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 6020

Inductively Coupled Plasma-Mass Spectrometry

Version 3.0

Month 2023

Written by the Connecticut DEEP QA/QC Workgroup

Revision	Comments	Date
1.0	First version for publication	7/05/2005
2.0	Final version based upon public comments	July 2006
3.0	Updates to reflect CAM method updates to improve consistency between different states.	Month 2023

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ACRONYNM LIST

ACRONYM	DEFINITION
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
IS	Internal standard
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 6020

1.1 Method Overview

Inductively coupled plasma-mass spectrometry ("ICP/MS") is applicable to the determination of sub-µg/L concentrations of many elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required. Refer to Chapter 3.0, SW-846 and Method 6020 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 6020

Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.1 of this RCP and Chapter 3.0 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.

Method 6020 describes the multi-elemental determination of analytes by ICP/MS in environmental samples. The instrument measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer.

The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed, and valid corrections applied, or the data flagged to indicate problems. Interference corrections must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

1.2.1 Sample Digestion

Except for filtered groundwater samples, analysis by Method 6020 requires samples be acid digestion by one of the following methods:

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

SW-846 Digestion/ Preparation Method	Matrix	Title/Description
3005	Aqueous: Surface Water/ Groundwater	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectrometry
3010	Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry

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SW-846 Digestion/ Preparation Method	Matrix	Title/Description
3015	Aqueous: Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Analysis by FLAA or ICP Spectrometry
3031	Solid: Oily Waste/Tar/ Wax/Paint/ Petroleum Product	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040	Solid: Oil/Grease/Wax	Dissolution Procedure for Oils, Greases, or Waxes
3050	Solid: Soil/Sediment/ Sludges	Acid Digestion of Sediments, Sludges, and Soils
3051	Solid: Soil/Sediment/ Sludge/Oil	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
3052	Solid: Biological Tissue/Oil/Ash Soil/Sediment/ Sludge	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
Digestion of samples is not required if the measured turbidity is <1.0 NTU. Laboratories must document turbidity readings for 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 6020 for further information on method interferences and contamination. Several common interferences and corrective measures are summarized as follows.

1.3.1 Isobaric Elemental Interferences

Isobaric elemental interferences are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio. A data system must be used to automatically correct for these interferences by determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal.

1.3.2 Isobaric Molecular Interferences

Isobaric molecular interferences and doubly-charged ion interferences are caused by ions consisting of more than one atom or charge, respectively. Isobaric interferences that affect ICP/MS results are identified in the

literature. A common example of isobaric molecular interference is from chloride on arsenic (specifically, $ArCl^+$ on 75As). Molecular isobaric interferences can be corrected using the natural isotope abundances from the literature. For most commercial ICP/MS instruments, this correction (based on the natural isotope abundances) is automatically performed by the data system. See Interferences Section of SW-846 Method 6020 for example isobaric corrections and for further information on isobaric interferences. The adequacy of corrections for isobaric interferences is partly evaluated using interference check solutions (ICSA and ICSAB, see Table 1A).

1.3.3 Physical Interferences

Physical interferences 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 using an internal standard ("IS"). An appropriate internal standard is required for each analyte determined by ICP/MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standards. Physical interferences can be minimized by using an IS. See Table 1A for further details on IS requirements.

1.3.4 Memory Interferences

Memory interferences are caused by a high concentration sample contributing to signals measured in a subsequent sample. Memory interferences can be minimized by using rinse blanks for appropriate rinse times between all sample analyses.

1.3.5 Chemical Interferences

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

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 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 6020

1.4.1 Reporting Limits/Lower Limits of Quantitation for Method 6020

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 various matrices utilizing ICP/MS. Solid matrices in this table assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation

Matrix	Typical Reporting Limit
Aqueous	
Antimony, Arsenic, Beryllium, Cadmium,	0.5 to 1 µg/L

Chromium, Lead, Silver, and Thallium	
Barium, Copper, Nickel, Selenium, Vanadium, and Zinc	1 to 10 µg/L
Soil and Sediment	
All RCP target analytes	0.05 to 0.5 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 Remediation Standard Regulation ("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/MS 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 6020. These requirements ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all inorganic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the instrument's operation may be found in SW-846 Chapter 3 and SW-846 6000 series and include evaluation of calibrations and performance of sample analyses. Instrument QC and method performance requirements for the ICP/MS system may be found in SW-846 Method 6020.

