State Of Connecticut Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Determination of Metals By SW-846 7000 Series

Methods for Flame and Graphite Furnace Atomic Absorption Spectroscopy

Version 2.0 July 2006

Written by the Connecticut DEP QA/QC Workgroup

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

Table of Contents

1.0 QA/QC Requirements for 7000 Series Methods	3
1.1 Method Overview	3
1.2 Reporting Limits for 7000 Series Methods	4
1.3 General Quality Control Requirements	4
Table 1.1 IDOC Requirements	5
1.4 Summary of 7000 Series Methods	
1.5 Method Interferences	7
1.6 Quality Control Requirements for 7000 Series Methods	10
TABLE 1A Specific QA/QC Requirements and Performance Standards for	r the 7000
Series Methods*	12
1.7 Analyte List for SW-846 7000 Series Methods	16
1.8 Routine Reporting Deliverables for 7000 Series Methods	16
Table 1.2 Report Deliverables	17
Table 1B Analyte List For SW-846 7000 Series Methods	18
Table 2A Sample Containers, Preservation and Holding Times	18

1.0 QA/QC Requirements for 7000 Series Methods

1.1 Method Overview

- 1.1.1 Metals in solution may be readily determined by atomic absorption spectroscopy. The method is simple, rapid, and applicable to a large number of metals in drinking, surface, and saline waters and domestic and industrial wastes. While drinking water free of particulate matter may be analyzed directly, ground water, other aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes require digestion prior to analysis for both total and acid leachable metals. Analysis for dissolved elements does not require digestion if the sample has been filtered and acidified.
- 1.1.2 Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. In general it should be noted that the detection limits obtained using of flame atomic absorption spectroscopy (FAAS) do not meet the requirements of the Connecticut Remediation Standards, and are therefore unsuitable for use in obtaining "Reasonable Confidence". Analysis by graphite furnace atomic absorption spectroscopy (GFAAS) will meet the detection limits required, but is generally limited to determination of antimony, arsenic, lead, selenium, and thallium. In the environmental laboratory arena metals, with the exception of mercury, are determined by inductively coupled plasma spectroscopy (either ICP-AES Method 6010 or ICP-MS Method 6020). Mercury is typically determined by the cold vapor technique (Methods 7470 or 7471) the requirements of which are described in their own method. Mercury may also be determined by ICP-MS, Method 6020.
- 1.1.3 Detection limits by FAAS may also be extended through concentration of the sample and/or through solvent extraction techniques. Detection is somewhat dependent on equipment (such as the type of spectrophotometer and furnace accessory, the energy source, the degree of electrical expansion of the output signal), and are greatly dependent on sample matrix. Detection limits should be established, empirically, for each matrix type analyzed. When using GFAAS techniques, however, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each sample for interference effects and, if detected, treat them accordingly, using either successive dilution, matrix modification, or method of standard additions.

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

1.2 Reporting Limits for 7000 Series Methods

Reporting Limits (RL), sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the wavelength, spectrometer, matrix and operating conditions. Consult Methods 7000 and the appropriate 7000 series method for information regarding each specific analyte regarding the recommended analytical wavelengths and estimated instrumental detection limits (IDLs).

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.

1.3 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 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	Table 1A
Duplicate	
Other Instrument QC Samples	Table 1A

1.4 Summary of 7000 Series Methods

1.4.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation procedure (see 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. The following table summarizes the preparation 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.		
3010	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.		

SW-846 Method	Description (continued)		
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.

- 1.4.2 In FAAS a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp or an electrodeless discharge lamp is directed through the flame into a monochromator, and onto a detector that measures the amount of absorbed light. Absorption depends upon the presence of free unexcited ground-state atoms in the flame. Because the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectroscopy.
- 1.4.3 When using GFAAS, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atoms is vaporized and dissociated for absorption in the tube rather than the flame, the use of smaller sample volumes or detection of lower concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption, except that a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground-state element in the vapor. The metal atoms to be measured are placed in the beam of radiation by increasing

the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp or electrodeless discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

- 1.4.4 When using GFAAS all samples and standards require at least two "burns. Laboratories shall report the average of all burns.
- 1.4.5 When using GFAAS the following matrix modifiers are required:

