State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Determination of Trace Metals By SW-846 Method 6010

Inductively Coupled Plasma-Atomic Emission Spectrometry

Version 3.0

Month 2023

Written by the Connecticut DEEP QA/QC Workgroup

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

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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
LCS/LCSD	Laboratory control sample / Laboratory control sample duplicate
LRD	Linear range determination
LLCV	Low-level calibration verification
LLOQ	Lower limit of quantitation
MB	Method blank
MD	Matrix duplicate
mg/L	Milligram per liter
mg/kg	Milligram per kilogram
mL	Milliliter
MS	Matrix spike
nm	Nanometer
%R	Percent recovery
r/r^2	Correlation coefficient
RL	Reporting limit
RPD	Relative percent difference
RSR/RSRs	Remediation Standard Regulations
SIC	Spectral-interference check
SRM	Standard reference material
QA	Quality assurance
QC	Quality control
µg/L	Microgram per liter
µm	Micrometer

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

1.1 Method Overview

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

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

1.2 Summary of SW-846 Method 6010

Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.~~3~~1 of this method and Chapter 3 of SW-846 and Method 8000). When analyzing groundwater for dissolved metals, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

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

Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would ~~actually degrade the analytical result. The possibility of additional interferences named in Section 1.3 should also be recognized and appropriate corrections made; tests for their presence are described in Section 8.5 of Method 6010. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.~~degrade the analytical result.

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

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1.2.1 Sample Digestion

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

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

SW-846 Digestion / Preparation Method	Matrix	Title/Description
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 HNO_3 prior to metal determination.~~ Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
3010	Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ aqueous waste	~~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~~. Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy
3015	Aqueous: Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure Extracts/ aqueous waste	~~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.~~ Microwave Assisted Acid Digestion of Aqueous Samples and Extracts

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SW-846 Digestion / Preparation Method	**Matrix**	**Title/Description**
3031	Solid: Oily Waste / Tar / Wax/ Paint/ Petroleum Product	~~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.~~ Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040	Solid: Oil/Grease/Wax	~~Method prepares oily waste samples for determination of soluble metals by FLAA, GFAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content~~ Dissolution Procedure for Oils, Greases, or Waxes
3050	Solid: Soil/Sediment/ Sludges	~~Method prepares waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples.~~ Acid Digestion of Sediments, Sludges, and Soils

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SW-846 Digestion / Preparation Method	Matrix	Title/Description
3051	Solid: Soil/Sediment/ Sludge/Oil	~~Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid is added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination.~~ Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
3052	Solid: Biological Tissue/Oil/Ash/ Soil/Sediment/ Sludge	~~Method prepares siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total analysis by FLAA, CVAA, GFAA, ICPAES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis~~ 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.

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

1.3.1 Spectral Interferences

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

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

~~1.3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.~~

~~1.3.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately~~

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~~describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.~~

~~1.3.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction **require** the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2 of Method 6010. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.~~

~~1.3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2 of Method 6010, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2 of Method 6010. The interference effects must be evaluated for each individual instrument since the intensities will vary.~~

~~1.3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.~~

~~1.3.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences~~

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~~(Table 2, Method 6010) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.~~

~~1.3.1.7 Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.~~

~~1.3.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.~~

~~1.3.1.9 When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.~~

~~1.3.1.10 When interelement corrections are not used, verification of absence of interferences is required.~~

~~1.3.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument~~

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~~detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).~~

~~1.3.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is > 20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.~~

1.3.2 Physical Interferences

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

~~1.3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 8.5.1 of Method 6010 will help determine if a physical interference is present.~~

~~1.3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.~~

1.3.3 Memory Interferences

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

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~~1.3.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.~~

1.3.4 Chemical Interferences

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

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

~~1.3.5 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.~~

~~1.3.6 The dashes in Table 2 of Method 6010 indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.~~

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1.4 Quality Control Requirements for SW-846 Method 6010

~~1.1.1 Reporting Limits for Method 6010~~

~~Reporting Limits (RL), sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1, SW-846 Method 6010B lists the recommended analytical wavelengths and estimated instrumental detection limits (IDLs) for numerous elements, including all analytes listed in Table 1B, in clean aqueous matrices. Elements and matrices other than those listed in Table 1, SW-846 Method 6010B may be analyzed by this method if performance at the concentration ranges of interest (see Section 8.0, SW-846 Method 6010B) is demonstrated.~~

~~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 SW-846 Method 6010

The reporting limit ("RL"), or lower limit of quantitation ("LLOQ"), for an individual analyte is dependent on the concentration of the lowest non-zero standard in the initial calibration or the low-level calibration verification ("LLCV"), sample weight/volume, preparation factors, percent solids, dilution factors, etc., as required. Table 2.0 lists approximate RL/LLOQs for various matrices utilizing ICP-OES. Solid matrices in this table assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation

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

Moisture content of soils and sediments will raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RL/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet RSR criteria. To meet the limits, it may be necessary to modify the analytical method to improve sensitivity. In such cases, the modifications must be noted in the laboratory report narrative.

