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Standard Practices for Extraction of Trace Elements from Sediments[1]

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1. Scope

1.1 These practices describe the partial extraction of soils, bottom sediments, suspended sediments, and waterborne materials to determine the extractable concentrations of certain trace elements.

1.1.1 *Practice A* is capable of extracting concentrations of aluminum, boron, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, sodium, strontium, vanadium, and zinc from the preceding materials. Other metals may be determined using this practice. This extraction is the more vigorous and more complicated of the two.

1.1.2 *Practice B* is capable of extracting concentrations of aluminum, cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, and zinc from the preceding materials. Other metals may be determined using this practice. This extraction is less vigorous and less complicated than Practice A.

1.2 These practices describe three means of preparing samples prior to digestion:

1.2.1 Freeze-drying.

1.2.2 Air-drying at room temperature.

1.2.3 Accelerated air-drying, for example, 95°C.

1.3 The detection limit and linear concentration range of each procedure for each element is dependent on the atomic absorption spectrophotometric or other technique employed and may be found in the manual accompanying the instrument used. Also see various ASTM test methods for determining specific metals using atomic absorption spectrophotometric techniques.

1.3.1 The sensitivity of the practice can be adjusted by varying the sample size (14.2) or the dilution of the sample (14.6), or both.

1.4 Extractable trace element analysis provides more information than total metal analysis for the detection of pollutants, since absorption, complexation, and precipitation are the methods by which metals from polluted waters are retained in sediments.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D887 Practices for Sampling Water-Formed Deposits
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water

3. Terminology

3.1 Refer to Terminology D1129.

4. Summary of Practices

4.1 The chemical portion of both practices involves acid digestion to disassociate the elements complexed in precipitated hydroxides, carbonates, sulfides, oxides, and organic materials. Surface but not interstitially bound elements will be desorbed in the case of clay mineral particulates. The silicate lattices of the minerals are not appreciably attacked (**1-5**).[3]

4.2 These practices provide samples suitable for analysis using flame or flameless atomic-absorption spectrophotometry, or other instrumental or colorimetric procedures.

5. Significance and Use

5.1 Industrialized and urban areas have been found to deposit a number of toxic elements into environments where those elements were previously either not present or were

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[1] These practices are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to the references at the end of these practices.

found in trace amounts. Consequently, it is important to be able to measure the concentration of these pollution-deposited elements to properly study pollution effects.

5.2 This procedure is concerned with the pollution-related trace elements that are described in 4.1 rather than those elements incorporated in the silicate lattices of the minerals from which the sediments were derived. These pollution-related trace elements are released into the water and readsorbed by the sediments with changes in general water quality, pH in particular. These elements are a serious source of pollution. The elements locked in the silicate lattices are not readily available in the biosphere (**1-8**).

5.3 When comparing the trace element concentrations, it is important to consider the particle sizes to be analyzed (**8, 9**).

5.3.1 The finer the particle the greater the surface area. Consequently, a potentially greater amount of a given trace element can be adsorbed on the surface of fine, particulate samples (**4**). For particle sizes smaller than 80 mesh, metal content is no longer dependent on surface area. Therefore, if this portion of the sediment is used, the analysis with respect to sample type (that is, sand, salt, or clay) is normalized. It has also been observed that the greatest contrast between anomalous and background samples is obtained when less than 80-mesh portion of the sediment is used (**4, 5**).

5.3.2 After the samples have been dried, care must be taken not to grind the sample in such a way to alter the natural particle-size distribution (14.1). Fracturing a particle disrupts the silicate lattice and makes available those elements which otherwise are not easily digested (**6**). Normally, aggregates of dried, natural soils, sediments, and many clays dissociate once the reagents are added (14.3 and 15.2).

6. Interferences

6.1 The only interferences are those encountered in the final determination of metals using atomic-absorption spectrophotometry or other instrumental or colorimetric procedures.

7. Apparatus

7.1 *Digestion Beakers*—Use only beakers made of borosilicate glass or TFE-fluorocarbons.

7.2 *Watchglasses*—Use ribbed watchglasses to cover the digestion beakers. These covers should fit loosely to allow evaporation of the digestion medium.

7.3 *Filter Paper*—The qualitative grade paper employed should be a fast filtering, hardened, ashless paper retaining both coarse and gelatinous precipitates.

7.4 *Sieves,* nylon, 10, 20, and 80-mesh.

7.5 *Petri Dishes,* large.

7.6 *Freeze-Drier.*

7.7 *Polyethylene or Polypropylene Bottles,* wide-mouth, 125-mL capacity.

7.8 *Suction Filtration Apparatus,* 0.45-µm filter.

7.9 *Automatic Shaker.*

7.10 *Volumetric Flasks,* 50 mL and 100-mL capacity.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in tests. Acids must have a low-metal content or should be doubly distilled and checked for purity. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[4]

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to D1193. Other reagent water types may be used provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the bias and precision of the test method. Type II water was specified at the time of round robin testing of this method. The water shall be free of metallic contaminants.

8.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). The acid must be low in metallic ions.

8.4 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3). The acid must be low in metallic ions.

