



Designation: E 887 – 88 (Reapproved 1996)

## Standard Test Method for Silica in Refuse-Derived Fuel (RDF) and RDF Ash<sup>1</sup>

This standard is issued under the fixed designation E 887; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of silica in RDF, RDF ash, fly ash, bottom ash, or slag.

1.2 The test method is an acid dehydration gravimetric procedure and is independent of interferences.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 statement, see Section 6.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

E 791 Test Method for Calculating Refuse-Derived Fuel Analysis Data from As-Determined to Different Bases<sup>3</sup>

E 829 Practice for Preparing Refuse-Derived-Fuel (RDF) Laboratory Samples for Analysis<sup>3</sup>

E 830 Test Method for Ash in the Analysis Sample of Refuse-Derived Fuel<sup>3</sup>

E 856 Definitions of Terms and Abbreviations Relating to Physical and Chemical Characteristics of Refuse-Derived Fuel<sup>3</sup>

### 3. Summary of Test Method

3.1 Silicon compounds in RDF ash, fly ash, bottom ash, or slag are dissolved by alkali fusion and dehydrated with hydrochloric acid (HCl). Dehydration is completed by ignition, and the silica is volatilized as silicon tetrafluoride.

### 4. Apparatus

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

4.2 *Muffle Furnace*—The furnace shall have an operating temperature of up to 1200°C.

4.3 *Hot Plate or Steam Bath*.

4.4 *Platinum Crucibles*, 35 to 85-mL capacity.

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

4.6 *Fused Quartz Dishes*, 35 to 85-mL capacity.

### 5. Reagents and Materials

5.1 *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.<sup>4</sup> 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.

5.2 *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.

5.3 *Sodium Carbonate* ( $\text{Na}_2\text{CO}_3$ ), anhydrous powder.

5.4 *Hydrochloric Acid* (HCl), concentrated, sp gr 1.19.

5.5 *Hydrochloric Acid* (1 + 3), Mix 1 volume of concentrated HCl with 3 volumes of water.

5.6 *Hydrochloric Acid* (1 + 1)—Mix 1 volume of concentrated HCl with 1 volume of water.

5.7 *Hydrochloric Acid* (1 + 99)—Mix 1 volume of concentrated HCl with 99 volumes of water.

5.8 *Sulfuric Acid* (1 + 1)—Mix 1 volume of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ , sp gr 1.84) with 1 volume of water.

5.9 *Hydrofluoric Acid* (HF), concentrated 48 to 51 %.

### 6. Hazards

6.1 Due to the origins of RDF in municipal waste, common sense dictates that precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF; wearing dust masks (NIOSH-approved type), especially while milling RDF samples; conducting tests under negative pressure hoods when possible; and washing hands before eating or smoking.

### 7. Sampling

7.1 *Refuse-Derived Fuel (RDF)*:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.03 on Treatment.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.04.

<sup>4</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

NOTE 1—ASTM Subcommittee E38.01 is currently in the process of developing procedures for sampling RDF.

7.1.1 RDF products are frequently nonhomogeneous. For this reason, significant care should be exercised to obtain a representative laboratory sample from the RDF lot to be characterized.

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

7.1.3 The laboratory sample must be air-dried and particle size reduced to pass a 0.5-mm screen for analysis. This procedure must be performed carefully to preserve the sample's representative characteristics (other than particle size) while preparing the analysis sample to be used in this procedure (see Practice E 829).

7.2 *Refuse-Derived Fuel Ash, Fly Ash, Bottom Ash, or Slag*—The method of sampling for this procedure should be based on agreement between involved parties.

## 8. Sample Preparation

### 8.1 *Refuse-Derived Fuel*:

8.1.1 Weigh accurately 30 to 50 g of RDF analysis sample as prepared in 7.16.1 into a conditioned and preweighed fused quartz dish.

8.1.2 Spread out the analysis sample of RDF in a layer not over 38.1 mm (1½ in.) in depth.

8.1.3 Place the dish in the muffle at a low temperature (not greater than 100°C) and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter.

