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SW846-6010B Inductively Coupled Plasma-Atomic Emission Spectroscopy-EPA Method 6010B

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1 Scope and Application

Method SW846-6010B is used to determine metals in ground water, aqueous matrices, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments and other solid wastes by ICP optical spectroscopy. An aliquot of the sample is accurately measured and refluxed with Hydrochloric and Nitric acids to solubilize analytes. Samples analyzed by this method must first be prepared by one of the following methods: SW846-3010A, SW846-3015, SW846-3050B, SW846-3051. The sample is centrifuged or allowed to settle overnight prior to analysis.

This method is used to analyze the following metals:

Compound	CAS No.
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Calcium	7440-70-2
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-58-8
Iron	7439-89-6
Lead	7439-92-1
Magnesium	7439-95-4
Manganese	7439-96-5
Molybdenum	7439-98-7
Nickel	7440-02-0
Potassium	7440-09-7
Selenium	7782-49-2
Silver	7440-22-4

Sodium	7440-23-5
Strontium	7440-24-6
Thallium	7440-28-0
Tin	7440-31-5
Vanadium	7440-62-2
Zinc	7440-66-6

1.2 Restricted Procedure

This procedure is restricted to use by an analyst experienced in the operation of an optical ICP. Additionally, the analyst must complete the requirements of the GaEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GaEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

3 Interferences

3.1 Method interferences may be caused by background emission, stray light from the line emission from high concentration elements, spectral overlap from another analyte, or unresolved overlap of molecular band spectra.

3.1.1 Subtracting the background adjacent to the analytical wavelength peak compensates for stray light and background emission. This is automatically performed by the instrument's software for every analytical scan. Background correction locations are chosen to be free of off-line spectral interference or reflect the same change in the background intensity as does the analytical peak.

3.1.2 Spectral overlaps are compensated for by the use of interelement correction factors (IECs). IECs are determined by analyzing potential interfering analytes individually at 500 ppm concentrations and monitoring the apparent concentrations (both positive and negative) of all other analytes. The correction equations and coefficients are determined by the instrument software and are automatically applied for every analytical scan.

3.1.3 Physical interferences are effects associated with the sample nebulization and transport such as changes in viscosity and surface tension. Physical interferences are corrected by using a peristaltic pump for sample delivery and maintenance to keep the sample introduction system clean.

3.1.4 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally these interferences are not a major concern with ICP spectroscopy, but are minimized by matrix matching the standard matrix to the sample matrix and by acid digesting all samples. The presence of chemical interferences is monitored for by the use of matrix spikes and matrix spike duplicates.

3.1.5 Memory interferences occur when analytes from a previous sample contribute to the signal measured in a new sample. Memory effects usually arise from a dirty sample introduction system and are minimized by proper instrument maintenance. Memory interferences are detected by analyzing a blank after a sample with an analyte concentration exceeding the linear dynamic range and by periodic analysis of the continuing calibration blank (CCB).

4 Safety

Refer to Laboratory Chemical Hygiene Plan, online revision

5 Apparatus and Equipment

5.1 Computer controlled ICP spectrometer with background correction.
5.2 Peristaltic pump for sample delivery.
5.3 Computer controlled autosampler.
5.4 High purity Argon gas supply at 50 PSI.

6 Reagents and Standards

All reagents and standards must be logged into the standard log notebook, the container must be labeled with the standard number, expiration date, name, and initials of the person who prepared the solution.

6.1 1000 mg/kg Yttrium solution for the "bullet" test.
6.2 1% (v/v) nitric acid, reagent grade, as running buffer for drinking water samples and the calibration blank.
6.3 6% (v/v) nitric acid, 5% (v/v) hydrochloric acid mixture as running buffer for all non-drinking water samples and the calibration blank.
6.4 18MΩ water.
6.5 Calibration standards (described in section 8).
6.6 Initial Calibration Verification standard (ICV) (described in section 8).