The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices 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 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 6020 for the procedure. The IDOC must include the following elements provided in Table 3.0:

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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, matrix spike/matrix spike duplicate 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 6020

Specific QA/QC requirements and performance standards for SW-846 Method 6020 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with “Reasonable Confidence” regarding the usability of analytical data to support environmental decisions. The concept of “Reasonable Confidence” is explained on the CT Department of Energy and Environmental Protection (“DEEP”) website.

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

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

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

3. Retain reported and unreported analytical data and information for a period of 10 years.

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Table 1A: Specific QA/QC Requirements and Performance Standards for Method 6020

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 SW-846 6020 and the applicable preparation method	No	Refer to SW-846 6020, the applicable preparation method requirements in SW-846 3000 series methods and Section 1.2.1 of this protocol.	NA	Group accepted MA language.
Preparation Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwater) and solid samples must be prepared (digested) prior to analysis. See Table 1.0 in Section 1.2 of this RCP for preparation method references.	No	NA	NA	Group accepted MA language.
Daily Performance Standard	Laboratory Analytical Accuracy	(1) Daily after tuning and prior to calibration. (2) Daily performance standard should be a 10µg/L standard of 3 or more elements representative of the analytical mass range. Analyze five replicates or five integrations. (3) Check sample introduction; sensitivity; oxide and double charge interferences. This is a multiple check on instrument performance suggested by the manufacturers. (4) Check manufacturer's requirements for acceptance criteria. (5) Criterion: RSD ≤5%, oxide and double charge levels ≤3%	No	Perform instrument maintenance and re-run standard.	If data are reported from an ICP/MS run in which the Daily Performance Standard exceeded the criterion, lab must narrate why the data are considered valid as sensitivity may be affected.	Group accepted additions of MA language in column 3, items 1 & 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)
Tuning	Laboratory Analytical Accuracy-Verify Operating Conditions	(1) Daily prior to calibration. (2) Tuning solution must contain elements representing all of mass regions of interest (See SW-846 6020). (3) Criteria- Mass calibration ≤0.1 amu difference from true amu; resolution <0.9 amu full width at 10% peak height. (4) RSD ≤5%	No	Re-optimize instrument operating conditions, re-tune.	Suspend all analyses until tuning non-compliance is rectified.	Group maintained existing RCP language as it matched MA language.
Initial Calibration	Laboratory Analytical Accuracy	(1) Daily following tuning and daily performance check of ICP/MS and prior to sample analysis. Also required if any modifications are made to the sample introduction system or detectors. (2) A minimum of 3 non-blank calibration points which may include the RL/LLCV standard. If LLCV standard is not included in calibration curve, then LLCV QC sample is required (see below). High level standard in calibration range. (3) Minimum of 3 integrations for calibration and sample analyses. (4) Linear regression with correlation coefficient r ≥0.995 and r^2 ≥ 0.990.	No	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6020.	Suspend all analyses until initial calibration meets criteria.	Group accepted of addition of MA language to Column 3 items 1 & 2. MA used LLCV acronym to follow EPA method. Column 3, Item 4: Did not adopt MA correlation values. Maintained r ≥ 0.995 from RCP and adopted r^2≥0.990 from EPA method 6020B. Column 6: removed RCP language referring to quadratic/non-linear calibration b/c labs generally do not use non-linear for metals.
Initial Calibration Verification ("ICV")	Laboratory Analytical Accuracy	(1) Daily immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. Matrix	No	(1) Reanalyze ICV: if acceptable, no further action required. (2) If reanalysis is still outside of criteria,	Suspend all analyses until ICV meets criteria.	Group accepted MA language additions in Column 3, items 3&4 and Column 5, item 2.