If Method 6020 is used to determine any analyte not listed in Section 1.5 of this RCP, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is

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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 ~~for Determinative Inorganic Methods~~

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

Refer to SW-846 Chapter One for general QC procedures for all ~~inorganic~~analytical methods, including SW-846 Method ~~6010B.~~ 6010. 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 ~~One, Section 2.0,~~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-~~AES~~OES system may be found in SW-846 Method ~~6010B, Sections 8.0 and 9.0, respectively~~6010.

~~1.1.2 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 6010 for the procedure. The Initial Demonstration of Proficiency must include the following elements:~~

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

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are generally discouraged. Field, rinsate, or other blanks should not be used for MS/MSD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative method being used. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform this method. These data must meet or fall within the performance standards as presented in Section 1.4 and Table 1A of this RCP. See SW-846 Chapter One and SW-846 Method 6010 for the procedure. The IDOC must include the following elements provided in Table 3.0:

Table 3.0: IDOC Requirements

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

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

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

1.4.3 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010

Specific QA/QC requirements and performance standards for SW-846 Method 6010 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with "Reasonable Confidence" regarding the usability of analytical data to support ~~DEP~~ environmental decisions. The concept of "Reasonable Confidence" is explained on the CT Department of Energy and Environmental Protection ("DEEP") website.

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

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

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, ~~compliance with~~required corrective actions and analytical response actions for all non-conforming analytical performance standards ~~prescribed in Table 1A for this test method~~; and

3. 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.~~

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~~1.4.3 Site Specific Matrix Spike (MS) and Matrix Duplicate (MD) Samples~~

~~It is strongly recommended that site specific MS/MD samples be analyzed from each site, and each matrix type sampled. Percent recovery data from site specific samples allow the EP 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 6010*

Required QC Parameter Column 1	Data Quality Objective Column 2	Required Performance Standard Column 3	Required Deliverable Column 4	Required Corrective Action Column 5	Required Analytical Response Action Column 6	Rationale for Changes
Initial Demonstration of Capability ("IDOC")	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedures in -846 6010 and the applicable preparation method (SW-846 3000 Series).	No	Refer to SW-846 6010, the applicable preparation method requirements in SW-846 3000 series method, and Section 1.2.1 of this RCP.	NA	Group accepted MA language.
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwaters) and solid samples must be prepared (digested prior to analysis. See Section 1.2.1 of this RCP for preparation method references.	No	NA	NA	Group accepted MA language, changed preparation reference to Digestion Section in RCP
Linear Range ~~Determination~~Check	Laboratory ~~Method~~ Analytical Accuracy	(1) Check linear range annually (SW-846 6010). ~~1) Performed at least annually~~ (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a	NO	If a linear range standard is not analyzed for any specific element, or fails, the highest standard in the calibration becomes the linear range. Concentrations above the linear range must be diluted. ~~N/A~~	Data must be on-file to document performance. Data must be on-file to document performance.	

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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
		minimum of different concentration standards across the range. See SW-846 Method 6010 for details. (3) At a minimum the LDR should be checked every year. A minimum of 3 different concentration standards across the ICP range one should be near the upper limit of the range. (4) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed. Percent recoveries must be within 90 – 110 % for each target analyte.~~2) Determine upper limit of linear dynamic range for each wavelength utilized as per method.~~				
Initial Calibration	Laboratory Analytical Accuracy	(1) Following profiling and optimization of ICP; daily prior to sample analysis. ~~1) Daily following instrument profiling and prior to sample analysis.~~ 2) Minimum of calibration blank plus one	NO	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6010. ~~Re-optimize instrument and recalibrate as necessary.~~	Suspend all analyses until initial calibration meets criteria. ~~Linear curve criteria~~	

