

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 [23.0](#)

[Month 2023](#)

Written by the Connecticut [DEP/DEEP](#) QA/QC Workgroup

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ACRONYM LIST

<u>ACRONYM</u>	<u>DEFINITION</u>
<u>AA</u>	<u>Atomic Absorption</u>
<u>CASN</u>	<u>Chemical Abstracts Service Number</u>
<u>CCB</u>	<u>Continuing calibration blank</u>
<u>CCV</u>	<u>Continuing calibration verification</u>
<u>%D</u>	<u>Percent difference</u>
<u>DEEP</u>	<u>CT Department of Energy and Environmental Protection</u>
<u>EP</u>	<u>Environmental Professional</u>
<u>FLAA</u>	<u>Flame atomic absorption spectrometry</u>
<u>g</u>	<u>Grams</u>
<u>GFAA</u>	<u>Graphite furnace atomic absorption spectrometry</u>
<u>HCl</u>	<u>Hydrochloric acid</u>
<u>HNO₃</u>	<u>Nitric acid</u>
<u>ICB</u>	<u>Initial calibration blank</u>
<u>ICSA/AB</u>	<u>Interelement interference check samples</u>
<u>ICV</u>	<u>Initial calibration verification</u>
<u>LCS/LCSD</u>	<u>Laboratory control sample / Laboratory control sample duplicate</u>
<u>LLCV</u>	<u>Low-level calibration verification</u>
<u>LLOQ</u>	<u>Lower limit of quantitation</u>
<u>LRD</u>	<u>Linear range determination</u>
<u>MB</u>	<u>Method blank</u>
<u>MD</u>	<u>Matrix duplicate</u>
<u>mg/L</u>	<u>Milligram per liter</u>
<u>mg/kg</u>	<u>Milligram per kilogram</u>
<u>mL</u>	<u>Milliliter</u>
<u>MS</u>	<u>Matrix spike</u>
<u>nm</u>	<u>Nanometer</u>
<u>%R</u>	<u>Percent recovery</u>
<u>r/r²</u>	<u>Correlation coefficient</u>
<u>RL</u>	<u>Reporting limit</u>
<u>RPD</u>	<u>Relative percent difference</u>
<u>RSD</u>	<u>Relative standard deviation</u>
<u>RSR/RSRs</u>	<u>Remediation Standard Regulations</u>
<u>QA</u>	<u>Quality assurance</u>
<u>QC</u>	<u>Quality control</u>
<u>µg/L</u>	<u>Microgram per liter</u>
<u>µm</u>	<u>Micrometer</u>

Connecticut ~~DEP~~DEEP RCPs
Quality Assurance and Quality Control Requirements
~~Determination of Metals By SW-846~~by Method 7000 and 7010 (7000 Series), SW-846
~~Methods for Flame and Graphite Furnace Atomic Absorption~~
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1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 7000 Series

1.1 Method Overview

~~1.1.1~~ Metals in solution may be readily determined by atomic absorption (“AA”) spectroscopy. The method is simple, rapid, and applicable to a large number of 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, EP 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.

~~1.1.2~~ Detection 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 detection limits RLs/LLOQs obtained using of flame atomic absorption spectroscopy (FAAS)(“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 detection RLs/LLOQs limits required, but is generally limited refer to determination SW-846 Method 7010 for a complete list of antimony, arsenic, lead, selenium, and thallium. In the environmental laboratory arena metals, with the exception of applicable metal analytes. The preferred analytical method(s) for mercury, are determined is by inductively coupled plasma spectroscopy (either ICP-AES Method 6010 or ICP-MS Method 6020). Mercury is typically determined by the cold vapor technique (atomic absorption described in SW-846 Methods 7470 or 7471) the requirements of which are described in their own method. Mercury may also be determined by ICP-MS, Method 6020.

