Designation: E 886 – 94

Standard Practices for Dissolution of Refuse-Derived Fuel (RDF) Ash Samples for Analyses of Metals[1]

This standard is issued under the fixed designation E 886; 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 These practices described herein cover the preparation of RDF ash, fly ash, bottom ash, or slag for analyses of metals by atomic absorption spectroscopy or inductively coupled plasma spectroscopy, or both (see Appendix X1).

1.2 These practices may be applicable to any waste material from which a laboratory analysis sample can be prepared.

1.3 Three practices are described in this standard:

1.3.1 *Practice A*—Lithium tetraborate ($Li_2B_4O_7$) fusion,

1.3.2 *Practice B*—Aqua regia dissolution, and

1.3.3 *Practice C*—Bomb, acid digestion method.

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 hazard statements, see Sections 4, 20.3, and 20.6.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 885 Test Methods for Analyses of Metals in Refuse-Derived Fuel by Atomic Absorption Spectroscopy[3]

3. Significance and Use

3.1 These practices are available to producers and users of RDF as methods of preparing RDF ashes for subsequent analyses of metals.

3.2 The chemical composition of laboratory prepared RDF ashes do not exactly represent the composition of mineral matter in the RDF or the composition of fly ash, bottom ash, and slag resulting from commercial-scale burning of RDF. These practices are for analyzing the various ash samples as received in the laboratory.

4. Hazards

4.1 The hot acidic solutions in these practices pose a significant potential hazard. Proper laboratory safety practices and equipment should be employed throughout these procedures.

5. Sampling

5.1 The method of sampling for these procedures should be based on agreement between involved parties.

PRACTICE A—LITHIUM TETRABORATE FUSION

6. Summary of Practice

6.1 The RDF ash, fly ash, bottom ash, or slag is fused with lithium tetraborate ($Li_2B_4O_7$) followed by a final dissolution of the melt in dilute hydrochloric acid (HCl). The solution is analyzed for metals by atomic absorption spectroscopy or inductively coupled plasma spectroscopy.

7. Apparatus

7.1 *Analytical Balance*, capable of weighing to 0.0001 g.

7.2 *Muffle Furnace*— The furnace shall have adequate air ventilation and shall be capable of temperature regulation up to at least 1000°C. An air change rate of 1 to 4 furnace volumes of air per minute has been found adequate.

7.3 *Platinum Dish*, 35 to 85-mL capacity, or

7.3.1 *Graphite Crucibles*, 35 to 85-mL capacity.

7.4 *Stirring Hotplate and Stirring Bars*, operating temperature of 200°C.

7.5 *Volumetric Flasks*, 50, 100, and 200-mL.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this test. 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.[4] 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.

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[1] These practices are under the jurisdiction of ASTM Committee D34 on Waste Management and are the direct responsibility of Subcommittee D34.06 on Recovery and Reuse.

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

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D 1193.

8.3 *Lithium Tetraborate* —($Li_2B_4O_7$), powder.

8.4 *Hydrochloric Acid* (HCl), (5+95)—Dilute 50 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1000 mL with water.

9. Preparation of Sample

9.1 Prepare the RDF ash, fly ash, bottom ash, or slag by grinding the sample in an agate mortar to a particle size to pass a No. 200 (74-µm) sieve.

10. Procedure

10.1 *Sample Fusion*:

NOTE 1—Stronger or weaker concentrations may be required to establish a suitable range for those metals of varying concentrations outside the range of the typical sample. Each analyst must determine the sensitivity and linear range of his own equipment and choose concentration ranges from standards, sample weight, and dilution factors all compatible with the instrument specific for his work. For very trace concentrations of any metal in the ash, multiple fusions may be made and the dissolved fusion beads pooled into one sample.

10.1.1 Weigh 0.1 ± 0.0001 g of sample as prepared in Section 9 into a platinum dish or graphite crucible.

10.1.2 Add 0.5 g of $Li_2B_4O_7$. Mix the sample and $Li_2B_4O_7$ powder well. Then add an additional 0.5 g of $Li_2B_4O_7$ to cover the mixture.

