



Designation: D5198 – 17

Standard Practice for Nitric Acid Digestion of Solid Waste¹

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1. Scope

1.1 This practice describes the partial digestion of solid waste using nitric acid for the subsequent determination of the total recoverable content of inorganic constituents.

1.2 This practice is to be used when the concentrations of total recoverable elements are to be determined from a waste sample. Total recoverable elements are often not equivalent to total elemental content, because of the solubility of the speciated forms of the element in the sample matrix. Recovery from refractory sample matrices, such as soils, is usually significantly less than total concentrations of the elements present.

NOTE 1—This practice has been used successfully for oily sludges and a municipal digested sludge standard [Environmental Protection Agency (EPA) Sample No. 397]. The practice may be applicable to some elements not listed above, such as arsenic, barium, selenium, cobalt, magnesium, and calcium. Refractory elements such as silicon, silver, and titanium, as well as organo-mercury, are not solubilized by this practice.

1.3 This practice has been divided into two methods, A and B, with Method A utilizing an electric hot plate and Method B utilizing an electric digestion block.

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

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.*

¹ This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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2. Referenced Documents

- 2.1 *ASTM Standards*:²
D1193 Specification for Reagent Water
D5681 Terminology for Waste and Waste Management

3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, refer to Terminology D5681.

4. Summary of Practice

4.1 A weighed portion of the waste sample is mixed with 1 + 1 nitric acid (HNO₃) in an Erlenmeyer flask. The flask is heated on an electric hot plate (Method A) or electric digestion block (Method B) for 2 h at 90 to 95 °C to dissolve the elements of interest. After cooling, the contents of the flask are diluted with reagent water and filtered, and the filtrate is made up to appropriate volume for subsequent analysis.

5. Significance and Use

5.1 A knowledge of the inorganic composition of a waste is often required for the selection of appropriate waste disposal practices. Solid waste may exist in a variety of forms and contain a range of organic and inorganic constituents. This practice describes a digestion procedure which dissolves many of the toxic inorganic constituents and produces a solution suitable for determination of total recoverable contents by such techniques as atomic absorption spectroscopy, atomic emission spectroscopy, and so forth. The relatively large sample size aids representative sampling of heterogeneous wastes. The relatively small dilution factor allows lower detection limits than most other sample digestion methods. Volatile metals, such as lead and mercury, are not lost during this digestion procedure, however organo-lead and organo-mercury may not be completely digested. Hydride-forming elements, such as arsenic and selenium, may be partially lost. Samples with total metal contents greater than 5 % may give low results. The analyst is responsible for determining whether this practice is applicable to the solid waste being tested.

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.

METHOD A – HOT PLATE

6. Apparatus

- 6.1 *Analytical Balance*, capable of weighing to 0.01 g.
- 6.2 *Erlenmeyer Flasks*, 125 mL.
- 6.3 *Graduated Cylinder*, 50 mL.
- 6.4 *Electric Hot Plate*, adjustable, capable of maintaining a temperature of 90 to 95 °C.
- 6.5 *Watch Glasses*.
- 6.6 *Thermometer*.
- 6.7 *Funnels*, glass or plastic.
- 6.8 *Volumetric Flasks*, glass-stoppered, 200 mL.
- 6.9 *Filter Paper*, quantitative, medium flow rate, Whatman No. 40 or equivalent.
- 6.10 *Fume Hood*.

7. Reagents

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ 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 determination.

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

7.3 *Nitric Acid*, concentrated, reagent grade.

7.4 *Nitric Acid (1 + 1)*—Add slowly, with stirring, 200 mL of concentrated nitric acid (HNO₃, sp gr 1.42) to 200 mL water. Cool the mixture and store in a clean glass bottle.

8. Procedure

8.1 Weigh 5 g of a thoroughly mixed waste sample to the nearest 0.01 g into a tared Erlenmeyer flask.

8.2 With a graduated cylinder, slowly add 25 mL of 1 + 1 nitric acid to the flask. Swirl the flask to wet the sample completely.

8.3 Carry a blank of 25 mL of 1 + 1 nitric acid through the procedure.

8.4 Place the flask on a cold hot plate, cover with a watch glass, and set the hot plate to maintain a temperature of 90 to 95 °C.

8.5 Heat the flask and contents for 2 h, occasionally swirling the flask to wash down any sample adhering to the walls.

Check the solution temperature with the thermometer and adjust the heat if necessary.

8.6 After 2 h, remove the flask from the hot plate and cool to room temperature. Add 50 mL of reagent water to the flask, washing down the flask walls during addition. Swirl the flask to mix the contents.

8.7 Filter the contents of the flask into a 200 mL volumetric flask. Rinse the flask and filter paper with several small portions of reagent water and add the rinsings to the volumetric flask.

8.8 Dilute the solution in the volumetric flask to the mark with reagent water and mix thoroughly. The solution is now ready for analysis.

METHOD B – DIGESTION BLOCK

9. Apparatus

- 9.1 *Analytical Balance*, capable of weighing to 0.01 g.
- 9.2 *Fume Hood*.
- 9.3 *Graduated Digestion Tubes*.
- 9.4 *Graduated Cylinder*, 50 mL.
- 9.5 *Digestion Tube Filters*.
- 9.6 *Digestion Block*, adjustable, capable of maintaining a temperature of 90 to 95 °C.

10. Reagents

10.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ 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 determination.

10.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

10.3 *Nitric Acid*, concentrated, reagent grade.

10.4 *Nitric Acid (1 + 1)*—Add slowly, with stirring, 200 mL of concentrated nitric acid (HNO₃, sp gr 1.42) to 200 mL water. Cool the mixture and store in a clean glass bottle.

11. Procedure

11.1 Weigh 5 g of a thoroughly mixed waste sample to the nearest 0.01 g and transfer into a graduated digestion tube.

11.2 With a graduated cylinder, slowly add 25 mL of 1 + 1 nitric acid to the digestion tube. Swirl the tube to wet the sample completely.

11.3 Carry a blank of 25 mL of 1 + 1 nitric acid through the procedure.

11.4 Place the digestion tube in a digestion block in a fume hood and set the digestion block to maintain a temperature of 90 to 95 °C.

³ *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 *Reagent Chemicals and Standards*, by Joseph Rosin, D., Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia*.