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EPA 200.3 Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements in Biological Tissues

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 Scope and Application

This method is used for preparation of samples for the determination of total recoverable metals in biological tissues.

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
Magnesium	7439-95-4
Manganese	7439-96-5
Mercury	7439-97-6
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

3.1 Contamination is the prime concern. The work area (including counters and hoods) is cleaned weekly to eliminate sources of contamination. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. Any glassware used must be triple washed in reagent grade 1:1 nitric acid followed by a triple rinse in 18 MΩ water. The acid rinse must be logged into the standard log. After washing, all glassware is dried in a clean environment then covered with parafilm and stored in a closed cabinet until used. Virgin plasticware (HDPE) is used as much as possible and discarded after use.

3.1.2 The use of high purity reagents helps to minimize interference problems. Reagent grade acids are used for this method.

4 Safety

Refer to Laboratory Chemical Hygiene Plan, Revision 1 February 28, 1999

5 Apparatus and Equipment

5.1 125 ml Erlenmeyer flasks.
5.2 Various size pipetters capable of delivering volumes ranging from 1.0 to 5000 µL and an assortment of high quality pipet tips.
5.3 Hot plate capable of maintaining a temperature of 95ºC.
5.4 Balance capable of measuring 0.5 g +/- 0.1 g.
5.5 Steel cabinet centrifuge with electric timer and brake.
5.6 Plastic wash bottles for cleaning.
5.7 Wood spatulas.

6. Reagents and Standards

6.1 Concentrated nitric acid (sp. gr. 1.41), reagent grade.
6.2 Concentrated hydrochloric acid, (sp. gr. 1.19), reagent grade.
6.3 30% Hydrogen Peroxide, reagent grade.
6.4 18 MΩ water.
6.5 Spiking solution for preparing all matrix spikes and laboratory control samples.

7 Sample Collection

Refer to Chapter 5 of the Georgia EPD Laboratory Quality Assurance Manual for Sample Container, Sample Preservation, and Sample Holding Times.

8 Calibration
Not applicable.

8.1 Calibration Curve
Not applicable.

8.2 Calibration Verification

Not applicable

9 Quality Control - Refer to analytical method

10 Procedure

10.1 Weigh out 0.5 +/- 0.1 g of ground, well mixed tissue into a labeled Erlenmeyer. Weigh out additional 0.5 +/- 0.1 g samples of Milli Q H_2O tissue for the Matrix Blank (MB), Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD). Prepare additional subsamples for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Add spiking solutions to LCS, LCSD, MS and MSD.

10.2 Add 5 ml concentrated nitric acid to each sample and place the samples on the hot plate. Heat at 95ºC +/- 10ºC until the samples are solubilized, swirling to assist in dissolving the tissue.

10.3 Continue heating until the solution begins to turn brown. Remove the samples from the hot plate and allow to cool. Add another 2.5 ml concentrated nitric acid and return the samples to the hot plate until the samples once again turn brown.

10.4 Remove the samples from the hot plate, cool, and add another 1 ml concentrated nitric acid. Return the samples to the hot plate and heat until the volume has been reduced to approximately 5 ml.

10.5 Remove the samples from the hot plate, cool, and add 1 ml of 30% hydrogen peroxide. Return the samples to the hot plate and heat until the volume has been reduced to approximately 5-10 ml.

10.6 Repeat section 10.5 until either the solution is clear or 5 ml of hydrogen peroxide has been added.

10.7 Remove the samples from the hot plate, cool, and add 1 ml of concentrated hydrochloric acid. Return the samples to the hot plate and heat the solution once again begins to turn brown.

10.8 Remove the samples from the hot plate, cool, transfer the samples to labeled 50 ml HDPE centrifuge tubes and bring the samples to 50 ml using 18 MΩ water.

10.8 Allow any undissolved material to settle overnight, or centrifuge a portion of the prepared sample until clear.

10.9 Record all sample weights, lot numbers, standard numbers, and volumes in the digestion log.

10.10 A 1:4 dilution may be prepared if needed to analyze samples by ICPMS.

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

12 References

12.1 *Methods for the Determination of Metals in Environmental Samples Supplement I;* U.S. EPA Office of Research and Development: Cincinnati, OH, 1994, EPA-600/R-94/111 May 1994

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