

State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Determination of Trace Metals By SW-846 Method 6010

Inductively Coupled Plasma-Optical Emission Spectrometry

Version 3.0

Month 2023

Written by the Connecticut DEEP QA/QC Workgroup

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1.0	First version for publication	7/2005
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3.0	Updates to reflect CAM method updates to improve consistency between different states.	Month 2023

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ACRONYMN LIST

<u>ACRONYM</u>	<u>DEFINITION</u>
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
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectrometry
ICP-MS	ICP-Mass Spectrometry
ICSA/AB	Interelement interference check samples
ICV	Initial calibration verification
LCS/LCSD	Laboratory control sample / Laboratory control sample duplicate
LRD	Linear range determination
LLCV	Low-level calibration verification
LLOQ	Lower limit of quantitation
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
RSR/RSRs	Remediation Standard Regulations
SIC	Spectral-interference check
SRM	Standard reference material
QA	Quality assurance
QC	Quality control
µg/L	Microgram per liter
µm	Micrometer

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1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 6010

1.1 Method Overview

Inductively coupled plasma-optical emission spectrometry (“ICP-OES”) determines trace elements, including metals, in solution. The method is applicable for all the analytes listed in Table 1B as well as numerous other elements (refer to the element table, SW-846 Method 6010). All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, Toxicity Characteristic Leaching Procedure (“TCLP”) extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not require acid digestion. Samples that are not digested must either use an internal standard or be matrix matched with the standards. Refer to the Chapter 3.0, SW-846 and SW-846 Method 6010 for the appropriate digestion procedures.

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 6010

Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.1 of this method and Chapter 3 of SW-846 and Method 8000). When analyzing groundwater for dissolved metals, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multi-elemental determinations by ICP-OES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radiofrequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would degrade the analytical result.

The possibility of additional interferences named in Section 1.3 of this RCP should also be recognized and appropriate corrections made.

1.2.1 Sample Digestion

Except for filtered groundwater samples, analysis by SW-846 Method 6010 requires samples be acid digestion by one of the following methods:

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

SW-846 Digestion / Preparation Method	Matrix	Title/Description
3005	<u>Aqueous:</u> Surface Water/Groundwater	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
3010	<u>Aqueous:</u> 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	<u>Aqueous:</u> Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure Extracts/ aqueous waste	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
3031	<u>Solid:</u> Oily Waste/Tar/ Wax/Paint/ Petroleum Product	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040	<u>Solid:</u> Oil/Grease/Wax	Dissolution Procedure for Oils, Greases, or Waxes
3050	<u>Solid:</u> Soil/Sediment/ Sludges	Acid Digestion of Sediments, Sludges, and Soils
3051	<u>Solid:</u> Soil/Sediment/ Sludge/Oil	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
3052	<u>Solid:</u> 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 review upon request.		

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.

Refer to SW-846 Method 6010 for further information on method interferences and contamination. Several common interferences and corrective measures are summarized as follows.

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1.3.1 Spectral Interferences

Spectral interferences (described in SW-846 Method 6010) are caused by background emission, stray light from high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. Common spectral interferents, which cause suppression or enhancement of other analytes present in a sample, include aluminum, calcium, iron, and magnesium (though other analytes can also contribute to spectral interference and should be monitored – see SW-846 Method 6010 for a list of potential interelement interferents and the analytes that they affect).

Spectral interferences are minimized by using background corrections and interelement corrections, which can be applied either automatically by the ICP data system or manually by the spectroscopist. It is recommended that automatic (computerized) corrections for both background and interelement interferences be utilized during analysis of all samples under this protocol. If not, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results. The acceptable analysis of interference check samples (ICSA and ICSAB, see Table 1A for acceptance criteria) provides evidence of acceptable background and interelement corrections.

Two types of spectral-interference checks (“SIC”) are used. Individual element SICs are performed when the instrument is initially setup, and periodically (at least once every 6 months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that and interference corrections applied are still valid (refer to Table 1A).

1.3.2 Physical Interferences

Physical interferences (described in SW-846 Method 6010) are caused by sample viscosity and surface tension effects on the sample nebulization. Samples with high dissolved solids or high acid content can exhibit physical interference. Physical interferences can be minimized by diluting the sample, using an internal standard, or using a high solids nebulizer to introduce the sample to the ICP. The common use of mass flow controllers also minimizes the effects of physical interferences and improves ICP performance.