Matrix Modifiers for GFAAS¹

Element	Matrix Modifier	
Antimony	Palladium + Magnesium Nitrate or Palladium + reducing agent such as	
	Citric Acid or Nickel Nitrate	
Arsenic	Palladium + Magnesium Nitrate or Palladium + reducing agent such as	
	Citric Acid or Palladium or Nickel Nitrate	
Lead	Palladium + reducing agent such as Citric Acid or Magnesium Nitrate	
	+ Phosphate or Lanthanum Nitrate ²	
Selenium	Palladium + Magnesium Nitrate or Nickel Nitrate	
Thallium	Palladium + Magnesium Nitrate	

- 1. See Method 3113A of <u>Standard Methods for the Examination of Water and Wastewater</u>, 20th ed. APHA-AWWA-WEF for details. If other elements are determined by GFAAS, the appropriate matrix modifier must be used.
- 2. See USEPA SOW ILMO4.0 Inorganic Contract Laboratory Protocols for details.

1.5 Method Interferences

- 1.5.1 Flame Atomic Absorption Spectroscopy
- 1.5.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or when the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome phosphate interference in magnesium, calcium, and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium.

- 1.5.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. Although complexing agents are employed primarily to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.
- 1.1.5.3 The presence of high dissolved solids in the sample may result in an interference from nonatomic absorbance such as light scattering. If background correction is not available, a nonabsorbing wavelength should be checked. Preferably, samples containing high solids should be extracted.
- 1.5.1.4 Ionization interferences occur when the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positively charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess (1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs.
- 1.5.1.5 Spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Interference can also occur when resonant energy from another element in a multielement lamp, or from a metal impurity in the lamp cathode, falls within the bandpass of the slit setting when that other metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.
- 1.5.1.6 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.
- 1.5.1.7 All metals are not equally stable in the digestate, especially if it contains only nitric acid, not nitric acid and hydrochloric acid. The digestate should be analyzed as soon as possible, with preference given to antimony, barium, molybdenum, silver, and tin.
- 1.5.2 Graphite Furnace Atomic Absorption Spectroscopy
- 1.5.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition

of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To verify the absence of matrix or chemical interference, all samples must be spiked at the instrument. Those samples which indicate the presence of an interference should be treated in one or more of the following ways:

- 1. Successively dilute and reanalyze the samples to eliminate interferences (detection limits requirements still must be met, if possible)
- 2. Modify the sample matrix either to remove interferences or to stabilize the analyte. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium phosphate to retain cadmium. The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.
- 3. Analyze the sample by method of standard additions while noticing the precautions and limitations of its use (see Step 8.7.2 of SW-846 Method 7000).
- 1.5.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, use either background correction or choose an alternate wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.
- 1.5.2.3 Continuum background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g., Zeeman background correction.
- 1.5.2.4 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.
- 1.5.2.5 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.
- 1.5.2.6 Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is

preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, a minimum amount should be used. This applies particularly to hydrochloric and, to a lesser extent, to sulfuric and phosphoric acids.

- 1.5.2.7 Carbide formation resulting from the chemical environment of the furnace has been observed. Molybdenum may be cited as an example. When carbides form, the metal is released very slowly from the resulting metal carbide as atomization continues. Molybdenum may require 30 seconds or more atomization time before the signal returns to baseline levels. Carbide formation is greatly reduced and the sensitivity increased with the use of pyrolytically coated graphite. Elements that readily form carbides include molybdenum and vanadium.
- 1.5.2.8 For comments on spectral interference, see 1.5.1.5.
- 1.5.2.9 Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as and acid rinsed prior to use. Pipet tips are a frequent source of contamination. If suspected, they should be acid soaked with 1:5 nitric acid and rinsed thoroughly with tap and reagent water. The use of a better grade of pipet tip can greatly reduce this problem. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become contaminated. As many as five to ten high-temperature burns may be required to clean the tube before use.

1.6 Quality Control Requirements for 7000 Series Methods

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 the 7000 series methods. 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 7000 and the specific analyte method.