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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
		calibration standard for each target analyte or a multi-point curve.. (3) Linear regression with correlation coefficient r ≥ 0.995 non-linear regression may be used if r^2 ≥ 0.990.~~3) Linear curve with "r" ≥ 0.995. Can use second order fit if r ≥ 0.995.~~			~~applicable to calibration curves with blank plus 2 or more calibration standards.~~	
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Prepare a second source standard different than the initial calibration. ~~2) 2nd source std~~ (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be between 90-110% for each target analyte. (6) Must use at least two replicates with RPD <5%.~~3) ICV ±10% of true value. Must use at least two replicates with RPD <5%~~	NO	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV. ~~Re-calibrate/Re-analyze ICV as required by method.~~	Suspend all analyses until ~~problem corrected and~~ ICV meets criteria.	
Initial Calibration Blank (ICB)	Evaluation of instrument drift,	(1) Immediately after ICV (2) Matrix matched with	NO	(1) Reanalyze ICB; if acceptable, no further action required.	Suspend all analyses until	

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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
	sensitivity, and contamination.	standards and samples. (3) Target analytes must be <½ RL/LLOQ.		(2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	ICB meets criteria.	
Low Level Calibration ~~Check Standard~~Verification (LLCV)	Laboratory Analytical Sensitivity (verify low-end of calibration range/verify RL/LLOQ)~~Instrument sensitivity to support RL~~	~~Only required if low calibration standard not at or below RL~~ 1) Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL/LLOQ for each target analyte. If initial calibration includes the RL/LLOQ as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Percent recoveries must be 80-120% for all target analytes.~~2) Std concentration ≤ RL for all analytes~~ ~~3) Recovery ±30% of true value except for antimony, arsenic, cobalt, and thallium which have a ± 50% limit.~~	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.~~Report non-conformances in narrative.~~	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL/LLOQ in the associated field samples.	
Interference Check Standards ("ICSA & ICSAB")	Laboratory Analytical Accuracy	(1) Daily prior to sample analysis. (2) ICSA and ICSAB	No	(1) Reanalyze ICSA/AB; if acceptable, no further action required.	~~Report non-conformances~~	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
	(Checks background points and interelement interference corrections on instrument)	must contain known amounts of interfering analytes (see SW-846 6010). (3) Percent recoveries must be 80-120% for all target analytes. (4) Non-spiked analytes in the ICSA must be <2x RL/LLOQ. ~~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.~~		(2) If ICSA/AB is still outside of criteria, adjust interference corrections, background corrections, and/or linear ranges, as needed and reanalyze ICSA/AB. (3) Recalibrate and reanalyze all samples since last compliant ICSA/AB. ~~May require adjustment of interelement,, correction factors, background correction and/or linear ranges~~	~~in narrative.~~ Suspend all analyses until ICSA/AB meet criteria. If automatic (computerized) corrections for background and IECs are not used during analysis, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results.	language in Column 3, item 4; Column 5, items 1 and Column 6.
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	1) Every 10 samples and at end of analytical ~~sequence~~run. (2) Prepared using same source as initial calibration standard ~~2) Can be same source~~	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	If (3) applies, include explanation in laboratory report narrative. ~~Report non-conformances~~	

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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
		~~or second source.~~ (3) Concentration level near midpoint curve. (4) Must contain all target analytes. (5) Precent recoveries mist be 90-110% for each target analyte, must use at least two replicates with RPD <5%. ~~3) Recovery ±10% of true value, Must use at least two replicates with RPD <5%.~~		non-detected, no corrective action required.~~Recalibrate/Re-analyze all samples since last compliant CCV~~	~~in narrative.~~ .	
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination) ~~Evaluation of instrument drift, sensitivity, and contamination.~~	1) Every 10 samples ~~immediately after~~following CCV. And at the end of the analytical run. 2) Target analytes must be <½ RL/LLOQ (positive and negative).~~2)) Matrix matched with standards and samples.~~ ~~3) ICB must be < RL~~	NO	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrated and reanalyze all associated samples since last compliant CCV-unless 3 applies. (3) If concentration of contaminant in CCB is >RL/LLOQ but all associated sample results are either non-detected or >10x concentration in CCB, no corrective action required. ~~Recalibrate/Re-analyze all samples since last compliant CCV~~	If (3) applies, include explanation in laboratory report narrative. ~~Report non-conformances in narrative.~~	
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)~~Laboratory Contamination~~	(1) Every 20 field samples, or every batch, whichever is more frequent prior to sample analysis and after	~~YES~~No	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrated and reanalyze all associated samples since last	If (3) applies, include explanation in laboratory report	

