



Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals¹

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1. Scope

1.1 This practice covers two procedures for the total digestion of sediments for subsequent determination of metals by such techniques as flame atomic absorption spectrophotometry, graphite-furnace atomic absorption spectrophotometry, atomic emission spectroscopy, etc.

1.2 This practice is applicable in the subsequent determination of volatile, semivolatile, and nonvolatile metals of sediments.

1.3 Actual metal quantitation can be accomplished by following the various test methods outlined under other appropriate ASTM standards for the metal(s) of interest. Before selecting either of the digestion techniques outlined in this practice, the user should consult the appropriate quantitation standard(s) for any special analytical considerations, and Practice D 3976 for any special preparatory considerations.

1.4 *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.* For a specific hazard statement, see Note 7.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits²

D 1193 Specification for Reagent Water²

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D 1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total digestion*—the dissolution of a sediment matrix such that quantitation will produce a measurement which is more than 95% of the constituent present in the sample.

3.2.2 *partial digestion*—the dissolution of a sediment matrix such that quantitation will produce a measurement of less than 95% of the constituent present in the sample. In such cases, recovery is operationally defined by the digestion procedure.

4. Summary of Practices

4.1 Many procedures are available for the total digestion of sediments prior to metal analysis, but almost all the methods fall into one of two main classes: fusion and subsequent dissolution of the bead, and wet digestion which directly dissolves the sample with mineral acids. Each of the classes has advantages and disadvantages, as do the individual procedures which fall under them. The two procedures outlined in this practice were selected because they are the least restricted, in terms of utility, for dealing with a wide variety of matrices. Before choosing a particular method, the user should consult the pertinent literature to determine the utility and applicability of either method, prior to final selection; or if a less rigorous digestion could be employed.^{4,5,6,7} Even then, experience with a particular sample type or digestion test method, or both, may have to be the final arbiter in test method selection.

4.2 Field collected samples should be treated according to the procedures outlined in Practice D 3976.

4.3 Dried samples are ground to finer than 100 mesh (150 μm) using an appropriate grinding device or system.

4.4 *Procedure A*—Fusion with lithium metaborate/tetraborate.

4.5 *Procedure B*—Wet digestion using a combination of hydrofluoric, perchloric, and nitric acids.

¹ This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D 19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.

⁴ Johnson, W., and Maxwell, J., *Rock and Mineral Analysis*, 2nd Edition, John Wiley & Sons, New York, 1981, p. 489.

⁵ Pinta, M., *Modern Methods for Trace Element Analysis*, Ann Arbor Science Publishers, Ann Arbor, 1982, pp. 133–264.

⁶ Dolezal, J., Povondra, C., and Sulcek, Z., *Decomposition Techniques in Inorganic Analysis*, Elsevier Publishing Co., New York, 1968, pp. 11–157.

⁷ Shapiro, L., “Rapid Analysis of Silicate, Carbonate, and Phosphate Rocks,” Revised Edition, *U.S. Geological Survey Bulletin*, 1401, 1975, p. 76.

5. Significance and Use

5.1 The chemical analysis of sediments, collected from such locations as streams, rivers, lakes, and oceans can provide information of environmental significance.

5.2 These practices can be used with either suspended sediment (material actively transported by water) or bed sediment (material temporarily at rest on the bed of a water body).

5.3 Standardized practices for digesting sediments, for subsequent chemical analysis, will facilitate inter- and intra-areal comparisons as well as comparison of data generated by different groups. The use of total digestions also eliminates the ambiguities and interpretational difficulties associated with partial digestions and the operational definitions that accompany them.

PROCEDURE A—FUSION

6. Scope

6.1 This procedure is effective for the total digestion of suspended and bottom sediments for the subsequent determination of aluminum, calcium, iron, magnesium, potassium, manganese, silicon, sodium, and titanium.

6.2 This practice may be appropriate for the subsequent determination of other metals provided the concentrations are high enough or if the instrumental sensitivity is sufficient.

