

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 ~~2~~3.0

[Month 2023](#)

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

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ACRONYM LIST

<u>ACRONYM</u>	<u>DEFINITION</u>
<u>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>ICP-AES</u>	<u>Inductively Coupled Plasma-Atomic Emission Spectrometry</u>
<u>ICP/MS</u>	<u>ICP/Mass Spectrometry</u>
<u>ICSA/AB</u>	<u>Interelement interference check samples</u>
<u>ICV</u>	<u>Initial calibration verification</u>
<u>IS</u>	<u>Internal standard</u>
<u>LCS/LCSD</u>	<u>Laboratory control sample / Laboratory control sample duplicate</u>
<u>LRD</u>	<u>Linear range determination</u>
<u>LLCV</u>	<u>Low-level calibration verification</u>
<u>LLOQ</u>	<u>Lower limit of quantitation</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>r²</u>	<u>Correlation coefficient</u>
<u>RL</u>	<u>Reporting limit</u>
<u>RPD</u>	<u>Relative percent difference</u>
<u>RSR/RSRs</u>	<u>Remediation Standard Regulations</u>
<u>SIC</u>	<u>Spectral-interference check</u>
<u>SRM</u>	<u>Standard reference material</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>

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 ~~a large number 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 ~~6020A~~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-846.1.1.2 ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability of Method 6020 in a multi-laboratory study on solid and aqueous wastes are listed below.

<u>Element</u>	<u>CAS No.</u>
Aluminum (Al)	7429-90-5
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-2
Barium (Ba)	7440-39-3
Beryllium (Be)	7440-41-7
Cadmium (Cd)	7440-43-9
Calcium (Ca)	7440-70-2
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Iron (Fe)	7439-89-6
Lead (Pb)	7439-92-1
Magnesium (Mg)	7439-95-4
Manganese (Mn)	7439-96-5
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Potassium (K)	7440-09-7
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Sodium (Na)	7440-23-5
Thallium (Tl)	7440-28-0

~~Vanadium (V) 7440-62-2~~
~~Zinc (Zn) 7440-66-6~~

1.4.1.2 Summary of SW-846 Method 6020

1.4.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.3.1 of this [methodRCP](#) 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.

1.4.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS in environmental samples. The [methodinstrument](#) 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 [correctioncorrections](#) must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

1.4.3.2.1 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 <u>Digestion/ Preparation Method</u>	<u>Matrix</u>	<u>Title/Description</u>
3005	Aqueous: Surface Water/ Groundwater	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. Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectrometry

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SW-846 Digestion/ Preparation Method	<u>Matrix</u>	<u>Title/Description</u>
3010 3010	<u>Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste</u>	Method prepares waste samples for total recoverable metal determinations by FLAA, ICPAES, 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 Spectrometry</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 or ICP Spectrometry</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,
3050	<u>Solid: Soil/Sediment/ Sludges</u>	Method prepares waste samples for total recoverable metals determinations by FLAA and
3051	<u>Solid: Soil/Sediment/ Sludge/Oil</u>	Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA,
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, GFAA, GFAA, ICPAES,

SW-846 <u>Digestion/ Preparation Method</u>	<u>Matrix</u>	<u>Title/Description</u>
Digestion of samples is not required if the measured turbidity is <1.0 NTU. Laboratories must document turbidity readings for review upon request.		

1.51.3 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.](#)

1.53.1 Isobaric Elemental Interferences

[Isobaric elemental interferences ~~in ICP-MS~~ are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio ~~\(m/z\)~~. A data system must be used to automatically correct for these interferences. ~~This involves by~~ determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. ~~Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.~~](#)

1.53.2 Isobaric Molecular Interferences

[Isobaric molecular interferences and doubly-charged ion interferences ~~in ICP-MS~~ are caused by ions consisting of more than one atom or charge, respectively. ~~Most isobaric~~ Isobaric interferences that ~~could affect ICP-/MS determinations have been~~ 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](#)

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~~partly evaluated using interference check solutions (ICSA and ICSAB, see Table 1A). Examples include $^{75}\text{ArCl}^+$ ion on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature (Reference 5), the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}\text{Ar}^{37}\text{Cl}^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}\text{Ar}^{35}\text{Cl}^+$ signal): Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = $(m/z$ 75 signal) - $(3.13)(m/z$ 77 signal) + $(2.73)(m/z$ 82 signal), where the final term adjusts for any selenium contribution at 77 m/z .~~