~~1.1.3~~ Detection limits RLs/LLOQs by FAAS FLAAS may also be extended through concentration of the process of concentrating the sample and/or through the process of solvent extraction techniques. Detection is somewhat 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. Detection limits 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 them 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.

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1.2 Reporting Limits for Summary of SW-846 Method 7000 Series Methods

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 ~~2A of 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.

When using GFAAS, An 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.

Matrix Modifiers for GFAAS¹

<u>Element</u>	<u>Matrix Modifier</u>
<u>Antimony</u>	<u>Palladium + Magnesium Nitrate or Palladium + reducing agent such as Citric Acid or Nickel Nitrate</u>
<u>Arsenic</u>	<u>Palladium + Magnesium Nitrate or Palladium + reducing agent such as Citric Acid or Palladium or Nickel Nitrate</u>
<u>Lead</u>	<u>Palladium + reducing agent such as Citric Acid or Magnesium Nitrate + Phosphate or Lanthanum Nitrate²</u>

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Selenium	Palladium + Magnesium Nitrate or Nickel Nitrate
Thallium	Palladium + Magnesium Nitrate

1. See Method 3113A of Standard Methods for the Examination of Water and Wastewater, 20th-ed. APHA-AWWA-WEF for details. If other elements are determined by GFAAS, the appropriate matrix modifier must be used.

2. See USEPA SOW ILMO4.0 Inorganic Contract Laboratory Protocols for details.

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	<u>Matrix</u>	<u>Title/Description</u>
3005	<u>Aqueous: Surface Water/ Groundwater</u>	Method prepares ground water and surface water samples for total recoverable and dissolved metal determinations by FLAA, ICP-AES, or ICP-MS. The unfiltered or filtered sample is heated with dilute HCl and HNO₃ prior to metal determination. <u>Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy</u>
3010	<u>Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste</u>	Method prepares waste samples for total recoverable metal determinations by FLAA, ICP-AES, or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid. The method is applicable to aqueous samples, EP and mobility procedure extracts. <u>Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy</u>

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SW-846 Digestion/ Preparation Method	<u>Matrix</u>	<u>Title/Description</u>
3015	<u>Aqueous: Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste</u>	Method prepares aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total recoverable metal determinations by FLAA, GFAA, ICP-AES, or ICP-MS. Nitric acid is added to the sample in a Teflon digestion vessel and heated in a microwave unit prior to metals determination. <u>Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Analysis by FLAA, GFAA, or ICP Spectrometry</u>
<u>3020</u>	<u>Aqueous: Surface Water/ Groundwater/ Mobility- procedure extracts/ aqueous waste</u>	<u>Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy</u>
3031	<u>Solid: Oily Waste/Tar/ Wax/Paint/ Petroleum Product</u>	Method prepares waste oils, oil sludges, tars, waxes, paints, paint sludges and other viscous petroleum products for analysis by FLAA, GFAA, and ICP-AES. The samples are vigorously digested with nitric acid, sulfuric acid, hydrochloric acid, and potassium permanganate prior to analysis. <u>Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry</u>
3040	<u>Solid: Oil/Grease/Wax</u>	Method prepares oily waste samples for determination of soluble metals by FLAA, GFAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content <u>Dissolution Procedure for Oils, Greases, or Waxes</u>

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SW-846 Digestion/ Preparation Method	<u>Matrix</u>	<u>Title/Description</u>
3050	<u>Solid: Soil/Sediment/ Sludges</u>	Method prepares waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples <u>Acid Digestion of Sediments, Sludges, and Soils</u>
3051	<u>Solid: Soil/Sediment/ Sludge/Oil</u>	Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid is added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination <u>Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils</u>
3052	<u>Solid: Biological Tissue/Oil/Ash Soil/Sediment/ Sludge</u>	Method prepares siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total analysis by FLAA, CVAA, GFAA, ICP-AES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis <u>Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices</u>

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.

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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.

