State Of Connecticut
Department of 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 2.0
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Written by the Connecticut DEP QA/QC Workgroup

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1.0	First version for publication	7/2005
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Table of Contents

1.0 QA/QC Requirements for Method 6010

1.1 Method Overview

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable for all of the analytes listed in Table 1B as well as numerous other elements (refer to Table 1, SW-846 Method 6010B). All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, 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 Method 6010B 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.

1.1.1 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.

1.1.2 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:

Table 1.1 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

1.2 Summary of Method 6010

1.2.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.3 of this method and Chapter 3 of SW-846). When analyzing groundwater for dissolved metals, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

1.2.2 This method describes multielemental determinations by ICP-AES 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 radio-frequency 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.

1.2.3 Sample Digestion

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

SW-846 Method	Description
3005	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.
30l0	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.
3015	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.

SW-846 Method	Description (continued)
3031	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.
3040	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
3050	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.
3051	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.
3052	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

Note: Digestion of samples is not required if the measured turbidity is <1.0 NTU. Laboratories must document turbidity readings for inspection.

1.3 Method Interferences

1.3.1 Spectral interferences 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.

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 scans 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 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 (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 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 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.

1.3.4 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.

1.3.5 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.

1.4 Quality Control Requirements for SW-846 Method 6010

1.4.1 General Quality Control Requirements for Determinative Inorganic Methods

Refer to SW-846 Chapter One for general quality control procedures for all inorganic methods, including SW-846 Method 6010B. These requirements ensure that each laboratory maintain a formal quality assurance program and records to document the quality of all inorganic data.

Quality Control procedures necessary to evaluate the instrument's operation may be found in Chapter One, Section 2.0, and include evaluation of calibrations and performance of sample analyses. Instrument quality control and method performance requirements for the ICP-AES system may be found in SW-846 Method 6010B, Sections 8.0 and 9.0, respectively.

1.4.2 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 environmental professional ("EP") with "Reasonable Confidence" regarding the usability of analytical data to support DEP decisions.

While optional, parties electing to utilize these protocols will be assured that "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, as necessary, compliance with performance standards prescribed in Table 1A for this test method; and

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

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.

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

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Initial Calibration	Laboratory Analytical Accuracy	1) Daily following instrument profiling and prior to sample analysis. 2) Minimum of calibration blank plus one standard. 3) Linear curve with "r" $\geq$ 0.995. Can use second order fit if r $\geq$ 0.995.	NO	Re-optimize instrument and recalibrate as necessary.	Linear curve criteria applicable to calibration curves with blank plus 2 or more calibration standards.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	1) Daily immediately after calibration and prior to sample analysis. 2) 2nd source std 3) ICV ±10% of true value. Must use at least two replicates with RPD <5%	NO	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, sensitivity, and contamination.	1) Daily immediately after ICV. 2) Matrix matched with standards and samples. 3) ICB must be < RL	NO	Re-calibrate/Re-analyze ICB as required by method.	
Low Level Calibration Check Standard	Instrument sensitivity to support RL	Only required if low calibration standard not at or below RL 1) Daily prior to sample analysis 2) Std concentration $\leq$ RL for all analytes 3) Recovery ±30% of true value except for antimony, arsenic, cobalt, and thallium which have a ± 50% limit.	NO	Recalibrate/Narrate	Report non-conformances in narrative.
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	1) Every 10 samples and at end of analytical sequence. 2) Can be same source or second source. 3) Recovery ±10% of true value, Must use at least two replicates with RPD <5%.	NO	Recalibrate/Re-analyze all samples since last compliant CCV	Report non-conformances in narrative. .