~~NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}\text{Se}^+$, (e.g., $^{81}\text{BrH}^+$ from bromine wastes). Similarly, Corrected cadmium signal (using natural isotopes abundances coefficient approximations) = $(m/z$ 114 signal) - $(0.027)(m/z$ 118 signal) - $(1.63)(m/z$ 108 signal), where last 2 terms adjust for any $^{114}\text{Sn}^+$ or $^{114}\text{MoO}^+$ contributions at m/z 114.~~

~~NOTE: Cadmium values will be biased low by this type of equation when $^{92}\text{ZrO}^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}\text{ZrOH}^+$) and indirect ($^{90}\text{ZrO}^+$) additive interferences when Zr is present.~~

~~NOTE: As for the arsenic equation above, the coefficients could be improved. The most appropriate coefficients for a particular instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision. The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary with operating conditions. If a correction for an oxide ion is based upon the ratio of parent to oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. For example, this type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed gas plasmas have been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.~~

~~1.5.3 Additionally, solid phase chelation may be used to eliminate isobaric interferences from both element and molecular sources. An on-line method has been demonstrated for~~

~~environmental waters such as sea water, drinking water and acid decomposed samples. Acid decomposed samples refer to samples decomposed by methods similar to methods 3052, 3051, 3050 or 3015. Samples with percent levels of iron and aluminum should be avoided. The method also provides a method for preconcentration to enhance detection limits simultaneously with elimination of isobaric interferences. The method relies on chelating resins such as iminodiacetate or other appropriate resins and selectively concentrates the elements of interest while eliminating interfering elements from the sample matrix. By eliminating the elements that are direct isobaric interferences or those that form isobaric interfering molecular masses, the mass region is simplified and these interferences can not occur. The method has been proven effective for the certification of standard reference materials and validated using SRMs. The method has the potential to be used on-line or off-line as an effective sample preparation method specifically designed to address interference problems.~~

~~1.5.4~~ 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 ⁶Li, ⁴⁵Sc, ⁸⁹Y, ¹⁰³Rh, ¹¹⁵In, ¹⁵⁹Tb, ¹⁶⁵Ho, and ²⁰⁹Bi. The lithium internal standard should have an enriched abundance of ⁶Li, 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. ~~associated with the sample nebulization and transport~~ Physical interferences can be minimized by using an IS. See Table 1A for further details on IS requirements.

~~processes as well as with ion transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When intolerable physical interferences are present in a sample, a significant suppression of the internal standard signals (to less than 30 % of the signals in the calibrations standard) will be observed. Dilution of the sample fivefold (1+4) will usually eliminate the problem.~~

~~1.5.5~~

1.3.4 Memory Interferences

Memory interferences ~~or carry-over can occur when there are large~~ are caused by a high concentration differences between samples or standards which are analyzed sequentially.

~~Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory sample contributing to signals measured in a subsequent sample. Memory interferences which are observed. They can be minimized by using rinse period between samples must be long enough to eliminate significant memory interference~~ blanks for appropriate rinse times between all sample analyses.

~~See SW-846 Method 6020A for references.~~

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.

1.6.4 Quality Control Requirements for SW-846 Method 6020

1.2 Reporting Limits for Method 6020

~~1.2.1 Reporting Limits (RL), sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the mass spectrometer, matrix and operating conditions. Table 2, SW-846 Method 6020A lists the recommended isotopic masses for quantitation.~~

~~If Method 6020 is used to determine any analyte not listed in Section 1.2, 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 (see Section 9.4 of Method 6020A). 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 (see Sec. 9.0 of Method 6020A).~~

~~1.2.2 Use of this method should be relegated to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.~~

~~An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ⁶Li, ⁴⁵Sc, ⁸⁹Y, ¹⁰³Rh, ¹¹⁵In, ¹⁵⁹Tb, ¹⁶⁵Ho, and ²⁰⁹Bi. The lithium internal standard should have an enriched abundance of ⁶Li, 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.~~

~~Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for trace metals are listed in Table 2A of this document. Moisture content of soils and sediments will raise the RL, 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.~~

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

<u>Matrix</u>	<u>Typical Reporting Limit</u>
<u>Aqueous</u>	
<u>Antimony, Arsenic, Beryllium, Cadmium, Chromium, Lead, Silver, and Thallium</u>	<u>0.5 to 1 µg/L</u>
<u>Barium, Copper, Nickel, Selenium, Vanadium, and Zinc</u>	<u>1 to 10 µg/L</u>
<u>Soil and Sediment</u>	
<u>All RCP target analytes</u>	<u>0.05 to 0.5 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/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.

