane by means of inductively coupled plasma atomic emission spectrometry," Royal Society of Chemistry. Journal of Analytical Atomic Spectrometry, 2000, 15, pp. 277-279. <http://pubs.rsc.org/ej/JA/2000/a908634j.pdf>

#### 4.2.73 Analytical Letters, 1994, 27 (14), pp. 2703-2718: Screening-Procedure for Sodium Fluoroacetate (Compound 1080) at Sub-Microgram/Gram Concentrations in Soils

This method should be used for **preparation** and **analysis** of solid and oily solid samples for the contaminant identified below and listed in Appendix A.

Contaminant	CAS RN
Fluoroacetate salts	NA

Sodium fluoroacetate is readily quantitated at sub-microgram per gram concentrations in small (ca. 1-g) soil samples. Samples are ultrasonically extracted with water, which is then partitioned with hexane, and acidified prior to re-extraction with ethyl acetate. The latter is taken to dryness in the presence of triethanolamine "keeper," and the resulting acid is derivatized with pentafluorobenzyl bromide. Quantitation is performed using a gas chromatograph equipped with an electron-capture detector. A standardized statistical protocol is used to verify a screening level of 0.2 µg sodium fluoroacetate/g soil. Difluoroacetic, trifluoroacetic, and naturally occurring formic acids do not interfere with the determination. The recovery for sodium fluoroacetate was 40% from soil fortified to 0.2 µg/g soil.

Source: Tomkins, B.A., "Screening-Procedure for Sodium Fluoroacetate (Compound 1080) at Sub-Microgram/Gram Concentrations in Soils," *Analytical Letters*. 27(14), 2703-2718 (1994).



## Section 5.0: Biological Methods

The purpose of this section is to provide analytical methods for the analysis of environmental samples for biological agents in response to a homeland security event.

Protocols from peer-reviewed journal articles have been identified for analyte-matrix pairs where standardized methods are not available. It should be noted that the limitations of these protocols are not the same as the limitations of the standardized methods that have been identified. Future steps include the development of standardized methods based on journal protocols. The literature references will be replaced with standardized, verified protocols as they become available.

Although culture-based methods have been selected for the bacterial pathogens, PCR techniques will be used for viruses because of the difficulty and time required to propagate these agents in host cell cultures. It should be noted that PCR techniques have inherent limitations with regard to the determination of viability or infectivity of the analyte.

Sample collection and handling protocols are not available for all analyte-matrix combinations included in this document. Future research will include the development and validation of sampling protocols and sample preparation procedures to support the specified analytical methods.

A list of analytical methods to be used in analyzing environmental samples for biological contaminants during homeland security events is provided in Appendix B. Methods are listed for each analyte and for each sample matrix that potentially may need to be measured and analyzed when responding to an environmental emergency. The methods tables in Appendix B-1, B-2, and B-3 are sorted alphabetically by analyte under each agent category (i.e., bacteria, biotoxins, viruses, and protozoa). Appendix B-1 lists methods to be used for waterborne analytes (both wastewater and drinking water). Appendix B-2 lists methods to be used for dustborne analytes and Appendix B-3 lists methods to be used for aerosolized analytes. Each appendix includes the following information:

- **Analyte(s).** The contaminant or contaminant(s) of interest.
- **Determinative technique.** An analytical instrument or technique used to determine the identity, quantity, and/or viability of a biological agent.
- **Determinative method identifier.** The unique identifier or number assigned to an analytical method by the method publisher.
- **Sample preparation procedure and/or sampling method.** The recommended sample preparation procedure and/or sample collection procedure for the analyte-matrix combination.
- **Identification Procedure.** A procedure used to establish the identity of a specific biological agent or a group of similar biological agents.
- **Viability Procedure.** For the purposes of this document, a procedure used to directly or indirectly evaluate the growth potential and/or replication of an organism under permissive conditions. For example, the proliferation of bacteria in culture is a direct indication of viability whereas the ability of some organisms (viruses and protozoa particularly) to infect, replicate, and cause a detectable alteration in a suitable host system (cell culture, animal or human) is an indirect measurement of viability.

## 5.1 General Guidance

The guidance summarized in this section provides a general overview of how to identify the appropriate biological method(s) for a given analyte-matrix combination as well as recommendations for quality control procedures.

For additional information on the properties of the biological agents 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. Additional information also can be found on CDC's Emergency Preparedness and Response Web site (<http://www.bt.cdc.gov/>). Further research on biological agents is ongoing within EPA and databases to manage this information are currently under development.

### 5.1.1 Standard Operating Procedures for Identifying Biological Methods

To determine the appropriate method and sample preparation procedure and/or sampling method that is to be used on the environmental samples, determine the matrix of interest and locate the appropriate biological appendix (B-1, B-2, or B-3).

After locating the correct appendix, find the analyte of interest and continue across the table to identify the appropriate identification determinative technique, viability determinative technique, determinative method, and sample preparation procedure and/or sampling method for the analyte of interest.

Sections 5.2.1 through 5.2.19 below provide summaries of the analytical methods listed in Appendix B. Where available, a direct link to the full text of the selected 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 2.

**Table 2. Sources of Biological Methods**

Name	Publisher	Reference
National Environmental Methods Index (NEMI)	EPA, USGS	<a href="http://www.nemi.gov">http://www.nemi.gov</a>
U.S. EPA Microbiology Methods	EPA	<a href="http://www.epa.gov/microbes/">http://www.epa.gov/microbes/</a>
USDA/FSIS Microbiology Laboratory Guidebook	USDA Food Safety and Inspection Service	<a href="http://www.fsis.usda.gov/OPHS/microlab/mlgbook.htm">http://www.fsis.usda.gov/OPHS/microlab/mlgbook.htm</a>
ICR Microbial Laboratory Manual	EPA Office of Research and Development	<a href="http://www.epa.gov/nerlcwww/icrmicro.pdf">http://www.epa.gov/nerlcwww/icrmicro.pdf</a>
Occupational Safety and Health Administration Methods	OSHA	<a href="http://www.osha-slc.gov/dts/sltc/methods/toc.html">http://www.osha-slc.gov/dts/sltc/methods/toc.html</a>
National Institutes for Occupational Safety and Health Methods	NIOSH	<a href="http://www.cdc.gov/niosh/nmam/">http://www.cdc.gov/niosh/nmam/</a>



Name	Publisher	Reference
Standard Methods for the Examination of Water and Wastewater, 20 <sup>th</sup> Edition, 1998*	American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF)	<a href="http://www.standardmethods.org">http://www.standardmethods.org</a>
Annual Book of ASTM Standards*	ASTM International	<a href="http://www.astm.org">http://www.astm.org</a>
Applied and Environmental Microbiology*	American Society for Microbiology	<a href="http://www.asm.org">http://www.asm.org</a>
Journal of Clinical Microbiology*	American Society for Microbiology	<a href="http://www.asm.org">http://www.asm.org</a>

\* Subscription and/or purchase required.

### 5.1.2 General Quality Control (QC) Guidance for Biological Methods

As with analysis of chemical analytes, the level or amount of quality control (QC) needed during sample analysis and reporting to address biological analytes depends on the intended purpose of the data. The specific decisions that will be made should be identified, and quality goals (including QC requirements) for data generation should be derived based on those decisions. Analytical data of known and documented quality is a critical factor in the accurate assessment of and appropriate response to emergency situations. The generation of data of sufficient quality requires that analytical laboratories: (1) conduct the appropriate QC procedures to ensure that all measurement systems are in control and operating properly, (2) properly document all analytical results, and (3) properly document analytical QC procedures and corrective actions.

The specific level and amount of QC procedures required during sample analysis and data reporting depends on the intended purpose of the data. Individual methods, sampling and analytical protocols, or contractual statements of work should be consulted to determine if additional QC procedures are required. Method-specific QC requirements are described in many of the individual methods cited in this manual and will be included in protocols developed to address specific analyte-matrix combinations of concern. In general, analytical QC requirements for biological methods include an initial demonstration of measurement system capability as well as ongoing analysis of control samples to ensure the continued reliability of the analytical results. At a minimum, the following QC analyses should be conducted on an ongoing basis for biological analytes:

- Media and reagent sterility checks
- Positive and negative controls
- Method blanks
- Matrix spikes to evaluate method performance in the matrix of interest
- Matrix spike duplicates (MSD) and/or sample replicates to assess method precision

QC procedures should be performed as frequently as necessary to ensure the reliability of analytical results. As with the identification of needed QC samples, frequency should be established based on an evaluation of data quality objectives.

**Please note:** The appropriate point of contact identified in Section 3 should be consulted regarding appropriate quality assurance and quality control (QA/QC) procedures prior to sample analysis. These contacts will consult with their respective QA/QC managers regarding QA/QC issues.

### 5.1.3 Safety and Waste Management

It is imperative that safety precautions are used during collection, processing, and analysis of environmental samples, particularly in emergency response situations that may include unknown hazards. Many of the methods summarized or cited in Section 5.2 contain specific requirements, guidance, 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. Additional resources that can be consulted for additional information include the following:

- Environmental Protection Agency's standards regulating hazardous waste (40 CFR parts 260 - 270)
- Biosafety in Microbiological and Biomedical Laboratories, 4<sup>th</sup> Edition, found at [www.cdc.gov/od/ohs/biosfty/bmb14toc.htm](http://www.cdc.gov/od/ohs/biosfty/bmb14toc.htm)
- Laboratory Security and Emergency Response Guidance for Laboratories Working with Select Agents, December 6, 2002 / 51 (RR19); 1-8, found at [www.cdc.gov/mmwr/preview/mmwrhtml/rr5119a1.htm](http://www.cdc.gov/mmwr/preview/mmwrhtml/rr5119a1.htm).

## 5.2 Method Summaries

Method summaries for the analytical methods listed in Appendix B are provided in Sections 5.2.1 through 5.2.19. Each method summary contains a table identifying the contaminants in Appendix B to which the method applies, provides a brief description of the analytical method, and includes a link (if available) to the full version of the method or source for obtaining a full version of the method.

**Please note:** Not all methods have been verified for the analyte/matrix combination listed in Appendix B. Please refer to the specified method to identify analyte/matrix combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 3.

### 5.2.1 Laboratory Response Network (LRN)

The agents identified below and listed in Appendix B should be analyzed in accordance with the appropriate LRN protocols.

Analyte(s)	Agent Category
<i>Bacillus anthracis</i> (Anthrax)	Bacteria
<i>Brucella</i> spp. (Brucellosis)	Bacteria
<i>Burkholderia mallei</i> (Glanders)	Bacteria
<i>Burkholderia pseudomallei</i> (Meliodosis)	Bacteria
<i>Coxiella burnetii</i> (Q-fever)	Bacteria
<i>Francisella tularensis</i> (Tularemia)	Bacteria
<i>Rickettsia prowazekii</i> (Epidemic Typhus)	Bacteria
<i>Yersinia pestis</i> (Plague)	Bacteria

These agents will be analyzed using restricted procedures available only through the Laboratory Response Network (LRN). These procedures are not available to the general laboratory community and thus are not discussed within this document. For additional information on the LRN, please use the contact information provided below or visit <http://www.bt.cdc.gov/lrn/>.

#### Centers for Disease Control and Prevention

Laboratory Response Branch  
Bioterrorism Preparedness and Response Program  
National Center for Infectious Diseases  
1600 Clifton Road NE, Mailstop C-18  
Atlanta, GA 30333  
Telephone: (404) 639-2790  
E-mail: [lrn@cdc.gov](mailto:lrn@cdc.gov)

Local public health laboratories, private laboratories, and commercial laboratories with questions about the LRN should contact their State public health laboratory director or the Association of Public Health Laboratories (contact information provided below).

#### Association of Public Health Laboratories

2025 M Street NW, Suite 550  
Washington, DC 20036  
Telephone: (202) 822-5227  
Fax: (202) 887-5098  
Web site: [www.aphl.org](http://www.aphl.org)  
E-mail: [info@aphl.org](mailto:info@aphl.org)

### 5.2.2 Biosafety Level 4 Viruses

Samples to be analyzed for the viruses identified below and listed in Appendix B should be analyzed under biosafety level (BSL) 4 conditions at the Centers for Disease Control and Prevention (CDC).

