State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Determination of Metals by SW-846 Methods 7000 and 7010 (7000 Series)

Flame and Graphite Furnace Atomic Absorption Spectroscopy

Version 3.0

May 2024

Written by the Connecticut DEEP QA/QC Workgroup

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1.0	First version for publication	July 2005
2.0	Final version based upon public comments	July 2006
3.0	Updates to reflect CAM method updates to improve consistency between different states.	May 2024

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Acronym List

ACRONYM	DEFINITION
AA	Atomic Absorption
CASN	Chemical Abstracts Service Number
CCB	Continuing calibration blank
CCV	Continuing calibration verification
%D	Percent difference
DEEP	CT Department of Energy and Environmental Protection
EP	Environmental Professional
FLAA	Flame atomic absorption spectrometry
g	Grams
GFAA	Graphite furnace atomic absorption spectrometry
HCl	Hydrochloric acid
HNO_3	Nitric acid
ICB	Initial calibration blank
ICSA/AB	Interelement interference check samples
ICV	Initial calibration verification
LCS/LCSD	Laboratory control sample / Laboratory control sample duplicate
LLCV	Low-level calibration verification
LLOQ	Lower limit of quantitation
LRD	Linear range determination
MB	Method blank
MD	Matrix duplicate
mg/L	Milligram per liter
mg/kg	Milligram per kilogram
mL	Milliliter
MS	Matrix spike
nm	Nanometer
%R	Percent recovery
r/r^2	Correlation coefficient
RL	Reporting limit
RPD	Relative percent difference
RSD	Relative standard deviation
RSR/RSRs	Remediation Standard Regulations
QA	Quality assurance
QC	Quality control
µg/L	Microgram per liter
µm	Micrometer

1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 7000 Series

1.1 Method Overview

Metals in solution may be readily determined by atomic absorption ("AA") spectroscopy. The method is simple, rapid, and applicable to many metals in drinking, surface, and saline waters and domestic and industrial wastes. While drinking water free of particulate matter may be analyzed directly, ground water, other aqueous samples, extracts, industrial wastes, soils, sludges, sediments, and other solid wastes require digestion prior to analysis for both total and acid leachable metals. Analysis for dissolved elements does not require digestion if the sample has been filtered and acidified.

Reporting limits/Lower limits of quantitation ("RLs"/ "LLOQs"), sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. In general, it should be noted that the RLs/LLOQs obtained using of flame atomic absorption spectroscopy ("FLAAS") do not meet the requirements of the Connecticut Remediation Standards ("RSRs"), and are therefore unsuitable for use in obtaining "Reasonable Confidence". Analysis by graphite furnace atomic absorption spectroscopy ("GFAAS") will meet the RLs/LLOQs limits required, refer to SW-846 Method 7010 for a complete list of applicable metal analytes. The preferred analytical method(s) for mercury is by cold vapor atomic absorption described in SW-846 Methods 7470 or 7471.

RLs/LLOQs by FLAAS may also be extended through the process of concentrating the sample and/or through the process of solvent extraction techniques. RLs/LLOQs are dependent on equipment (such as the type of spectrophotometer and furnace accessory, the energy source, the degree of electrical expansion of the output signal) and are greatly dependent on sample matrix. RLs/LLOQs should be established, empirically, for each matrix type analyzed. When using GFAAS techniques, however, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each sample for interference effects and, if detected, treat the samples accordingly, using either successive dilution, matrix modification, or method of standard additions.

All method references are to the latest promulgated version of the method found in *Test Methods for Evaluating Solid Waste, SW-846.*

1.2 Summary of SW-846 Method 7000 Series

1.2.1 Summary of SW-846 Method 7000

When using FLAAS, a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp or an electrodeless discharge lamp is directed through the flame into a monochromator, and onto a detector that measures the amount of absorbed light. Absorption depends upon the presence of free unexcited ground-state atoms in the flame. Because the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of AA spectroscopy.

1.2.2 Summary of SW-846 Method 7010

GFAA spectrometry is used to determine trace elements in solution. The method is applicable for all the analytes listed in Table 1B in this RCP, as well as numerous other elements listed in SW-846 Method 7010. All aqueous matrices (except filtered groundwater samples) and solid matrices require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not require acid digestion.

An aliquot of the sample solution (digestate) is deposited into a graphite tube in the furnace, where it is evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atoms is vaporized and dissociated (atomized) in the graphite tube as compared to a flame, the use of smaller sample volumes and detection of lower concentrations of elements is possible with GFAA than with flame AA. Radiation from the "excited" elements passes through a vapor containing ground-state atoms of that 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 hallow cathode lamp or electrodeless discharge lamp and a photosensitive device measures the transmitted
radiation.