~~1.3 General Quality Control Requirements~~

~~Each laboratory is required to operate a formal quality assurance program and be certified by the Connecticut Department of Public Health for the analysis performed. The minimum requirements include initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples (LCS) to assess precision and accuracy. The use of site specific matrix spikes and matrix duplicates is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making good decisions.~~

~~Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Section 1.5 and Table 1A. See Section 4.4.1 of SW-846 Chapter One and Section 8.0 of Method 6020A for the procedure. The Initial Demonstration of Proficiency must include the following elements:~~

1.4.2 General Quality Control Requirements ~~for Determinative Inorganic Methods~~

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 ~~quality control~~QC procedures for all ~~inorganic 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. Quality Control 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 One, Section 2.0,3 and SW-846 6000 series and include evaluation of calibrations and performance of sample analyses. Instrument ~~quality control~~QC and method performance requirements for the ICP-/MS system may be found in SW-846 Method ~~6020A, Sections 9.0 and 10.0, respectively.~~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 ~~be analyzed~~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 ~~LEP~~environmental professional (“EP”) to make ~~intelligent~~informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific

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matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field ~~blanks~~, rinsate, or other 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.

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:

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.

1.4.3-1.6.2 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 ~~DEP decisions~~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 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 performance~~ required corrective and analytical response actions for all non-conforming analytical performance standards; and prescribed in Table 1A for this test method; and

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

~~3. Adopt the reporting formats and elements specified in Section 1.7 of this method.~~

~~1.6.3 Site Specific Matrix Spike (MS) and Matrix Duplicate~~

~~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 LEP 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.~~

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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
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.	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
		(4) Check manufacturer's requirements for acceptance criteria. (5) Criterion: RSD ≤5%, oxide and double charge levels ≤3%				
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) (see Method 6020A Section 5.8). (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 $r \geq 0.995$	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 $r \geq 0.995$ from RCP and adopted $r^2 \geq 0.990$ from EPA method 6020B. Column 6: removed RCP language referring to quadratic/non-linear calibration b/c labs generally do not use

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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
		and $r^2 \geq 0.990$.				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 matched. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be between 90-110%.	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 additions in Column 3, items 3&4 and Column 5, item 2.
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 $\leq \frac{1}{2}$ 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. Group added <1/2 RL to match general lab accepted practice for blank criteria.
Low-Level Calibration Verification ("LLCV") Reporting Limit (RL) Calibration Check Standard	Laboratory Analytical Sensitivity (verify low end of calibration range/verify RL/LLOQ) Instrument sensitivity to support RL	(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. 3) Recovery $\pm 30\%$ of true value.	No	Recalibrate/reanalyze or narrate if affected elements >10x the RL (1) Reanalyze LLCV: if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are $\leq 10x$ RL/LLOQ in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes	Report non-conformances in narrative 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 70-130% range appropriate b/c in line with promulgated EPA Method.

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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
				are >10x RL/LLOQ in associated field samples, include explanation in laboratory report narrative; no further action required.		
Interference Check Standards ("ICSA" and "ICSAB")	Laboratory Analytical Accuracy <u>(verify adequacy of isobaric interference corrections)</u>	(1) Daily prior to sample analysis. (2) ICSA and ICSAB must contain known amounts of interfering analytes <u>(See Method SW-846 6020)</u> . (3) Percent recoveries must be 80-120% for all target analytes. 3) Recoveries for all analytes ±20% of true value or 2x the RL, whichever is greater. If analyte not present, its true value is zero.	No	<u>This is a method requirement of SW-846 6020. No corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed.</u> Method requirement. No corrective action.	Narrate non-conformance. <u>If in compliance, then data are considered acceptable.</u>	<u>Group accepted MA language additions in Column 3, item 3 and Column 5 & 6 for clarification within the intent of the method.</u>
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Every 10 samples and at the end of analytical run. <u>(2) Prepared using same source as initial calibration standard.</u> 2) Can be same source or second source. <u>(3) Concentration level near midpoint of curve.</u> <u>(4) Must contain all target analytes.</u> (5) Percent recoveries must be 90-110% for each target analyte.	No	(1) Reanalyze CCV; if acceptable, no further action required. <u>(2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV - unless (3) applies.</u> <u>(3) If recovery is high (>110%) and all associated sample results are non-detected, no corrective action required.</u>	<u>If 3 applies, include explanation in laboratory report narrative.</u>	<u>Group accepted MA language additions in Column 3, items 2-4; Column 5, items 2&3; and Column 6.</u>
Continuing Calibration Blank ("CCB")	<u>Laboratory Analytical Sensitivity (instrument drift & contamination)</u>	(1) Every 10 samples following CCV <u>and at the end of the analytical run.</u> (2) Matrix matched with	No	<u>(1) Reanalyze CCB; if acceptable, no further action required.</u> <u>(2) If reanalysis is still</u>	<u>If 3 applies, include explanation in laboratory report narrative.</u>	<u>Group accepted MA language additions in Column 3, item 3; Column 5, items 1 &</u>