Table 1A Specific QA/QC Requirements and Performance Standards for Method 6010* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Continuing Calibration Blank (CCB)	Evaluation of instrument drift, sensitivity, and contamination.	1) Every 10 samples immediately after CCV. 2)) Matrix matched with standards and samples. 3) ICB must be < RL	NO	Recalibrate/Re-analyze all samples since last compliant CCV	Report non-conformances in narrative.
Interference Check Standards (ICSA & ICSAB)	Laboratory Analytical Accuracy	1) Daily prior to sample analysis and at the end of the analytical sequence. 2) ICSA and ICSAB containing known amounts of analytes and/or interferents per method. 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	May require adjustment of interelement,, correction factors, background correction and/or linear ranges	Report non-conformances in narrative.
Method Blanks	Laboratory Contamination Evaluation	1) Digested every 20 or every batch, whichever is greater. If no digestion, ICB = blank 2) Matrix specific and matrix matched 3) Target analytes must be <RL	YES	Locate source of contamination and correct problem. Reanalyze method blank. Reprepare samples unless all analyte concentration >10x method blank level	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. If samples not digested, ICV = LCS 2) Matrix specific (solid, aqueous, etc). 3) LCS recoveries ±20% for aqueous media and within vendor control (95% confidence limits) for solids.	YES	Redigest and reanalyze all samples.	Report non-conformances in narrative.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 6010* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Site Specific Matrix Spike	Accuracy in Sample Matrix	1) Every 20 samples or batch per matrix* 2) Percent recovery limits must be between 75-125%.	Yes* (*If analyzed)	If recoveries >30% and LCS in limits note in narrative If MS recoveries <30%, reprepare and reanalyze samples	Note outliers in narrative
Site Specific Matrix Duplicate (Lab may elect to analyze MSD instead)	Precision in Sample Matrix	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* (*If analyzed)	If LCS in criteria, narrate outliers.	Note outliers in narrative
Linear Range Determination	Laboratory Method Accuracy	1) Performed at least annually 2) Determine upper limit of linear dynamic range for each wavelength utilized as per method.	NO	N/A	Data must be on-file to document performance.
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.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method 6010* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	N/A	1) The laboratory should report only concentrations detected above the sample specific RL. 2) Concentrations below the reporting limit (RL) should be reported as "ND" with the sample specific RL also reported 3) Dilutions: If analytes above linear range, dilute and reanalyze for those analytes. 4) Soils/sediments reported on a dry weight basis.	N/A	N/A	

Notes for Table 1A

* Refers to latest promulgated version of SW-846 Method 6010.
RPD = Relative Percent Difference
%RSD = Relative Percent Standard Deviation
r = Correlation Coefficient
N/A = Not Applicable

1.5 Analyte List for SW-846 Method 6010

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

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 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 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) 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.

1.6.1 Reporting and Flagging of Results

The following rules apply to reporting results:

Non-Detects: Report all non-detects and results below the reporting limit as "ND" (Not Detected at the specified Reporting Limit). The reporting limit for each compound in each sample must be listed on the report and take into account the exact sample mass, any dilution factors, percent moisture, etc.

Compounds detected above the reporting limit in blanks and found in samples, also above the reporting limit, shall be flagged with a "B" suffix (e.g. 25B).

All soil/sediment results shall be reported on a dry weight basis.

Elements not listed in Table 1B and identified and quantified in the course of analysis to evaluate inter-element correction factors need not be reported as contaminants.

Table 1.2 Report Deliverables

PARAMETER	DELIVERABLE	COMMENTS
Initial Calibration	NO	
Initial Calibration Verification Standard (ICV)	NO	ICV must pass
Initial Calibration Blank (ICB)	NO	Note non-conformances in narrative
Low Level Calibration Check Std	NO	Not required if low standard at RL
Continuing Calibration Verification (CCV)	NO	CCV must pass
Interference Check Stds (ICSA & ICSAB)	NO	
Method Blanks	YES	Note non-conformances in narrative. Flag all positive sample results above RL with "B" flag.
Lab Control Sample (LCS)	YES	Note non-conformances in narrative
Site Specific Matrix Spike/ Matrix Duplicate	YES (If requested)	Note non-conformances in narrative
Linear Range Determination	NO	Data on file at laboratory
Inter-element Correction Factors (IEC's)	NO	Data on file at laboratory
General Reporting Issues	YES	Note non-conformances in narrative
QA/QC Certification Form	YES	Signed by laboratory director or his/her designee.

Table 1B Analyte List for SW-846 Method 6010

ANALYTE	CAS NUMBER	NOTES
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	

Table 2A Sample Containers, Preservation, and Holding Times

MATRIX	CONTAINER	PRESERVATIVE	HOLDING TIME
Aqueous	1-liter plastic† or glass. (1)	Nitric Acid to pH <2	180 days (2)
Soil/Sediment samples.	250 mL plastic or glass jar with Teflon or plastic lined cap.	Cool to 4 ± 2º C	180 days (2)
High Concentration Waste Samples	Collect in glass jar with Teflon or plastic lined cap.	Cool 4 ± 2º C.	180 days (2)

Notes:
The number of sample containers is optional. Laboratories should supply enough containers to allow for any reanalysis or breakage.
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.
2. If mercury is to be determined, the holding time for mercury is 28 days from collection.
† Plastic bottles must be acid rinsed and either high-density polyethylene, or Teflon.