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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)
		matched. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be between 90-110%.		recalibrate and reanalyze ICV.		
Initial Calibration Blank ("ICB")	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Daily immediately after ICV. (2) Matrix matched with standards and samples. (3) Target analytes must be ≤ ½ RL/LLOQ (positive and negative).	No	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.	Group accepted MA language additions in Column 5, item 2 and Column 6.
Low-Level Calibration Verification ("LLCV")	Laboratory Analytical Sensitivity (verify low end of calibration range/verify RL/LLOQ)	(1) Daily prior to sample analysis after initial calibration. (2) Prepared using same source as initial calibration standards. (3) Concentration levels must be at the level of the RL/LLOQ for all target analytes. (4) 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 the criteria unless the concentrations of affected target analytes are >10x RL/LLOQ in the associated field samples.	Group accepted MA language additions in Column 3, items 2 & 4; Column 5, items 1-3; and Column 6. Group decided 80-120% recovery range rather than 90-110% range appropriate b/c in line with promulgated EPA Method.
Interference	Laboratory Analytical	(1) Daily prior to sample	No	This is a method	Narrate non-	Group accepted 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)
Check Standards ("ICSA" and "ICSAB")	Accuracy (verify adequacy of isobaric interference corrections)	analysis. (2) ICSA and ICSAB must contain known amounts of interfering analytes (See Method SW-846 6020). (3) Percent recoveries must be 80-120% for all target analytes.		requirement of SW-846 6020. No corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed.	conformance. If in compliance, then data are considered acceptable.	language additions in Column 3, item 3 and Column 5 & 6 for clarification within the intent of the method.
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Every 10 samples and at the end of analytical run. (2) Prepared using same source as initial calibration standard. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte.	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 additions in Column 3, items 2-4; Column 5, items 2&3; and Column 6.
Continuing Calibration Blank ("CCB")	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Every 10 samples following CCV and at the end of the analytical run. (2) Matrix matched with standards and samples. (3) Target analytes must be ≤½ RL/LLOQ (positive and negative).	No	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB-unless 3	If 3 applies, include explanation in laboratory report narrative.	Group accepted MA language additions in Column 3, item 3; Column 5, items 1 & 2; 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)
				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.		
Linear Range Standard	Laboratory Analytical Accuracy	(1) Performed at least annually. (2) Determine upper limit of linear dynamic range for each mass charge ratio utilized as per method SW-846 6020. (3) At a minimum the linear range should be checked every year. A minimum of 3 different concentration standards across the ICP range one should be near the upper limit of the range. (4) Should concentrations be reported above the curve, a daily linear range verification standard must be analyzed. Percent recoveries must be within 90 – 110 % for each target analyte.	No	Re-optimize instrument. Recalibrate as required by method. 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.	Report non-conformances.	The linear range study was not included in the CAM table. Linear range is required by EPA 6020B, group agreed this language should be maintained from RCP and edited language to improve clarity of purpose and process for linear range check.
Method Blank ("MB")	Laboratory Method Sensitivity (contamination evaluation)	(1) One per digestion of ≤ 20 field samples or every batch. (2) Must be digested with the samples using the same preparation method as the samples. (3) Target analytes must be ≤½ RL/LLOQ.	Yes	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, re-digest and reanalyze MB and all associated field	If (3) applies, include explanation in laboratory report narrative.	Group accepted MA language additions in Column 3, items 2&3; Column 5, item 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)
		(4) Matrix specific and matrix matched.		samples in batch – unless (3) applies. (3) If concentration of contamination in MB is > RL/LLOQ but all associated sample results are either non-detected or >10x concentration in MB, no corrective action required.		
Laboratory Control Sample ("LCS")	Laboratory Analytical Accuracy	(1) One per digestion of ≤20 field sample or one per batch. (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." A SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte, no corrective action required. (3) If LCS and LCSD are both outside of criteria, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.	Report non-conformances in laboratory report narrative.	Group accepted MA language additions in Column 3, items 2-5; Column 5, items 1&2.
LCS Duplicate	Laboratory Analytical	(1) One per digestion batch of	Yes (ONLY	(1) reanalyze LCSD;	Report recovery and	Group accepted 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)
("LCSD")	Accuracy & Precision	≤ 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. 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.	if no MD)	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.	RPD non-compliances in laboratory report narrative.	language for entire row.
Matrix Spike ("MS") (site-specific)	Method Accuracy in Sample Matrix	(1) One per digestion batch of ≤ 20 field samples per matrix (at discretion of lab or at request of data user). (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by data user	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required. (3) If MS recovery is <30% associated with non-detected results, re-digest (homogenize sample well) and reanalyze sample/MS pair.	Report MS non-compliance 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, items 1&2; 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)
				Report results and narrate.		
Matrix Duplicate ("MD") (site-specific)	Method Precision in Sample Matrix	(1) One per digestion batch of ≤ 20 field samples per matrix is strongly recommended (at discretion of lab or at request of data user). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by data user	If LCS in criteria, narrate outliers.	Report non-conformances in laboratory report narrative.	Group accepted MA language additions in Column 3, 2&3, and Column 6. Removed RCP language referring to acceptance criteria from past EPA method revisions to maintain consistency with promulgated method.
Dilution Test	Analytical Accuracy in Sample Matrix	(1) One dilution test per 20 samples per matrix- only if project-specific MS outside of acceptance limits and analyte concentration is *at minimum* >25x RL/LLOQ. (2) Perform 1:5 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 ±20% for all matrices.	No	Narrate.	Report non-compliance in laboratory report narrative.	Group accepted MA language additions in Column 3, items 2&3, Column 5, and Column 6. Group adopted EPA 6020B 25x RL/LLOQ requirement to maintain consistency with the intent of the Method.
Internal Standards ("IS")	Analytical Accuracy in Sample Matrix	(1) IS must be added to all field and QC samples. (2) Relative Intensity (RI) of IS must be 70-130% of IS in midpoint standard of the initial calibration curve. (3) Optimize mass and ionization potential match of IS to elements to be quantitated	No	(1) Perform dilution and reanalyze until IS criteria are met. (2) If IS in QC samples still not met, terminate analysis, re-calibrate, verify new calibration, and reanalyze affected	Report non-compliances in laboratory report narrative.	Group accepted MA language additions in Column 3, items 2&3, Column 5, item 3, 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)
		by ICP/MS. IS should generally be within 50 AMU of the element. See SW-846 6020 for recommended IS elements and further details.		samples. (3) If still not met for field samples, narrate non-compliance as matrix interference.		
General Reporting Issues	N/A	(1) Non-detected values must be reported with the sample-specific RL/LLOQ for each target analyte using all preparation/dilution factors. (2) The lab must only report values ≥ the sample-specific RL/LLOQ. (3) Sample concentrations that exceed the highest calibration standard must be diluted and reanalyzed to fall within the linear calibration range, measured at an alternate (less abundant) isotope to fall within the linear range when reanalyzed, or reported with narration. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to RSR regulatory standards. (5) Results must be reported with 2 or more “significant figures” if ≥ RL/LLOQ. (6) All concentration calculations must include appropriate interference corrections as described in SW-846 Method 6020, internal standard normalization, and appropriate compensations for	N/A	N/A	(1) The performance of dilutions must be documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target analytes, reasons for dilutions must be explained in the laboratory report narrative. (2) If samples are not preserved properly or are not received with an acceptable cooler temperature, not 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.	Group accepted MA language additions in Column 3, items 1, 3, & 5; Colum 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)
		isotopic abundances. (7) Concentrations below RL/LLOQ should be reported as "ND" with the sample specific RL/LLOQ also reported.				
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.						

