United States Environmental Protection Agency | Office of Solid Waste and Emergency Response | OSWER Document 9240.1-39FS
EPA Publication 540-F-02-008
October 2002

Multi-Media, Multi-Concentration, Inorganic Analytical Service for Superfund (ILM05.2)

Office of Emergency and Remedial Response
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to EPA at such sites is a series of inorganic analytes and cyanide that are analyzed using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), Cold Vapor Atomic Absorption (CVAA), and colorimetric techniques. The Analytical Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) offers an analytical service that provides data from the analysis of water/aqueous and soil/sediment samples for inorganic analytes for use in the Superfund decision making process. Through a series of standardized procedures and a strict chain-of-custody, the inorganic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The inorganic analytical service provides a technical and contractual framework for laboratories to utilize EPA/CLP analytical methods. These methods are used in the isolation, detection and quantitative measurement of 23 target analyte metals (including mercury) and cyanide in both water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data.

Three data delivery turnaround times are available to CLP customers: 7, 14, and 21-day turnaround after receipt of the last sample in the set. A 72-hour preliminary data submission option also is available for all turnaround times. The data associated with these Preliminary Results is due within 72 hours after receipt of each sample at the laboratory. In addition, data users may include, but are not limited to, additional analytes and/or lower quantitation limits.

DATA USES

This analytical service provides data that EPA uses for a variety of purposes. Examples include determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including: site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET ANALYTES

The inorganic analytes and quantitation limits for which this service is applicable are listed in **Table 1**. Specific detection limits are method and matrix dependent.

The list of target analytes for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, analytes have been added to and deleted from the Target Analyte List (TAL), based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program.

METHODS AND INSTRUMENTATION

The Contractor will demonstrate the ability to meet certain program data quality objectives prior to analyzing field samples. The Contractor must document methods used to generate analytical results and determine Method Detection Limits (MDLs). ICP-Atomic Emission Spectroscopy (ICP-AES) is used to analyze water, sediment, sludge, and soil samples.

Table 1. Inorganic Target Analyte List and Contract Required Quantitation Limits (CRQLs)

	Analyte	ICP-AES CRQL for Water (µg/L)	ICP-AES CRQL for Soil (mg/kg)	ICP-MS CRQL for Water (µg/L)
1.	Aluminum	200	40	30
2.	Antimony	60	12	2
3.	Arsenic	15	3	1
4.	Barium	200	40	10
5.	Beryllium	5	1	1
6.	Cadmium	5	1	1
7.	Calcium	5000	1000	- -
8.	Chromium	10	2	2
9.	Cobalt	50	10	0.5
10.	Copper	25	5	2
11.	Iron	100	20	- -
12.	Lead	10	2	1
13.	Magnesium	5000	1000	- -
14.	Manganese	15	3	0.5
15.	Mercury	0.2	0.1	- -
16.	Nickel	40	8	1
17.	Potassium	5000	1000	- -
18.	Selenium	35	7	5
19.	Silver	10	2	1
20.	Sodium	5000	1000	- -
21.	Thallium	25	5	1
22.	Vanadium	50	10	1
23.	Zinc	60	12	1
24.	Cyanide	10	1	- -

Water and soil samples are treated with acids and heat or microwave energy. The digestates are then analyzed for trace metals by an atomic emission optical spectroscopic technique. The samples are nebulized and the aerosol is transported to a plasma torch. The atomic-line emission spectra are dispersed and a photosensitive device monitors line intensities.

ICP-Mass Spectrometry (ICP-MS) is used to determine the concentration of dissolved and total recoverable elements in water/aqueous samples. The sample material is introduced, by nebulization, into radio frequency plasma where desolvation, atomization, and ionization take place. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated based on their mass-to-charge ratio.

Cold Vapor Atomic Absorption (CVAA) is used to analyze water, sediment, sludge, and soil samples for total mercury. Organomercury compounds may also be present and will need to be broken down and converted to mercuric ions to respond to the CVAA techniques. For water samples, organic compounds are oxidized and then reacted with a strong reducing agent. The volatile free mercury is then driven from the reaction flask by bubbling air through the solution. The air stream carries the mercury atoms to an absorption cell, which is then placed in the light path of the AA spectrophotometer. For soil/sediment, the samples undergo acid digestion/oxidation followed by reduction and measurement by conventional cold vapor technique.

Various water types, sediment, sludge, and soil samples are also analyzed for total cyanide. Hydrocyanic acid (HCN) is released through a reflux-distillation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion is determined colorimetrically by converting it to cyanogen chloride (CNCl).

Table 2 summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include both hardcopy/electronic data reporting forms and supporting raw data. The laboratory must submit data to EPA within 7-, 14- or 21-days, or preliminary data must be submitted within 72 hours after laboratory receipt of each sample in the set, if requested. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) review to provide EPA Regions with electronic reports (PC-compatible reports, spreadsheets, and electronic files) within 24 to 48 hours from the receipt of the data. This automated tool facilitates the transfer of analytical data

Table 2. Methods and Instruments

Analyte	Instrument	Method
Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn	Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES)	ICP analysis of atomic-line emission spectra.
Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, Zn	ICP - Mass Spectrometry (ICP-MS)	ICP analysis of ions separated on basis of mass-to-charge ratio.
Mercury (Hg)	Cold Vapor Atomic Absorption (CVAA)	Acid digestion/oxidation followed by reduction and CVAA analysis.
Cyanide (CN)	Colorimeter or Spectrophotometer	Distillation followed by colorimetric analysis.

Table 3. Quality Control

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is set up.
Initial Calibration Verification	Following each instrument calibration for each wavelength or mass used.
Initial Calibration Blank	Following each instrument calibration, immediately after the Initial Calibration Verification (ICV).
Continuing Calibration Verification	For each wavelength or mass used, at a frequency of 10% or every two hours of a run, whichever is more frequent, and at the beginning and end of each run.
Continuing Calibration Blank	10% or every two hours of a run, whichever is more frequent, and at the beginning and end of each run. Performed immediately after the last Continuing Calibration Verification (CCV).
CRQL Check Standard (CRI)	Every 20 analytical samples and at the beginning and end of each run, but not before the ICV. Performed before the Interference Check Sample.
Interference Check Sample	For ICP-AES, every 20 analytical samples and at the beginning and end of each run, immediately after the CRI. For ICP-MS, at the beginning of the run.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples.
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, except aqueous mercury and cyanide.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post Digestion/Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
ICP-MS Tune	Prior to calibration.
Method Detection Limit Determination	Prior to contract, annually thereafter, and after major instrument maintenance.
Interelement Corrections	Prior to contract, quarterly thereafter, and after major instrument adjustment.
Linear Range Analysis	Quarterly.

into Regional databases. DAT can also be used to assist in the data validation process at the Region. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has four business days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE

The Quality Assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity and ensures that the data provided are of the quality required. During the data collection effort, QA activities ensure that the Quality Control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions. Each contract laboratory will establish a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are shown in **Table 3**.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, and evaluates laboratory performance with blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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