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SW846-3050B: Acid Digestion of Sediments, Sludges, and Soils for Total Metals Analysis by ICP Spectroscopy

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at https://epd.georgia.gov/about-us/epd-laboratory-operations. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1.1 Scope and Application

Method SW846-3050B is used for the preparation of sediments, sludges, and soils for analysis by ICP spectroscopy. Separate digestates are prepared for ICP-OES and ICP-MS analysis; these digestates are not interchangeable. Samples digested by this method are analyzed by SW846-6010B or SW846-6020.

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 handling of hazardous materials. 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
Interferences are discussed in the analytical method.

4 Safety
Refer to Laboratory Chemical Hygiene Plan, online revision

5 Apparatus and Equipment
5.1 Hot block capable of maintaining a constant temperature of 95ºC.
5.2 50 flat bottom HDPE digestion tubes with caps.
5.3 Ribbed HDPE watch glasses.
5.4 Electronic top-loading balance capable of weighing 1.0 g +/- 0.01 g.
5.5 Various size pipetters capable of delivering volumes ranging from 1.0 to 5000 µL and an assortment of high quality pipet tips.
5.6 Graduated cylinders capable of measuring volumes ranging from 10 to 50 mL.
5.7 Wood scrapers.
5.8 Mortar and pestle.

6 Reagents and Standards
6.1 Concentrated nitric acid (sp. gr. 1.41), reagent grade.
6.2 1:1 nitric acid.
6.3 concentrated hydrochloric acid.
6.4 30% hydrogen peroxide.
6.5 18 MΩ water.
6.6 Spiking solution for preparing all matrix spikes and laboratory control samples.

7 Sample Collection
Soil and sediment samples for metal analysis are collected in 500 ml wide mouth plastic (HDPE) bottles. Samples must be cooled to 4^0C after sample collection. Analysis must be performed within 180 days.

8 Calibration
Not applicable.

9 Quality Control
Refer to analytical method

10 Procedure

10.1 Break up the sample if needed using either the wood scrapers or a mortar and pestle. Discard the wood scrapers after each sample, clean the mortar and pestle by washing with 1:1 nitric acid followed by rinsing with 18 MΩ water between samples.

10.2 Weigh 0.5 g +/- 0.01 g into the digestion tube. Do not use a dried sample for wastes, all other samples must be dried. Prepare a matrix blank, LCS, and LCSD for each batch using 0.5g ± 0.01g 18 MΩ water instead of solid. Prepare a sample spike and sample spike duplicate sample for every 10 samples in the batch.

10.3 Add 5 ml 1:1 nitric acid, mix with the sample, cover with a watch glass, heat to 95ºC +/- 5ºC and reflux for 10-15 minutes. Add spiking solution to the appropriate QC samples prior to heating.

10.4 Cool the sample and add 2.5 mL concentrated nitric acid to each sample, replace the watch glass and reflux for 30 minutes. Repeat this step until no brown fumes are generated. Record any additional amounts of nitric acid used in this step on the digestion sheet.

10.5 Cover the samples with a ribbed watch glass and either allow the sample to evaporate to about 5 mL or heat for 2 hours at 95ºC. Keep the digestion vessels covered at all times and do not allow any sample to boil. If a sample boils, it must be redigested.

10.6 Cool the samples, add 1.0 mL 18 MΩ water and 1.5 mL 30% hydrogen peroxide. Cover the samples and return them to the Hotblock. Heat until the effervescence subsides; then cool the samples. Continue adding 1.5 mL aliquots of 30% hydrogen peroxide followed by heating until the effervescence no longer occurs. Do not add more than 5 mL 30% hydrogen peroxide. Care must be taken to ensure that sample loss does not occur during the effervescence. Record the volume of hydrogen peroxide added to each sample on the digestion sheet.

10.7 Cover the samples with a ribbed watch glass and heat the nitric acid/hydrogen peroxide digestate until the volume has been reduced to about 5 mL or heat at 95ºC without boiling for 2 hours.

10.8 Cool the samples and bring up to 50 mL with 18 MΩ water. Allow the samples to sit overnight to allow any suspended solids to settle. The sample is ready for analysis by ICP-MS.

10.9 For analysis by ICP-OES, add 5 mL concentrated hydrochloric acid to the cooled samples from step 10.7, cover with a watch glass and reflux at 95ºC +/- 5ºC for 15 minutes. Proceed to step 10.8.

10.10 Cool the samples. Either filter through Whatman 41 (or equivalent) filter paper, collect the filtrate in 50 mL centrifuge tubes and bring up to 50 mL with 18 MΩ water and cap the samples, or allow the samples to stand overnight to settle any suspended solids. The sample is now ready for analysis by ICP-OES.

10.11 Optional digestion method: This procedure may be used to increase the solubilities and recoveries of antimony, barium, lead, and silver when necessary. This procedure is optional and not required on a routine basis.

10.11.1 Break up the sample if needed using either the wood scrapers or a mortar and pestle. Discard the wood scrapers after each sample; clean the mortar and pestle by rinsing with 1:1 nitric acid followed by rinsing with 18 MΩ water between samples.

10.11.2 Weigh 0.5 g +/- 0.01 g into the digestion tube. Do not use a dried sample for wastes; all other samples must be dried. Prepare the matrix blank, LCS, and LCSD by weighing out 0.5 g +/- 0.01 g clean Ottowa Sand and placing in digestion tubes prepare a sample spike and sample spike duplicate sample for every 10 samples in the batch.

10.11.3 All 1.25 mL concentrated nitric acid and 5 mL concentrated hydrochloric acid to the samples. Cover with a watch glass and reflux at 95ºC +/- 5ºC for 15 minutes.

10.11.4 Cool the samples and filter the digestate through Whatman 40 (or equivalent) filter paper. Collect the filtrate in 50 mL digestion tubes. Wash the filter paper while still in the funnel with no more than 2.5 mL of hot (95ºC) concentrated hydrochloric acid followed by 10 mL of hot (95ºC) 18 MΩ water into the same 50 mL digestion tube.

10.11.5 Remove the filter and residue from the funnel and place in a new digestion tube. Heat at 95ºC +/- 5ºC until the paper dissolves. Remove the sample from the Hotblock and cool; wash the watch glass and digestion tube sides with 18 MΩ water. Filter the digestate into the same 50 mL digestion tube used earlier. Bring up to 50 mL with 18 MΩ water.

11 Evaluation of the Linearity of the Initial Calibration
Not applicable.

12 References

12.1 *Test Methods for Evaluating Solid Waste, SW846*, USEPA Office of Solid Waste, revision 2, 1996.

13 Practical Quantitation Limits (PQLs), Precision and Accuracy Criteria, and Quality Control Approach
Not applicable