7 Samples Collection

Water samples and liquid waste samples for analysis by method 6010B are collected in a 500 ml narrow mouth plastic (HDPE) bottle. Samples are preserved with sufficient HNO_3 to lower the pH below 2. Solid samples are collected in 500 ml wide mouth HDPE bottle and preserved by cooling to 4 Cº ± 2 Cº. Samples must be analyzed within 180 days of collection.

8 Calibration

8.1 Calibration Standards: The ICP-OES is calibrated daily prior to analysis. The calibration curve consists of two calibration standards; a correlation coefficient of 0.995 is required or the instrument is recalibrated.

8.2 Calibration Verification: The calibration is verified by analyzing the initial calibration verification (ICV) standard. The ICV recoveries must by between 90% and 110% of the true value or the instrument must be recalibrated.

8.3 Continuing calibration check (CCC): after analysis of the ICV and after every ten samples, a continuing calibration check (CCC) and continuing calibration blank (CCB) are analyzed. The initial CCC recoveries must be between 90% and 110% of the true value, subsequent recoveries must be between 90% and 110% of the true value. All CCB concentrations must be below the reporting limit. If either the CCC or CCB fails, the instrument must be recalibrated and all samples analyzed after the last successful CCC and CCB.

8.4 Interference check: Must be reanalyzed. All spiked elements must have recoveries between 80% and 120% of the true value, unspiked elements must have the absolute value of the apparent concentration less than the reporting limit. The Interference Check Standard (INT-AB) concentrations are as follows:

Table 8.4 Interference Check Standard concentrations.

Element	**Solids Concentration (mg/kg)**	**Liquids Concentration (mg/L)**
Aluminum	500	500
Antimony		
Arsenic		
Barium	1	1
Beryllium	1	1
Cadmium	2	2
Calcium	500	500
Chromium	1	1
Cobalt	1	1
Copper	1	1
Iron	200	200
Lead	2	2
Magnesium	500	500
Manganese	1	1
Molybdenum		
Nickel	2	2

Element	**Solids Concentration (mg/kg)**	**Liquids Concentration (mg/L)**
Potassium		
Selenium		
Silver	2	2
Sodium		
Strontium		
Thallium		
Tin		
Vanadium		
Zinc	2	2

9 Quality Control - Refer to Table 13.1 for Reporting Limits (PQLs), Table 13.2 for Quality Control Acceptance Criteria, and Table 13.3 for Quality Control Procedures associated with this method.

10 Procedure

10.1 All samples analyzed by method SW846-6010B must be prepared for analysis by the appropriate method prior to analysis.
10.2 Start the instrument and run for at least 45 minutes to warm up.
10.3 Profile the instrument.
10.4 Calibrate the instrument using first the low standard (calibration blank) followed by the high standard.
10.5 Analyze the ICV.
10.6 Analyze the CCC and CCB. A CCC and CCB must be analyzed at the start of each batch, after every ten samples in a batch, and as the last samples analyzed in a batch. The CCC and CCB do not count towards the sample total in a batch.
10.7 Determine the instrument detection limits (IDLs).
10.7.1 Change the number of repetitions from 3 to 10.
10.7.2 Aspirate the running matrix and analyze for 10 repetitions.
10.7.3 Calculate the IDL for each element by multiplying the standard deviation (SDEV) by 3. If any of the IDLs exceed the RL end the analysis, correct the problem, and start from step 10.4.
10.7.4 Change the number of repetitions back to 3.
10.8 Analyze the instrument detection limit standard.
10.9 Analyze the interference check standard INT-AB..
10.10 Analyze the matrix blank. Some batches (such as TCLP) may have more than one matrix blank per batch. All matrix blanks must be analyzed and used for QC purposes.

10.11 Analyze the LCS and LCSD.
10.12 Begin analyzing samples.
10.12.1 A matrix spike must be analyzed for every ten samples in a batch.
10.12.2 A matrix spike duplicate must be analyzed for every ten samples in a batch.
10.12.3 TCLP batches will have a sample duplicate in addition to the spike duplicate. The purpose of the sample duplicate is to provide QC for the tumbling process.