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1.5 Analyte List for SW-846 Method 6020

The DEEP analyte list for SW-846 Method 6020 is presented in Table 1B. The elements listed are readily determined by Method 6020. 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 6020

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 6020

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

1.6 Routine Reporting Deliverables for Method 6020

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

Table 4.0: Report Deliverables

Parameter	Deliverable	Comments
Initial Calibration	NO	Correlation coefficient must meet QA/QC requirements
Initial Calibration Verification Standard	NO	ICV must pass
Initial Calibration Blank	NO	Note non-conformances in laboratory report narrative
Low Level Calibration Check Std	NO	Not required if low standard at RL/LLOQ

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Parameter	Deliverable	Comments
Continuing Calibration Verification	NO	CCV must pass
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.
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory

1.6.1 Reporting and Flagging of Results

The following rules apply to reporting results:

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

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

Table 5.0: Sample Containers, Preservation, and Holding Times

Matrix	Container[1,2]	Preservative[3]	Holding Time[4]
Aqueous Total Metals	500 mL plastic or glass.	Nitric Acid to pH <2	180 days
Aqueous	500 mL plastic or	Filter (0.45 µm) on site	180 days

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Matrix	Container[1,2]	Preservative[3]	Holding Time[4]
Dissolved Metals (Filtered)	glass	or at the laboratory (prior to acid preservation) within 24 hours of collection; then preserve with Nitric Acid to pH <2	
Soil/Sediment samples.	250 mL plastic or glass jar with Teflon or plastic lined cap.	Cool to 4 ± 2º C	180 days
High Concentration Waste Samples	Collect in glass jar with Teflon or plastic lined cap.	Cool 4 ± 2º C.	180 days

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

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

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

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