7. Interferences

7.1 Numerous inter-element interferences, both positive and negative, exist for this procedure and have been amply documented elsewhere.^{4,5}

7.2 Interferences are eliminated or compensated for, or both, through the use of cesium chloride (CsCl), orthoboric acid (H₃BO₃), lithium metaborate (LiBO₂), lithium tetraborate (Li₂B₄O₇), and the use of mixed salt standards during quantitation by flame atomic absorption spectrophotometry.

8. Apparatus

8.1 *Graphite Crucibles*, drill point, with a 7.5-mL capacity and a 1-in. (25.4 mm) outside diameter, 3/4-in. (19.05 mm) inside diameter, and total depth of 1 3/8 in. (34.925 mm).

8.2 *Magnetic Stirrer*.

8.3 *Muffle Furnace*, capable of reaching a temperature of at least 1000°C.

9. Reagents

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all digestions. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁸ Other grades may be used, provided it is first ascertained that the reagent is of

sufficiently high purity to permit its use without lessening the accuracy of the subsequent quantitation.

9.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

9.3 *Mixed Salt Standards*—The mixed salt standards are provided as a guide to the user for use with atomic absorption analyses to reduce matrix and interelement interferences. They have been found effective for the constituents listed in 6.1. They may have to be modified to accommodate others.

9.4 *Cesium Chloride, Solution* (4 g/L)—Dissolve 4 g of CsCl in water and dilute to 1 L.

9.5 *Diluent Solution*—Dissolve 6 g of flux mixture in 500 mL of water. Add 12.5 mL concentrated nitric acid (sp gr 1.41), and dilute to 1 L with water.

9.6 *Flux Mixture*—Thoroughly mix 1 part powdered anhydrous lithium metaborate, LiBO₂, and 2 parts anhydrous lithium tetraborate, Li₂B₄O₇. Store in a tightly closed bottle.

NOTE 1—It is possible to purchase pre-mixed fusion fluxes from several suppliers, and provided they are of sufficient purity, have been found quite satisfactory.

9.7 *Mixed Metals Solution, Stock*—Dissolve by appropriate means, the following compounds, elements, or both: aluminum metal (1.500 g), calcium carbonate (1.249 g), iron metal (1.000 g), magnesium metal (0.200 g), manganese metal (0.040 g), KCl (0.668 g), ammonium hexafluorosilicate (18.987 g), NaCl (0.636 g), and ammonium titanyl oxalate (1.227 g), and dilute to 1000 mL with diluent solution (9.5). This solution will contain the following concentrations: aluminum (1500 mg/L), calcium (500 mg/L), iron (1000 mg/L), magnesium (200 mg/L), manganese (40 mg/L), potassium (350 mg/L), silica (3000 mg/L), sodium (250 mg/L), and titanium (200 mg/L). Store in a plastic or TFE-fluorocarbon bottle.

9.8 *Mixed Metals Solutions, Standards 1, 2, and 3*—Take respectively, a 10-, 6-, and 2-mL aliquot of the mixed metals stock solution (9.7), and dilute to 100 mL in volumetric glassware with standard diluent solution (9.5). Concentrations are given in Table 1.

9.9 *Nitric Acid*, concentrated (sp gr 1.41).

9.10 *Nitric Acid* (1 + 1)—Add 250 mL of concentrated nitric acid (sp gr 1.41) to 250 mL water. Store in a plastic bottle.

9.11 *Orthoboric Acid Solution* (50 g/L)—Dissolve 50 g of H₃BO₃ in water and dilute to 1 L. Heat may be required to complete dissolution. Prepare fresh daily because orthoboric acid may precipitate within 12 to 18 h.

TABLE 1 Concentrations of Mixed Metals Solutions 1, 2, and 3

	Standard 1, mg/L	Standard 2, mg/L	Standard 3, mg/L
Volume (mL)	10	6	2
Iron	100	60	20
Magnesium	20	12	4
Silicon	300	180	60
Aluminum	150	90	30
Titanium	20	12	4
Calcium	50	30	10
Sodium	25	15	5
Potassium	35	21	7
Manganese	4	2	1

⁸ *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 *Analar 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.

