Designation: D 4503 – 86 (Reapproved 2003)

Standard Practice for Dissolution of Solid Waste by Lithium Metaborate Fusion[1]

This standard is issued under the fixed designation D 4503; 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 the drying, ashing, and solubilization of solid waste using a lithium metaborate ($LiBO_2$) fusion for the subsequent determination of inorganic constituents by argon plasma emission spectroscopy or atomic absorption spectroscopy.

1.2 The following elements may be solubilized by this practice:

aluminum
barium
cadmium
calcium
copper
chromium
iron
magnesium
manganese
nickel
silicon
titanium
vanadium
zinc

1.3 This practice has been used successfully with a bauxite ore and a neutralized metal treatment sludge. The practice may be applicable to other elements not listed above. Some metals, such as cadmium and zinc, may volatilize from some samples during the drying, ashing, or fusion steps. The analyst is responsible for determining whether the practice is applicable to the solid waste being tested.

1.4 This practice is intended for the solubilization of nonvolatile inorganic constituents in solid waste. The $LiBO_2$ fusion is appropriate for a silicate matrix or acid resistant samples.

1.5 *This standard does not purport to address all of the safety problems 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 specific hazard statements see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1193 Specification for Reagent Water
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water
D 3682 Test Method for Major and Minor Elements in Coal and Coke Ash by Atomic Absorption
E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals

3. Summary of Practice

3.1 The solid waste is weighed, dried, and ashed at 550°C to remove water and organic constituents, and reweighed. A known portion of the ground ash is mixed with $LiBO_2$ in a graphite crucible and fused at 1000°C. Immediately after fusion, the molten mass is poured directly into stirred dilute HNO_3 solution, dissolved, filtered, and made to appropriate volume for subsequent analysis.

4. Significance and Use

4.1 A knowledge of the inorganic constituent composition in 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 drying and ashing step that may be applied to remove moisture and volatile and nonvolatile organic constituents prior to determining nonvolatile metals. Generation of a dry ash concentrates the inorganic constituents of interest and makes the $LiBO_2$ fusion feasible for a greater variety of waste samples. Acidification of the $LiBO_2$ fusion mix results in a solution amenable to inductively coupled plasma (ICP) or atomic absorption spectrometry (AAS) analysis.

5. Apparatus

5.1 *Analytical Balance*, sensitive to 0.1 mg.

5.2 *Fusion Muffle Furnace*, electrically heated, capable of maintaining a temperature of 1000°C.

5.3 *Ashing Muffle Furnace*, electrically heated, capable of maintaining a temperature of 550°C ± 30°C and with an adequate air circulation. This may be accomplished by connecting rubber tubing to a controlled source of clean dry air. Then, via a ceramic tube inserted into a convenient muffle opening, flow approximately 4 L/min of air into the furnace.

5.4 *Drying Oven*, capable of operating at a temperature up to 150°C.

5.5 *Evaporating/Ashing Dish*, 50 to 100-mL capacity, made of platinum, silica, or porcelain.

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[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] 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.

5.6 *Fusion Crucibles*, graphite, 28 to 30-mL capacity.

5.7 *Stirring Hot Plate*, capable of operating at a surface temperature up to 300°C with TFE-fluorocarbon-coated stir magnet.

5.8 *Mortar and Pestle*, agate or mullite type.

5.9 *Sieve and Pan*, ASTM U.S. Standard Testing Sieve, 200 m (75 µm opening).

5.10 *Desiccator*.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intendedthat 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.

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

6.3 *Lithium Metaborate*—$LiBo_2$, anhydrous powder.

6.4 *Nitric Acid, Solution (5 ± 95)*—Add while stirring 50-mL of nitric acid (HNO_3, sp. gr. 1.42) to 900 mL of water. Make to 1 L volume and store in a polyethylene bottle.

6.5 *Dessicant*—Anhydrous calcium sulfate ($CaSO_4$) or equivalent.

7. Hazards

7.1 Samples known or suspected of containing toxic or hazardous materials must be handled in a fume hood. Safety information relative to the handling of any known toxic materials must be obtained and implemented prior to any sample handling.