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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~~	calibration standards. ~~1) Digested every 20 or every batch, whichever is greater. If no digestion, ICB = blank~~ (2) Must be digested with the samples using the same preparation method as the samples. ~~2) Matrix specific and matrix matched~~ 3) Target analytes must be ≤ ½ ~~<~~RL		compliant MB-unless 3 applies. (3) If concentration of contaminant in MB is >RL/LLOQ but all associated sample results are either non-detected or >10x concentration in MB, no corrective action required. (4) Locate source of contamination and correct problem. ~~Locate source of contamination and correct problem. Reanalyze method blank. Reprepare samples unless all analyte concentration >10x method blank level~~	narrative. ~~1) Report non-conformances in case narrative.~~	
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	1) Every 20 samples or each batch, whichever is more frequent. prior to sample analysis and after calibration standards. ~~If samples not digested, ICV = LCS~~ (2) Must be matrix-matched by digesting with the samples using the same preparation method. It is recommended a solid SRM be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains the analytes of	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, redigest and reanalyze LCS/LCSD and all associated field samples in batch. ~~Redigest and reanalyze all samples.~~	Report recovery exceedances and non-conformances in laboratory report narrative. ~~Report non-conformances in narrative.~~	

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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
		interest at known concentrations and with 95% confidence limits. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits for solids (the SRMs). 2) Matrix specific (solid, aqueous, etc). ~~3) LCS recoveries ±20% for aqueous media and within vendor control (95% confidence limits) for solids.~~				
Laboratory Control Sample Duplicate ("LCSD")	Laboratory Analytical Accuracy & Precision	(1) One per digestion batch of ≤20 field sample ONLY if not performing project-specific Matrix Duplicate (MD). (2) Must be matrix-matched by digesting with the samples using the same preparation methods. Use of a SRM is recommended. (3) Concentration levels must be same as LCS.	Yes (ONLY if no MD)	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory report narrative.	Group accepted MA language

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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) 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.				
Matrix Spike ("MS") (Site-specific)~~Site Specific Matrix Spike~~	Method Accuracy in Sample Matrix ~~Accuracy in Sample Matrix~~	(1) Solid samples- one per 20 field samples per matrix (at discretion of lab or at request of data user). Aqueous Samples- one per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). ~~1) Every 20 samples or batch per matrix*~~ (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. ~~2~~4) Percent recoveries~~y~~	Yes ONLY when requested by data user* ~~(*If analyzed)~~	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action required. (3) If MS recovery is <30% and associated with non-detected results, re-digest (homogenize sample well) and reanalyze sample/MS pair.~~If recoveries >30% and LCS in limits note in narrative~~ ~~If MS recoveries <30%, reprepare and reanalyze samples~~	Report MS non-conformances in laboratory report narrative. If re-digest due to recoveries <30%, report both sets of sample/MS data. ~~Note outliers in narrative~~	

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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
		for all target analytes ~~limits~~ must be between 75-125%.				
Matrix Duplicate ("MD") (site-specific)~~Site Specific Matrix Duplicate (Lab may elect to analyze MSD instead)~~	Precision in Sample Matrix	(1) One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤20 for aqueous and ≤35 for solids.~~1) Every 20 samples or batch per matrix*~~ ~~2) For aqueous samples, if concentration >5x the RL, RPD <20%. If concentration <5x RL, difference ±RL.~~ ~~3) For solids if conc >5x RL, RPD <35%. If conc. < 5x RL, difference ± 2x RL~~	Yes ONLY when requested by data user * (~~*If analyzed)~~	If LCS in criteria, narrate outliers.	Report non-conformances in laboratory report narrative. ~~Note outliers in narrative~~	

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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
Inter-element correction factors (IEC's)	Laboratory Method Accuracy	1) Verify every six months 2) Routine analysis of ICSA and ICSAB verifies inter-element spectral interference corrections – See method for details	NO	Adjust software settings.	Data must be on-file to document performance.	
Dilution Test	Accuracy in Sample Matrix	(1) One per ≤20 field samples per matrix; only if project-specific MS requested and analyte concentration is >50x RL/LLOQ. (2) Perform 5x serial dilution on same sample. (3) %D of the Sample and dilution results for target analytes at levels >50x RL/LLOQ must be ±10% for all matrices.	Yes ONLY if project-specific MS requested by data user.	Narrate.	Report non-conformances in laboratory report narrative.	Group accepted MA language
General Reporting Issues	N/A	(1) Sample-specific RL/LLOQ for each target analyte using all preparation/dilution factors should be reported. (2) Concentrations below the reporting limit should be reported as "ND" with the sample specific RL/LLOQ also reported. (3) The laboratory must	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	Group accepted MA language in Column 3, items 1-4; and Column 6 with the exception of language

