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 6020

Inductively Coupled Plasma-Mass Spectrometry
Version 2.0
July 2006

Written by the Connecticut DEP QA/QC Workgroup

Revision	Comments	Date
1.0	First version for publication	7/05/2005
2.0	Final version based upon public comments	July 2006

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1.0 QA/QC Requirements for Method 6020

1.1 Method Overview

1.1.1 All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, SW-846. Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-μg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required. Refer to Chapter 3.0, SW-846 and Method 6020A for the appropriate digestion procedures.

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

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

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Element	CAS No (continued)
Sodium (Na)	7440-23-5
Thallium (Tl)	7440-28-0
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

1.2 Reporting Limits for Method 6020

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

If Method 6020 is used to determine any analyte not listed in Section 1.2, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality (see Section 9.4 of Method 6020A). Other elements and matrices may be analyzed by this method if performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest in the same manner as the listed elements and matrices (see Sec. 9.0 of Method 6020A).

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

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standards.

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.

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1.3 General Quality Control Requirements

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

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

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

1.4 Summary of Method 6020

1.4.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see Section 1.2.3 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.

1.4.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS in environmental samples. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the

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resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer.

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

1.4.3 Sample Digestion
    Except for filtered groundwater samples, analysis by Method 6020 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.
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.

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SW-846 Method	Description
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.

1.5 Method Interferences

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

1.5.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $^{75}ArCl+$ ion on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature (Reference 5), the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal): Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = (m/z 75 signal) - (3.13) (m/z 77 signal) +

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(2.73) (m/z 82 signal),where the final term adjusts for any selenium contribution at 77 m/z,

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

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

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

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

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be used on-line or off-line as an effective sample preparation method specifically designed to address interference problems.

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

1.5.5 Memory interferences or carry-over can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

See SW-846 Method 6020A for references.

1.6 Quality Control Requirements for SW-846 Method 6020

1.6.1 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 6020. 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-MS system may be found in SW-846 Method 6020A, Sections 9.0 and 10.0, respectively.

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1.6.2 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6020

Specific QA/QC requirements and performance standards for SW-846 Method 6020 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the environmental professional and the LEP 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.

1.6.3 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 LEP to make intelligent decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field blanks, rinsate blanks, etc. should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD.

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

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Daily Performance Standard	Accuracy and Representativeness	1) Frequency – Daily after tuning and when instrument modifications performed. 2) Daily performance standard should be a 10 µg/L standard of 3 or more elements representative of the analytical mass range. Analyze five replicates or 5 integrations. 3) Purpose – check of sample introduction; sensitivity; oxide and double charge interferences. This is a multiple check on instrument performance suggested by the manufacturers. 4) Criterion: RSD ≤5%, oxide and double charge levels ≤ 3%	NO	If Daily Performance Standard exceeds criterion; perform instrument maintenance and re-run standard.	If data are reported from an ICP-MS run in which the Daily Performance Standard exceeded the criterion, lab must narrate why the data are considered valid.
Tuning	Accuracy – Verify Operating Conditions	1) Frequency – Daily prior to calibration 2) Tuning solution must contain elements representing all of the mass regions of interest (see Method 6020A Section 5.8) 3) Criteria: Mass Calibration ≤ 0.1 amu difference from true; Resolution < 0.9 amu full width at 10% peak ht; 4) Relative Standard Deviation (RSD) ≤ 5%	NO	Re-optimize instrument operating conditions, re-tune	Suspend all analyses until tuning non-compliance is rectified
Initial Calibration	Laboratory Analytical Accuracy	1) Daily following tuning of MS and prior to sample analysis. 2) Minimum of calibration blank plus three standards. Curve may include RL and LR standards. If not see below. 3) Minimum of 3 integrations for calibration and sample analysis 4) Linear curve with "r" ≥ 0.995.	NO	Re-optimize instrument and recalibrate as necessary.	1) Sample analysis cannot proceed without a valid initial calibration for all analytes being reported. 2) If linear regression not used (e.g. quadratic equation) must note all affected analytes in narrative.