~~1.5.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or when the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome phosphate interference in magnesium, calcium, and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium.~~

~~1.5.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. Although complexing agents are employed primarily to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.~~

~~1.1.5.3 The presence of high dissolved solids in the sample may result in an interference from nonatomic absorbance such as light scattering. If background correction is not available, a nonabsorbing wavelength should be checked. Preferably, samples containing high solids should be extracted.~~

~~1.5.1.4 Ionization interferences occur when the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positively charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess (1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs.~~

~~1.5.1.5 Spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal.~~

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~~Interference can also occur when resonant energy from another element in a multielement lamp, or from a metal impurity in the lamp cathode, falls within the bandpass of the slit setting when that other metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.~~

~~1.5.1.6 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.~~

~~1.5.1.7 All metals are not equally stable in the digestate, especially if it contains only nitric acid, not nitric acid and hydrochloric acid. The digestate should be analyzed as soon as possible, with preference given to antimony, barium, molybdenum, silver, and tin.~~

~~1.5.2 Graphite Furnace Atomic Absorption Spectroscopy~~

~~1.5.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To verify the absence of matrix or chemical interference, all samples must be spiked at the instrument. Those samples which indicate the presence of an interference should be treated in one or more of the following ways:~~

- ~~1. Successively dilute and reanalyze the samples to eliminate interferences (detection limits requirements still must be met, if possible)~~
- ~~2. Modify the sample matrix either to remove interferences or to stabilize the analyte. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium phosphate to retain cadmium. The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.~~
- ~~3. Analyze the sample by method of standard additions while noticing the precautions and limitations of its use (see Step 8.7.2 of SW-846 Method 7000).~~

~~1.5.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, use either background correction or choose an alternate wavelength. Background correction may also compensate for nonspecific~~

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~~broad band absorption interference.~~

~~1.5.2.3 Continuum background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g., Zeeman background correction.~~

~~1.5.2.4 Interference from a smoke producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.~~

~~1.5.2.5 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad band absorption will be minimized.~~

~~1.5.2.6 Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, a minimum amount should be used. This applies particularly to hydrochloric and, to a lesser extent, to sulfuric and phosphoric acids.~~

~~1.5.2.7 Carbide formation resulting from the chemical environment of the furnace has been observed. Molybdenum may be cited as an example. When carbides form, the metal is released very slowly from the resulting metal carbide as atomization continues. Molybdenum may require 30 seconds or more atomization time before the signal returns to baseline levels. Carbide formation is greatly reduced and the sensitivity increased with the use of pyrolytically coated graphite. Elements that readily form carbides include molybdenum and vanadium.~~

~~1.5.2.8 For comments on spectral interference, see 1.5.1.5.~~

~~1.5.2.9 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 as and acid rinsed prior to use. Pipet tips are a frequent source of contamination. If suspected, they should be acid soaked with 1:5 nitric acid 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~~

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~~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.~~

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 (“HNO₃”), 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 HNO₃ 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.

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1.4 Quality Control Requirements for SW-846 Method 7000 Series

~~Reporting Limits (RL), sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the wavelength, spectrometer, matrix and operating conditions. Consult Methods 7000 and the appropriate 7000 series method for information regarding each specific analyte regarding the recommended analytical wavelengths and estimated instrumental detection limits (IDLs).~~

~~Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for trace metals are~~

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 this table assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation

<u>Matrix</u>	<u>Typical Reporting Limit</u>
<u>Aqueous</u>	
<u>Method 7000 (FLAAS)</u>	<u>1 to 800 µg/L</u>
<u>Method 7010 (GFAAS)</u>	<u>0.5 to 10 µg/L</u>
<u>Soil and Sediment</u>	
<u>Method 7000</u>	<u>1 to 800 mg/kg</u>
<u>Method 7010</u>	<u>0.1 to 5 mg/kg</u>

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's to be raised~~RL/LLOQs to be raised. It is the responsibility of the ~~EP data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet RSR criteria.~~ To meet the limits, 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.

