



Designation: **E775 – 87 (Reapproved 2008)^{ε1} E775 – 15**

Standard Test Methods for Total Sulfur in the Analysis Sample of Refuse-Derived Fuel¹

This standard is issued under the fixed designation E775; 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.

^{ε1} NOTE—Terms were removed and reference to Terminology D 5681 was added editorially to Section 3 in December 2008. Also, units statement was added.

1. Scope

1.1 These test methods ~~cover~~^{represent} two alternative procedures for the determination of total sulfur in prepared analysis samples of solid forms of refuse-derived fuel (RDF). Sulfur is included in the ultimate analysis of RDF.

1.2 The test methods appear in the following order:

Test	Sections
Eschka Method	8 – 11
Bomb Washing Method	12 and 13

1.3 These test methods may be applicable to any waste material from which a laboratory analysis sample can be prepared.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard as standard. Inch-pound units are provided for information.

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 and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements see Section 6.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D5681 Terminology for Waste and Waste Management

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E711 Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter (Withdrawn 2011)³

E829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis (Withdrawn 2002)³

3. Terminology

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

4. Summary of Test Methods

4.1 *Eschka Method*—A weighed sample and Eschka mixture are ignited together and the sulfur is precipitated from the resulting solution as barium sulfate (BaSO_4). The precipitate is filtered, ashed, and weighed.

4.2 *Bomb Washing Method*—Sulfur is precipitated as BaSO_4 from the oxygen-bomb calorimeter washings and the precipitate is filtered, ashed, and weighed.

¹ These test methods are under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.03-02D34.03 on Municipal Treatment, Recovery and Reuse (Disbanded 06/09).

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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 The last approved version of this historical standard is referenced on www.astm.org.

5. Significance and Use

5.1 ~~The standards are available to~~ These procedures are used by producers and users of RDF for determining the total sulfur content of the fuel.

6. Precautions

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

7. Sampling

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

7.2 The sampling method for this procedure should be based on agreement between the involved parties.

7.3 The laboratory sample must be air-dried and particle size reduced to pass through a 0.5-mm screen as described in Practice **E829**. This procedure must be performed carefully to preserve the sample's ~~representative characteristics (other than particle size)~~ representativeness beyond just particle size while preparing the analysis sample to be ~~used in the~~ analyzed according to these procedures.

TEST METHOD A—ESCHKA METHOD

8. Apparatus

8.1 *Gas (Note 1) or Electric Muffle Furnace or Burners*, for igniting the sample with Eschka mixture and for igniting the barium sulfate (BaSO_4).

NOTE 1—~~Gas may used can~~ contain sulfur compounds in sufficient quantities to ~~affect the results~~ positively bias the results. The gas may require sulfur compound removal prior to use.

8.2 *Crucibles or Capsules*—Porcelain capsules, $\frac{7}{8}$ in. (22 mm) in depth and $1\frac{3}{4}$ in. (44 mm) in diameter, or porcelain crucibles of 30-mL capacity, high or low-form, or platinum crucibles of similar size shall be used for igniting the sample with the Eschka mixture. Porcelain, platinum, Alundum, or silica crucibles of 10 to 15-mL capacity shall be used for the final ignition step (see **10.3.8**).

9. Reagents

9.1 *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 American Chemical Society, where such specifications are available. Other grades⁴ may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without ~~lessening~~ impacting the accuracy of the determination.

9.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type III conforming to Specification **D1193**.

9.3 *Barium Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) and dilute to 1 L with water.

9.4 *Bromine Water* (saturated)—Add an excess of bromine to 1 L of water.

9.5 *Eschka Mixture*—Thoroughly mix 2 parts by weight of light calcined magnesium oxide (MgO) with 1 part of anhydrous sodium carbonate (Na_2CO_3). Both materials should be as free as possible from sulfur.

9.6 *Hydrochloric Acid* (1 + 1)—Mix equal volumes of concentrated HCl (sp gr 1.19) and water.

9.7 *Hydrochloric Acid* (1 + 9)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 9 volumes of water.

9.8 *Methyl Orange Indicator Solution* (0.2 g/L)—Dissolve 0.2 g of methyl orange in 1000 mL of hot water and filter.

9.9 *Sodium Carbonate* (saturated solution)—Dissolve approximately 60 g of crystallized sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$) or 20 g of anhydrous sodium carbonate (Na_2CO_3) in 100 mL of water, using a sufficient excess of Na_2CO_3 to ensure a saturated solution.

9.10 *Sodium Hydroxide Solution* (100 g/L)—Dissolve 100 g of sodium hydroxide (NaOH) in 1 L of water. This solution may be used in place of Na_2CO_3 solution.

⁴ "Reagent Chemicals, American Chemical Society Specification," *American Chemical Society*, Washington, DC. For suggestions on 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."