10.1.3 Place the dish in a clean silica or refractory tray and place in a muffle furnace preheated to 1000°C; 15 min at 1000°C is sufficient to fuse the mixture completely.

10.1.4 Remove the tray and dish from the muffle furnace, and cool to room temperature.

10.2 *Dissolution*:

10.2.1 Carefully rinse the bottom and outside of the platinum dish or graphite crucible to remove possible contamination. Then place it in a clean 250 or 400-mL beaker.

10.2.2 Place a clean TFE-fluorocarbon-coated stirring magnet inside the dish and add 150 mL of HCl (5+95) to the beaker and dish, and place immediately on the stirring hotplate.

10.2.3 Heat the solution to just below boiling temperature and maintain for not more than 30 min with constant stirring. This time and temperature is sufficient to completely dissolve the metal. If stirring is not maintained constantly, some of the ash constituents are apt to precipitate and the analysis must be repeated.

10.2.4 Remove the beaker from the hotplate, and permit to cool to room temperature.

10.2.5 Quantitatively transfer the solution to a 200-mL volumetric flask, wash the platinum dish or graphite crucible and beaker with small amounts of HCl (5+95), and dilute to the 200-mL mark with the HCl solution. This solution (200 mL) contains those metals as were contained in 0.1 g of the ash (500 ppm) and 5 grams per litre of $Li_2B_4O_7$ solution.

10.2.6 This solution (10.2.5) can be either concentrated or diluted depending upon the magnitude of concentration of the metal in question (see Note 1).

10.3 Analyze this solution for the metal analyte in question in accordance with Test Methods E 885.

10.4 Prepare reagent blanks using the procedure outlined in 10.1.2 through 10.3.

PRACTICE B—AQUA REGIA DISSOLUTION

11. Summary of Practice

11.1 The RDF ash, fly ash, bottom ash, or slag is dissolved in mineral acids. The resulting solution is analyzed for metals by atomic absorption spectroscopy or inductively coupled plasma spectroscopy.

12. Apparatus

12.1 *Analytical Balance*, capable of weighing to 0.0001 g.

12.2 *Bottles*, polyethylene or polytetrafluoroethylene, 125-mL capacity with screw-cap lids, capable of withstanding 130°C.

12.3 *Steam Bath*.

12.4 *Volumetric Flasks*, 50, 100, and 200-mL.

12.5 *Crucibles*, 50-mL quartz or high silica.

13. Reagents

13.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this test. 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.[4] 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.

13.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D 1193.

13.3 *Aqua Regia Solution*—Mix 1 part concentrated nitric acid (HNO_3, sp gr 1.42), 3 parts concentrated hydrochloric acid (HCl, sp gr 1.19), and 1 part water.

13.4 *Hydrofluoric Acid* (HF)—Concentrated sp gr 1.15.

13.5 *Boric Acid* (H_3BO_3)—Saturated solution.

14. Preparation of Sample

14.1 Prepare the RDF ash, fly ash, bottom ash, or slag by grinding the sample in an agate mortar to a particle size to pass a No. 200 (74-µm) sieve.

15. Procedure

15.1 *Dissolution*:

15.1.1 Weigh accurately about 0.2 g of ash sample as prepared in 14.1, and add to a 125-mL plastic bottle with a screw cap.

15.1.2 Add 3 mL of aqua regia solution and 5 mL of HF to the sample.

15.1.3 Tighten the screw-cap, and place bottle on a steam bath for at least 2 h.

15.1.4 Add 50 mL of saturated H_3BO_3 solution to the resultant solution (see Note 2). If a residue remains, the mixture may be reheated for about 1 h to help dissolve it (see Note 3).

NOTE 2—Boric acid (H_3BO_3) not only complexes fluoride (F^-), but it also has been shown to have good flame properties and acts as a flame buffer.