



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

This standard is issued under the fixed designation D 4698; 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 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.

Current edition approved Oct. 15, 1992. Published December 1992. Originally published as D 4698 – 87. Last previous edition D 4698 – 87.

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.