8.1.4 Complete the conversion to ash at a temperature of 800 to 900°C (1470 to 1650°F).

8.1.5 Cool in a desiccator and stir the ash to ensure homogeneity of particle sizes. Be careful not to lose any ash from the dish during this stirring.

8.1.6 Spread the ash in a thin layer in the dish, and ignite in a stream of oxygen for 1½ h at 800 to 850°C (1470 to 1560°F) to ensure complete and uniform oxidation of the ash.

8.1.7 Cool the ash to room temperature in a desiccator.

8.1.8 Weigh the dish and the ash.

8.1.9 Calculate the percent ash as follows:

$$\% \text{ Ash} = (C - A)/(B - A) \times 100 \quad (1)$$

where:

*A* = weight of fused quartz, g,

*B* = weight of fused quartz dish and sample, g, and

*C* = weight of fused quartz dish and ash, g.

### 8.2 *Refuse-Derived Fuel Ash, Fly Ash, Bottom Ash, or Slag*:

8.2.1 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 (75-µm) sieve.

8.2.2 Weigh accurately 6 to 10 g of RDF ash, fly ash, bottom ash, or slag as prepared in 8.2.1 into a conditioned preweighed fused quartz dish.

8.2.3 Spread out the analysis sample of the ash to be analyzed in a layer not over 6.4 mm (¼ in.) in depth.

8.2.4 Place the dish in the muffle at a low temperature (not greater than 100°C), and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter.

8.2.5 Complete the conversion to ash at a temperature of 800 to 900°C (1470 to 1659°F).

8.2.6 Cool in a desiccator and stir the ash to ensure homogeneity of particle sizes. Be careful not to lose any ash from the dish during this stirring.

8.2.7 Spread the ash in a thin layer in the dish and ignite in a stream of oxygen for 1½ h at 800 to 850°C (1470 to 1560°F) to ensure complete and uniform oxidation of the ash.

8.2.8 Cool the ash to room temperature in a desiccator.

8.2.9 Weigh the dish and the ash.

8.2.10 Calculate the percent residue after ignition as follows:

$$\% \text{ Residue after ignition} = (F - D)/(E - D) \times 100 \quad (2)$$

where:

*D* = weight of fused quartz dish, g,

*E* = weight of fused quartz dish and sample, of RDF ash, fly ash, bottom ash or slag, g, and

*F* = weight of fused quartz dish and residue after ignition of RDF ash, fly ash, bottom ash, or slag, g.

## 9. Procedure

### 9.1 *Sample Fusion*:

9.1.1 Weigh accurately 0.010 to 0.100 g of the prepared RDF ash as prepared in 8.1 or the residue of RDF ash, fly ash, bottom ash, or slag as prepared in 8.2 into a platinum or graphite crucible.

9.1.2 Add 1.0 g of Na<sub>2</sub>CO<sub>3</sub>. Mix the ash and Na<sub>2</sub>CO<sub>3</sub> well, then add an additional 0.5 g of Na<sub>2</sub>CO<sub>3</sub> to cover the mixture.

9.1.3 Place the crucible into a clean silica or refractory tray, and place in a muffle furnace preheated to 1000°C and maintain until the mass is quiescent (about 45 min).

9.1.4 Set the crucible aside to cool.

9.1.5 Rinse off the outside of the crucible, and place it on its side in a 300-mL casserole or beaker about one-third full of water. Warm and stir until the cake disintegrates and can be easily removed.

9.1.6 By means of platinum tipped tongs, lift the crucible out of the liquid, rinsing it thoroughly with water followed by rinsing with dilute hydrochloric acid (HCl, 1 + 3); adding the rinse to the casserole or beaker.

### 9.2 *Acid Dehydration*:

9.2.1 Very slowly and cautiously add 20 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to the covered casserole or beaker (see Note 2). Remove the cover and rinse. If any gritty particles are present, the fusion is incomplete and must be repeated, using a new sample and a larger amount of Na<sub>2</sub>CO<sub>3</sub>.