When using GFAAS all samples and standards require at least two "burns". Laboratories shall report the average of all burns.

It is recommended that all graphite furnace analyses be carried out using an appropriate matrix modifier. The choice of matrix modifier is dependent on analytes, conditions, and instrumentation and should be chosen by the analyst as the situation dictates. Analysts should consult the instrument manufacturer's instructions and also refer to SW-846 Method 7010 for further details. Analysts may also refer to Standards Methods 3113 and 3500 series for matrix modifier's if appropriate.

1.2.3 Sample Digestion

Prior to analysis, samples must be solubilized, or digested, using the appropriate sample preparation procedure (refer to Chapter 3 of SW-846). When analyzing groundwater for dissolved metals, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis. Refer to Table 1.0 below for appropriate digestion methods:

Table 1.0: Methods for Sample Digestion/Preparation for Trace Metals Analysis

SW-846 Digestion/ Preparation Method	Matrix	Title/Description
3005	Aqueous: Surface Water/ Groundwater	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
3010	Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy
3015	Aqueous: Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Analysis by FLAA, GFAA, or ICP Spectrometry
3020	Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy
3031	Solid: Oily Waste/Tar/ Wax/Paint/ Petroleum Product	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry

SW-846 Digestion/ Preparation Method	Matrix	Title/Description
3040	Solid: Oil/Grease/Wax	Dissolution Procedure for Oils, Greases, or Waxes
3050	Solid: Soil/Sediment/ Sludges	Acid Digestion of Sediments, Sludges, and Soils
3051	Solid: Soil/Sediment/ Sludge/Oil	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
3052	Solid: Biological Tissue/Oil/Ash Soil/Sediment/ Sludge	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
Digestion of samples is not required if the measured turbidity is <1.0 NTU. Laboratories must document turbidity readings for inspection.		

1.3 Method Interferences

Samples submitted to a laboratory for trace metal analysis may become contaminated by numerous routes during both sampling and analysis. Potential sources of contamination may include:

- Metallic or metal-containing containers and sampling equipment;
- Laboratory acids or reagents;
- Improperly cleaned or stored equipment; and
- Atmospheric inputs such as dirt and dust.

1.3.1 Spectral Interferences

Spectral interferences may cause high biased results due to interelement interferences, matrix interferences with non-target compounds that absorb light at the same wavelength as the target analyte, and other chemical interferences. These interferences can be minimized by using continuum Zeeman background correction (important, for example, in analyzing arsenic in the presence of aluminum and analyzing selenium in the presence of iron), modifying the sample charring and atomization program for the specific matrix, using a graphite platform, and/or using a matrix modifier during char and atomization steps in the graphite furnace.

1.3.2 Memory Interferences

Memory interferences may be caused by incomplete volatilization of the sample contributing to signals measured in a subsequent sample. These interferences can be minimized by using “blank burns” at regular intervals during the analytical run.

1.3.3. High Salt Concentrations

High salt concentrations (e.g., seawater samples) may cause analyte signal suppression or enhancement, dependent upon the element. Samples with high salt content can cause both physical interference and molecular interferences and may require high dilutions and/or alternate preparation procedures for accurate quantitation.

1.3.4 Analyte-Specific Interferences

Metals including antimony, arsenic, barium, beryllium, cadmium, chromium, lead, nickel, selenium, silver, thallium, and vanadium can each cause interferences due to the wavelengths they absorb. Procedures recommended to minimize these interference effects are detailed in SW-846 Method 7010.

All metals are not equally stable in the digestate, especially if it contains only nitric acid ("HNO_3"), rather than nitric acid and hydrochloric acid ("HCl"). The digestate should be analyzed as soon as possible, with preference given to antimony, barium, molybdenum, silver, and tin.

1.3.5 Cross Contamination

Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned and acid rinsed prior to use. Pipet tips are a frequent source of contamination. If suspected, they should be acid soaked with 1:5 HNO_3 and rinsed thoroughly with tap and reagent water. The use of a better grade of pipet tip can greatly reduce this problem. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become contaminated. As many as five to ten high-temperature burns may be required to clean the tube before use.

Analysis of blanks provides information about the presence of contaminants. When potential interfering peaks or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the contamination and eliminate it. **Subtracting blank concentrations from sample results is not permitted.** Any method blank exceedances should be fully documented in the laboratory report narrative.