Analyte(s)	Agent Category
Arenaviruses (Hemorrhagic fever)	Viruses
Bunyaviruses (Hemorrhagic fever)	Viruses
Filoviruses (Hemorrhagic fever)	Viruses
Flaviviruses (Hemorrhagic fever)	Viruses
Orthopoxvirus: Monkey pox	Viruses
Orthopoxvirus: Variola major (Smallpox)	Viruses

For additional information on the BSL 4 laboratories, please use the contact information provided below or visit <http://www.bt.cdc.gov/lrn/>.

#### Centers for Disease Control and Prevention

Laboratory Response Branch  
Bioterrorism Preparedness and Response Program  
National Center for Infectious Diseases  
1600 Clifton Road NE, Mailstop C-18  
Atlanta, GA 30333  
Telephone: (404) 639-2790  
E-mail: [lrn@cdc.gov](mailto:lrn@cdc.gov)

### 5.2.3 Standard Methods 9260 B: *Salmonella typhi*

This method should be used for the **identification** and **viability** assessment of *Salmonella typhi* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Salmonella typhi</i> (Typhoid fever)	Bacteria

Concentrated samples are enriched in either selenite cystine, selenite-F, or tetrathionate broths and incubated at 35°C - 37°C for up to five days. An aliquot from each turbid tube is streaked onto bismuth sulfite (BS) plates and incubated at 35°C - 37°C for 24 - 48 hours. Presumptive positive colonies are then subjected to biochemical characterization and serological confirmation using polyvalent O and Vi antiserum.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. This method was originally developed for water matrices; further research will be required to develop and standardize sample processing protocols for other matrices.

**Source:** American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

### 5.2.4 Standard Methods 9260 E: *Shigella* species

This method should be used for the **identification** and **viability** assessment of *Shigella* species in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Shigella</i> species (Shigellosis)	Bacteria

This method contains two options for sample concentration: membrane filtration (liquid matrices) and centrifugation (liquid and solid matrices) for analyses. Both options include inoculation of an enrichment medium (Selenite F broth). Isolation of the target analyte is achieved by plating onto XLD and/or MacConkey agar. Biochemical identification consists of inoculating TSI and LIA slants. Serological grouping is done by slide agglutination tests using polyvalent antisera. Serotyping for *S. dysenteriae*, *S. flexneri*, and *S. boydii* may be performed at State Health laboratories or CDC.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. This method was originally developed for water matrices; further research will be required to develop and standardize sample processing protocols for other matrices.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

### 5.2.5 Standard Methods 9260 F: Pathogenic *Escherichia coli*

This method should be used for the **identification** and **viability** assessment of *Escherichia coli* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Escherichia coli</i> ( <i>E. coli</i> ) O157:H7	Bacteria

This method allows for two options, one being a modification of SM 9221 B followed by plating and biochemical identification. The second option, modification of a food method, allows for the analysis of large sample volumes. A 200-mL water sample is centrifuged, resuspended in *E. coli* enrichment broth (EEB) and incubated for 6 hours. Tellurite Cefixime SMAC (TC SMAC) plates are inoculated with the enriched EEB culture. The TC SMAC plates are incubated for up to 24 hours. Colorless colonies on TC SMAC are tested for indole production. Additional biochemical tests and serotyping are also done to confirm identification.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. This method was originally developed for liquid matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

### 5.2.6 Standard Methods 9260 G: *Campylobacter jejuni*

This method should be used for the **identification** and **viability** assessment of *Campylobacter jejuni* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Campylobacter jejuni</i>	Bacteria

Water samples (1 to several liter volumes) are filtered using a cellulose nitrate membrane filter. Filters are placed face down on either Skirrow's medium or Campy-BAP and incubated for 24 hours at 42°C. Filters are then transferred to another selective medium face-down and incubated for a total of 5 days at 42°C under microaerophilic conditions. Identification is made by culture examination, microscopy, motility test, and biochemical testing. Biochemical tests include oxidase, catalase, nitrite and nitrate reduction, H<sub>2</sub>S production, and hippurate hydrolysis. Serotyping is done using commercially available rapid test kits. Skirrow's and other selective media containing antibiotics may prevent the growth of injured organisms.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

### 5.2.7 Standard Methods 9260 H: *Vibrio cholerae*

This method should be used for the **identification** and **viability** assessment of *Vibrio cholerae* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Vibrio cholerae</i> (Cholera)	Bacteria

Samples are enriched in alkaline peptone broth and incubated for up to 8 hours. Thiosulfate-citrate-bile salts-sucrose TCBS agar plates are inoculated with the incubated broth and incubated for 24 hours. *Vibrio cholerae* isolates are plated on tryptic soy agar with 0.5% NaCl. Biochemical confirmation is done using multiple tests including but not limited to ONPG, Indole, and Voges-Proskauer. Slide agglutination assays are used for serological identification.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

Source: American Public Health Association, American Water Works Association, and Water Environment Federation. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20<sup>th</sup> Edition. (<http://www.standardmethods.org/>)

### 5.2.8 Literature Reference for Enteric Viruses (Applied and Environmental Microbiology. 69(6): 3158-3164)

This method should be used for the **identification** of Enteroviruses, Hepatitis A virus, and Rotavirus (Group A) in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Picornaviruses: Enteroviruses	Viruses
Picornaviruses: Hepatitis A virus (HAV)	Viruses
Reoviruses: Rotavirus (Group A)	Viruses

This method is used to detect human enteric viruses (enteroviruses, HAV, rotavirus) in groundwater samples. It is a multiplex reverse-transcription PCR (RT-PCR) procedure optimized for the simultaneous detection of enteroviruses, hepatitis A virus (HAV), reoviruses, and rotaviruses. Water samples are collected by filtration (1 MDS filter) and viruses are eluted using a beef extract solution (1.5%, pH 9.5). Viruses are concentrated using celite adsorption (pH 4.0), filtration, and celite-elution with sodium phosphate (0.15 M, pH 9.0), followed by further concentration and processing to remove inhibitors (ultracentrifugation, solvent extraction, and MW-exclusion filtration). Concentrated samples are analyzed by a two-step multiplex RT-PCR (RT followed by PCR) using virus-specific primer sets. Detection of amplicons is by gel electrophoresis with subsequent confirmation by hybridization (dot-blot) using digoxigenin-labeled internal (nested) probes.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

*Source:* Fout, G. S., Martinson, B. C., Moyer, M. W. N., and Dahling, D. R. 2003. "A Multiplex Reverse Transcription-PCR Method for Detection of Human Enteric Viruses in Groundwater." *Applied and Environmental Microbiology*. 69(6): 3158-3164. (<http://aem.asm.org/cgi/reprint/69/6/3158.pdf>)

### 5.2.9 Literature Reference for Noroviruses (Applied and Environmental Microbiology. 69(9): 5263-5268)

This method should be used for the **identification** of noroviruses in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Caliciviruses: Noroviruses	Viruses

This method is for the detection of human noroviruses (Genogroups I and II) in groundwater samples. Water samples are collected by filtration using a 1 MDS filter and subsequently eluted using a beef extract solution (1.5%, pH 9.0). Viruses are further concentrated using celite adsorption (pH 4.0), filtration, and celite-elution with sodium phosphate (0.15 M, pH 9.0). The concentrated material is processed to remove PCR inhibitors (density sedimentation, solvent extraction, and MW-exclusion filtration). RNA is extracted from concentrated samples and subjected to reverse-transcription (RT) using one or both genogroup-specific cDNA-sense primer sets and the resulting cDNA is then amplified using one or both genogroup-specific RNA-sense primer sets. The end-point RT-PCR products are analyzed by agarose gel electrophoresis and SYBR Green I staining. Amplicons can also be verified by cloning and sequencing.



**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated: positive control, negative control, and blank reactions. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for ground water matrices; further research should be conducted to develop and standardize sample processing protocols for other matrices.

*Source:* Parshionaker, S. U., Willian-True, S., Fout, G. S., Robbins, D. E., Seys, S. A., Cassady, J. D., and Harris, R. 2003. "Waterborne Outbreak of Gastroenteritis Associated with a Norovirus." *Applied and Environmental Microbiology*. 69(9): 5263-5268.

#### 5.2.10 Literature Reference for Hepatitis E virus (Journal of Virological Methods. 101: 175-188)

This method should be used for the **identification** of Hepatitis E virus in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Hepatitis E virus (HEV)	Viruses

This molecular detection method is used for the detection of several hepatitis E virus (HEV) classes (Asian/African, Mexico, and US clusters) in environmental water samples using a reverse transcription-polymerase chain reaction (RT-PCR) approach. Water samples are collected by filtration using a 1 MDS filter; and viruses are eluted using a beef extract solution (1.5%, pH 9.5). Viruses are concentrated using celite adsorption (pH 4.0), filtration, and celite-elution with sodium phosphate (0.15 M, pH 9.0), followed by further concentration and processing to remove inhibitors (ultracentrifugation, solvent extraction, and MW-exclusion filtration). Concentrated samples are analyzed by target-specific, two-step RT-PCR (RT followed by PCR) assays using single- and multiplexed virus-specific primer sets. End point detection of amplicons is by gel electrophoresis and subsequent confirmation by hybridization analysis (Southern or dot-blot) using digoxigenin-labeled internal (nested) probes.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

*Source:* Grimm, A. C. and G. S. Fout, 2002. "Development of a Molecular Method to Identify Hepatitis E Virus in Water." *Journal of Virological Methods*, Vol. 101: 175-188.



### 5.2.11 Literature Reference for Astroviruses (Canadian Journal of Microbiology. 50: 269-278)

This method should be used for the **identification** of Astroviruses in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Astroviruses	Viruses

This method is used to detect all eight astrovirus serotypes in clinical specimens (stool) and water samples. It is a reverse transcription-polymerase chain reaction (RT-PCR) procedure optimized for use in a real-time PCR assay and can be integrated with sample-cell culture (CaCo-2 cells) to enhance sensitivity (ICC/RT-PCR). Water samples are collected by filtration (1 MDS filter), and viruses are eluted using a beef extract solution (1.5%, pH 9.5). Viruses are concentrated using celite adsorption (pH 4.0), filtration, and celite-elution with sodium phosphate (0.15 M, pH 9.0), followed by further concentration and processing to remove inhibitors (ultracentrifugation, solvent extraction, and MW-exclusion filtration). Concentrated samples are analyzed directly or indirectly (following cell culture) by a two-step RT-PCR (RT followed by PCR) assay using astrovirus-specific primer sets. Detection of amplicons is by gel electrophoresis with subsequent confirmation by hybridization (dot-blot) using digoxigenin-labeled internal (nested) probes or by real-time detection using fluorogenic probes.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

*Source:* Grimm, A. C., Cashdollar, J. L., Williams, F. P., and Fout, G. S. 2004. "Development of an Astrovirus RT-PCR Detection Assay for use with Conventional, Real-Time, and Integrated Cell Culture/RT-PCR." *Canadian Journal of Microbiology*. 50(4): 269-268.

### 5.2.12 Literature Reference for Togaviruses (Journal of Clinical Microbiology. 38(4): 1527-1535)

This method should be used for the **identification** of Venezuelan Equine Encephalitis Virus in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Togaviruses: Venezuelan Equine Encephalitis Virus (VEEV)	Viruses

The VEEV-specific RT-PCR assay is applied to human sera viruses that are isolated in either Vero cells, C6/36 cells, or newborn mice. VEEV is identified using an indirect immunofluorescence assay. QIAmp viral RNA kit (Qiagen) is used to extract RNA without the use of TaqExtender and amplification is done using gene-specific RT-PCR–seminested PCR. Annealing temperature is 55°C in the thermocycler.

**Please note:** This procedure has not been fully verified for matrices other than human sera. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for*

Laboratories Performing PCR Analyses on Environmental Samples document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for clinical matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

Source: Linssen, B., Kinney, R. M., Aguilar, P., Russell, K. L., Watts, D. M., Kaaden, O., and Pfeffer M. 2000. "Development of Reverse Transcription-PCR Assays Specific for Detection of Equine Encephalitis Viruses." *Journal of Clinical Microbiology*. 38(4):1527-1535. (<http://jcm.asm.org/cgi/reprint/38/4/1527.pdf>)

### 5.2.13 Literature Reference for Adenoviruses (Applied and Environmental Microbiology. 71(6): 3131-3136)

This method should be used for the **identification** of Adenoviruses: enteric and non-enteric (A-F) in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Adenoviruses: enteric and non-enteric (A-F)	Viruses

This method uses a broadly reactive fluorogenic 5' nuclease (TaqMan) quantitative real-time polymerase chain reaction (PCR) assay for the detection of all six species (A-F) of human adenoviruses (HAdV). Sensitive detection and discrimination of adenovirus F species (AdV40 and AdV41) can be achieved by using a real-time fluorescence resonance energy transfer (FRET)-based PCR assay.