11.1 Evaluation of the Linearity of the Initial Calibration
Since a two point calibration is used, all calibrations curves are linear.

11.2 Sample Concentration

Liquid Sample Concentration: Concentration = CD_f Where C = concentration from instrument D_f = dilution factor $D_f = \frac{D}{S}$ Where D = dilution volume in liters. S = Sample aliquot volume in liters.	Concentration = $\frac{CVD_f}{DW}$ Where C = concentration from instrument in mg/L. V = final digestion volume in L. D_f = dilution factor. DW = sample weight in kg after drying to constant weight at 60°C ± 5°C.
Use the following formula if there is insufficient sample to dry for digestion: Concentration = $\frac{CVD_F P_s}{WW}$ Where C = concentration from instrument in mg/L V = final digestion volume in L. D_f = dilution factor. WW = wet weight of sample (not dried). P_s = percent solids Percent Solids = $\frac{DW}{WW}$ Where DW = sample weight in kg after drying to constant weight at 60°C ± 5°C WW = wet weight of sample (not dried).	Waste Concentration Calculation: Concentration= $\frac{CVD_F}{WW}$ Where C= concentration from instrument in mg/L V = final digestion volume in L. D_f= dilution factor WW = wet weight of sample.

Aqueous sample results are expressed in ug/L.
TCLP extracts results are expressed in mg/L.
Waste sample results are expressed in mg/kg wet weight

Solid sample results are expressed in mg/kg dry weight

12 References

12.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (*SW-846)*;* U.S. EPA Office of Research and Development: Cincinnati, OH, 1988, Revision 2, 1995.

13 Practical Quantitation Limits (PQLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 1 RLs for EPA Method SW846-6010B

Method	Analyte	Matrix (Aqueous and Extracts)		Matrix (Soils, Solids and Wastes)	
		RL	Unit	RL	Unit
6010B	Aluminum	0.06	mg/L	0.06	mg/kg
	Antimony	0.12	mg/L	0.12	mg/kg
	Arsenic	0.08	mg/L	0.08	mg/kg
	Barium	0.01	mg/L	0.01	mg/kg
	Beryllium	0.01	mg/L	0.01	mg/kg
	Bismuth	0.09	mg/L	0.09	mg/kg
	Cadmium	0.01	mg/L	0.01	mg/kg
	Calcium	1.0	mg/L	1.0	mg/kg
	Chromium	0.02	mg/L	0.02	mg/kg
	Cobalt	0.01	mg/L	0.01	mg/kg
	Copper	0.02	mg/L	0.02	mg/kg
	Iron	0.02	mg/L	0.02	mg/kg
	Lead	0.09	mg/L	0.09	mg/kg
	Magnesium	1.0	mg/L	1.0	mg/kg
	Manganese	0.01	mg/L	0.01	mg/kg
	Molybdenum	0.04	mg/L	0.04	mg/kg
	Nickel	0.02	mg/L	0.02	mg/kg
	Potassium	5.0	mg/L	0.05	mg/kg
	Selenium	0.19	mg/L	0.19	mg/kg
	Silver	0.01	mg/L	0.01	mg/kg
	Strontium	0.01	mg/L	0.01	mg/kg
	Thallium	0.20	mg/L	0.02	mg/kg
	Titanium	0.01	mg/L	0.01	mg/kg
	Vanadium	0.01	mg/L	0.01	mg/kg
	Zinc	0.02	mg/L	0.02	mg/kg

Table 13.1 RLs for EPA Method SW846-6010B for Target Analyte List (TAL)

Method	Analyte	Matrix (Water)		Matrix (Solids andWastes)	
		RL	Unit	RL	Unit
6010B	Silver			10000	ug/kg
	Aluminum	200	ug/L	200000	ug/kg
	Arsenic			80000	ug/kg
	Barium			200000	ug/kg
	Beryllium			5000	ug/kg
	Calcium	5000	ug/L	5000000	ug/kg

Table 13.1 RLs for EPA Method SW846-6010B for Target Analyte List (TAL)