10. Procedure

10.1 Immediately before each use, clean all glassware by rinsing first with $\text{HNO}_3(1 + 1)$, and then with water.

10.2 Dry the sediment sample by an appropriate procedure such as freeze-drying, or oven drying at 105°C (see Practice D 3976).

10.3 If the sediment sample is greater than 100 g, split it to less than 100 g by the use of a nonmetallic sample splitter (riffle sampler) or by coning and quartering.

10.4 Grind the sample with an appropriate system until all material is finer than 100 mesh (150 μm).

10.5 Transfer approximately 1.2 g of flux mixture to a waxed or plastic-coated weighing paper (6 in. by 6 in. (152.4 mm by 152.4 mm)). Weigh and transfer 0.2000 g of finely ground sample to the flux mixture and mix by rolling successive corners of the paper about 30 times. Carefully transfer the combined sample/flux to a graphite crucible, and tamp down by gently tapping the crucible on a tabletop.

10.6 Weigh appropriate sediment or rock standards and treat as in 10.5.

10.7 Carry several blanks through the procedure by using only flux and treat as in 10.5.

10.8 Fuse the mixtures in a muffle furnace, preheated to 1000°C , for 30 min.

NOTE 2—When the crucibles, samples, and crucible racks are placed in the muffle furnace, the temperature may drop as much as 200°C . Time is still measured from the time of insertion in the furnace.

10.9 Remove the crucibles from the furnace and allow to cool; dislodge the beads by gentle tapping or with a spatula.

NOTE 3—The beads can be dissolved immediately after cooling, or can be stored in plastic vials for dissolution at a later time.

10.10 Place the bead in an acid-washed 250-mL plastic bottle and add a $\frac{3}{4}$ to 1 in. (19.05 to 25.4 mm) magnetic stirring bar. Add approximately 50-mL boiling water using a plastic graduate, place the bottle on a magnetic stirrer, and mix. Add 5 mL of $\text{HNO}_3(1 + 1)$ to each bottle and stir rapidly for about 60 min. Cap the bottle lightly to prevent both contamination and possible spattering.

10.11 Immediately after 60 min, remove the bottles from the stirrers, and add about 100 mL of water to prevent the polymerization of silica.

NOTE 4—The solutions may contain small amounts of graphite from the crucibles which can be ignored. However, if the solution is cloudy, this indicates a very high concentration of silica in the original sample and that it has polymerized. Such a solution must be discarded, and a new fusion performed using a smaller quantity of sample.

10.12 Pour each solution into a 200-mL volumetric flask, using a funnel, in order to retain the stirring bar. Rinse the bottle and cap, and bring to the mark with water. Pour the solution back into the plastic bottle for storage.

10.13 Add 10 mL of CsCl solution and 20 mL of H_3BO_3 solution to each bottle.

NOTE 5—The CsCl acts as an ionization suppressant and the H_3BO_3 stabilizes the silica; these are used when quantitation is by flame atomic absorption spectrophotometry.

10.14 Prepare the mixed metals standard solutions (see 9.8) and to each 100 mL, add 5 mL of CsCl solution, and 10 mL of

H_3BO_3 solution (Note 5).

10.15 See the appropriate ASTM test methods for subsequent quantitation.

PROCEDURE B—WET DIGESTION

11. Scope

11.1 This procedure is effective for the total digestion of suspended and bottom sediments for the subsequent determination of aluminum, calcium, iron, magnesium, manganese, potassium, sodium, titanium, strontium, lithium, copper, zinc, cadmium, lead, cobalt, nickel, chromium, arsenic, antimony, and selenium.

11.2 This practice may be appropriate for the subsequent determination of other metals provided the concentrations are high enough or if the instrumental sensitivity is sufficient.