**Please note:** This procedure has not been fully verified for matrices other than human sera. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for clinical matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

Source: Jothikumar, N., Cromeans, T. L., Hill, V. R., Lu, X., Sobsey, M., and Erdman, D. D. 2005. "Quantitative Real-Time PCR Assays for Detection of Human Adenoviruses and Identification of Serotypes 40 and 41". *Applied and Environmental Microbiology*. 71 (6): 3131-3136.

### 5.2.14 Literature Reference for Coronaviruses (SARS) (Journal of Virological Methods. 122: 29-36)

This method should be used for the **identification** of SARS-associated human coronavirus in water, dust, and aerosol samples.

Analyte(s)	Agent Category
Coronaviruses: SARS-associated human coronavirus	Viruses

This method uses a conventional single-tube reverse transcription-polymerase chain reaction (RT-PCR) procedure based on consensus primer sequences targeting conserved regions of coronavirus genome sequences. End-point amplicon analysis is by electrophoresis and subsequent visualization. The assay can detect the severe acute respiratory syndrome (SARS) - associated human coronavirus (SARS-HCoV) as well as several other human respiratory coronaviruses (HCoV-OC43 and HCoV-229E). Species identification is provided by amplicon sequencing or by rapid restriction enzyme analysis.

**Please note:** This procedure has not been fully verified for matrices other than human sera. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) or call the point of contact identified in Section 3. This method was originally developed for clinical matrices, further research will be conducted to develop and standardize sample processing protocols for other matrices.

**Source:** Adachi, D., Johnson, G., Draker, R., Ayers, M., Mazzulli, T., Talbot, P. J., and Tellier, R. 2004. “Comprehensive detection and identification of human coronaviruses, including the SARS-associated coronavirus, with a single RT-PCR assay.” *Journal of Virological Methods*. 122: 29-36.

#### 5.2.15 EPA Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA

This method should be used for the **identification** of *Cryptosporidium* species in drinking water (source and finished), dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Cryptosporidium</i> species (Cryptosporidiosis)	Protozoa

A water sample is filtered and the oocysts and extraneous materials are retained on the filter. Materials on the filter are eluted, the eluate is centrifuged to pellet the oocysts, and the supernatant fluid is aspirated. The oocysts are magnetized by attachment of magnetic beads conjugated to anti-*Cryptosporidium* antibodies. The magnetized oocysts are separated from the extraneous materials using a magnet, and the extraneous materials are discarded. The magnetic bead complex is then detached from the oocysts. The oocysts are stained on well slides with fluorescently labeled monoclonal antibodies and 4',6-diamidino-2-phenylindole (DAPI). The stained sample is examined using fluorescence and differential interference contrast (DIC) microscopy. Qualitative analysis is performed by scanning each slide well for objects that meet the size, shape, and fluorescence characteristics of *Cryptosporidium* oocysts. Quantitative analysis is performed by counting the total number of objects on the slide confirmed as oocysts. This method is not intended to determine viability of the oocysts.

**Please note:** This method was originally developed for water matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

**Source:** USEPA. 2003. *Cryptosporidium* in Water by Filtration/IMS/FA (document is currently on Web site as draft for comment). United States Environmental Protection Agency, Washington, D.C. ([http://www.epa.gov/safewater/lt2/pdfs/guide\\_lt2\\_mlmanual\\_appendix-b\\_method-1622-June-2003.pdf](http://www.epa.gov/safewater/lt2/pdfs/guide_lt2_mlmanual_appendix-b_method-1622-June-2003.pdf))

#### 5.2.16 Draft EPA Method 1693: *Cryptosporidium* and *Giardia* in Disinfected Wastewater and Combined Sewer Overflows (CSOs) by Concentration/IMS/IFA

This method should be used for the **identification** of *Cryptosporidium* species in wastewater samples.

Analyte(s)	Agent Category
<i>Cryptosporidium</i> species (Cryptosporidiosis)	Protozoa

A disinfected wastewater or CSO sample is concentrated either by filtration using the Envirochek™ HV capsule or by direct centrifugation. The filtration option of Draft Method 1693 is a modification of the filter elution procedures used in EPA Method 1623 and includes a rinse of the filtered sample with sodium hexametaphosphate and reagent water prior to filter elution. The second concentration option

concentrates highly turbid or "unfilterable" matrices using direct centrifugation. The concentrated oocysts and cysts are recovered by immunomagnetic separation (IMS) using magnetic beads conjugated to anti-*Cryptosporidium* and anti-*Giardia* antibodies. The magnetic bead-sample complexes are separated from the extraneous materials using a magnet, and the extraneous materials are discarded. For samples concentrated by direct centrifugation, this separation step includes the addition of kaolin. Kaolin is included to adsorb fats, oils, organics, and heavy particulates which may be present in a sample deemed to be unfilterable. The magnetic bead-sample complex is then rinsed prior to dissociation and recovery of the oocysts and cysts. The magnetic bead complex is then detached from the oocysts. The oocysts are stained on well slides with fluorescently labeled monoclonal antibodies and 4',6-diamidino-2-phenylindole (DAPI). The stained sample is examined using fluorescence and differential interference contrast (DIC) microscopy. Qualitative analysis is performed by scanning each slide well for objects that meet the size, shape, and fluorescence characteristics of *Cryptosporidium* oocysts. Quantitative analysis is performed by counting the total number of objects on the slide confirmed as oocysts. This method is not intended to determine viability of the oocysts.

**Please note:** This method was originally developed for wastewater matrices; further research will be conducted to develop and standardize sample processing protocols for other matrices.

**Source:** USEPA. 2005. Draft Method 1693: *Cryptosporidium* and *Giardia* in Disinfected Wastewater and Combined Sewer Overflows (CSOs) by Concentration/IMS/IFA. United States Environmental Protection Agency, Washington, D.C.

### 5.2.17 Literature References for *Toxoplasma gondii* (Applied and Environmental Microbiology. 70(7): 4035-4039)

This method should be used for the **identification** and **viability** assessment of *Toxoplasma gondii* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Toxoplasma gondii</i> (Toxoplasmosis)	Protozoa

This method uses a fluorogenic 5' nuclease (TaqMan) real-time polymerase chain reaction (PCR) assay for the detection of *T. gondii* oocyst DNA using gene-specific (B1 gene) primers and probe. Water samples (10 - 100 L) are filtered (Envirochek™) to concentrate oocysts. Filters are eluted and recovered oocysts are further purified and concentrated by differential flotation and centrifugation. Final sample pellets are split and subjected to PCR detection and mouse bioassay.

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* ([www.epa.gov/nerlcwww/qa\\_qc\\_pcr10\\_04.pdf](http://www.epa.gov/nerlcwww/qa_qc_pcr10_04.pdf)) document or call the point of contact identified in Section 3. This method was originally developed for water matrices; further research will be required to develop and standardize sample processing protocols for other matrices.

**Source:** Villena, I., Aubert, D., Gomis, P., Ferte, H., Ingland, J-C., Denise-Bisiaux, H., Dondon, J-M., Pisano, E., Ortis, N., and Pinon, J-M. 2004. "Evaluation of a Strategy for *Toxoplasma gondii* Oocyst Detection in Water". *Applied and Environmental Microbiology*. 70(7): 4035-4039.

### 5.2.18 *Entamoeba histolytica*: PCR

This method should be used for the **identification** of *Entamoeba histolytica* in water, dust, and aerosol samples.

Analyte(s)	Agent Category
<i>Entamoeba histolytica</i>	Protozoa

A standardized method/protocol for the analysis of *Entamoeba histolytica* has not been identified. However, a real-time PCR assay has recently been developed by CDC and is expected to be published during 2006. In the interim, PCR test kits may be obtained for analysis of this organism. Please contact the appropriate point of contact identified in Section 3 if the need to analyze samples for *Entamoeba histolytica* arises.

### 5.2.19 Literature Reference for Shiga Toxin Gene (Journal of Clinical Microbiology. 39(1): 370-374)

This method should be used to **detect** Shiga toxin genes from isolated bacterial colonies derived from water, dust, and aerosol samples.

Analyte(s)	Agent Category
Shiga toxin	Biotoxin

This method is used to detect bacterial genes ( $stx_1$  and  $stx_2$ ) encoding the two major classes of Shiga toxins, Shiga toxin 1 and Shiga toxin 2. Detection of  $stx$  genes ( $stx_1$ ,  $stx_2$ , and  $stx_{2e}$ ) is performed using a multiplexed polymerase chain reaction (PCR) assay and fluorescence-based melting curve analysis of amplicons in a single capillary tube format (LightCycler).

**Please note:** This procedure has not been fully verified. At a minimum, the following sample processing quality control checks should be performed and evaluated before using this protocol: positive control, negative control, and blank. PCR quality control checks should be performed according to *EPA Draft Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples* document or call the point of contact identified in Section 3.

Source: Bellin, T., Pulz, M., Matussek, A., Hempen, H-G., and Gunzer, F. 2001. "Rapid Detection of Enterohemorrhagic *Escherichia coli* by Real-Time PCR with Fluorescent Hybridization Probes." *Journal of Clinical Microbiology*. 39(1): 370-374.



## Section 6.0: 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 C. Methods are listed for each isotope and for each sample matrix that potentially may need to be measured and analyzed when responding to an emergency. The methods table in Appendix C is sorted alphabetically by analyte and includes the following information:

- **Analyte(s).** The compound or compound(s) of interest.
- **Chemical Abstract Survey 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. In this section (Section 6.0) and Appendix C, the CAS RNs correspond to the specific radionuclide identified.
- **Determinative technique.** An analytical instrument or technique used for qualitative and quantitative determination of compounds or components in a sample.
- **Drinking water sample analysis procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in drinking water samples. Methods have been identified for gross determination and confirmation of specific isotopes.
- **Aqueous and liquid phase sample analysis procedure.** The recommended method/procedure 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 gross determination and confirmation of specific isotopes.
- **Soil and sediment phase sample analysis procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in soil and sediment samples. Methods have been identified for gross determination and confirmation of specific isotopes.
- **Surface wipe sample analysis procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in surface wipe samples. Methods have been identified for gross determination and confirmation of specific isotopes.
- **Air filter sample analysis procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in air filter samples. Methods have been identified for gross determination and confirmation of specific isotopes.
- **Gross determination method identifier.** A unique identifier or number assigned to an analytical method by the method publisher. The identified method is for measurement of the specific activity (i.e., gamma, alpha or beta) from all radioisotopes of the targeted radiological element.
- **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 specific activity (i.e., gamma, alpha or beta) from a particular target radioisotope of a radiological element.

### 6.1 General Guidance

The guidance summarized in this section provides a general overview of how to identify the appropriate radiochemical method(s) for a given analyte-matrix combination as well as recommendations for quality control procedures.

For additional information on the properties of the radionuclides listed in Appendix C, 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 Information (<http://www.epa.gov/radiation/radionuclides/index.html>) and the Multi-Agency Radiological



Laboratory Analytical Protocols Manual (<http://www.epa.gov/radiation/marlap/manual.htm>) Web sites provide some additional information pertaining to radionuclides of interest and radiochemical methods.

### 6.1.1 Standard Operating Procedures for Identifying Radiochemical Methods

To determine the appropriate method that is to be used on the environmental samples, locate the analyte of concern in Appendix C: Radiochemical Methods under the “Analyte(s)” column. After locating the analyte of concern, continue across the table to identify the appropriate determinative technique and determinative (gross and/or confirmatory) method applicable to the matrix 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 6.2.20 below provide summaries of the gross and confirmatory determinative methods listed in Appendix C. Where available, a direct link to the full text of the selected analytical method is provided in the method summary. For additional information on sample preparation and analysis procedures and on methods available through consensus standards organizations, please use the contact information provided in Table 3.