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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
		only report values ≥ the sample-specific RL/LLOQ. The lab must report results for samples and blanks in a consistent manner. (4) Sample concentrations that exceed the LDR must be diluted and reanalyzed to fall within the linear dynamic range. (5) Results for soils/sediments must be reported on a dry-weight basis.			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, note the non-conformances in the laboratory report narrative. (3) If samples are digested and/or analyzed outside of the holding time, note the non-conformances in the	re: reporting estimated values <RL/LLOQ as values <RL/LLOQ should be reported as ND.

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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
					laboratory report narrative. (4) Narrate any additional method non-compliance or sample-specific anomaly.	

~~Notes for Table 1A~~

~~* Refers to latest promulgated version of SW-846 Method 6010.~~ ~~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 6010

The ~~Connecticut DEP (DEP)~~DEEP analyte list for SW-846 Method 6010 is presented in Table 1B. The ~~compounds~~elements listed are readily determined by Method SW-846 6010. Most of the ~~compounds~~elements listed have Connecticut ~~Remediation Standard Criteria~~RSR criteria or are listed in the Approved Criteria for Additional Polluting Substances.

Table 1B: Analyte List for SW-846 Method 6010

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

1.5.1 Additional Reporting Requirements for SW-846 Method 6010

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. ~~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 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. taking into account all dilutions, sample weight/volume, etc. 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 EP 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~~

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~~increased sample volume or mass, concentration of the digestate, etc. In such cases the modifications must be noted in the narrative.~~

1.6 Routine Reporting Deliverables for Method 6010

The following table (Table ~~1.2~~4.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report all items in special circumstances.

Table 4.0: Report Deliverables

Parameter	Deliverable	Comments
Initial Calibration	NO	Correlation coefficient must meet QA/QC requirements prior to analysis
Initial Calibration Verification Standard	NO	ICV must pass to continue analysis
Initial Calibration Blank	NO	Note non-conformances in laboratory report narrative
Low Level Calibration Verification	NO	Not required if low standard at RL/LLOQ
Continuing Calibration Verification	NO	CCV must pass to continue analysis
Continuing Calibration Blank	NO	Note non-conformances in laboratory report narrative
Interference Check Standards	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report laboratory report narrative. Flag all positive sample results above RL/LLOQ with “B” flag.
Lab Control Sample/Lab Control Sample Duplicate	YES	Note non-conformances in laboratory report narrative
Site Specific Matrix Spike/ Matrix Duplicate	YES (If requested)	Note non-conformances in laboratory report narrative
Linear Range Determination	NO	Data on file at laboratory
Inter-element Correction Factors	NO	Data on file at laboratory
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Serial Dilution Test	YES (if MS requested by data user)	Note non-conformances in laboratory report narrative
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory

1.6.1 Reporting and Flagging of Results

The following rules apply to reporting results:

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- Non-Detects: Report all non-detects and results below the reporting limit as "ND" (Not Detected at the specified RL/LLOQ). The ~~reporting limit~~RL/LLOQ for each ~~compound~~element in each sample must be listed on the report and ~~take into account~~based upon the lowest calibration standard, the exact sample mass, any dilution factors, percent moisture, etc.

- ~~Compounds~~Elements detected above the ~~reporting limit~~RL/LLOQ in blanks and found in samples, also above the reporting limit, 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 ~~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]	Preservative[3]	Holding Time[4]
Aqueous Total Metals	500 mL plastic[1] or glass.	Nitric Acid to pH <2	180 days
Aqueous Dissolved Metals (Filtered)	500 mL plastic[1] or glass	Filter (0.45 µm) on site or at the laboratory (prior to acid preservation) within 24 hours of collection; then preserve with Nitric Acid to pH <2	180 days
Soil/Sediment samples.	250 mL plastic[1] or glass jar with Teflon or plastic lined cap.	Cool to 4 ± 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. If dissolved metals are to be determined, the samples must be filtered within 24 hours of collection through a 0.45 µm membrane filter prior to acidification.~~

[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).