1.4 Quality Control Requirements for SW-846 Method 7000 Series

1.4.1 Reporting Limits/Lower Limits of Quantitation for Method 7000 Series

The reporting limit ("RL"), or lower limit of quantitation ("LLOQ"), for an individual analyte is dependent on the concentration of the lowest non-zero standard in the initial calibration or the low-level calibration verification ("LLCV"), analyzed under identical conditions as the sample, with adjustments made for the sample size, preparation factors, percent solids, dilution factors, etc., as required. Table 2.0 lists approximate RL/LLOQs for the applicable methods and matrices using FLAAS or GFAA. Solid matrices in Table 2.0 assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation[1]

Matrix	**Typical Reporting Limit**
Aqueous	
Method 7000 (FLAAS)	1 to 800 µg/L
Method 7010 (GFAAS)	0.5 to 10 µg/L
Soil and Sediment	
Method 7000	1 to 800 mg/kg
Method 7010	0.1 to 5 mg/kg
[1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.	

Moisture content of soils and sediments will raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RL/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet the project Data Quality Objectives ("DQOs"). To meet the RLs/LLOQs

applicable to project DQOs, it may be necessary to modify the analytical method to improve sensitivity. In such cases, the modifications must be noted in the laboratory report narrative.

1.4.2 General Quality Control Requirements

This protocol is restricted to use by, or under the supervision of, analysts who are experienced in using FLAAS and/or GFAAS as a quantitative tool and skilled in the correction of spectral, chemical, and physical interferences described in this method.

Refer to SW-846 Chapter One for general quality control ("QC") procedures for all analytical methods, including the 7000 series methods. These requirements ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all inorganic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the instrument's operation may be found in SW-846 Chapter 3 and SW-846 7000 Series and include evaluation of calibrations and performance of sample analyses. Instrument QC and method performance requirements for the FLAAS and GFAAS systems may be found in SW-846 Methods 7000 and 7010.

The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Field, rinsate, or other blanks should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative method being used. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform the referenced Methods. These data must meet or fall within the performance standards as presented in Section 1.4 and Table 1A of this RCP. See SW-846 Chapter One and SW-846 Method 7000 and/or 7010 for the procedure. The IDOC must include the following elements provided in Table 3.0:

Table 3.0: IDOC Requirements

QC Element	Performance Criteria
Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Percent Recovery for MS/LCS	Table 1A
Relative Percent Difference of Matrix Duplicate	Table 1A
Other Instrument QC Samples	Table 1A

Because of the extensive analyte list and number of QC elements associated with the IDOC it should be expected that one or more analytes may not meet the performance standards for one or more QC elements. The laboratory should make every effort to find and correct the problem and repeat the analysis. All non-conforming analytes along with the laboratory acceptance criteria should be noted in the IDOC data.

Laboratories are required to generate laboratory specific performance criteria for LCS element recovery limits, matrix spike/matrix spike duplicate element recovery and relative percent different ("RPD") limits. These limits must be equal to or fall within the limits specified in Table 1A of this RCP.

1.4.3 Specific QA/QC Requirements and Performance Standards for the SW-846 Method 7000 Series

Specific QA/QC requirements and performance standards for the SW-846 Method 7000 Series are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with “Reasonable Confidence” regarding the usability of analytical data to support environmental decisions. The concept of "Reasonable Confidence" is explained on the CT Department of Energy and Environmental Protection (“DEEP”) website.

While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally, accept “Reasonable Confidence” data. To achieve “Reasonable Confidence” parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming analytical performance standards; and

3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

Table 1A: Specific QA/QC Requirements and Performance Standards for Methods 7000 and 7010