7.2 Flammable materials must be kept from heat, sparks, or flames.

7.3 Drying should be conducted on an explosion proof electrical heater in a fume hood if use of a conventional drying apparatus may result in toxic, flammable, or irritating vapors.

7.4 Ashing at 550°C must be conducted in a hood with adequate ventilation and shielding. Refer to Practices E 50 for additional information.

8. Procedure

8.1 Prepare the analytical sample from a thoroughly mixed representative laboratory sample.

8.2 Weigh sufficient sample to the nearest 1 mg into a tared ashing dish so that after drying and ashing at least 2 g remains for use in 8.6. Typically 5 to 10 g of waste is sufficient unless moisture and organic content are a major portion of the sample.

8.3 Dry the sample at 110 to 150°C. If appreciable free moisture or volatiles are present, continue drying until the sample is suitable for ashing.

8.4 Place the sample into an ashing furnace set at about 300°C and increase heat gradually so the furnace reaches 550°C in 1 h. Ash at 550°C until no carbonaceous matter is apparent. Stirring the sample once an hour may increase the oxidation of carbonaceous matter. The ashing time required will depend on the nature of the sample. Several hours, or even overnight, may be required by difficult-to-ash samples.

8.5 Remove the ashing dish and sample from the muffle, cool in a desiccator, and weigh to determine the combined loss on ashing and drying.

8.6 Quantitatively transfer the ash to a mortar and grind to pass a No. 200 sieve, if necessary. Transfer back to the ashing dish and reheat the ground ash at 550°C for 1 h, remove from the ashing furnace and cool in a desiccator. Transfer quantitatively to a weighing bottle. Weigh approximately 0.3 g of sample to the nearest 0.0001 g by difference into a graphite crucible containing 1.5 g of $LiBO_2$. Mix the ash and $LiBO_2$ well, then add an additional 0.5 g of $LiBO_2$ on top of the mix.

NOTE 1—Ashing at 550°C typically gives a free flowing or friable ash, so quantitative transfer is possible with careful brushing. Should a portion of the ash melt or stick to the dish so quantitative transfer is impossible, the analyst should use a lower ashing temperature or consider an alternative dissolution practice.

8.7 Place the crucible in a muffle furnace preheated to 1000°C and fuse for 20 min. Remove the crucible from the muffle, swirl to consolidate the molten bead, and pour into a 250-mL beaker containing 150 mL of HNO_3 solution (5 ± 95). The acid solution should be warm (50 to 70°C) and stirred with a stirring hot plate. Complete dissolution of the melt, other than traces of graphite particles from the crucible, should occur in 10 to 15 min.

8.8 Gravity filter the solution through a medium filter, such as Whatman No. 41, into a 250-mL volumetric flask. Quantitatively wash the beaker and filter with water. Add the wash water to the filtrate, cool, dilute to volume with water, and mix. This solution is ready for ICP or AAS analysis. Refer to Test Method D 3682 for an AAS analysis method.

8.9 Carry a $LiBO_2$ blank as in 8.6-8.8 for use as a method blank in the analytical step.

9. Precision and Bias

9.1 Six laboratories participated in a collaborative test program. Two wastes were tested with a single operator at each laboratory performing the practice once on each of 3 days. A central laboratory analyzed all the prepared solutions using an inductively coupled argon plasma spectrometer.

9.1.1 The mean, single-operator precision (S_o), and overall precision (S_T) for the elements solubilized by this practice are shown in Table 1. Practice D 2777 was used in developing these precision estimates.

9.2 Determination of the bias of this practice is not possible, as no suitable standard reference material exists.

9.2.1 Comparison of the analytical values obtained using this practice with those obtained by a single laboratory using classical dissolution and analytical methods is shown in Table 2. The data indicate a suitable degree of agreement between independent methods.

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[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*, Joseph Rosin, D. Van Nostrand and Co., Inc., New York, NY, and the *United States Pharmacopeia.*