1.34.2 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 ~~Each~~ (“QC”) procedures for all analytical methods, including the 7000 series methods. These requirements ensure that each laboratory ~~is required to operate/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 1, Section 2.0 Chapter 3 and SW-846 7000 Series and include evaluation of calibrations and performance of

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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 laboratory proficiency, 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 and accuracy. The use of site-specific matrix spikes and matrix duplicates is highly-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 compound element recovery is key to making good 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 and are in general discouraged. 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 Initial Demonstration of Proficiency 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 exceed fall within the performance standards as presented in Section 1.54 and Table 1A of this RCP. See Section 4.4.1 of SW-846 Chapter One and Section 8.0 of SW-846 Method 6010/7000 and/or 7010 for the procedure. The Initial Demonstration of Proficiency IDOC must include the following elements provided in Table 1-13.0:

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.

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

Specific QA/QC requirements and performance standards for the ~~SW-846 Method~~ 7000 Series ~~Methods~~ 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 ~~environmental professional~~ “EP” with “Reasonable Confidence” regarding the usability of analytical data to support ~~DEP 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, ~~will be generally accepted by agency reviewers. In order to, 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, ~~compliance with required corrective actions and analytical response actions for all non-conforming analytical performance standards prescribed in Table 1A for this test method;~~ and
3. Adopt the reporting formats and elements specified in Section 1.7 of this method. Retain reported and unreported analytical data and information for a period of 10 years.

1.6.3 Site Specific Matrix Spike (MS) and Matrix Duplicate (MD) Samples

~~It is strongly recommended that site specific MS/MD samples be analyzed from each site, and each matrix type sampled. Percent recovery data from site specific samples allow the to make intelligent 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 and are in general discouraged. Field blanks, rinsate blanks, etc. should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD.~~TABLE

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

Required QA/QC Parameter <u>Column 1</u>	Data Quality Objective <u>Column 2</u>	Required Performance Standard <u>Column 3</u>	Required Deliverable <u>Column 4</u>	Recommended Corrective Action <u>Column 5</u>	Required Analytical Response Action <u>Column 6</u>	Rationale for Changes
<u>Initial Demonstration of Capability ("IDOC")</u>	<u>Laboratory Analytical Accuracy & Precision</u>	<u>(1) Must be performed prior to using method on samples.</u> <u>(2) Must be performed for each matrix.</u> <u>(3) Must contain all target analytes.</u> <u>(4) Must follow procedures in SW-846 7000/7010 and the applicable preparation method.</u>	No	<u>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.</u>	N/A	<u>Group accepted MA language.</u>
<u>Sample Preparation of Samples</u>	Accuracy and Representativeness	All (1) All aqueous (except dissolved/filtered GWs) and solid samples must be digested prior to analysis with the exception. See Digestion Section of aqueous samples filtered and acidified this RCP for dissolved metals preparation method references.	No	N/A	N/A	<u>Group accepted MA language, changed preparation reference to "Digestion Section in RCP"</u>
<u>Duplicate Injections (For GFAA)</u>	<u>Method Precision</u>	<u>(1) Each calibration standard, QC sample, and field sample must</u>	No	<u>(1) Calibration/QC: Reanalyze; if duplicate injection</u>	<u>Report duplicate injection RPD non-conformances in</u>	<u>Group accepted MA language.</u>