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Capability ("IDOC")	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedures in SW-846 7000/7010 and the applicable preparation method.	No	Refer to SW-846 7000/7010, the applicable preparation method requirements in the SW-846 3000 series methods and Section 1.2.3 of this protocol.	N/A
Preparation of Samples	Accuracy & Representativeness	(1) All aqueous (except dissolved/filtered GWs) and solid samples must be digested prior to analysis. See Digestion Section of this RCP for preparation method references.	No	N/A	N/A
Duplicate Injections (For GFAA)	Method Precision	(1) Each calibration standard, QC sample, and field sample must be analyzed (injected) twice. (2) RPD must be ≤10% for calibration standards and ≤20% for all other detected results. (3) Report the average results of duplicate injections for all target metals.	No	(1) Calibration/QC: Reanalyze; if duplicate injection RPD meet criteria, no further action required. (2) Calibration/QC: If duplicate injection still outside criteria, recalibrate instrument and reanalyze all QC and associated samples. (3) Field Samples: Reanalyze; if duplicate injection RPD meet criteria, no further action required. (4) Field Samples: If RPD still outside of criteria, dilute sample re-analyze diluted sample with duplicate injections.	Report duplicate injection RPD non-conformances in laboratory report narrative - potential sample matrix interference.
Initial Calibration ("ICAL")	Laboratory Analytical Accuracy	(1) Daily and prior to sample analysis. (2) Minimum calibration blank plus 3 calibration standards (multi-point) which may include the RL/LLCV standard; if LLCV standard is not included in calibration curve, then LLCV QC sample is required. High level standard in calibration defines the upper end of the linear calibration range. (3) Linear regression with correlation coefficient $r \geq 0.995$.	No	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 7000/7010.	Suspend all analyses until initial calibration meets criteria.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification ("ICV")	Laboratory Analytical Accuracy	(1) Daily, immediately after each initial calibration. (2) Prepared using standard source different than use for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be between 90-110% for each target analyte.	No	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.
Initial Calibration Blank ("ICB")	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Immediately after ICV. (2) Target analytes must be <RL/LLOQ. (3) Matrix matched with standards and samples.	No	(1) Reanalyze ICB; f acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.
Low-Level Calibration Verification ("LLCV")	Laboratory Analytical Sensitivity (very low-end of calibration range/verify RL/LLOQ)	(1) Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL/LLOQ. If initial calibration includes the RL/LLOQ as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL/LLOQ for all target analytes. (4) Percent recoveries must be 70-130% for all target analytes.	No	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are ≤10x RL/LLOQ in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes are >10x RL/LLOQ in associated field samples, include explanation in laboratory report narrative no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL/LLOQ in associated field samples.
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Every 10 samples and at the end of analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) GFAAS: Recovery ± 10% of true value, must use at least two burns with RPD <5%. (6) FLAAS: Recovery ±10% of true value.	No	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV- unless (3) applies. (3) If recovery is high (>110%) and all associated sample results are non-detect no corrective action is required.	If (3) applies, include explanation in laboratory report narrative.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Blank ("CCB")	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Every 10 samples following CCV and at the end of the analytical run. (2) Target analytes must be ≤½ RL/LLOQ. (3) Matrix matched with standards and samples.	No	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside criteria, recalibrate and reanalyze all associated samples since last compliant CCB- unless (3) applies. (3) If concentration of contaminant in CCB is >RL/LLOQ but all associated sample results are either non-detected or >10x concentration of contaminant in CCB; no corrective action required.	If (3) applies, include explanation in laboratory report narrative.
Method Blank ("MB")	Laboratory Method Sensitivity (contamination evaluation)	(1) One per digestion batch of ≤20 field samples or every batch. If no digestion, ICB/CCB = method blank. (2) Must be digested with the samples using the same preparation method as the samples. (3) Matrix specific and matrix matched. (4) Target analytes must be <RL/LLOQ.	Yes	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, re-digest and reanalyze MB and all associated samples in batch- unless (3) applies. (3) If concentration of contaminant in MB is >RL/LLOQ but all associated sample results are either non-detected or >10x concentration in MB; no corrective action required.	If (3) applies, include explanation in laboratory report narrative.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample ("LCS")	Laboratory Method Accuracy	(1) One per digestion of ≤20 field samples or each batch**.** If samples not digested, ICV/CCV = LCS. (2) Must be matrix-matched by digesting with the samples using the same preparation method. It is recommended that a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) Prepared using second source standard.	Yes	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte; no corrective action required. (3) If LCS and LCSD are both outside of criteria, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.	Report non-conformances in laboratory report narrative.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
LCS Duplicate ("LCSD")	Laboratory Analytical Accuracy & Precision	(1) One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. It is recommended that a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels must be same as LCS. (4) Must contain all target analytes; analyze immediately following LCS. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) RPDs must be ≤20% for aqueous LCS/LCSD and ≤30% for solid LCS/LCSD.	Yes If analyzed	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.	Report non-conformances in laboratory report narrative.
Matrix Spike ("MS") (site-specific)	Method Accuracy in Sample Matrix	(1) Solid samples: One per ≤20 field samples per matrix or one per batch; designated by data user on COC or at project set-up. Aqueous Samples: One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by data user	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective is required. (3) If MS recovery is <30% and associated with non-detected results, re-digest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS non-conformances in laboratory report narrative. If re-digested due to recoveries <30%, report both sets of sample/MS data.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Matrix Duplicate ("MD") ("site-specific")	Method Precision in Sample Matrix	(1) One per digestion batch of ≤20 field samples or every batch. (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤ 20% for aqueous and ≤ 35% for solids.	Yes ONLY when requested by data user	Narrate non-conformances in laboratory report narrative.	Report non-conformances in laboratory report narrative.
Dilution Test	Accuracy in Sample Matrix	(1) One per ≤20 field samples per matrix; only if project-specific MS requested and analyte concentration is >50x RL/LLOQ. (2) Perform 5x serial dilution on same sample used for MS/MD. (3) %D of the sample and dilution results for target analytes at levels >50x RL/LLOQ must be ±10% for all matrices.	Yes ONLY if project-specific MS requested by data user	Narrate.	Report non-conformances in laboratory report narrative.

