Designation: D4698 – 21

Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals[1]

This standard is issued under the fixed designation D4698; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 D3976 for any special preparatory considerations.

1.4 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 15.5.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)[3]

D1193 Specification for Reagent Water

D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *partial digestion, n*—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.

3.2.2 *total digestion, n*—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.

4. Summary of Practice

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

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is 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 last approved version of this historical standard is referenced on www.astm.org.

of either method, prior to final selection; or if a less rigorous digestion could be employed (**1-4**).[4] 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 D3976.

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.

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 (**1, 2**).

7.2 Interferences are eliminated or compensated for, or both, through the use of cesium chloride (CsCl), orthoboric acid (H_3BO_3), lithium metaborate ($LiBO_2$), lithium tetraborate ($Li_2B_4O_7$), 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, ¾-in. (19.05 mm) inside diameter, and total depth of 1⅜ 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.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without reducing 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 D1193.

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 constit-uents 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, $LiBO_2$, and 2 parts anhydrous lithium tetraborate, $Li_2B_4O_7$. 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.

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[5] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.