12. Interferences

12.1 Numerous inter-element interferences, both positive and negative, exist for this procedure and have been documented elsewhere.^{4, 5, 9}

12.2 Interferences are eliminated, compensated for, or both, through the use of cesium chloride (CsCl), the use of mixed salt standards, and background correction if quantitation is by atomic absorption spectroscopy.

13. Apparatus

13.1 *TFE-Fluorocarbon Beakers*, 100-mL capacity, thick wall, capable of withstanding temperature up to 260°C .

13.2 *Hot Plate*, electric or gas, capable of reaching at least 250°C .

13.3 *Perchloric Acid Hood*, with appropriate washdown facility and gas or electric outlets.

14. Reagents

14.1 *Purity of Reagents*—See 9.1.

14.2 *Purity of Water*—See 9.2.

14.3 The mixed salt standards are provided as a guide to the user for use with atomic absorption analyses to reduce matrix and interelement interferences. They have been found effective for the constituents listed in 11.1. They may have to be modified to accommodate others.

14.4 *Standard Solution, Aluminum* [1.00 mL = 1.00 mg Al]—Dissolve 1.000 g of aluminum metal in 20 mL of HCl (sp gr 1.19) with a trace of a mercury salt to catalyze the reaction, and dilute to 1000 mL with water.

14.5 *Cesium Chloride Solution* (CsCl) (4 g/L)—See 9.4.

14.6 *Hydrochloric Acid* (HCl), concentrated (sp gr 1.19).

14.7 *Hydrochloric Acid*, (1 + 1)—Add 250 mL concentrated hydrochloric acid (sp gr 1.19) to 250 mL water. Store in a plastic bottle.

14.8 *Hydrochloric Acid*, (1 + 49)—Add 10 mL concentrated hydrochloric acid (sp gr 1.19) to 490 mL water. Store in a plastic bottle.

14.9 *Hydrofluoric Acid* (HF), concentrated (48–51%) (sp gr 1.19).

⁹ Walsh, J., "Interferences in the Determination of Titanium in Silicate Rocks and Minerals by Flame Atomic Absorption Spectrophotometry," *Analyst*, Vol 102, 1977, pp. 972–976.

14.10 *Standard Solution, Iron*, [1.00 mL = 1.00 mg Fe]—Dissolve 1.000 g iron metal in 20 mL HCl (1 + 1) and dilute to 1000 mL with water.

14.11 *Mixed Metals Solution, Stock (Minors)*—Dissolve by appropriate means, the following compounds or elements: cadmium metal (0.200 g), chromium metal (0.800 g), cobalt metal (1.200 g), copper metal (0.800 g), lead metal (2.000 g), lithium carbonate (2.130 g), manganese metal (2.000 g), nickel metal (1.200 g), strontium carbonate (1.685 g), and zinc metal (0.320 g), add 20 mL of HCl (sp gr 1.19), and dilute to 1000 mL with water. This solution will contain the following concentrations: cadmium (200 mg/L), chromium (800 mg/L), cobalt (1200 mg/L), copper (800 mg/L), lead (2000 mg/L), lithium (400 mg/L), manganese (2000 mg/L), nickel (1200 mg/L), strontium (1000 mg/L), and zinc (320 mg/L). Store in a plastic or TFE-fluorocarbon bottle.

14.12 *Mixed Metals Solution, Stock (Majors)*—Dissolve by appropriate means, the following compounds or elements: aluminum metal (1.500 g), calcium carbonate (1.249 g), iron metal (1.000 g), magnesium metal (0.200 g), manganese metal (0.040 g), potassium chloride (0.668 g), sodium chloride (0.636 g), and ammonium titanyl oxalate (1.227 g). Add 20 mL HCl (sp gr 1.19), and dilute to 1000 mL with water. This solution will contain the following concentrations: aluminum (1500 mg/L), calcium (500 mg/L), iron (1000 mg/L), magnesium (200 mg/L), manganese (40 mg/L), potassium (350 mg/L), sodium (250 mg/L), and titanium (200 mg/L). Store in a plastic or TFE-fluorocarbon bottle.