1.3.3 Memory Interferences

Memory interferences (described in SW-846 Method 6010) are caused by a high concentration sample contributing to signals measured in a subsequent sample. Optimizing rinse times between sample analyses (including both field and quality control (“QC”) samples) will minimize the potential for memory interferences.

1.3.4 Chemical Interferences

High salt concentrations (described in SW-846 Method 6010) are cause analyte signal suppression (e.g., seawater samples). Samples with high salt content can cause both physical interferences, by salting-over the torch, and significant suppression of analyte response in the sample. Samples should be diluted to bring the sodium (and other analytes) within the linear range of the instrument; note, however, this approach may raise the sample-specific reporting limit for analytes of interest above the Remediation Standard Regulations (“RSR”) criteria requirements. Therefore, it is recommended that alternate digestion/preparation methods be used to remove the salt interference prior to ICP analysis.

Analysis of blanks provides information about the presence of contaminants. When potential interferences or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the

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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 6010

1.4.1 Reporting Limits/Lower Limits of Quantitation for SW-846 Method 6010

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”), sample weight/volume, preparation factors, percent solids, dilution factors, etc., as required. Table 2.0 lists approximate RL/LLOQs for various matrices utilizing ICP-OES. Solid matrices in this table assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation

Matrix	Typical Reporting Limit
Aqueous	
Arsenic, Beryllium, Cadmium, Chromium, Lead, Silver, and Vanadium	5 to 10 µg/L
Antimony, Barium, Nickel, Selenium, Thallium, and Zinc	25 to 50 µg/L
Soil and Sediment	
Beryllium, Cadmium, Chromium, Silver, and Vanadium	0.5 to 1 mg/kg
Antimony, Arsenic, Barium, Lead, Nickel, Selenium, Thallium, and Zinc	1 to 7 mg/kg

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 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.

If Method 6020 is used to determine any analyte not listed in Section 1.5 of this RCP, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality using SW-846 Method 6020. Other elements and matrices may be analyzed by this method if performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest in the same manner as the listed elements and matrices.

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 ICP-OES 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 QC procedures for all analytical methods, including SW-846 Method 6010. These requirements ensure that each laboratory maintain a formal quality assurance (“QA”)

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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 6000 Series and include evaluation of calibrations and performance of sample analyses. Instrument QC and method performance requirements for the ICP-OES system may be found in SW-846 Method 6010.

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 is required for solid 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/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are generally discouraged. Field, rinsate, or other blanks should not be used for MS/MSD’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 this method. 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 6010 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. This information should be kept on-file at the laboratory.

Laboratories are required to generate laboratory specific performance criteria for LCS element recovery limits, MS/MSD element recovery and relative percent difference (“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 SW-846 Method 6010

Specific QA/QC requirements and performance standards for SW-846 Method 6010 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”

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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 10 years.

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Table 1A: Specific QA/QC Requirements and Performance Standards for Method 6010

Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
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 - 846 6010 and the applicable preparation method (SW-846 3000 Series).	No	Refer to SW-846 6010, the applicable preparation method requirements in SW-846 3000 series method, and Section 1.2.1 of this RCP.	NA	Group accepted MA language.
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwaters) and solid samples must be prepared (digested prior to analysis. See Section 1.2.1 of this RCP for preparation method references.	No	NA	NA	Group accepted MA language, changed preparation reference to Digestion Section in RCP
Linear Range Check	Laboratory Analytical Accuracy	(1) Check linear range annually (SW-846 6010). (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a minimum of different concentration standards across the range. See SW-846 Method 6010 for details. (3) At a minimum the LDR should be checked every year. A minimum of 3 different concentration standards across the ICP range one should be	No	If a linear range standard is not analyzed for any specific element, or fails, the highest standard in the calibration becomes the linear range. Concentrations above the linear range must be diluted.	Data must be on-file to document performance.	Group accepted additions of MA language in Column 3 items 3 & 4.