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		<p><u>be analyzed (injected) twice.</u> <u>(2) RPD must be ≤10% for calibration standards and ≤20% for all other detected results.</u> <u>(3) Report the average results of duplicate injections for all target metals.</u></p>		<p><u>RPD meet criteria, no further action required.</u> <u>(2) Calibration/QC: If duplicate injection still outside criteria, recalibrate instrument and reanalyze all QC and associated samples.</u> <u>(3) Field Samples: Reanalyze; if duplicate injection RPD meet criteria, no further action required.</u> <u>(4) Field Samples: If RPD still outside of criteria, dilute sample re-analyze diluted sample with duplicate injections.</u></p>	<p><u>laboratory report narrative - potential sample matrix interference.</u></p>	
Initial Calibration	Laboratory Analytical Accuracy	<p><u>(1) Daily and prior to sample analysis.</u> <u>(2) Minimum calibration blank plus 3 calibration standards (multi-point) which may include the RL/LLCV standard; if</u></p>	No	<p><u>Recalibrate as necessary. Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as</u></p>	<p><u>Analyses cannot proceed without a valid</u>Suspend all analyses until <u>initial calibration meets criteria.</u></p>	<p><u>Group accepted additions of MA language in column 3, item 2.</u></p>

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		<p><u>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.</u> (3) Linear curveregression with correlation coefficient $r \geq 0.995$. Can use second order fit if $r \geq 0.995$. 4) Low level std at RL.</p>		<p><u>required by SW-846 7000/7010.</u></p>		
Initial Calibration Verification ("ICV")	Laboratory Analytical Accuracy	<p>(1) Daily, immediately after <u>each initial calibration and prior to sample analysis.</u> (2) 2nd<u>Prepared using standard source std different than use for initial calibration.</u> (3) <u>Concentration level near midpoint of curve.</u> (4) <u>Must contain all target analytes.</u> (5) Percent recoveries must be between 90-110% for each target</p>	No	<p>Re-calibrate/Re-analyze as required by method (1) <u>Reanalyze ICV; if acceptable, no further action required.</u> (2) <u>If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.</u></p>	Suspend all analyses until problem corrected and ICV meets criteria.	<p><u>Group accepted additions of MA language in column 3, items 3, 4, and 5 and in column 5.</u></p>

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Initial Calibration Blank ("ICB")	Evaluation of Laboratory Analytical Sensitivity (instrument drift, sensitivity, and & contamination.)	analyte. (1) Daily Immediately after ICV. (2) Target analytes must be ≤½ RL/LLOQ. (3) Matrix matched with standards and samples.	No	Re-calibrate/Re-analyze (1) Reanalyze ICB as; f acceptable, no further action required by method. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.	Group accepted additions of MA language in column 5.
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	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	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL/LLOQ in associated field samples.	Group accepted MA language.

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Required QA/QC Parameter <u>Column 1</u>	Data Quality Objective <u>Column 2</u>	Required Performance Standard <u>Column 3</u>	Required Deliverable <u>Column 4</u>	Recommended Corrective Action <u>Column 5</u>	Required Analytical Response Action <u>Column 6</u>	Rationale for Changes
		<u>target analytes.</u> <u>(4) Percent recoveries must be 70-130% for all target analytes.</u>		<u>samples, include explanation in laboratory report narrative no further action required.</u>		
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Every 10 samples and at <u>the</u> end of analytical <u>sequence</u> run. (2) Can be Prepared using same source or second source as initial calibration standards. (3) <u>Concentration level near midpoint of curve.</u> (4) <u>Must contain all target analytes.</u> (5) GFAAS: Recovery \pm 10% of true value, must use at least two burns with RPD <5%. (6) FLAAS: Recovery \pm <u>15</u> \pm 10% of true value.	No	Recalibrate/Re-analyze (1) <u>Reanalyze CCV; if acceptable, no further action required.</u> (2) <u>If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV_ unless (3) applies.</u> (3) <u>If recovery is high (>110%) and all associated sample results are non-detect no corrective action is required.</u>	Report non-conformances (3) <u>applies, include explanation in laboratory report narrative.</u>	<u>Group accepted MA language additions in column 3, items 2, 3, & 4 and column 5, items 2 &3, and column 6.</u>
Continuing Calibration Blank ("CCB")	<u>Evaluation of instrument drift, sensitivity, and</u>	(1) Every 10 samples following CCV <u>and at the end of the analytical run.</u>	No	Recalibrate/Re-analyze all samples since last compliant	<u>If (3) applies, include explanation in laboratory report narrative.</u>	<u>Group accepted MA language additions in column 3, item 1; column 5, items 1, 2,</u>