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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
	Evaluation of instrument drift, sensitivity, and contamination.	standards and samples. (3) Target analytes must be $\leq \frac{1}{2}$ RL/LLOQ (positive and negative).		outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB-unless 3 applies. (3) If concentration of contaminant in CCB is > RL/LLOQ but all associated sample results are either non-detected or >10x concentration in CCB, no corrective action required.		2; Column 6.
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) <u>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.</u> (4) <u>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.</u>	No	Re-optimize instrument. Recalibrate as required by method. <u>If a linear range standard is not analyzed for any specific element, or fails, the highest standard in the calibration becomes the linear range.</u> <u>Concentrations above the linear range must be diluted.</u>	Report non-conformances.	<u>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.</u>
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	Yes	(1) Reanalyze MB; if acceptable, no further action required. (2) <u>If reanalysis is still outside of criteria, re-digest</u>	<u>If (3) applies, include explanation in laboratory report narrative.</u>	<u>Group accepted MA language additions in Column 3, items 2&3; Column 5, item 2; and Column 6.</u>

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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
		<p>samples. (3) Target analytes must be $\leq \frac{1}{2}$ RL/LLOQ. (4) Matrix specific and matrix matched.</p>		<p>and reanalyze MB and all associated field 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.</p>		
Laboratory Control Sample (“LCS”)	Laboratory Analytical Accuracy	<p>(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.</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, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.</p>	Report non-conformances in laboratory report narrative.	Group accepted MA language additions in Column 3, items 2-4; Column 5, items 1&2.
LCS Duplicate (“LCSD”)	Laboratory Analytical Accuracy & Precision	(1) One per digestion batch of ≤ 20 field samples ONLY if	Yes (ONLY if no MD)	(1) reanalyze LCSD; if acceptable, no further	Report recovery and RPD non-compliances	Group accepted MA language for entire

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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
		<p><u>not performing project-specific MD.</u> (2) <u>Must be matrix-matched by digesting with the samples using the same preparation method. Use of a SRM is recommended.</u> (3) <u>Concentration levels must be same as LCS.</u> (4) <u>Must contain all target analytes; analyze immediately following LCS.</u> (5) <u>Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.</u> (6) <u>RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.</u></p>		<p><u>action required.</u> (2) <u>If reanalysis is still outside of recovery criteria and LCS is in-control for same analyte, no corrective action required.</u> (3) <u>If LCSD and LCS are both outside of recovery criteria, re-digest and reanalyze LCS/LCSD and all associated field samples in batch.</u></p>	<p><u>in laboratory report narrative.</u></p>	<p><u>row.</u></p>
Matrix Spike ("MS") (site-specific)	Method Accuracy in Sample Matrix	<p>(1) <u>One per digestion batch of ≤ 20 field samples per matrix (at discretion of lab or at request of data user).</u> (2) <u>Concentration levels near midpoint of curve.</u> (3) <u>Must contain all target analytes.</u> (4) <u>Percent recoveries for all target analytes must be 75-125%.</u></p>	<p>Yes</p> <p><u>ONLY when requested by data user</u></p>	<p>(1) <u>Reanalyze MS; if acceptable, no further action required.</u> (2) <u>After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required.</u> (3) <u>If MS recovery is <30% associated with non-detected results, re-digest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.</u></p>	<p><u>Report MS non-compliance laboratory report narrative. If re-digested due to recoveries <30%, report both sets of sample/MS data.</u></p>	<p><u>Group accepted MA language additions in Column 3, items 1-3; Column 5, items 1&2; Column 6.</u></p>
Matrix Duplicate	Method Precision in Sample Matrix	<p>(1) <u>One per digestion batch of ≤ 20 field samples per matrix</u></p>	<p>Yes</p>	<p>If LCS in criteria, narrate outliers.</p>	<p>Report non-conformances in</p>	<p><u>Group accepted MA language additions in</u></p>