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
		near the upper limit of the range. (4) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed. Percent recoveries must be within 90 – 110 % for each target analyte.				
Initial Calibration (“ICAL”)	Laboratory Analytical Accuracy	(1) Following profiling and optimization of ICP; daily prior to sample analysis. (2) Minimum calibration blank plus one calibration standard for each target analyte or a multi-point curve. (3) Linear regression with correlation coefficient $r \geq 0.995$ non-linear regression may be used if $r^2 \geq 0.990$.	No	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6010.	Suspend all analyses until initial calibration meets criteria.	Group adopted 6010D correlation coefficient updates and accepted MA language.
Initial Calibration Verification (“ICV”)	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Prepare a second source standard different than the 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. (6) Must use at least two replicates with RPD <5%.	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.	Group accepted MA language in Column 3, items 3-5
Initial	Laboratory Analytical	(1) Immediately after ICV.	No	(1) Reanalyze ICB; if	Suspend all analyses until	Group adopted MA

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
Calibration Blank ("ICB")	Sensitivity (instrument drift & contamination)	(2) Matrix matched with standards and samples. (3) Target analytes must be <½ RL/LLOQ.		acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICB & ICB.	ICB meets criteria.	language in Column 3, item 3 and column 5, item 1.
Low-Level Calibration Verification ("LLCV")	Laboratory Analytical Sensitivity (verify 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 for each target analyte. 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) Percent recoveries must be 80-120% 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 the associated field samples.	Group accepted MA language in Column 3, item 2. The percent recovery range in Item 3 was adjusted to match method 6010D as it is more conservative. Group accepted MA language in Columns 5 & 6.
Interference Check Standards ("ICSA & ICSAB")	Laboratory Analytical Accuracy (Checks background points and	(1) Daily prior to sample analysis. (2) ICSA and ICSAB must contain known amounts of interfering analytes (see SW-	No	(1) Reanalyze ICSA/AB; if acceptable, no further action required.	Suspend all analyses until ICSA/AB meet criteria. If automatic (computerized) corrections for background and IECs are	Group accepted MA language in Column 3, item 4; Column 5, items 1 and Column 6.

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
	interelement interference corrections on instrument)	846 6010). (3) Percent recoveries must be 80-120% for all target analytes. (4) Non-spiked analytes in the ICSEA must be <2x RL/LLOQ.		(2) If ICSEA/AB is still outside of criteria, adjust interference corrections, background corrections, and/or linear ranges, as needed and reanalyze ICSEA/AB. (3) Recalibrate and reanalyze all samples since last compliant ICSEA/AB.	not used during analysis, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results.	
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Every 10 samples and at the end of an analytical run. (2) Prepared using same source as initial calibration standard (3) Concentration level near midpoint curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte, must use at least two replicates with RPD <5%.	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-detected, no corrective action required.	If (3) applies, include explanation in laboratory report narrative.	Group accepted MA language in Column 3, items 2-5; Column 5, items 1 & 3, and Column 6.
Continuing Calibration	Laboratory Analytical Sensitivity (instrument	(1) Every 10 samples following CCV and at the end of the	No	(1) Reanalyze CCB; if acceptable, no	If (3) applies, include explanation in laboratory	Group accepted MA language in Column

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
Blank ("CCB")	drift & contamination)	analytical run. (2) Target analytes must be <1/2 RL/LLOQ (positive and negative). (3) Matrix matched with standards and samples.		further action required. (2) If reanalysis is still outside of criteria, recalibrated and reanalyze all associated samples since last compliant CCV-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 in CCB, no corrective action required.	report narrative.	5, items 1 & 3, and Column 6.
Method Blank ("MB")	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 field samples, or every batch, whichever is more frequent prior to sample analysis and after calibration standards. (2) Must be digested with the samples using the same preparation method as the samples. (3) Target analytes must be ≤1/2 RL/LLOQ.	No	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrated and reanalyze all associated samples since last compliant MB-unless 3 applies. (3) If concentration of contaminant in MB is >RL/LLOQ but all associated sample	If (3) applies, include explanation in laboratory report narrative.	Group accepted MA language in Column 3, item 2; Column 5; and Column 6.

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
				<p>results are either non-detected or >10x concentration in MB, no corrective action required. (4) Locate source of contamination and correct problem.</p>		
Laboratory Control Sample (“LCS”)	Laboratory Method Accuracy	<p>(1) Every 20 field samples, or every batch, whichever is more frequent prior to sample analysis and after calibration standards. (2) Must be matrix-matched by digesting with the samples using the same preparation method. It is recommended a solid 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) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits for solids (the SRMs). (5) Matrix specific and matrix matched.</p>	Yes	<p>(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, redigest and reanalyze LCS/LCSD and all associated field samples in batch.</p>	Report recovery exceedances and non-conformances in laboratory report narrative.	Group accepted MA language in Column 3, items 2 & 3; Column 5, items 1-3.

