Designation: E887 – 21

Standard Test Method for
Silica in Refuse-Derived Fuel (RDF) and RDF Ash[1]

This standard is issued under the fixed designation E887; 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 test method covers the determination of silica in refuse-derived fuel (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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For hazard statement, see Section 7.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
D5681 Terminology for Waste and Waste Management
D6044 Guide for Representative Sampling for Management of Waste and Contaminated Media
E791 Test Method for Calculating Refuse-Derived Fuel Analysis Data from As-Determined to Different Bases
E829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D5681.

4. Summary of Test Method

4.1 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.

5. Apparatus

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

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

5.3 *Hot Plate, Microwave Digester, or Steam Bath.*

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

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

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

6. Reagents and Materials

6.1 *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.[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, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D1193.

6.3 *Sodium Carbonate* (Na_2CO_3), anhydrous powder.

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

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[1] 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, Recovery and Reuse.

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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.

[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 *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

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

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

6.8 *Sulfuric Acid* (1 + 1)—Mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 1 volume of water.

6.9 *Hydrofluric Acid* (HF), concentrated 48 to 51 %.

7. Hazards

7.1 Due to the origins of RDF in municipal waste, precautions should be observed when conducting tests on samples. Recommended safety practices include use of suitable 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 upon completion of activity and before eating or smoking.

8. Sampling

NOTE 1—See Guide D6044.

8.1 *Refuse-Derived Fuel (RDF):*

8.1.1 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.

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

8.1.3 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 E829).

8.2 *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.

9. Sample Preparation

9.1 *Refuse-Derived Fuel:*

9.1.1 Weigh accurately 30 to 50 g of RDF analysis sample as prepared in 7.1 into a conditioned and preweighed fused quartz dish.

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

9.1.3 Place the dish in the muffle furnace 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.

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

9.1.5 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.

9.1.6 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.

9.1.7 Cool the ash to room temperature in a desiccator.

9.1.8 Weigh the dish and the ash.

9.1.9 Calculate the percent ash as follows:

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

where:

A = weight of fused quartz dish, g,
B = weight of fused quartz dish and sample, g, and
C = weight of fused quartz dish and ash, g.

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

9.2.1 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.

9.2.2 Weigh accurately 6 to 10 g of RDF ash, fly ash, bottom ash, or slag as prepared in 9.2.1 into a conditioned preweighed fused quartz dish.

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

9.2.4 Place the dish in the muffle furnace 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.

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

9.2.6 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.

9.2.7 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.

9.2.8 Cool the ash to room temperature in a desiccator.

9.2.9 Weigh the dish and the ash.

9.2.10 Calculate the percent residue after ignition as follows:

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

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.

10. Procedure

10.1 *Sample Fusion:*

10.1.1 Weigh 0.010 to 0.100 g of the prepared RDF ash as prepared in 9.1 or the residue of RDF ash, fly ash, bottom ash, or slag as prepared in 9.2 into a platinum or graphite crucible.

10.1.2 Add 1.0 g of Na_2CO_3. Mix the ash and Na_2CO_3 well, then add an additional 0.5 g of Na_2CO_3 to cover the mixture.

10.1.3 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).

10.1.4 Set the crucible aside to cool.

10.1.5 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.

10.1.6 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.

10.2 *Acid Dehydration:*

10.2.1 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_2CO_3.

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.

10.2.2 Evaporate the solution to dryness on a steam bath or hot plate.

10.2.3 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.

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

10.2.5 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.

10.2.6 Reserve the residue.

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

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

10.2.9 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.

10.2.10 Transfer the paper containing the residues (from 10.2.6 and 10.2.9) to a weighed conditioned platinum crucible.

10.2.11 Dry and ignite the paper, 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 the weight of residue after ignition.

10.3 *Volatilization of Silicon Tetrafluoride:*

10.3.1 Treat the silica (SiO_2) thus obtained, which will contain impurities, in the crucible with 0.5 to 1 mL of water, two drops of H_2SO_4 (1 + 1), and 10 mL of HF (48 to 51 %).

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

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

10.3.4 Cool in a desiccator and weigh.

10.3.5 The difference between this weight and the weight previously obtained in 10.2.11 represents the amount of SiO_2.

10.4 *Blank*—Make a blank determination, following the same procedures as used in 10.1.2 through 10.3.5 using the same amounts of reagents and correct the obtained in the analysis accordingly.

11. Calculation

11.1 Calculate the concentration of SiO_2 as follows:

11.1.1 Percent SiO_2 on prepared sample as used in 10.1.1.

$$\%\ SiO_2 = (W_1 - W_2) - (W_3 - W_4)/W_5 \times 100 \qquad (3)$$

where:

W_1 = weight of residue after first ignition as in 10.2.11, g,
W_2 = weight of blank after first ignition, g,
W_3 = weight of residue after treatment with HF and second ignition as in 10.3.4, 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 10.1.1, g.

11.1.2 Percent SiO_2 in RDF (7.1).

$$\%\ SiO_2 \text{ in RDF} = (G \times H)/(100) \qquad (4)$$

where:

G = ash as found in 9.1.9, %, and
H = SiO_2 as found in 11.1.1, %.

11.1.3 Percent SiO_2 in RDF ash, fly ash, bottom ash, or slag (9.2).

$$\%\ SiO_2 \text{ in RDF ash, fly ash, bottom ash, or slag} = (I \times H)/(100) \qquad (5)$$

where:

I = residue after ignition as found in 9.2.10, %, and
H = SiO_2 as found in 11.1.1, %.

See Test Method E791 for procedures to convert values to other bases.

12. Precision and Bias

12.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method E887 for measuring SiO_2 because a suitable reference is unavailable.

12.2 *Bias*—The bias of this test method has not been determined due to a lack of an accepted standard reference material.