NOTE 2—This solution will tend to “creep” over the rim of the casserole unless the rim is kept hot and dry. This can be accomplished by applying heat from above by means of infrared lamps.

9.2.2 Evaporate the solution to dryness on a steam bath.

9.2.3 Without heating the residue any further, treat it with 5 to 10 mL of HCl (concentrated), wait at least 2 min, then add an equal amount of water.

9.2.4 Cover the casserole or beaker, and digest for 10 min on the steam bath or hot plate.

9.2.5 Dilute the solution with an equal volume of hot water, immediately filter through medium-textured ashless paper, and wash the residue thoroughly with hot HCl (1 + 99), then with hot water.

9.2.6 Reserve the residue.

9.2.7 Again, evaporate the filtrate to dryness and bake the residue in an oven for 1 h at 105 to 110°C.

9.2.8 Cool, add 10 to 15 mL HCl (1 + 1) and digest on the steam bath or hot plate for 10 min.

9.2.9 Dilute with an equal volume of water, filter immediately on a fresh filter paper, and wash the small amount of residue thoroughly with hot HCl (1 + 99), then with hot water.

9.2.10 Transfer the papers containing the residues (from 9.2.6 and 9.2.9) to a weighed conditioned platinum crucible.

9.2.11 Dry and ignite the papers, first at a low heat until the carbon of the filter paper is completely consumed without flaming, and finally ignite at 1100 to 1200°C until the weight becomes constant. Record weight of residue after ignition.

9.3 *Volatilization of Silicon Tetrafluoride:*

9.3.1 Treat the silica (SiO<sub>2</sub>) thus obtained, which will contain impurities, in the crucible with 0.5 to 1 mL of water, 2 drops of H<sub>2</sub>SO<sub>4</sub> (1 + 1) and 10 mL of HF.

9.3.2 Cautiously evaporate to dryness on a hot plate or hot sand bath.

9.3.3 Finally, ignite the small residue at 1050 to 1100°C (1922 to 2012°F) for 5 min.

9.3.4 Cool in a desiccator and weigh.

9.3.5 The difference between this weight and the weight previously obtained in 9.2.11 represents the amount of SiO<sub>2</sub>.

9.4 *Blank*—Make a blank determination, following the same procedures as used in 9.1.2 through 9.3.5 using the same amounts of reagents and correct the obtained in the analysis accordingly.

## 10. Calculation

10.1 Calculate the concentration of SiO<sub>2</sub> as follows:

10.1.1 Percent SiO<sub>2</sub> on prepared sample as used in 9.1.1.

$$\% \text{SiO}_2 = (W_1 - W_2) - (W_3 - W_4)/W_5 \times 100 \quad (3)$$

where:

$W_1$  = weight of residue after first ignition as in 9.2.11, g,

$W_2$  = weight of blank after first ignition, g,

$W_3$  = weight of residue after treatment with HF and second ignition as in 9.3.4, g,

$W_4$  = weight of blank after treatment with HF and second ignition, g, and

$W_5$  = weight of sample of prepared RDF, RDF ash, fly ash, bottom ash, or slag as used in 9.1.1, g.

10.1.2 Percent SiO<sub>2</sub> in RDF (6.1).

$$\% \text{SiO}_2 \text{ in RDF} = (G \times H)/(100) \quad (4)$$

where:

$G$  = ash as found in 8.1.9, %, and

$H$  = SiO<sub>2</sub> as found in 10.1.1, %.

10.1.3 Percent SiO<sub>2</sub> in RDF ash, fly ash, bottom ash, or slag (8.2).

$$\% \text{SiO}_2 \text{ in RDF Ash, fly ash, bottom ash, or slag} = (I \times H)/(100) \quad (5)$$

where:

$I$  = residue after ignition as found in 8.2.10, %, and

$H$  = SiO<sub>2</sub> as found in 10.1.1, %.

See Method E 791 for procedures to convert values to other bases.

## 11. Precision and Bias

11.1 Precision and bias statements cannot be made at this time for the measurement of SiO<sub>2</sub> by this test method.

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