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Table 1A Specific QA/QC Requirements and Performance Standards for Method 6020* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	1) Daily immediately after calibration and prior to sample analysis. 2) 2nd source std, matrix matched. 3) ICV ±10% of true value.	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.	
Reporting Limit (RL) Calibration Check Standard	Instrument sensitivity to support RL	Only required if RL standard is not part of the initial calibration curve. 1) Daily prior to sample analysis 2) Std concentration at RL for all analytes 3) Recovery ±30% of true value.	NO	Recalibrate/reanalyze or narrate if affected elements >10x the RL	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.	NO	Recalibrate/Re-analyze all samples since last compliant CCV	Report non-conformances in narrative.
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 (positive & negative drifts)	NO	If associated analyte results are >10x the CCB level, accept results and narrate. If not, recalibrate/re-analyze all samples since last compliant CCB	Report non-conformances in narrative.

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Table 1A Specific QA/QC Requirements and Performance Standards for Method 6020* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Linear Range (LR) Standard	Laboratory Analytical Accuracy	1) Performed at least annually 2) Determine upper limit of linear dynamic range for each mass charge ratio utilized as per method.	NO	Re-optimize instrument. Recalibrate as required by method.	Report non-conformances in narrative.
Interference Check Standards (ICSA & ICSAB)	Laboratory Analytical Accuracy	1) Daily prior to sample analysis . 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	Method requirement. No corrective action.	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	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.

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Table 1A Specific QA/QC Requirements and Performance Standards for Method 6020* (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	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
Dilution Test	Analytical Accuracy in sample matrix	1) One dilution test per 20 samples, but only analytes >100x RL. 2) Dilute digestate 1:5 3) % Difference ±15 %	NO	None	Note outliers in narrative
Internal Standards	Analytical Accuracy in Sample Matrix	1) IS must be added to all field and QC samples. 2) For field samples, relative intensity (RI) of IS must be 30-120% of initial calibration. 3) For QC samples, RI 80-120% 4) Optimize mass and ionization potential of IS to analytes to be quantitated – See Method 6020A of SW-846.	NO	Perform dilution and reanalyze until criteria is met. If still not met terminate analysis, recalibrate, and reanalyze samples.	Note outliers in narrative

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Table 1A Specific QA/QC Requirements and Performance Standards for Method 6020* (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. 5) All concentration calculations must include appropriate interference corrections as described in SW-846 Method 6020A, Section 4.2, internal standard normalization, and appropriate compensations for isotopic abundances (e.g., requirements for lead).	N/A	N/A	N/A

Notes for Table 1A:
* Refers to latest promulgated version of SW-846 Method 6020.   r = Correlation Coefficient   RPD = Relative Percent Difference
%RSD = Relative Percent Standard Deviation   N/A = Not Applicable

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1.7 Analyte List for SW-846 Method 6020

The Connecticut DEP (DEP) analyte list for SW-846 Method 6020 is presented in Table 1B. The compounds listed are readily determined by Method 6020. Most of the compounds listed have Connecticut Remediation Standard Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

1.7.1 Additional Reporting Requirements for SW-846 Method 6020

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP 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. Alternatively, if the instrument does not allow for multi-standard calibration due to software limitations, the RL may be verified by analysis of a check standard at or below the RL. The found value must be within 30% of the true concentration.

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

1.8 Routine Reporting Deliverables for Method 6020

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.

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1.8.1 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.

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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.

Connecticut DEP RCPs
Quality Assurance and Quality Control Requirements
Determination of Trace Metals By SW-846 Method 6020
Inductively Coupled Plasma-Mass Spectrometry
Version 2.0
July 2006

Table 1B Analyte List For SW-846 Method 6020

ANALYTE	CAS NUMBER	NOTES
Antimony	7440360	
Arsenic	7440382	
Barium	7440393	
Beryllium	7440417	
Cadmium	7440439	
Chromium (total)	7440473	
Copper	7440508	
Lead	7439921	
Mercury	7439976	
Nickel	7440020	
Selenium	7782492	
Silver	7440224	
Thallium	7440280	
Vanadium	7440622	
Zinc	7440666	

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