**Table 3. Sources of Radiochemical Methods**

Name	Publisher	Reference
National Environmental Methods Index (NEMI)	U.S. Environmental Protection Agency (USEPA), United States Geological Survey (USGS)	<a href="http://www.nemi.gov">http://www.nemi.gov</a>
Prescribed Procedures for Measurement of Radioactivity in Drinking Water (EPA-600 4-80-032 August 1980)	U.S. Environmental Protection Agency (USEPA), Office of Research and Development (ORD), Environmental Monitoring and Support Laboratory (EMSL)	Available from National Technical Information Service (NTIS). NTIS, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161 (703) 605-6000.
Annual Book of ASTM Standards, Vol. 11.02*	American Society for Testing and Materials (ASTM) International	<a href="http://www.astm.org">http://www.astm.org</a>
EML Procedures Manual, HASL-300, 28 <sup>th</sup> Edition, February, 1997	U.S. Department of Energy (DOE), Environmental Measurements Laboratory (EML) / Now, U.S. Department of Homeland Security (DHS),	<a href="http://www.eml.doe.gov/publications/pr ocman/">http://www.eml.doe.gov/publications/pr ocman/</a>  Also available from National Technical Information Service (NTIS). 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 1978. EMSL-LV-0539-17	United States Environmental Protection Agency (U.S. EPA), Environmental Monitoring and Support Laboratory (EMSL)	Available from National Technical Information Service (NTIS). NTIS, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. (703) 605-6000



Name	Publisher	Reference
Standard Methods for the Examination of Water and Wastewater, 20 <sup>th</sup> Edition, 1998*	American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF)	<a href="http://www.standardmethods.org">http://www.standardmethods.org</a>

\* Subscription and/or purchase required.

### 6.1.2 General Quality Control (QC) Guidance for Radiochemical Methods

Having data of known and documented quality is critical in order for public officials to accurately assess the activities that may be needed in responding to emergency situations. 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 screening determinations versus quantitative analytical analyses. The specific needs for data generation should be identified. Quality control requirements and data quality objectives should be derived based on those needs. 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 risk assessment and site release, such as those identified in this document, might require increased QC requirements (demonstrations of method sensitivity, precision, and accuracy).

Some method-specific QC requirements are described in many of the individual methods that are cited in this manual, and will be referenced in any standardized analytical protocols developed to address specific analytes and matrices of concern. 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 samples are required to assess the precision, accuracy, and independence of sample results. All QC results are tracked on control charts for prescribed parameters of their results and reviewed for acceptability and trends in analysis or instrument operation. These quality assurance (QA) parameters are measured as required per method at the prescribed frequency. QA of laboratory analyses using radiochemical methods includes an initial demonstration of proficiency or capability as well as ongoing analysis of standards and other samples to ensure continued reliability of the analytical results.

- Method blank
- Lab fortified blank recovery for samples that are chemically prepared
- Calibration check
- Sample and sample duplicate
- Laboratory control sample recovery for samples that are not chemically prepared, OR
- Matrix spike and matrix spike duplicate for samples that are chemically prepared
- Tracer recovery

**Please note:** The appropriate point of contact identified in Section 3 should be consulted regarding appropriate quality assurance and quality control (QA/QC) procedures prior to sample analysis. These contacts will consult with their respective QA/QC managers regarding QA/QC issues.

### 6.1.3 Safety and Waste Management

It is imperative that safety precautions are used during collection, processing, and analysis of environmental samples, particularly in emergency response situations that may include unknown hazards. Many of the methods summarized or cited in Section 6.2 contain specific requirements, guidance, 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:

- Occupational Health and Safety Administration's standard for Occupational Exposure to Hazardous Chemicals in Laboratories (29 CFR 1910.1450)
- Environmental Protection Agency's standards regulating hazardous waste (40 CFR parts 260 - 270)
- Standards for protection against radiation (10 CFR part 20)
- U.S. Department of Energy (DOE). Order O 435.1: Radioactive Waste Management. July 1, 1999. Available at: [www.directives.doe.gov/pdfs/doe/doetext/neword/435/o4351.html](http://www.directives.doe.gov/pdfs/doe/doetext/neword/435/o4351.html)
- U.S. Department of Energy (DOE). M 435.1-1. *Radioactive Waste Management Manual*. Office of Environmental Management. July 9, 1999. Available at: <http://www.directives.doe.gov/pdfs/doe/doetext/neword/435/m4351-1.html>
- U.S. Department of Energy (DOE). *Compendium of EPA-Approved Analytical Methods for Measuring Radionuclides in Drinking Water*. Prepared by the Office of Environmental Policy and Assistance Air, Water and Radiation Division (EH-412). June 1998
- U.S. Environmental Protection Agency (EPA). 1996. *Profile and Management Options for EPA Laboratory Generated Mixed Waste*. Office of Radiation and Indoor Air, Washington, DC
- EPA 402-R-96-015. August. Available at: [http://www.epa.gov/radiation/mixed-waste/mw\\_pg7.htm#lab\\_mix](http://www.epa.gov/radiation/mixed-waste/mw_pg7.htm#lab_mix)
- U.S. Environmental Protection Agency (EPA). 2001. Changes to 40 CFR 266 (Storage, Treatment, Transportation, and Disposal of Mixed Waste), *Federal Register* 66:27217-27266, May 16
- U.S. Environmental Protection Agency (EPA). 2002. *RCRA Orientation Manual*. Office of Solid Waste, Washington, DC. EPA530-R-02-016. 259 pp. Available at: <http://www.epa.gov/epaoswer/general/orientat/>
- Waste Management in a Radioanalytical Laboratory 17-18 Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual, July 2004
- National Research Council. 1995. *Prudent Practices in the Laboratory; Handling and Disposal of Chemicals*, National Academy Press, Washington, DC
- National Council on Radiation Protection and Measurements (NCRP). 2002. *Risk-Based Classification of Radioactive and Hazardous Chemical Wastes*, 7910 Woodmont Avenue, Suite 400, Bethesda, MD 20814-3095
- U.S. Nuclear Regulatory Commission/U.S. Environmental Protection Agency (NRC/EPA). 1995. Low-Level Mixed Waste Storage Guidance, *Federal Register* 60:40204-40211, August 7

## 6.2 Method Summaries

Summaries for the analytical methods listed in Appendix C are provided in Sections 6.2.1 through 6.2.20. These summaries contain information that has been extracted from the selected methods. Each method summary contains a table identifying the contaminants in Appendix C 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.

**Please note:** Not all methods have been verified for the analyte/matrix combination listed in Appendix C. Please refer to the specified method to identify analyte/matrix combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 3.

### 6.2.1 EPA Method 901.1: Gamma Emitting Radionuclides in Drinking Water

This method should be used for **gross determination** and **confirmatory analysis** of drinking water samples for the contaminants identified below and listed in Appendix C.

Contaminant	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

\* The identified method will measure decay products of these isotopes

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, using a Ge(Li) detector (preferred) or a NaI(Tl) detector. 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. Significant interference can occur using the NaI(Tl) detector when counting a sample containing radionuclides that emit gamma photons of nearly identical energies. Detection limits for this method are dependent on sample volume, geometry (physical shape), and counting time.

**Source:** "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/4/80/032, August 1980, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

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

This method should be used for **gross determination analysis** of drinking water samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Radium-226	13982-63-3

This method covers measurement of the total soluble alpha emitting radioisotopes 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 co-precipitation 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 alpha activity in radium-226 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 levels for this method is 0.5 pCi/L.

*Source:* “Prescribed Procedures for Measurement of Radioactivity in Drinking Water,” National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/4/80/032, August 1980, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

### 6.2.3 EPA Method 903.1: Radium-226 in Drinking Water - Radon Emanation Technique

This method should be used for **confirmatory analysis** of drinking water samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Radium-226	13982-63-3

This method is specific for radium-226, and is based on the emanation and scintillation counting of radon-222, a daughter product of radium-226. Radium-226 is concentrated and separated from the water sample by co-precipitation 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 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:* “Prescribed Procedures for Measurement of Radioactivity in Drinking Water,” National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/4/80/032, August 1980, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847. Also at [http://web1.er.usgs.gov/nemi/method\\_summary.jsp?param\\_method\\_id=4732](http://web1.er.usgs.gov/nemi/method_summary.jsp?param_method_id=4732)

#### 6.2.4 EPA Method 905.0: Radioactive Strontium in Drinking Water

This method should be used for **gross determination** and **confirmatory analysis** of drinking water samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Strontium-90	10098-97-2

This method measures total strontium and soluble strontium-89 and strontium-90 in drinking water samples. Some naturally insoluble (and probably suspended) forms of strontium-89 and strontium-90 would also be measured by this method when samples are acid preserved before analysis. Stable strontium carrier is added to the sample and strontium-89 and strontium-90 are precipitated from solution as insoluble carbonates. The yttrium-90 daughter of strontium-90 is removed by a hydroxide precipitation step and the separated combined strontium-89 and strontium-90 are counted for beta particle activity. The counting result, immediately ascertained, represents total strontium activity (strontium-90 + strontium-89). To determine the amount of strontium-90, the yttrium-90 daughter is allowed to grow for two weeks as the strontium-90 decays. Yttrium-90 is then separated with stable yttrium carrier as hydroxide and finally precipitated as oxalate and beta counted. The strontium-90 concentration is equal to the yttrium-90 activity. Strontium-89 activity is determined by subtracting the yttrium-90 activity from the strontium-89 and strontium-90 total activity. Interferences from calcium and some radionuclides are removed by one or more precipitations of the strontium carrier as strontium nitrate. Barium and radium are removed as chromate. Based on a 1000-mL sample and 100-minute counting time, the minimum detectable level for this method is 0.5 pCi/L.

*Source:* “Prescribed Procedures for Measurement of Radioactivity in Drinking Water,” National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/4/80/032, August 1980, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

#### 6.2.5 EPA Method 908.0: Uranium in Drinking Water - Radiochemical Method

This method should be used for **gross determination analysis** of drinking water samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Uranium-238	7440-61-1

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 co-precipitated 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.

*Source:* “Prescribed Procedures for Measurement of Radioactivity in Drinking Water,” National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/4/80/032, August 1980, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

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

This method should be used for **confirmatory analysis** of soil/sediment, surface wipe, and air filter samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Radium-226 *	13982-63-3

\* The identified method will measure decay products of this isotope

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 solution and transferred to an emanation tube. The radon is allowed to come to equilibrium for approximately 30 days. Radium-226 decays by alpha 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:* "Radiochemical Analytical Procedures for Analysis of Environmental Samples," United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory (EMSL), March 1979, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

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

This method should be used for **confirmatory analysis** of drinking water, aqueous/liquid, soil/sediment, surface wipe, and/or air filter samples for the contaminants identified below and listed in Appendix C.

Contaminant	CAS RN
Plutonium-238	13981-16-3
Uranium-238	7440-61-1

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 co-precipitation 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 picocuries per sample. To avoid possible cross-contamination, sample activities should be limited to 25 picocuries or less.

*Source:* "Radiochemical Analytical Procedures for Analysis of Environmental Samples," United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory (EMSL), March 1979, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Phone: 800-553-6847.

### 6.2.8 ASTM Method D3084: Standard Practice for Alpha Spectrometry in Water

This method should be used for **gross determination analysis** of drinking water and aqueous/liquid samples for the contaminants identified below and listed in Appendix C.

Contaminant	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4
Plutonium-238	13981-16-3

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 followed 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 and D3972) 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 disk). Electrodeposition and microprecipitation are preferred. 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.

*Source:* "Annual Book of ASTM Standards, Vol. 11.02," American Society for Testing and Materials (ASTM), 1996, ASTM International, 100 Barr Harbor Drive West, Conshohocken, PA 19428. Phone: 610-832-9500. Web: <http://www.astm.org>. Use Method number when ordering.

### 6.2.9 ASTM Method D3972: Standard Test Method for Isotopic Uranium in Water by Radiochemistry

This method should be used for **confirmatory analysis** of drinking water samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Uranium-238	7440-61-1

This method covers the determination of uranium isotopes in water by means of chemical separations and alpha pulse height analysis. Uranium is chemically separated from a water sample by co-precipitation with ferrous hydroxide, 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 co-precipitated 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 resins, followed by elution with hydrochloric acid. The uranium is finally electrodeposited onto a stainless steel disk for alpha pulse analysis with a silicon-surface barrier detector.

*Source:* "Annual Book of ASTM Standards, Vol. 11.02," American Society for Testing and Materials (ASTM), 2002, ASTM International, 100 Barr Harbor Drive West, Conshohocken, PA 19428. Phone: 610-832-9500. Web: <http://www.astm.org>. Use Method number when ordering.