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
Laboratory Control Sample Duplicate (“LCSD”)	Laboratory Analytical Accuracy & Precision	(1) One per digestion batch of ≤20 field sample ONLY if not performing project-specific Matrix Duplicate (MD). (2) Must be matrix-matched by digesting with the samples using the same preparation methods. Use of a SRM is recommended. (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 ONLY if no MD	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory report narrative.	Group accepted MA language
Matrix Spike (“MS”) (Site-specific)	Method Accuracy in Sample Matrix	(1) <u>Solid samples</u> - one per 20 field samples per matrix (at discretion of lab or at request of data user). <u>Aqueous Samples</u> - 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	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 action required. (3) If MS recovery is	Report MS non-conformances in laboratory report narrative. If re-digest due to recoveries <30%, report both sets of sample/MS data.	Group accepted MA language in Column 3, items 1-3; Column 5, items 1 & 2; and Column 6.

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
		midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be between 75-125%.		<30% and associated with non-detected results, re-digest (homogenize sample well) and reanalyze sample/MS pair.		
Matrix Duplicate ("MD") (site-specific)	Method Precision in Sample Matrix	(1) One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (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 request by data user.	If LCS in criteria, narrate outliers.	Report non-conformances in laboratory report narrative.	Group accepted MA language in Column 3, items 1-3
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. (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.	Group accepted MA language
Inter-element correction factors	Laboratory Method Accuracy	(1) Verify every six months. (2) Routine analysis of ICSA and ICSAB verifies inter-	No	Adjust software settings.	Data must be on-file to document performance.	Group maintained this language from the RCP to maintain

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Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes (this column will be removed from the final version)
("IEC")		element spectral interference corrections- see method for details				the intent of the method.
General Reporting Issues	N/A	<p>(1) Sample-specific RL/LLOQ for each target analyte using all preparation/dilution factors should be reported.</p> <p>(2) Concentrations below the reporting limit should be reported as "ND" with the sample specific RL/LLOQ also reported.</p> <p>(3) The laboratory must only report values \geq the sample-specific RL/LLOQ. The lab must report results for samples and blanks in a consistent manner.</p> <p>(4) Sample concentrations that exceed the LDR must be diluted and reanalyzed to fall within the linear dynamic range.</p> <p>(5) Results for soils/sediments must be reported on a dry-weight basis.</p>	N/A	N/A	<p>(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 the laboratory report narrative.</p> <p>(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.</p> <p>(3) If samples are digested and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative.</p> <p>(4) Narrate any additional method non-compliance or sample-specific anomaly.</p>	Group accepted MA language in Column 3, items 1-4; and Column 6 with the exception of language re: reporting estimated values <RL/LLOQ as values <RL/LLOQ should be reported as ND.
<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>						

Connecticut DEEP RCPs
Quality Assurance and Quality Control Requirements
Trace Metals by Method 6010, SW-846
Version 3.0
Month 2023

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1.5 Analyte List for SW-846 Method 6010

The DEEP analyte list for SW-846 Method 6010 is presented in Table 1B. The elements listed are readily determined by Method SW-846 6010. 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 6010

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 Method 6010

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 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 control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.6 Routine Reporting Deliverables for Method 6010

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 prior to analysis
Initial Calibration Verification Standard	NO	ICV must pass to continue analysis
Initial Calibration Blank	NO	Note non-conformances in laboratory report

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Parameter	Deliverable	Comments
		narrative
Low Level Calibration Verification	NO	Not required if low standard at RL/LLOQ
Continuing Calibration Verification	NO	CCV must pass to continue analysis
Continuing Calibration Blank	NO	Note non-conformances in laboratory report narrative
Interference Check Standards	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
Site Specific Matrix Spike/ Matrix Duplicate	YES (If requested)	Note non-conformances in laboratory report narrative
Linear Range Determination	NO	Data on file at laboratory
Inter-element Correction Factors	NO	Data on file at laboratory
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Serial Dilution Test	YES (if MS requested by data user)	Note non-conformances in laboratory report narrative
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 based upon the lowest calibration standard, 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 reporting limit, shall be flagged with a “B” suffix (e.g., 25B).
- All soil/sediment results shall be reported on a dry weight basis.
- Elements not listed in Table 1B and identified and quantified during analysis to evaluate inter-element correction factors need not be reported as contaminants.

1.7 Sample Containers, Preservations, and Holding Times

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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
Aqueous Total Metals	500 mL plastic ¹ or glass.	Nitric Acid to pH <2	180 days
Aqueous Dissolved Metals (Filtered)	500 mL plastic ¹ 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 ¹ 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).