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Required QA/QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Recommended Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes
	contamination. <u>Laboratory Analytical Sensitivity (instrument drift & contamination)</u>	(2) Target analytes must be $\leq \frac{1}{2}$ RL/LLOQ. (3) Matrix matched with standards and samples.		CCV. <u>(1) Reanalyze CCB; if acceptable, no further action required.</u> <u>(2) If reanalysis is still outside criteria, recalibrate and reanalyze all associated samples since last compliant CCB- unless (3) applies.</u> <u>(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.</u>		<u>& 3, and column 6.</u>
Method Blank ("MB")	<u>Laboratory Contamination Evaluation Laboratory Method Sensitivity (contamination)</u>	(1) One per digestion batch of ≤ 20 field samples or every batch. <u>. If no digestion, ICB/CCB = method blank.</u>	Yes	Locate source of contamination and correct problem. Reanalyze method blank.	1) Report non-conformances in case narrative. <u>If (3) applies, include explanation in laboratory report</u>	<u>Group accepted MA language additions in column 3, items 1 & 2; column 5, items 2 & 3, and column 6.</u>

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	evaluation)	(2) <u>Must be digested with the samples using the same preparation method as the samples.</u> (3) Matrix specific and matrix matched. (4) Target analytes must be RL/LLOQ.		Reprepare samples unless all analyte concentration >10x method blank level (1) <u>Reanalyze MB; if acceptable, no further action required.</u> (2) <u>If reanalysis is still outside of criteria, re-digest and reanalyze MB and all associated samples in batch- unless (3) applies.</u> (3) <u>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.</u>	<u>narrative.</u>	
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.	Yes	Redigest and reanalyze all samples. (1) <u>Reanalyze LCS;</u>	Report non-conformances in laboratory report narrative.	<u>Group accepted MA language additions in column 3, items 1-5; and column 5, items 1-3.</u>

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		<p>(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.</p> <p>(3) Concentration levels for aqueous LCS near midpoint of curve.</p> <p>(4) Must contain all target analytes.</p> <p>(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.</p> <p>(6) Same source as</p>		<p>if acceptable, no further action required.</p> <p>(2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte; no corrective action required.</p> <p>(3) If LCS and LCSD are both outside of criteria, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.</p>		

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		initial calibration source. 2) Standard source can be initial calibration source.				
LCS Duplicate ("LCSD")	<u>Laboratory Analytical Accuracy & Precision</u>	(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	Yes (ONLY if no MD)	(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.	<u>Group accepted MA language</u>

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		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.				
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.	Yes ONLY when requested by data user (*If analyzed)	If recoveries >30% and LCS in limits note in narrative If MS recoveries <30%, reprepare and reanalyze samples 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	Report MS non-conformances in laboratory report narrative. If re-digested due to recoveries <30%, report both sets of sample/MS data.	Group accepted MA language additions in column 3, items 1-3; column 5; and column 6.

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		(4) Percent recoveries for all target analytes must be 75-125%.		<u>required.</u> (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.		
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. 2) For aqueous samples, if concentration >5x the RL, RPD ±20%, if concentration <5x RL,	Yes <u>ONLY when requested by data user</u> (*If analyzed)	Narrate non-conformances in laboratory report narrative. If LCS in criteria, narrate outliers. If LCS out of criteria, reprepare and reanalyze samples.	Report non-conformances in laboratory report narrative.	<u>Group accepted MA language additions in column 3, items 2 & 3; column 5. Removed RCP reference to LCS criteria. Not appropriate to compare LCS to MD since there are not analytes added to the duplicate.</u>