#### 6.2.10 U.S. DHS EML Method Am-01-RC: Americium in Soil

This method should be used for **confirmatory analysis** of soil/sediment samples for the contaminants identified below and listed in Appendix C.

Contaminant	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4

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 Procedure Pu-11-RC. Americium is collected with a calcium oxalate precipitation and finally isolated and purified by ion exchange. Californium is expected to be eluted at a point after americium is stripped off the column. After source preparation by microprecipitation, americium-241 and californium-252 are determined by separate alpha spectrometry analysis. The lower limit of detection (LLD) for americium-241 is 0.3 mBq when counted for 5000 minutes.

*Source:* AM-01-RC and Pu-11-RC. "EML Procedures Manual, HASL-3000," 28<sup>th</sup> Edition, Environmental Measurements Laboratory (EML), Department of Energy (EML is currently part of the U.S. Department of Homeland Security), February 1997. Web: <http://www.eml.doe.gov/publications/procman/>

#### 6.2.11 U.S. DHS EML Method Am-02-RC: Americium-241 in Soil-Gamma Spectrometry

This method should be used for **gross determination analysis** of soil/sediment samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Americium-241	14596-10-2

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 attenuation corrections are required if the calibration source and the sample are in a different matrix or are of different densities. The lower limit of detection (LLD) for 600 to 800 g of soil in a Martinelli beaker is 0.74 mBq for a 1000-minute count.

*Source:* "EML Procedures Manual, HASL-3000," 28<sup>th</sup> Edition, Environmental Measurements Laboratory (EML), Department of Energy (EML is currently part of the U.S. Department of Homeland Security), February 1997. Web: <http://www.eml.doe.gov/publications/procman/>



### 6.2.12 U.S. DHS EML Method Am-04-RC: Americium in QAP Water and Air Filters - Eichrom's TRU Resin

This method should be used for **confirmatory analysis** of drinking water and aqueous/liquid samples for the contaminants identified below and listed in Appendix C.

Contaminant	CAS RN
Americium-241	14596-10-2
Californium-252	13981-17-4

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. 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 TRU Resin extraction column. Americium (and curium, if present) is separated and purified on the column and finally stripped with dilute nitric acid stripping solution. Californium is expected to be eluted at a point after americium is stripped off the column. Microprecipitation is used to prepare for alpha-precipitation. The method involves sample preparation steps from U.S. DHS EML Method Pu-10-RC for water samples. The lower limit of detection (LLD) for total americium is 0.1 mBq when counted for 5000 minutes.

*Source:* "EML Procedures Manual, HASL-3000," 28<sup>th</sup> Edition, Environmental Measurements Laboratory (EML), Department of Energy (EML is currently part of the U.S. Department of Homeland Security), February 1997. Web: <http://www.eml.doe.gov/publications/procman/>

### 6.2.13 U.S. DHS EML Method Ga-01-R: Gamma Radioassay

This method should be used for **gross determination** and/or **confirmatory analysis** of soil/sediment, surface wipes, and/or air filter samples for the contaminants identified below and listed in Appendix C.

Contaminant	CAS RN
Cesium-137 *	10045-97-3
Cobalt-60	10198-40-0
Europium-154	15585-10-1
Iridium-192	14694-69-0
Radium-226 **	13982-63-3
Ruthenium-103	13968-53-1
Ruthenium-106 *	13967-48-1

\* The identified method will measure decay products of these isotopes

\*\* Method selected for gross determination of radium-226 in soil and sediment samples only

This method uses gamma spectroscopy for the measurement of gamma photons emitted from radionuclides without separating them from the sample matrix. The method is applicable for analysis of samples that contain radionuclides emitting gamma photons with energies ranging from about >40 keV for Ge(Li) and 100 keV for NaI(Tl) detector. The sample is placed into a standard geometry (physical shape) for gamma counting. Soil samples and sludge are placed into an appropriately sized Martinelli beaker after drying and grinding the sample for homogenization. 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.

*Source:* “EML Procedures Manual, HASL-3000,” 28<sup>th</sup> Edition, Environmental Measurements Laboratory (EML), Department of Energy (EML is currently part of the U.S. Department of Homeland Security), February, 1997. Web: <http://www.eml.doe.gov/publications/procman/>

#### 6.2.14 U.S. DHS EML Method Sr-03-RC: Strontium-90 in Environmental Samples

This method should be used for **gross determination** and **confirmatory analysis** of soil/sediment, surface wipes, and air filter samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Strontium-90 *	10098-97-2

\* The identified method will measure decay products of these isotopes

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 Procedures Manual, HASL-3000,” 28<sup>th</sup> Edition, Environmental Measurements Laboratory (EML), Department of Energy (EML is currently part of the U.S. Department of Homeland Security), February, 1997. Web: <http://www.eml.doe.gov/publications/procman/>

#### 6.2.15 Standard Method 7120: Gamma-Emitting Radionuclides

This method should be used for **gross determination** and **confirmatory analysis** of aqueous/liquid samples for the contaminants identified below and listed in Appendix C.

Contaminant	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

\* The identified method will measure decay products of these isotopes

The method uses gamma spectroscopy for measurement of gamma photons emitted from radionuclides in water samples, using either germanium (Ge) diodes or thallium-activated sodium iodide (NaI(Tl)) crystals. The method is applicable to samples that contain radionuclides emitting gamma photons with energies ranging from about 60 to 2000 KeV, and can be used for qualitative and quantitative determinations (using Ge detectors) or for screening and semi-quantitative determinations (using NaI(Tl))

detectors). Exact quantitation 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 about 100 to 2000 KeV is used for energy calibration.

*Source:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>

#### 6.2.16 Standard Method 7500-Ra B: Radium: Precipitation Method

This method should be used for **gross determination analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Radium-226 *	13982-63-3

\* The identified method will measure decay products of this isotope

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 reprecipitating 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 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:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>

#### 6.2.17 Standard Method 7500-Ra C: Radium: Emanation Method

This method should be used for **confirmatory analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Radium-226 *	13982-63-3

\* The identified method will measure decay products of this isotope

This method is for determination of radium-226 by alpha counting. Radium in water is concentrated and separated from sample solids by co-precipitation 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 four 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:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>

#### **6.2.18 Standard Method 7500-Sr B: Total Radioactive Strontium and Strontium-90: Precipitation Method**

This method should be used for **gross determination** and **confirmatory analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Strontium-90 *	10098-97-2

\* The identified method will measure decay products of this isotope

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 daughter 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:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>

#### **6.2.19 Standard Method 7500-U B: Uranium: Radiochemical Method**

This method should be used for **gross determination analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Uranium-238	7440-61-1

The sample is acidified with hydrochloric or nitric acid and boiled to eliminate carbonate and bicarbonate ions. Uranium is co-precipitated 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.

*Source:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>

### 6.2.20 Standard Method 7500-U C: Uranium: Isotopic Method

This method should be used for **confirmatory analysis** of aqueous/liquid samples for the contaminant identified below and listed in Appendix C.

Contaminant	CAS RN
Uranium-238	7440-61-1

This method is a radiochemical procedure for determination of the isotopic content of uranium alpha activity; it is consistent with determining the differences among naturally occurring, depleted, and enriched uranium. The sample is acidified with hydrochloric or nitric acid, and uranium-232 is added as an isotopic tracer. Uranium is co-precipitated 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, and the residual salt is converted to nitrate and electrodeposited onto a stainless steel disk for counting by alpha pulse height analysis using a silicon surface barrier detector.

*Source:* “Standard Methods for Examination of Water and Wastewater,” 20<sup>th</sup> Edition, American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1998. Web: <http://www.standardmethods.org/>



## Section 7.0: Biotoxin Methods

A list of analytical methods to be used in analyzing environmental samples for biotoxin contaminants during homeland security events is provided in Appendix D. Methods are listed for each analyte and for each sample matrix that potentially may need to be measured and analyzed when responding to an emergency. The methods table in Appendix D is sorted alphabetically by analyte and includes the following information:

- **Analyte(s).** The compound or compound(s) of interest.
- **Chemical Abstract Survey 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.
- **Determinative method identifier.** The unique identifier or number assigned to an analytical method by the method publisher.
- **Solid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in solid phase samples.
- **Oily solid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in oily phase samples.
- **Aqueous/Liquid sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in aqueous and/or liquid phase samples.
- **Drinking water sample preparation procedure.** The recommended method/procedure for sample preparation to measure the analyte of interest in drinking water samples.
- **Air sample preparation procedure.** The recommended method/procedure for sample preparation and analysis to measure the analyte of interest in air samples.

### 7.1 General Guidance

The guidance summarized in this section provides a general overview of how to identify the appropriate biotoxin method(s) for a given analyte-matrix combination as well as recommendations for quality control 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:

- A U.S. Army Medical Research Institute of Infectious Diseases' document at <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.
- The U.S. Centers for Disease Control has information regarding biotoxins, including 42 CFR Part 1003 regulations for possession, use, and transfer of select agents and toxins, on the following Web site: <http://www.cdc.gov/od/sap/toxinamt.htm>
- Syracuse Research Corporation's Physprop and Chemfate, part of the Environmental Fate Database supported by EPA. <http://www.syrres.com/esc/databases.htm>

- 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/vz72d288.html#JWIDAW> for toxicity information.
- EPA's Integrated Risk Information System (IRIS): <http://www.epa.gov/iris/> contains toxicity information.
- The Forensic Science and Communications Journal published by the Laboratory Division of the Federal Bureau of Investigation. <http://www.fbi.gov/hq/lab/fsc/current/backissu.htm>.

Additional research on biotoxin contaminants is ongoing within EPA.

### 7.1.1 Standard Operating Procedures for Identifying Biotoxin Methods

To determine the appropriate method and sample preparation technique that is to be used on the environmental samples, locate analyte of concern in Appendix D: Biotoxin Methods under the "Analyte" column. After locating the analyte of concern, continue across the table to identify the determinative technique and determinative method for that particular compound. To determine the sample preparation technique select the appropriate matrix column (Solid, Oily Solid, Aqueous/Liquid, Drinking Water, or Air) for that particular analyte.

Sections 7.2.1 through 7.2.4 below provide summaries of the determinative and sample preparation methods listed in Appendix D. Where available, a direct link to the full text of the selected analytical 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 4.

**Table 4. Sources of Biotoxin Methods**

Name	Publisher	Reference
National Environmental Methods Index (NEMI)	EPA, USGS	<a href="http://www.nemi.gov">http://www.nemi.gov</a>
U.S. EPA Office of Water (OW) Methods	EPA Office of Water	<a href="http://www.epa.gov/safewater/methods/sourcalt.html">http://www.epa.gov/safewater/methods/sourcalt.html</a>
U.S. EPA SW-846 Methods	EPA Office of Solid Waste	<a href="http://www.epa.gov/epaoswer/hazwaste/test/main.htm">http://www.epa.gov/epaoswer/hazwaste/test/main.htm</a>
U.S. EPA Office of Research and Development Methods	EPA Office of Research and Development	<a href="http://www.epa.gov/nerlcwww/ordmeth.htm">http://www.epa.gov/nerlcwww/ordmeth.htm</a>
U.S. EPA Air Toxics Methods	EPA Office of Air and Radiation	<a href="http://www.epa.gov/ttn/amtic/airtox.html">http://www.epa.gov/ttn/amtic/airtox.html</a>
Occupational Safety and Health Administration Methods	OSHA	<a href="http://www.osha-slc.gov/dts/sltc/methods/toc.html">http://www.osha-slc.gov/dts/sltc/methods/toc.html</a>
National Institutes for Occupational Safety and Health Methods	NIOSH	<a href="http://www.cdc.gov/niosh/nmam/">http://www.cdc.gov/niosh/nmam/</a>



Name	Publisher	Reference
Standard Methods for the Examination of Water and Wastewater, 20 <sup>th</sup> Edition, 1998*	American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF)	<a href="http://www.standardmethods.org">http://www.standardmethods.org</a>
Annual Book of ASTM Standards*	ASTM International	<a href="http://www.astm.org">http://www.astm.org</a>
International Organization for Standardization Methods*	ISO	<a href="http://www.iso.org">http://www.iso.org</a>
Official Methods of Analysis of AOAC International*	AOAC International	<a href="http://www.aoac.org">http://www.aoac.org</a>
Journal of Clinical Microbiology*	American Society for Microbiology	<a href="http://aem.asm.org">http://aem.asm.org</a>

\* Subscription and/or purchase required.