Method	Analyte	Matrix (Water)		Matrix (Solids andWastes)	
		RL	Unit	RL	Unit
6010B	Cadmium			5000	ug/kg
	Cobalt			50000	ug/kg
	Chromium			100000	ug/kg
	Copper			25000	ug/kg
	Iron	100	ug/L	100000	ug/kg
	Potassium	5000	ug/L	5000000	ug/kg
	Magnesium	5000	ug/L	5000000	ug/kg
	Manganese	15	ug/L	15000	ug/kg
	Sodium	5000	ug/L	5000000	ug/kg
	Nickel			40000	ug/kg
	Lead			90000	ug/kg
	Antimony			60000	ug/kg
	Selenium			190000	ug/kg
	Thallium			200000	ug/kg
	Vanadium			50000	ug/kg
	Zinc			20000	ug/kg

Table 13.2 Acceptance Criteria for EPA Method SW846-6010B

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)	Accuracy Solids (%R)	Precision Solids (RPD)
6010B	Aluminum	85-115	≤ 15	85-115	≤ 15
	Antimony	85-115	≤ 15	85-115	≤ 15
	Arsenic	85-115	≤ 15	85-115	≤ 15
	Barium	85-115	≤ 15	85-115	≤ 15
	Beryllium	85-115	≤ 15	85-115	≤ 15
	Bismuth	85-115	≤ 15	85-115	≤ 15
	Cadmium	85-115	≤ 15	85-115	≤ 15
	Calcium	85-115	≤ 15	85-115	≤ 15
	Chromium	85-115	≤ 15	85-115	≤ 15
	Cobalt	85-115	≤ 15	85-115	≤ 15
	Copper	85-115	≤ 15	85-115	≤ 15
	Iron	85-115	≤ 15	85-115	≤ 15
	Lead	85-115	≤ 15	85-115	≤ 15
	Magnesium	85-115	≤ 15	85-115	≤ 15
	Manganese	85-115	≤ 15	85-115	≤ 15
	Molybdenum	85-115	≤ 15	85-115	≤ 15
	Nickel	85-115	≤ 15	85-115	≤ 15
	Potassium	85-115	≤ 15	85-115	≤ 15
	Selenium	85-115	≤ 15	85-115	≤ 15
	Silver	85-115	≤ 15	85-115	≤ 15
	Sodium	85-115	≤ 15	85-115	≤ 15
	Strontium	85-115	≤ 15	85-115	≤ 15
	Thallium	85-115	≤ 15	85-115	≤ 15
	Titanium	85-115	≤ 15	85-115	≤ 15
	Vanadium	85-115	≤ 15	85-115	≤ 15
	Zinc	85-115	≤ 15	85-115	≤ 15

Table 13.3 Summary of Calibration and QC Procedures for EPA Method 6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
6010B	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc	Analyst Initial Demonstration.	Once per analyst	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 75%-125%, TCLP limits are between 50%-150%	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		MDL study.	Once every 12 months.	All analyte MDLs must be < reporting limits.	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem	
		Initial Calibration. Minimum of 2 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient ≥ 0.995	Correct the problem and recalibrate	
		Interference Check Sample (ICS)	Daily after calibration.	Spiked element recoveries between 80-120% recovery, the absolute value of the other element concentrations must be < RL	Correct the problem, calculate new interelement correction factors and recalibrate.	
		MDL Check	Daily after calibration.	All analyte recoveries between 50-150%.	Correct the problem and recalibrate.	
		IDL Calculation	Daily after calibration.	All analyte IDLs < RL.	Correct the problem, clean the torch, recalibrate.	
		Initial Calibration Verification (ICV)	Daily after calibration.	All analyte recoveries between 90% and 110% of true value.	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte concentrations < RL.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	If unable to re-analyze, flag with a "B"