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		<p>difference \pm RL. 3) For solids if conc $>$ 5x RL, RPD \pm 35%. If conc. $<$ 5x RL, difference \pm 2x RL</p>				
<u>Dilution Test</u>	<u>Accuracy in Sample Matrix</u>	<p>(1) One per \leq 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 \pm 10% for all matrices.</p>	<p>Yes</p> <p>ONLY if project-specific MS requested by data user</p>	<u>Narrate.</u>	<u>Report non-conformances in laboratory report narrative.</u>	<u>Group accepted MA language.</u>
General Reporting Issues	N/A	<p>(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 \geq the</p>	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,	<u>Group accepted MA language additions in column 3, items 1, 3, & 5; and column 6.</u>

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		<p>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. <u>(5) Results must be reported with 2 or more "significant figures" if ≥ RL/LLOQ.</u> (6) Concentrations below the reporting limit should be reported as "ND" with the sample specific RL/LLOQ also reported.</p>			<p>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.</p>	

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Required QA/QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Recommended Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes
						<p>If the RL/LLOQ is estimated due to unacceptable recovery of the lowest standard, the RL/LLOQ has not been achieved; Question 5b of the "Reasonable Confidence Protocol Laboratory Analysis QA/QC Certification Form" must be answered "NO" and this must be addressed in the laboratory report narrative.</p> <p>Notes for Table 1A:</p> <p>* Refers to latest promulgated version of SW-846 Methods.</p> <p>_____ r = _____</p> <p>Correlation Coefficient RPD = Relative Percent Difference %RSD = Relative</p>

Connecticut ~~DEP~~DEEP RCPs
 Quality Assurance and Quality Control Requirements
~~Determination of Metals By SW-846~~by Method 7000 and 7010 (7000 Series), SW-846
 Methods for Flame and Graphite Furnace Atomic Absorption
 Version 23.0
 July 2006
 Month 2023

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Required QA/QC Parameter <u>Column 1</u>	Data Quality Objective <u>Column 2</u>	Required Performance Standard <u>Column 3</u>	Required Deliverable <u>Column 4</u>	Recommended Corrective Action <u>Column 5</u>	Required Analytical Response Action <u>Column 6</u>	<u>Rationale for Changes</u>
						<u>Percent Standard Deviation</u> _____ _____ N/A = Not Applicable

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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 compounds listed are readily determined by SW-846 Methods* 7000 and 7010. Most of the elements compounds 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

1.71.5.1 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. ~~DEP~~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 quality controlQC requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.86 Routine Reporting Deliverables for 7000 Series Methods

The following table (Table 1-24.0) 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	<u>Correlation coefficient must meet QA/QC</u>

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Parameter	Deliverable	Comments
		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.
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
Interference check analytical spike	NO	Note non-conformances in narrative.
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

1.86.1 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 [Reporting Limit RL/LLOQ](#)). The [reporting limit RL/LLOQ](#) for each [compound element](#) in each sample must be listed on the report and [take into account/consider](#) the exact sample mass, any dilution factors, percent moisture, etc.
- [Compounds Elements](#) detected above the [reporting limit RL/LLOQ](#) in blanks and found in samples, also above the [reporting limit RL/LLOQ](#) shall be flagged with a “B” suffix (e.g., 25B).
- All soil/sediment results shall be reported on a dry weight basis.

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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}	Preservative³	Holding Time⁴
<u>Aqueous Total Metals</u>	500 mL plastic or glass	Nitric Acid to pH <2	180 days
<u>Aqueous Dissolved Metals (Filtered)</u>	<u>500 mL plastic or glass</u>	<u>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</u>	<u>180 days</u>
Soil/Sediment samples.	250 mL plastic or glass jar with Teflon or plastic lined cap.	Cool to 4 ± 2° C	180 days
High Concentration Waste Samples	Collect in glass jar with Teflon or plastic lined cap.	Cool 4 ± 2° C.	180 days

¹The 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.

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

³If 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 6°C are acceptable.

⁴If 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).