14.13 *Mixed Metals Solution, Standard (Minors)*—Take 100 mL of mixed metals stock solution (minors) (14.11), add 20 mL HCl (sp gr 1.19), and dilute to 1000 mL in volumetric glassware with water. This solution will contain the following concentrations: cadmium (20 mg/L), chromium (80 mg/L), cobalt (120 mg/L), copper (80 mg/L), lead (200 mg/L), lithium (40 mg/L), manganese (200 mg/L), nickel (120 mg/L), strontium (100 mg/L), and zinc (32 mg/L). Store in a plastic or TFE-fluorocarbon bottle. Solution is stable for 3 months.

14.14 *Mixed Metals Solutions, Standards 1, 2, and 3*—Take respectively, a 10-, 5-, and 1-mL aliquots of mixed metals standard solution (14.13), and add to each 4 mL HCl (sp gr 1.19), 20 mL of mixed metals standard stock solution (14.12). Dilute to 200 mL in volumetric glassware with water. Concentrations are given in Table 2. Store in plastic or TFE-fluorocarbon bottles. Prepare fresh for each analysis.

14.15 *Mixed Metals Solutions, Standards 4, 5, and 6*—Take respectively, a 10-, 6-, and 2-mL aliquots of mixed metals stock solution (14.12), and add 2 mL HCl (sp gr 1.19), and 10 mL of the CsCl solution (14.5). Dilute to 100 mL in volumetric glassware with water. Concentrations are given in Table 2.

14.16 *Mixed Metals Solutions, Standards 7, 8, and 9*—Take

a 10-mL aliquot of standard solutions 4, 5, and 6 (14.5), add 2 mL HCl (sp gr 1.19), and add 10 mL of the CsCl solution (14.5). Dilute to 100 mL in volumetric glassware with water. Concentrations are given in Table 3. Store in plastic or TFE-fluorocarbon bottles. Prepare fresh for each analysis.

14.17 *Nitric Acid* (HNO₃), concentrated (sp gr 1.41).

14.18 *Perchloric Acid* (HClO₄), concentrated (70 to 72%) (sp gr 1.67).

14.19 *Standard Solution, Sodium* [1.00 mL = 1.00 mg Na]—Dissolve 2.542 g NaCl, in water, add 20 mL HCl (sp gr 1.19), and dilute to 1000 mL with water.

14.20 *Standard Solution, Titanium* [1.00 mL = 1.00 mg Ti]—Dissolve 6.135 g ammonium titanyl oxalate in water, and dilute to 1000 mL with water.

14.21 *Working Solution, Titanium*—Take respectively, a 20-, 10-, and 5-mL aliquot of the titanium standard solution (14.20), and add 100 mL of the aluminum standard solution (14.4), 50 mL of the iron standard solution (14.10), 35 mL of the sodium standard solution (14.19), 200 mL of the CsCl solution (14.5), and 20 mL HCl (sp gr 1.19). Dilute to 1000 mL in volumetric glassware with water. The standards contain, respectively, 20, 10, and 5 mg/L titanium.

15. Procedure

15.1 See 10.1 to 10.4.

15.2 Weigh and transfer 0.5000 g of finely ground sample to a 100 mL TFE-fluorocarbon beaker; weigh out appropriate rock or sediment standards as well.

NOTE 6—This practice can be used with sample weights of between 0.2500 to 1.000 g, with appropriate adjustments to the final solution volumes and acid strengths (15.2 and 15.9). Larger weights (greater than 1.000 g) may be used, but will require an extra digestion with HF and HClO₄ (see 15.6 and 15.7).

15.3 Carry several blanks through the procedure by using empty beakers.

15.4 Place the hot plate in a perchloric acid hood, and turn on the hood and hotplate. Adjust the hot plate to produce a surface temperature of 200°C.