8.5 *Metal Solutions, Stock*—Prepare metal stock solutions, each containing 1000 mg/L of a metal of interest and either negligible or known concentrations of interfering metals.

9. Precautions

9.1 Digest the samples only in a laboratory ventilation hood.

10. Sampling

10.1 Collect the sediments using an appropriate technique (see Practice D887).

10.2 Retain and store that portion of sediment which passes through a nylon, 10-mesh sieve, 1-mm particle size (5.3).

10.3 Store the sample in plastic bags or plastic bottles that can be tightly sealed. Immediately pack and cool the samples for shipping.

10.4 Store samples at 4°C if analysis is to be performed within 1 week. Otherwise, store the samples at – 20°C until analyzed.

11. Glassware Cleaning

11.1 Immerse all glassware and implements in a hot solution of HCl (1 + 1) for 3 to 5 min.

11.2 Second, immerse all glassware and implements in HNO_3 (1 + 1) for 3 to 5 min.

11.3 Rinse all glassware and implements repeatedly with water following the acid washes.

12. Sample Preparation

12.1 Completely thaw frozen samples before unsealing.

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

12.2 Preparation of the samples for analysis may involve compositing, splitting, or subsampling (5.3.2).

12.3 *Drying Samples:*

12.3.1 *Preparation I*—Freeze-drying is rapid and results in loose samples. See the apparatus instruction manuals for further information.

12.3.2 *Preparation II*—Air-drying at room temperature is accomplished by spreading the samples out on petri dishes and drying to constant weight. This procedure requires a long time to complete and the sample must be covered to avoid dust contamination.

12.3.3 *Preparation III*—Accelerated air-drying the samples, for example, 95°C is followed by allowing the samples to cool in a desiccator. Dry to constant weight. Often constant weight may be achieved by drying overnight, however, constant weight must be obtained for consecutive measurements. When analyzing for more volatile metals, use the temperature recommended for that specific method.

13. Percent Solids Determination

13.1 Using desiccated, tared-beakers, weigh the well-mixed wet samples from 12.2 or 12.3. Record the weights.

13.2 Dry overnight or to constant weight at 105°C and allow the samples to cool in a desiccator.

13.3 Reweigh the beakers and samples. Determine the percent solids as described in 16.1.

14. Digestion Practice A

14.1 Crush the sample to facilitate weighing if a hard aggregate is formed during drying (5.3 and 12.3).

14.2 Weigh 4 g of dried sample into a 250-mL beaker. Record the sample weight to the nearest 0.1 mg. Include an empty beaker in each analysis set as a reagent/glassware blank.

14.3 Add the following to each sample and the blank:

14.3.1 *Water,* 100.0 mL.

14.3.2 *Concentrated* HNO_3(sp gr 1.42), 1.0 mL. A foaming reaction indicates the presence of carbonates; in this case add the acids slowly.

14.3.3 *Concentrated HCl* (sp gr 1.19), 10 mL.

14.4 Cover the beakers with ribbed watchglasses and heat on a hot plate at 95°C. To prevent splattering do not allow the solutions to boil or bump.

14.5 Remove each beaker from the hot plate when the solution remaining is 10 to 15 mL. Allow the contents to cool to room temperature.

14.6 Filter each solution and quantitatively transfer the solution to a 50-mL volumetric flask and dilute to volume.

15. Digestion Practice B

15.1 Weigh 1.0000 g of dried sediment and place in a 125-mL polypropylene wide-mouth bottle. For low-level trace elements use up to 10 g of sediment sample. Include an empty bottle as a reagent/glassware blank with each set of samples.

15.2 Add 95 mL of water and 5.0 mL of HCl (sp gr 1.19) to the sample and to the blank bottle and cap tightly. In the case of a foaming reaction, which indicates the presence of carbonates, add the acidic solution slowly.

15.3 Shake at room temperature in a mechanical shaker 16 h (overnight).

15.4 Filter solution by suction filtration or filter paper. Quantitatively transfer the solution to a 100-mL volumetric flask and dilute to volume.

16. Calculation

16.1 Use the following equation to determine the percent solids of the sample (Section 13):

$$B = \frac{M}{N} \times 100 \quad (1)$$

where:

B = percent solids of the sample,
M = dry weight of the sample (13.3), and
N = wet weight of the sample (13.1).

16.2 Calculate the concentration of the element for each dry sample as follows:

$$C = \frac{(Q - S)V}{U} \quad (2)$$

where:

Q = concentration of the element in the digested solution µg/mL,
S = concentration of the trace element found in the reagent/glassware blank (14.2 and 15.1), µg/mL,
V = volume of extract from 14.6 and 15.4,
U = weight of the sample corrected to a dried sample at 105°C, g, and
C = trace element per gram of dry sample, µg.

16.3 Use the following equation to determine the concentration of the metal in the wet sample (13.1):

$$A = C \times \frac{B}{100} \quad (3)$$

where:

A = metal per gram of wet sample, µg,
B = percent solids of the sample, and
C = trace element per gram of dry sample (16.2), µg.

17. Keywords

17.1 extraction; sediments; toxic; trace elements

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