### 7.1.2 General Quality Control (QC) Guidance for Biotoxin Methods

Having data of known and documented quality is critical in order for public officials to accurately assess the activities that may be needed in responding to 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 contaminant presence/absence screening determinations versus quantitative analytical analyses. The specific needs for data generation should be identified. Quality control requirements and data quality objectives should be derived based on those needs. 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 risk assessment and site release, such as those identified in this document, might require increased QC requirements (demonstrations of method sensitivity, precision, and accuracy).

While method-specific QC requirements are described in many of the individual methods that are cited in this manual, and will be referenced in any standardized analytical protocols developed to address specific analytes and matrices of concern, the following describes a minimum set of QC procedures that shall be conducted for all chemical testing. Individual methods, sampling and analysis protocols, or contractual statements of work also should be consulted to determine any additional QC that may be needed. These QC requirements generally consist of analysis of laboratory control samples and or matrix spikes to identify and quantify measurement system accuracy at the levels of concern, blanks as a measure of freedom from contamination, and matrix spike duplicates (MSD) or sample replicates to assess data precision. 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 analysis of standards and other samples to ensure the continued reliability of the analytical results. Examples of sufficient quality control includes:

- Demonstration that 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/matrix/level of concern-specific QC samples (verify that measurement system has adequate sensitivity at levels of concern)
- Demonstration of continued measurement system reliability
  - Matrix spike/matrix spike duplicates (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 3 should be consulted regarding appropriate quality assurance and quality control (QA/QC) procedures prior to sample analysis. These contacts will consult with their respective QA/QC managers regarding QA/QC issues.

### 7.1.3 Safety and Waste Management

It is imperative that safety precautions are used during collection, processing, and analysis of environmental samples, particularly in emergency response situations that may include unknown hazards. Laboratories should have a documented health and safety plan for handling samples that may contain the target chemical, biological, or radiological 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 4.2 contain some specific requirements, guidance, 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:

- Occupational Health and Safety Administration's (OSHA) standard for Occupational Exposure to Hazardous Chemicals in Laboratories (29 CFR 1910.1450)
- OSHA regulations for hazardous waste operations and emergency response (29 CFR Part 1910)
- Environmental Protection Agency's standards regulating hazardous waste (40 CFR Parts 260 - 270)
- U.S. Department of Transportation (DOT) regulations for transporting hazardous materials (49 CFR Part 172)
- U.S. Department of Health and Human Services, Centers for Disease Control and Prevention's requirements for possession, use, and transfer of select agents and toxins (42 CFR Part 1003)

## 7.2 Method Summaries

Method summaries for the analytical methods listed in Appendix D, including methods for sample preparation and determinative techniques, are provided in Section 7.2.1 through 7.2.4. Information provided in these sections contains summary information only, extracted from the selected methods. The full version of the method should be consulted prior to sample analysis.

Each method summary contains a table identifying the contaminants in Appendix D to which the method applies, a brief description of the analytical method, and a link to the full version of the method or source for obtaining a full version of the method.

**Please note:** Not all methods have been verified for the analyte/matrix combination listed in Appendix D. Please refer to the specified method to identify analyte/matrix combinations that have been verified. Any questions regarding information discussed in this section should be addressed to the appropriate contact(s) listed in Section 3.

### 7.2.1 Laboratory Response Network (LRN)

The agents identified below and listed in Appendix D should be analyzed in accordance with the appropriate LRN protocols.

Contaminants	CAS RN
Alpha amanitin	NA
Botulinum toxin	NA
Microcystin	NA
Ricin	9009-86-3
Tetanus toxin	NA

These agents will be analyzed using restricted procedures available only through the Laboratory Response Network (LRN). These procedures are not available to the general laboratory community and thus are not discussed within this document. For additional information on the LRN, please see the contact information listed below or visit <http://www.bt.cdc.gov/lrn/>.

#### Centers for Disease Control and Prevention

Laboratory Response Branch  
Bioterrorism Preparedness and Response Program  
National Center for Infectious Diseases  
1600 Clifton Road NE, Mailstop C-18  
Atlanta, GA 30333  
Telephone: (404) 639-2790  
E-mail: [lrn@cdc.gov](mailto:lrn@cdc.gov)

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 (contact information provided below).

#### Association of Public Health Laboratories

2025 M Street NW, Suite 550  
Washington, DC 20036  
Telephone: (202) 822-5227  
Fax: (202) 887-5098  
Website: [www.aphl.org](http://www.aphl.org)  
E-mail: [info@aphl.org](mailto:info@aphl.org)

### 7.2.2 AOAC Official Method 994.08: Aflatoxin in Corn, Almonds, Brazil Nuts, Peanuts, and Pistachio Nuts

This method should be used for **preparation** and **analysis** of solid, oily solid, aqueous/liquid, and drinking water samples for the contaminants identified below and listed in Appendix D.

Contaminant	CAS RN
Aflatoxin	1402-68-2
Brevetoxin *	NA
Picrotoxin **	124-87-8
Saxitoxin *	35523-89-8
T-2 Mycotoxin *	NA

\* Alternative derivitization chemistries, chromatographic conditions, and fluorometric calibration of standards may be required

\*\* The U.S. Department of Agriculture (USDA) is currently developing an HPLC procedure specifically for detection of this toxin

Samples are extracted using an acetonitrile-water (9 + 1) solution. Sample extracts are then run through a multifunctional cleanup column. The purified extract and standards are derivatized with trifluoroacetic acid, and then analyzed using a high performance liquid chromatography (HPLC) system with a fluorescence detector. Specific aflatoxins can be identified by their retention time and quantified using standard curves. Method performance was characterized using various commodities (e.g., corn) at aflatoxin levels over a range of 5 to 30 ng/g. This method was originally designed for the analysis of aflatoxins (B<sub>1</sub>, B<sub>2</sub>, G<sub>1</sub>, and G<sub>2</sub>) in commodities where cleanup was necessary to remove other food components, such as fats and proteins; the cleanup procedure may not be necessary with water analyses. Coupling the procedures, or a modification of the procedures, included in this method with an immunoassay and/or viability test (where available) will provide more information regarding the specificity and toxicity of each target biotoxin.

Source: AOAC International. 1998. *Official Methods of Analysis of AOAC International*. 16<sup>th</sup> Edition, 4<sup>th</sup> Revision; Vol II.

### 7.2.3 Shiga Toxin Genes (Stx<sub>1</sub>, Stx<sub>2</sub>)

Contaminant	CAS RN
Shiga toxin	NA

Bacterial genes encoding Shiga toxin 1 and Shiga toxin 2 are detected using a real-time polymerase chain reaction (PCR) assay. Please see Section 5 for method summary.

### 7.2.4 Staphylococcal Enterotoxin (Method to be determined)

Contaminant	CAS RN
Staphylococcal enterotoxin	NA

## Section 8.0: Conclusions

Methods listed in Appendix A (chemical methods), Appendix B (biological methods), Appendix C (radiochemical methods), and Appendix D (biotoxin methods) are recommended for use in assessment of the extent of contamination and the effectiveness of decontamination in response to a homeland security event.

As stated in the introduction, the primary objective of this document is not necessarily to identify the “best” method for use during homeland security events, but rather to provide a balanced approach between leveraging existing and available determinative procedures and providing consistent analytical results. The method selected for each analyte/matrix pair was deemed the most general, appropriate, and broadly applicable of available methods. This is a living document and recommended methods are subject to change based on advances in technology.

Any questions concerning the information in this document should be directed to the appropriate point(s) of contact listed in Section 3.



# **Appendix A: Chemical Methods**





## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Aldicarb (Temik)	116-06-3	HPLC	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	531.2 (OW)	Not of concern in this matrix
Allyl alcohol	107-18-6	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	1402 (NIOSH)
Ammonia	7664-41-7	Spectrophotometry / ISE	4500-NH3 G (SM)	Not of concern in this matrix	Not of concern in this matrix	4500-NH3 B (SM)	350.3 (OW)	6015 (NIOSH) / ID-188 (OSHA)
Arsenic III compound	22569-72-8	ICP-MS/ICP-AES	6020A/6010C (SW-846)	3050B (SW-846)	3031/3050B (SW-846)	200.8 (OW)	200.8 (OW)	IO-3.1/IO-3.4/IO-3.5 (ORD)
Arsenic trichloride (analyze for Arsenic)	7784-34-1	ICP-MS/ICP-AES	6020A/6010C (SW-846)	3050B (SW-846)	3031/3050B (SW-846)	200.8 (OW)	200.8 (OW)	IO-3.1/IO-3.4/IO-3.5 (ORD)
Arsine	7784-42-1	GFAA/ICP-MS	7010 (SW-846)	3050B (SW-846)	Not of concern in this matrix	200.8 (OW)	200.8 (OW)	6001 (NIOSH)
Asbestos	1332-21-4	TEM	ASTM(dust) / ISO-10312 (air)	ASTM D5755-03 (soft surfaces-microvac) or D6480-99 (hard surfaces wipies)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	ISO-10312 (filter)
Boron trichloride	10294-34-5	ICP-AES	Journal Article: J. Anal. At. Spectrom., 2000, 15, 277-279	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Journal Article: J. Anal. At. Spectrom., 2000, 15, 277-279
Boron trifluoride	7637-07-2	ISE	ID-216SG (OSHA)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	ID - 216SG (OSHA)
Bromadiolone	28772-56-7	HPLC-UV	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	Not of concern in this matrix

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Cadmium	7440-43-9	ICP-MS/ICP-AES	6020A/6010C (SW-846)	3050B (SW-846)	3031/3050B (SW-846)	200.8 (OW)	200.8 (OW)	IO-3.1/IO-3.4/IO-3.5 (ORD)
Carbofuran (Furadan)	1563-66-2	HPLC / GC/MS	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	531.2 (OW)	TO-10A / ISO-12884
Carbon disulfide	75-15-0	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	524.2 (OW)	TO-15
Chlorine	7782-50-5	IC	6011 (NIOSH) / 4500-Cl G (SM)	Not of concern in this matrix	Not of concern in this matrix	4500-Cl G (SM)	4500-Cl G (SM)	6011 (NIOSH)
2-Chloroethanol	107-07-3	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	2513 (NIOSH)
3-Chloro-1,2-propanediol	96-24-2	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	TO-15
Chloropicrin	76-06-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Chlorosarin	1445-76-7	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Chlorosoman	7040-57-5	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
2-Chlorovinylarsonous acid (CVAA) (degradation product of Lewisite)	85090-33-1	ICP-MS / GC/MS	6020A (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Cyanide	57-12-5	Spectrophotometry (colorimetric)	CLP ILM05.3 CN	CLP ILM05.3 CN	Not of concern in this matrix	CLP ILM05.3 CN	335.4 (OW)	7904 (NIOSH)
Cyanogen chloride	506-77-4	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	TO-15
Cyclohexyl sarin (GF)	329-99-7	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
1,2-Dichloroethane (degradation product of HD)	107-06-2	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	524.2 (OW)	TO-15
Dichlorvos	62-73-7	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	525.2 (OW)	TO-10A / ISO-12884
Dicrotophos	141-66-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Diesel Range Organics	NA	GC-FID	8015C (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	Not of concern in this matrix
Diisopropyl methylphosphonate (DIMP) (degradation product of GB)	1445-75-6	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Dimethylphosphite	868-85-9	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Dimethylphosphoramidic acid (degradation product of GA)	33876-51-6	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
1,4-Dithiane (degradation product of HD)	505-29-3	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	TO-15
EA2192 (hydrolysis product of VX)	73207-98-4	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Ethylchloroarsine (ED)	598-14-1	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846) Note: For liquid matrices use 3520C (SW-846)	3520C/3535A (SW-846) Note: For liquid matrices use 3520C (SW-846)	TO-15
Ethylene oxide	75-21-8	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	TO-15
Ethylmethyl phosphonate (EMPA) (degradation product of VX)	1832-53-7	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Fenamiphos	22224-92-6	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	525.2 (OW)	TO-10A / ISO-12884
Fluoroacetate salts	NA	Ion Chromatography/ GC-ECD	300.1 (OW)	Journal Article: Analytical Letters, 1994, 27 (14), 2703- 2718	Journal Article: Analytical Letters, 1994, 27 (14), 2703- 2718	300.1 (OW)	300.1 (OW)	S301-1 (NIOSH)
Formaldehyde	50-00-0	HPLC	8315A (SW-846)	8315A (SW-846)	Not of concern in this matrix	8315A (SW-846)	8315A (SW-846)	ISO-16000-3