15.5 To each beaker, add 6 mL HNO₃ (sp gr 1.41), and place it on the hot plate for approximately 30 min.

NOTE 7—**Warning:** This step is designed to oxidize organic matter in the sample. It is imperative that this step be carried out prior to the addition of perchloric acid, otherwise a violent explosion could occur. Resistant organics, such as coals, may require a second treatment with nitric acid.

15.6 Remove the beakers from the hot plate and wait 5 min. Add 6 mL of HF (sp gr 1.19), 2 mL of HClO₄ (sp gr 1.67), and return the beakers to the hot plate. Continue heating the beakers until the evolution of white perchloric fumes and the solution has reached incipient dryness; however, do not bake the solutions.

TABLE 2 Concentrations of Mixed Metals Solutions 4, 5, and 6

	Standard 4, mg/L	Standard 5, mg/L	Standard 6, mg/L
Volume (mL)	10	6	2
Aluminum	150	90	30
Iron	100	60	20
Magnesium	20	12	4
Manganese	4	2	1

TABLE 3 Concentrations of Mixed Metal Solutions 7, 8, and 9

	Standard 7, mg/L	Standard 8, mg/L	Standard 9, mg/L
Volume (mL)	10, Standard 1	10, Standard 2	10, Standard 3
Calcium	5	3	1
Potassium	3.5	2.1	0.7
Sodium	2.5	1.5	0.5

15.7 Repeat 15.6.

15.8 Remove the beakers from the hot plate and wait 5 min. Add 2 mL of HClO_4 (sp gr 1.67), and return the beakers to the hot plate. Continue heating until the solution has reached incipient dryness; however, do not bake the solutions.

15.9 Remove the beakers from the hot plate, and lower the temperature to 100°C . Add 2 mL of HCl (1 + 1) and swirl the beaker; add 10 mL of water and return to the hot plate to dissolve the residue.

NOTE 8—If quantitation is to be by graphite furnace atomic absorption spectrophotometry, substitute 2 mL of HNO_3 (1 + 1) for HCl (1 + 1). If quantitation is to be by hydride generation (for example, arsenic, antimony, or selenium), then substitute 25 mL of HCl (sp gr 1.19).

15.10 Cool the beakers, and pour each solution into a 50-mL volumetric flask. Rinse the beaker several times and bring to the mark with water (Note 9). Pour the solution into an acid-rinsed plastic bottle for storage. This solution represents a concentration of 10 g of sample per litre of solution (a dilution factor of 100).

NOTE 9—If a sample contains a large amount of organic matter, it is not unusual to have a final solution which contains black flecks in it. These can be ignored provided that when the solutions are aspirated into an atomic absorption spectrophotometer, they are allowed to settle first.

15.11 Remove a 5-mL aliquot from the 100x solution

(15.10), add 1 mL of HCl (sp gr 1.19) (substitute HNO_3 , sp gr 1.41 if quantitation is by graphite furnace), and 5 mL of CsCl solution (14.5) (Note 10), place in a 50-mL volumetric flask and bring to the mark with water. Pour the solution into an acid-rinsed plastic bottle for storage. This solution represents a concentration of 1 g of sample per litre of solution (a dilution factor of 1000).

NOTE 10— CsCl acts as an ionization suppressant and is used for flame atomic absorption spectrophotometry.

15.12 Remove a 5-mL aliquot from the 1000x solution (15.11), add 1 mL HCl (sp gr 1.19) (substitute HNO_3 , sp gr 1.41 if quantitation is by graphite furnace), and 5 mL of CsCl solution (14.5) (Note 10), place in a 50-mL volumetric flask, and bring to the mark with water. Pour the solution into an acid-rinsed plastic bottle for storage. This solution represents a concentration of 0.1 g of sample per litre of solution (a dilution factor of 10 000).

15.13 See the appropriate ASTM test methods for subsequent quantitation.

16. Keywords

16.1 chemical analysis; metal; sediment samples; total; total digestion

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