1.6.2 Specific QA/QC Requirements and Performance Standards for the 7000 Series Methods

Specific QA/QC requirements and performance standards for the 7000 Series Methods 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.
- 1.6.3 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 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 the 7000 Series Methods*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverabl e	Recommended Corrective Action	Analytical Response Action
Sample Preparation	Accuracy and Representativeness	All samples must be digested prior to analysis with the exception of aqueous samples filtered and acidified for dissolved metals.	NO		
Initial Calibration	Laboratory Analytical Accuracy	 Daily and prior to sample analysis. Minimum of calibration blank plus three standards. Linear curve with "r" ≥ 0.995. Can use second order fit if r ≥ 0.995. Low level std at RL. 	NO	Recalibrate as necessary.	Analyses cannot proceed without a valid initial calibration.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	1) Daily immediately after calibration and prior to sample analysis. 2) 2 nd source std 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.	Daily immediately after ICV. Matrix matched with standards and samples. ICB must be < RL	NO	Re-calibrate/Re-analyze ICB as required by method.	
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	 Every 10 samples and at end of analytical sequence. Can be same source or second source. GFAAS: Recovery ±10% of true value, must use at least two burns with RPD <5%. FAAS: Recovery ±15% of true value. 	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 7000 Series Methods*

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.	Every 10 samples immediately after CCV. Matrix matched with standards and samples. ICB must be < RL	NO	Recalibrate/Re- analyze all samples since last compliant CCV	Report non-conformances in narrative.
Interference Check Analytical Spike (GFAAS Only)	Laboratory Analytical Accuracy	 Every sample must be spiked at instrument. Spike recovery must be ±15% If recovery out dilute and reanalyze or perform method of standard additions. 	NO	See Sections 8.6 and 8.7 of Method 7000.	Report non-conformances in narrative. Report all samples requiring MSA.
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< td=""><td>YES</td><td>Locate source of contamination and correct problem. Reanalyze method blank. Reprepare samples unless all analyte concentration >10x method blank level</td><td>1) Report non-conformances in case narrative.</td></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.

Table 1A Specific QA/QC Requirements and Performance Standards for 7000 Series Methods * (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
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) Standard source can be initial calibration source. 3) Matrix specific (solid, aqueous, etc). 4) 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.
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. If LCS out of criteria, reprepare and reanalyze samples.	Note outliers in narrative

Table 1A Specific QA/QC Requirements and Performance Standards for 7000 Series Methods * (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:

RPD = Relative Percent Difference

%RSD = Relative Percent Standard Deviation

r = Correlation Coefficient

N/A = Not Applicable

^{*} Refers to latest promulgated version of SW-846 Methods.

1.7 Analyte List for SW-846 7000 Series Methods

The Connecticut DEP (DEP) analyte list for SW-846 7000 Series Methods is presented in Table 1B. The compounds listed are readily determined by these methods. 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 7000 Series Methods

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 or by analysis of a low standard after calibration., taking into account exact sample weight or volume, any dilutions, percent moisture, etc. 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.

1.8 Routine Reporting Deliverables for 7000 Series Methods

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.

1.8.1 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.

Table 1.2 Report Deliverables

PARAMETER	DELIVERABLE	COMMENTS
Initial Calibration	NO	
Initial Calibration Verification	NO	ICV must pass
Standard (ICV)		
Initial Calibration Blank (ICB)	NO	Note non-conformances in narrative
Continuing Calibration	NO	CCV must pass
Verification (CCV)		
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
Interference check analytical	NO	Note non-conformances in narrative.
spike		
Site Specific Matrix Spike/ Matrix	YES (If	Note non-conformances in narrative
Duplicate	requested)	
Linear Range Determination	NO	Data on file at laboratory
Inter-element Correction Factors	NO	Data on file at laboratory
(IEC's)		
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 7000 Series Methods

ANALYTE	CAS	NOTES
	NUMBER	
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	Nitric Acid to pH <2	180 days
	glass (1)		
Soil/Sediment	250 mL plastic or	Cool to $4 \pm 2^{\circ}$ C	180 days
samples.	glass jar with		
	Teflon or plastic		
	lined cap.		
High	Collect in glass jar	Cool $4 \pm 2^{\circ}$ C.	180 days
Concentration	with Teflon or		
Waste Samples	plastic lined cap.		

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 $0.45~\mu m$ membrane filter prior to acidification.
- † Plastic bottles must be acid rinsed and either high density polyethylene or Teflon