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Gasoline Range Organics	NA	GC-FID	8015C (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	Not of concern in this matrix
GE (1-methylethyl ester ethyl-phosphonofluoric acid)	1189-87-3	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Hydrogen bromide	10035-10-6	IC	4110 B (SM) / 7903 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	4110 B (SM)	4110 B (SM)	7903 (NIOSH)
Hydrogen chloride	7647-01-0	IC	4110 B (SM) / 7903 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	4110 B (SM)	4110 B (SM)	7903 (NIOSH)
Hydrogen cyanide	74-90-8	Spectrophotometry / ISE	6010 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	6010 (NIOSH)
Hydrogen fluoride	7664-39-3	IC	7906/7903 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	7906/7903 (NIOSH)
Hydrogen sulfide	7783-06-4	IC	6013 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	6013 (NIOSH)
Isopropyl methylphosphonic acid (IMPA) (degradation product of GB)	1832-54-8	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Kerosene	64742-81-0	GC-FID	8015C (SW-846)	5035A (SW-846)	3545A/3541/3580A (SW-846)	5030C (SW-846) Note: For liquid matrices use 3520C/3535A (SW-846)	5030C (SW-846)	Not of concern in this matrix

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Lewisite 1 (L-1) [2-chlorovinyl]dichloroarsine]	541-25-3	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Lewisite 2 (L-2) [bis(2-chlorovinyl)-chloroarsine]	40334-69-8	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Lewisite 3 (L-3) [tris(2-chlorovinyl)-arsine]	40334-70-1	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Lewisite Oxide (degradation product of Lewisite)	1306-02-1	ICP-MS	6020A (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Mercury	7439-97-6	CVAA/CVAFS	7471B (s) / 7470A (aq) (SW-846)	7471B (SW-846)	Not of concern in this matrix	7470A (SW-846)	245.2 (OW)	IO-5 (ORD)
Metals, NOS	NA	ICP-MS/ICP-AES	6020A/6010C (SW-846)	3050B (SW-846)	3031/3050B (SW-846)	200.8 (OW)	200.8 (OW)	See specific metals methods
Methyl hydrazine	60-34-4	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	3510 (NIOSH)
Methyl isocyanate	624-83-9	HPLC	207-2 (OAQPS)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	207-2 (OAQPS)
Methyl parathion	298-00-0	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Methylamine	74-89-5	GC	2010 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	2010 (NIOSH)

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Methylphosphonic acid (MPA) (degradation product of VX, GB, & GD)	993-13-5	HPLC-MS / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Mevinphos	7786-34-7	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	525.2 (OW)	TO-10A / ISO-12884
Mustard, nitrogen (HN-2) [unstable compound]	51-75-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Mustard, sulfur (HD) / Mustard Gas (H)	505-60-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Nicotine	54-11-5	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	Not of concern in this matrix
Osmium tetroxide (analyze for Osmium)	20816-12-0	ICP-AES / FAA	6010C (SW-846)	3050B (SW-846)	Not of concern in this matrix	252.2 (OW)	252.2 (OW)	IO-3.4 (ORD)
Oxamyl	23135-22-0	HPLC	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	8318A (SW-846)	531.2 (OW)	TO-15
Paraquat	4685-14-7	HPLC-UV	549.2 (OW)	Problematic	Problematic	549.2 (OW)	549.2 (OW)	Not of concern in this matrix
Perfluoroisobutylene (PFIB)	382-21-8	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Phencyclidine	77-10-1	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Phenol	108-95-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Phorate	298-02-2	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Phosgene	75-44-5	GC/MS / HPLC	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	Not of concern in this matrix	Not of concern in this matrix	TO-6/TO-15
Phosphine	7803-51-2	UV-VIS	6002 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	6002 (NIOSH)
Phosphorus trichloride	7719-12-2	Spectrophotometry	6402 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	6402 (NIOSH)
Polychlorinated biphenyls (PCBs)	1336-36-3	GC/MS / GC-ECD / GC	8082A (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	508 (OW)	TO-10A / ISO-12884
Propylene oxide	75-56-9	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	1612 (NIOSH)
Red Phosphorus (RP) (analyze for total Phosphorus)	7723-14-0	Spectrophotometry (colorimetric)	365.1 (NERL)	Not of concern in this matrix	Not of concern in this matrix	365.1 (NERL)	365.1 (NERL)	Not of concern in this matrix
Sarin (GB)	107-44-8	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Semivolatile Organic Compounds, NOS	NA	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	525.2 (OW)	TO-10A / ISO-12884



## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Soman (GD)	96-64-0	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Strychnine	57-24-9	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	Not of concern in this matrix
Sulfur Dioxide	7446-09-5	IC	6004 (NIOSH)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	6004 (NIOSH)
Tabun (GA)	77-81-6	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
Tear gas (CS) [chlorobenzylidene malonitrile]	2698-41-1	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Tetraethyl pyrophosphate	107-49-3	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Tetramethylene-disulfotetramine	80-12-6	HPLC-UV / GC/MS	8321B (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Thiodiglycol (TDG) (degradation product of HD)	111-48-8	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-15
1,4-Thioxane (degradation product of HD)	15980-15-1	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	5030C (SW-846)	TO-15
Titanium tetrachloride (analyze for total Titanium)	7550-45-0	ICP-MS/ICP-AES	6020A/6010C (SW-846)	3050B (SW-846)	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix	Not of concern in this matrix

## Appendix A: Chemical Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Trimethyl phosphite	121-45-9	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846) Note: For liquid matrices use 3520C (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
VE	21738-25-0	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
VG	78-53-5	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
VM	21770-86-5	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884
Volatile Organic Compounds, NOS	NA	GC/MS	8260B (SW-846)	5035A (SW-846)	3585 (SW-846)	5030C (SW-846)	524.2 (OW)	TO-15
VX [O-ethyl-S-(2-diisopropylaminoethyl) methyl phosphonothiolate]	50782-69-9	GC/MS	8270D (SW-846)	3545A/3541 (SW-846)	3545A/3541/3580A (SW-846)	3520C/3535A (SW-846)	3520C/3535A (SW-846)	TO-10A / ISO-12884

# **Appendix B: Biological Methods**



## Appendix B-1: Waterborne Biological Methods

Waterborne									
Agent Category	Analyte(s)	Identification Procedures				Viability Procedures			
		Identification determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method
Bacteria									
Bacteria	<i>Bacillus anthracis</i> (Anthrax)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Brucella</i> spp. (Brucellosis)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Burkholderia mallei</i> (Glanders)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Burkholderia pseudomallei</i> (Meliodiosis)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Campylobacter jejuni</i>	Culture	SM 9260 G	Filtration of large volumes of water using 0.45 or 0.22 micron filter	Ultrafiltration device	Culture	SM 9260 G	Filtration of large volumes of water using 0.45 or 0.22 micron filter	Ultrafiltration device
Bacteria	<i>Coxiella burnetii</i> (Q-fever)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Escherichia coli</i> ( <i>E. coli</i> ) O157:H7	Culture	SM 9260 F	Collect 100 mL sample in sterile container	Ultrafiltration device	Culture	SM 9260 F	Collect 100 mL sample in sterile container	Ultrafiltration device
Bacteria	<i>Francisella tularensis</i> (Tularemia)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Rickettsia prowazekii</i> (Epidemic Typhus)	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol
Bacteria	<i>Salmonella typhi</i> (Typhoid fever)	Culture	SM 9260 B	As specified by SM protocol	As specified by SM protocol	Culture	SM 9260 B	As specified by SM protocol	As specified by SM protocol
Bacteria	<i>Shigella</i> spp. (Shigellosis)	Culture	SM 9260 E	Filtration of large volumes of water using 0.45 or 0.22 micron filter	Ultrafiltration device	Culture	SM 9260 E	Filtration of large volumes of water using 0.45 or 0.22 micron filter	Ultrafiltration device
Bacteria	<i>Vibrio cholerae</i> (Cholera)	Culture	SM 9260 H	Concentration by placing Moore swabs in flowing wastewater for 1 week	Ultrafiltration device	Culture	SM 9260 H	Concentration by placing Moore swabs in flowing wastewater for 1 week	Ultrafiltration device
Bacteria	<i>Yersinia pestis</i> (Plague)	Culture / PCR / TRF	LRN	As specified by LRN protocol	As specified by LRN protocol	Culture	LRN	As specified by LRN protocol	As specified by LRN protocol

Waterborne									
Agent Category	Analyte(s)	Identification Procedures				Viability Procedures			
		Identification determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method
Biotoxin									
Biotoxin	Shiga toxin	PCR	Journal of Clinical Microbiology Vol. 39 No. 1: 370-374	Analysis conducted on isolated bacteria	Analysis conducted on isolated bacteria	NA	NA	NA	NA
Hemorrhagic Fever Viruses									
Viruses	Arenaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC
Viruses	Bunyaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC
Viruses	Filoviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC
Viruses	Flaviviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC
Encephalomyelitis / Encephalitis Viruses									
Viruses	Togaviruses: Venezuelan Equine Encephalitis Virus (VEEV)	RT-PCR	Journal of Clinical Microbiology Vol. 38 No. 4: 1527-1535	TBD	Filtration (1 MDS filter)	Tissue Culture	TBD	TBD	Filtration (1 MDS filter)
Poxviruses									
Viruses	Orthopoxvirus: Monkeypox virus	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC
Viruses	Orthopoxvirus: Variola major (Smallpox)	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	As specified by CDC

Waterborne									
Agent Category	Analyte(s)	Identification Procedures				Viability Procedures			
		Identification determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Wastewater sample preparation <sup>1</sup> procedure and/or sampling method	Drinking water sample preparation <sup>1</sup> procedure and/or sampling method
Enteric viruses									
Viruses	Adenoviruses: enteric and non-enteric (A-F)	Real-time PCR	AEM Vol. 71 No. 6: 3131-3136	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	Tissue Culture	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Astroviruses	RT-PCR ICC/RT-PCR	Canadian Journal of Microbiology Vol. 50: 269-278	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	Tissue Culture	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Caliciviruses: Noroviruses	RT-PCR	AEM Vol. 69 No. 9: 5263-5268	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	TBD	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Caliciviruses: Sapovirus	RT-PCR	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	TBD	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Coronaviruses: SARS-associated human coronavirus	RT-PCR	Journal of Virological Methods Vol. 122: 29-36	TBD	TBD	Tissue Culture	TBD	TBD	TBD
Viruses	Hepatitis E virus (HEV)	RT-PCR	Journal of Virological Methods Vol. 101: 175-188	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	TBD	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Picornaviruses: Enteroviruses	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	Tissue Culture	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Picornaviruses: Hepatitis A virus (HAV)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	Tissue Culture	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Viruses	Reoviruses: Rotavirus (Group A)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	Tissue Culture	TBD	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001
Protozoa									
Protozoa	<i>Cryptosporidium</i> species ( <i>Cryptosporidiosis</i> )	FA / IFA	Method 1622 / Method 1693	Filtration/Centrifugation per Method 1693	Filtration per Method 1622	TBD	TBD	Filtration/centrifugation	Filtration
Protozoa	<i>Entamoeba histolytica</i>	PCR	TBD	TBD	TBD	Mouse Bioassay/Tissue culture	TBD	TBD	TBD
Protozoa	<i>Toxoplasma gondii</i> (Toxoplasmosis)	PCR	AEM Vol. 70 No. 7: 4035-4039	TBD	TBD	Mouse Bioassay	AEM Vol. 70 No. 7: 4035-4039	TBD	TBD

<sup>1</sup> A dechlorinating agent (Sodium thiosulfate) should be added to treated water samples to remove any residual chlorine.