General Reporting Issues	N/A	(1) Non-detected values must be reported with the sample-specific RL/LLOQ for each target analyte using all preparation/dilution factors. (2) The lab must only report values ≥ the sample-specific RL/LLOQ. (3) Sample concentrations that exceed the highest calibration standard must be diluted and re-analyzed to fall within the linear calibration range. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to RSR regulatory standards. (5) Results must be reported with 2 or more "significant figures" if ≥RL/LLOQ. (6) Concentrations below the reporting limit should be reported as "ND" with the sample specific RL/LLOQ also reported.	N/A	N/A	(1) The performance of dilutions must be documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target analytes, reasons for dilutions must be explained in laboratory report narrative. (2) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report narrative. (3) If samples are digested and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative. (4) Narrate any additional method non-compliance or sample-specific anomaly.

1.5 Analyte List for SW-846 Method 7000 Series

The DEEP analyte list for SW-846 Method 7000 Series is presented in Table 1B. The elements listed are readily determined by SW-846 Methods 7000 and 7010. Most of the elements listed have Connecticut RSR Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

Table 1B: Analyte List for SW-846 Method 7000 Series

Analyte	CASN
Antimony	7440360
Arsenic	7440382
Barium	7440393
Beryllium	7440417
Cadmium	7440439
Chromium (total)	7440473
Copper	7440508
Lead	7439921
Nickel	7440020
Selenium	7782492
Silver	7440224
Thallium	7440280
Vanadium	7440622
Zinc	7440666

1.5.1 Additional Reporting Requirements for SW-846 7000 Series Methods

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEEP strongly recommends that the full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific QC requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.6 Routine Reporting Deliverables for 7000 Series Methods

The following table (Table 4.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report all items in special circumstances.

Table 4.0: Report Deliverables

Parameter	Deliverable	Comments
Initial Calibration	NO	Correlation coefficient must meet QA/QC requirements
Initial Calibration Verification Standard	NO	ICV must pass
Initial Calibration Blank	NO	Note non-conformances in laboratory report narrative
Continuing Calibration Verification	NO	CCV must pass
Continuing Calibration Blank	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive sample results above RL/LLOQ with “B” flag.

Parameter	Deliverable	Comments
Lab Control Sample / Lab Control Sample Duplicate	YES	Note non-conformances in laboratory report narrative
Interference Check Standards	NO	Note non-conformances in laboratory report narrative.
Site-Specific Matrix Spike/ Matrix Duplicate	YES (Only if requested by data user)	Note non-conformances in laboratory report narrative
Linear Range Determination	NO	Data on file at laboratory
Dilution Test	Yes (ONLY if project-specific MS requested by data user.)	Note non-conformances in laboratory report narrative
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory

1.6.1 Reporting and Flagging of Results

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as “ND” (Not Detected at the specified RL/LLOQ). The RL/LLOQ for each element in each sample must be listed on the report and consider the exact sample mass, any dilution factors, percent moisture, etc.

- Elements detected above the RL/LLOQ in blanks and found in samples, also above the RL/LLOQ, shall be flagged with a “B” suffix (e.g., 25B).

- All soil/sediment results shall be reported on a dry weight basis.

1.7 Sample Containers, Preservations, and Holding Times

Table 5.0 identifies the type of containers, preservation requirements, and holding times dependent upon analyte and matrix.

Table 5.0: Sample Containers, Preservation, and Holding Times

Matrix	**Container[1,2]**	**Preservative[3]**	**Holding Time[4]**
Aqueous Total Metals	500 mL plastic or glass	Nitric Acid to pH <2	180 days
Aqueous Dissolved Metals (Filtered)	500 mL plastic or glass	Filter (0.45 µm) on site or at the laboratory (prior to acid preservation) within 24 hours of collection; then preserve with Nitric Acid to pH <2	180 days
Soil/Sediment samples.	250 mL plastic or glass jar with Teflon or plastic lined cap.	Cool to 4 ± 2º C	180 days

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]Plastic bottles must be acid rinsed and either high-density polyethylene, or Teflon.

[3]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6ºC are acceptable.

[4]If mercury is to be determined, the holding time for mercury is 28 days from collection. The preferred analytical method for mercury is SW-846 Methods 7470 and 7471 (cold vapor atomic absorption).