Table 13.3 Summary of Calibration and QC Procedures for EPA Method 6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
6010B	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc	Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte recoveries between 90%-110%.	Correct the problem; recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85%-115%.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J"
		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J"
		Matrix Blank	Once per batch.	All analyte concentrations < RL.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "B"
		Matrix Spike	Every 10 samples.	All analyte recoveries between 75%-125%, TCLP recoveries between 50%-150%	If recovery is outside QC limits but CCC, CCB, IPC, and LCS are acceptable, matrix effect is suspected.	
		TCLP Matrix Spike	Every 10 samples	All analyte recoveries between 50% and 150%.	If recovery is outside QC limits but CCC, CCB, IPC, and LCS are acceptable, matrix effect is suspected	
		Matrix Spike Duplicate.	Every 10 samples.	≤ 20 RPD	Sample commented..	
		TCLP duplicate	One every batch	≤ 20 RPD	Sample Commented.	

Appendix A for Method SW846-6010B Inductively Coupled Plasma-Atomic Emission Spectroscopy-EPA Method 6010

Table A.1 TAL ($TAL_L)				
QC Type	**Analyte**	**Accuracy (%R)** **LCL**	 **UCL**	**Precision (%RPD)**
LCS/LCSD	**Aluminum**	**85 -**	**115**	**15**
	Calcium	**85**	**115**	**15**
	Iron	**85**	**115**	**15**
	Potassium	**85**	**115**	**15**
	Magnesium	**85**	**115**	**15**
	Manganese	**85**	**115**	**15**
	Sodium	**85**	**115**	**15**
MS/MSD	**Metals(Aluminum-Sodium)**	**75**	**125**	**20**
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				

Table A.2 TAL ($TAL_S)				
QC Type	**Analyte**	**Accuracy (%R)** **LCL**	 **UCL**	**Precision (%RPD)**
LCS/LCSD	**Silver**	**85 -**	**115**	**15**
	Aluminum	**85**	**115**	**15**
	Arsenic	**85**	**115**	**15**
	Barium	**85**	**115**	**15**
	Beryllium	**85**	**115**	**15**
	Calcium	**85**	**115**	**15**

	Cadmium	**85**	**115**	**15**
	Cobalt	**85**	**115**	**15**
	Chromium	**85**	**115**	**15**
	Copper	**85**	**115**	**15**
	Iron	**85**	**115**	**15**
	Potassium	**85**	**115**	**15**
	Magnesium	**85**	**115**	**15**
	Manganese	**85**	**115**	**15**
	Sodium	**85**	**115**	**15**
	Nickel	**85**	**115**	**15**
	Lead	**85**	**115**	**15**
	Antimony	**85**	**115**	**15**
	Selenium	**85**	**115**	**15**
	Thallium	**85**	**115**	**15**
	Vanadium	**85**	**115**	**15**
	Zinc	**85**	**115**	**15**
MS/MSD	**Metals(Silver-Zinc)**	**75**	**125**	**20**
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				

Table A.3 ($ICP_E)

QC Type	**Analyte**	**Accuracy (%R)** **LCL**	 **UCL**	**Precision (%RPD)**
LCS/LCSD	**Silver**	**85 -**	**115**	**15**
	Arsenic	**85**	**115**	**15**
	Barium	**85**	**115**	**15**
	Cadmium	**85**	**115**	**15**
	Chromium	**85**	**115**	**15**
	Lead	**85**	**115**	**15**
	Selenium	**85**	**115**	**15**
MS/MSD	**Metals(Silver-Selenium)**	**50**	**150**	**20**
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				

Table A.4 ($ICP_H)				
QC Type	**Analyte**	**Accuracy (%R)** **LCL**	 **UCL**	**Precision (%RPD)**
LCS/LCSD	**Silver**	**85 -**	**115**	**15**
	Arsenic	**85**	**115**	**15**
	Barium	**85**	**115**	**15**
	Cadmium	**85**	**115**	**15**
	Chromium	**85**	**115**	**15**
	Lead	**85**	**115**	**15**
	Selenium	**85**	**115**	**15**
MS/MSD	**Metals(Silver-Selenium)**	**75**	**125**	**20**
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				