## Appendix B-2: Dustborne Biological Methods

Dustborne							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Bacteria							
Bacteria	<i>Bacillus anthracis</i> (Anthrax)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Brucella</i> spp. (Brucellosis)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Burkholderia mallei</i> (Glanders)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Burkholderia pseudomallei</i> (Melioidosis)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Campylobacter jejuni</i>	Culture	SM 9260 G	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	SM 9260 G	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Coxiella burnetii</i> (Q-fever)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Escherichia coli</i> ( <i>E. coli</i> ) O157:H7	Culture	SM 9260 F	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	SM 9260 F	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Francisella tularensis</i> (Tularemia)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Rickettsia prowazekii</i> (Epidemic Typhus)	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Salmonella typhi</i> (Typhoid fever)	Culture	SM 9260 B	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	SM 9260 B	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Shigella</i> spp. (Shigellosis)	Culture	SM 9260 E	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	SM 9260 E	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Vibrio cholerae</i> (Cholera)	Culture	SM 9260 H	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	SM 9260 H	Swabs, socks, swipes CDC/NIOSH Sampling techniques
Bacteria	<i>Yersinia pestis</i> (Plague)	Culture / PCR / TRF	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques	Culture	LRN	Swabs, socks, swipes CDC/NIOSH Sampling techniques

Dustborne							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Biotoxin							
Biotoxin	Shiga toxin	PCR	Journal of Clinical Microbiology Vol. 39 No. 1: 370-374	Analysis conducted on isolated bacteria	NA	NA	NA
Hemorrhagic Fever Viruses							
Viruses	Arenaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Bunyaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Filoviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Flaviviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Encephalomyelitis / Encephalitis Viruses							
Viruses	Togaviruses: Venezuelan Equine Encephalitis Virus (VEEV)	RT-PCR	Journal of Clinical Microbiology Vol. 38 No. 4: 1527-1535	TBD	Tissue Culture	TBD	TBD
Poxviruses							
Viruses	Orthopoxvirus: Monkeypox virus	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Orthopoxvirus: Variola major (Smallpox)	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC

Dustborne							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Enteric viruses							
Viruses	Adenoviruses: enteric and non-enteric (A-F)	Real-time PCR	AEM Vol. 71 No. 6: 3131-3136	TBD	Tissue Culture	TBD	TBD
Viruses	Astroviruses	RT-PCR ICC/RT-PCR	Canadian Journal of Microbiology Vol. 50: 269-278	TBD	Tissue Culture	TBD	TBD
Viruses	Caliciviruses: Noroviruses	RT-PCR	AEM Vol. 69 No. 9: 5263-5268	TBD	TBD	TBD	TBD
Viruses	Caliciviruses: Sapovirus	RT-PCR	TBD	TBD	TBD	TBD	TBD
Viruses	Coronaviruses: SARS-associated human coronavirus	RT-PCR	Journal of Virological Methods Vol. 122: 29-36	TBD	Tissue Culture	TBD	TBD
Viruses	Hepatitis E virus (HEV)	RT-PCR	Journal of Virological Methods Vol. 101: 175-188	TBD	TBD	TBD	TBD
Viruses	Picornaviruses: Enteroviruses	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	TBD
Viruses	Picornaviruses: Hepatitis A virus (HAV)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	TBD	TBD
Viruses	Reoviruses: Rotavirus (Group A)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	TBD	TBD
Protozoa							
Protozoa	<i>Cryptosporidium</i> species ( <i>Cryptosporidiosis</i> )	FA	Method 1622	TBD	TBD	TBD	TBD
Protozoa	<i>Entamoeba histolytica</i>	PCR	TBD	TBD	Mouse Bioassay/Tissue culture	TBD	TBD
Protozoa	<i>Toxoplasma gondii</i> (Toxoplasmosis)	PCR	AEM Vol. 70 No. 7: 4035-4039	TBD	Mouse Bioassay	AEM Vol. 70 No. 7: 4035-4039	TBD



## Appendix B-3: Aerosol Biological Methods

Aerosol							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Bacteria							
Bacteria	<i>Bacillus anthracis</i> (Anthrax)	Culture / PCR / TRF	LRN	XMZ / Anderson Button Sampler / DFU	Culture	LRN	XMZ / Anderson Button Sampler / DFU
Bacteria	<i>Brucella</i> spp. (Brucellosis)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Burkholderia mallei</i> (Glanders)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Burkholderia pseudomallei</i> (Melioidosis)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Campylobacter jejuni</i>	Culture	SM 9260 G	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	SM 9260 G	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Coxiella burnetii</i> (Q-fever)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Escherichia coli</i> ( <i>E. coli</i> ) O157:H7	Culture	SM 9260 F	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	SM 9260 F	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Francisella tularensis</i> (Tularemia)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Rickettsia prowazekii</i> (Epidemic Typhus)	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Salmonella typhi</i> (Typhoid fever)	Culture	SM 9260 B	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	SM 9260 B	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Shigella</i> spp. (Shigellosis)	Culture	SM 9260 E	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	SM 9260 E	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Vibrio cholerae</i> (Cholera)	Culture	SM 9260 H	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	SM 9260 H	Wetted-wall cyclones, CDC/NIOSH Sampling techniques
Bacteria	<i>Yersinia pestis</i> (Plague)	Culture / PCR / TRF	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques	Culture	LRN	Wetted-wall cyclones, CDC/NIOSH Sampling techniques

Aerosol							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Biotoxin							
Biotoxin	Shiga toxin	PCR	Journal of Clinical Microbiology Vol. 39 No. 1: 370-374	Analysis conducted on isolated bacteria	NA	NA	NA
Hemorrhagic Fever Viruses							
Viruses	Arenaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Bunyaviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Filoviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Flaviviruses	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Encephalomyelitis / Encephalitis Viruses							
Viruses	Togaviruses: Venezuelan Equine Encephalitis Virus (VEEV)	RT-PCR	Journal of Clinical Microbiology Vol. 38 No. 4: 1527-1535	TBD	Tissue Culture	TBD	TBD
Poxviruses							
Viruses	Orthopoxvirus: Monkeypox virus	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC
Viruses	Orthopoxvirus: Variola major (Smallpox)	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC	Biosafety Level 4 - Ship directly to CDC laboratory	CDC	As specified by CDC

Aerosol							
Agent Category	Analyte(s)	Identification Procedures			Viability Procedures		
		Identification determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method	Viability determinative technique	Determinative method identifier	Sample preparation procedure and/or sampling method
Enteric viruses							
Viruses	Adenoviruses: enteric and non-enteric (A-F)	PCR	AEM Vol. 71 No. 6: 3131-3136	TBD	Tissue Culture	TBD	TBD
Viruses	Astroviruses	RT-PCR ICC/RT-PCR	Canadian Journal of Microbiology Vol. 50: 269-278.	TBD	Tissue Culture	TBD	TBD
Viruses	Caliciviruses: Noroviruses	RT-PCR	AEM Vol. 69 No. 9: 5263-5268	TBD	TBD	TBD	TBD
Viruses	Caliciviruses: Sapovirus	RT-PCR	TBD	TBD	TBD	TBD	TBD
Viruses	Coronaviruses: SARS-associated human coronavirus	RT-PCR	Journal of Virological Methods Vol. 122: 29-36	TBD	Tissue Culture	TBD	TBD
Viruses	Hepatitis E virus (HEV)	RT-PCR	Journal of Virological Methods Vol. 101: 175-188	TBD	TBD	TBD	TBD
Viruses	Picornaviruses: Enteroviruses	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	As specified in USEPA Manual of Methods for Virology EPA/600/4-84/013, April 2001	TBD
Viruses	Picornaviruses: Hepatitis A virus (HAV)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	TBD	TBD
Viruses	Reoviruses: Rotavirus (Group A)	RT-PCR	AEM Vol. 69 No. 6: 3158-3164	TBD	Tissue Culture	TBD	TBD
Protozoa							
Protozoa	<i>Cryptosporidium</i> species ( <i>Cryptosporidiosis</i> )	FA	Method 1622	TBD	TBD	TBD	TBD
Protozoa	<i>Entamoeba histolytica</i>	PCR	TBD	TBD	Mouse Bioassay/Tissue culture	TBD	TBD
Protozoa	<i>Toxoplasma gondii</i> (Toxoplasmosis)	PCR	AEM Vol. 70 No. 7: 4035-4039	TBD	Mouse Bioassay	AEM Vol. 70 No. 7: 4035-4039	TBD





## **Appendix C: Radiochemical Methods**



## Appendix C: Radiochemical Methods

Analyte(s)	CAS RN	Determinative Technique	Drinking Water Samples		Aqueous and Liquid Phase Samples		Soil and Sediment Samples		Surface Wipes		Air Filters	
			Gross Determination	Confirmatory	Gross Determination	Confirmatory	Gross Determination	Confirmatory	Gross Determination	Confirmatory	Gross Determination	Confirmatory
Americium-241	14596-10-2	Alpha/Gamma spectrometry	D3084 (ASTM)	Am-04-RC (DHS)	D3084 (ASTM)	Am-04-RC (DHS)	Am-02-RC (DHS)	Am-01-RC (DHS)	TBD	TBD	TBD	TBD
Californium-252	13981-17-4	Alpha spectrometry	D3084 (ASTM)	Am-04-RC (DHS)	D3084 (ASTM)	Am-04-RC (DHS)	TBD	Am-01-RC (DHS)	TBD	TBD	TBD	TBD
Cesium-137 **	10045-97-3	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Cobalt-60	10198-40-0	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Europium-154	15585-10-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Iridium-192	14694-69-0	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Plutonium-238	13981-16-3	Alpha spectrometry	D3084 (ASTM)	EMSL-33 (EPA)	D3084 (ASTM)	EMSL-33 (EPA)	TBD	EMSL-33 (EPA)	TBD	EMSL-33 (EPA)	TBD	EMSL-33 (EPA)
Radium-226	13982-63-3	Alpha Counting	903.0 (EPA)	903.1 (EPA)	7500-Ra B (SM)	7500-Ra C (SM)	Ga-01-R (DHS)	EMSL-19 (EPA)	TBD	EMSL-19 (EPA)	TBD	EMSL-19 (EPA)
Ruthenium-103	13968-53-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Ruthenium-106 **	13967-48-1	Gamma spectrometry	901.1 (EPA)	901.1 (EPA)	7120 (SM)	7120 (SM)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)	Ga-01-R (DHS)
Strontium-90	10098-97-2	Beta counting by low-background gas flow proportional detector	905.0 (EPA)	905.0 (EPA)	7500-Sr B (SM)	7500-Sr B (SM)	Sr-03-RC (DHS)	Sr-03-RC (DHS)	Sr-03-RC (DHS)	Sr-03-RC (DHS)	Sr-03-RC (DHS)	Sr-03-RC (DHS)
Uranium-238	7440-61-1	Alpha Counting	908.0 (EPA)	D3972 (ASTM)	7500-U B (SM)	7500-U C (SM)	TBD	EMSL-33 (EPA)	TBD	EMSL-33 (EPA)	TBD	EMSL-33 (EPA)

\*\* Methods identified will measure decay product of these isotopes



# **Appendix D: Biotoxin Methods**



## Appendix D: Biotoxins Methods

Analyte(s)	CAS RN	Determinative Technique	Determinative Method Identifier	Solid Sample Prep / Procedure	Oily Solid Sample Prep / Procedure	Aqueous/Liquid Sample Prep / Procedure	Drinking Water Sample Prep / Procedure	Air Sample Prep / Procedure
Aflatoxin	1402-68-2	HPLC-FL	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	Not of concern in this matrix
Alpha amanitin	NA	Immunoassay	LRN	LRN	LRN	LRN	LRN	LRN
Botulinum toxin	NA	Immunoassay	LRN	LRN	LRN	LRN	LRN	LRN
Brevetoxin	NA	HPLC-FL	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	Not of concern in this matrix
Microcystin	NA	Immunoassay	LRN	LRN	LRN	LRN	LRN	LRN
Picrotoxin	124-87-8	HPLC-FL	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	Not of concern in this matrix
Ricin	9009-86-3	Immunoassay	LRN	LRN	LRN	LRN	LRN	LRN
Saxitoxin	35523-89-8	HPLC-FL	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	Not of concern in this matrix
T-2 Mycotoxin	NA	HPLC-FL	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	994.08 (AOAC)	Not of concern in this matrix
Tetanus toxin	NA	Immunoassay	LRN	LRN	LRN	LRN	LRN	LRN
Shiga toxin	NA	PCR	Refer to Appendix B for appropriate methods					
Staphylococcal enterotoxin	NA	Immunoassay	TBD	TBD	TBD	TBD	TBD	TBD