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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
("MD") (site-specific)		is strongly recommended (<u>at discretion of lab or at request of data user</u>). (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.	<u>ONLY when requested by data user</u>		laboratory report narrative.	<u>Column 3, 2&3.</u>
Dilution Test	Analytical Accuracy in Sample Matrix	(1) One dilution test per 20 samples per matrix- <u>only if project-specific MS outside of acceptance limits and analyte concentration is at minimum >25x RL/LLOQ. but only analytes >100x RL.</u> (2) Perform 1:5 dilution <u>on same sample used for MS/MD.</u> (3) %D of the sample and dilution results for target analytes at levels >50x RL/LLOQ must be $\pm 20\%$ for all matrices.	No	Narrate.	Report non-compliance in laboratory report narrative.	<u>Group accepted MA language additions in Column 3, items 2&3, Column 5, and Column 6.</u> <u>Group adopted EPA 6020B 25x RL/LLOQ requirement to maintain consistency with the intent of the Method.</u>
Internal Standards ("IS")	Analytical Accuracy in Sample Matrix	(1) IS must be added to all field and QC samples. (2) Relative Intensity (RI) <u>of IS must be 70-130% of IS in midpoint standard of the initial calibration curve.</u> (3) Optimize mass and ionization potential match of IS to elements to be quantitated <u>by ICP/MS. IS should generally be within 50 AMU of the element. See SW-</u>	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 samples. (3) <u>If still not met for field samples, narrate non-compliance as matrix</u>	Report non-compliances in laboratory report narrative.	<u>Group accepted MA language additions in Column 3, items 2&3, Column 5, item 3.</u>

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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
		846 6020 for recommended IS elements and further details.		interference.		
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.</p> <p>(2) The lab must only report values \geq the sample-specific RL/LLOQ.</p> <p>(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.</p> <p>(4) Results for soils/sediments must be reported on a dry-weight basis for comparison to RSR regulatory standards.</p> <p>(5) Results must be reported with 2 or more "significant figures" if \geq RL/LLOQ.</p> <p>(6) All concentration calculations must include appropriate interference corrections as described in SW-846 Method 6020, internal standard normalization, and appropriate compensations for isotopic abundances.</p> <p>(7) Concentrations below</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, not 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 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
		RL/LLOQ should be reported as "ND" with the sample specific RL/LLOQ also reported.				

Notes for Table 1A:

~~* Refers to latest promulgated version of SW 846 Method 6020. r = Correlation Coefficient RPD = Relative Percent Difference~~

~~%RSD = Relative Percent Standard Deviation N/A = Not Applicable~~

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 ~~Connecticut DEP (DEP)~~DEEP analyte list for SW-846 Method 6020 is presented in Table 1B. The ~~compounds~~elements listed are readily determined by Method 6020. Most of the ~~compounds~~elements listed have Connecticut ~~Remediation Standard~~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
Mercury	7439976
Nickel	7440020
Selenium	7782492
Silver	7440224
Thallium	7440280
Vanadium	7440622
Zinc	7440666

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

~~The Reporting Limit (RL) is based upon the lowest standard in the initial calibration. Alternatively, if the instrument does not allow for multi-standard calibration due to software limitations, the RL may be verified by analysis of a check standard at or below the RL. The found value must be within 30% of the true concentration.~~

~~It is the responsibility of the environmental professional or LEP to specify to the laboratory the detection limits required for the samples. In order to meet the limits it may be necessary to modify the analytical method by using increased sample volume or mass, concentration of the digestate, etc. In such cases the modifications must be noted in the narrative.~~

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1.86 Routine Reporting Deliverables for Method 6020

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	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
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

1.86.1 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 in blanks and found in samples, also above the reporting limit, shall be flagged with a “B” suffix (e.g., [25B](#)).

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- All soil/sediment results shall be reported on a dry weight basis.
- Elements not listed in Table 1B and identified and quantified ~~in the course of~~ 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}	Preservative³	Holding Time⁴
Aqueous <u>Total Metals</u>	500 mL plastic or glass.	Nitric Acid to pH <2	180 days
Aqueous <u>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
<p>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.</p> <p>²Plastic bottles must be acid rinsed and either high-density polyethylene, or Teflon.</p> <p>³<u>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 6°C are acceptable.</u></p> <p>⁴If mercury is to be determined, the holding time for mercury is 28 days from collection. <u>The preferred analytical method for mercury is SW-846 Methods 7470 and 7471 (cold vapor atomic absorption).</u></p>			