



Designation: E775 – 15

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

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1. Scope

1.1 These test methods present two alternative procedures for the determination of total sulfur in prepared analysis samples of solid 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 SI units are to be regarded 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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³
 - E711 Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter (Withdrawn 2011)³

¹ These test methods are 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 The last approved version of this historical standard is referenced on www.astm.org.

E829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis (Withdrawn 2002)³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 oxygen-bomb calorimeter washings and the precipitate is filtered, ashed, and weighed.

5. Significance and Use

5.1 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, precautions should be observed when conducting tests on samples. Recommended 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 inhomogeneous. For this reason significant care should be exercised to obtain a representative 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 representativeness beyond just particle size while preparing the analysis sample to be 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 used can contain sulfur compounds in sufficient quantities to 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 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.

10. Procedure

10.1 *Preparation of Sample and Eschka Mixture*—Weigh to the nearest 0.1 mg about 1 g of mixed air-dried analysis sample and 3 g of Eschka mixture on glazed paper. Mix thoroughly. The amount of sample to be taken will depend on the amount of BaCl_2 solution required (see 10.3.5 and Note 2).

10.1.1 Quantitatively transfer the mixture to a porcelain capsule or porcelain crucible or platinum crucible, and cover with about 1 g of Eschka mixture.

10.2 *Ignition*—Heat the crucible over a gas flame as described in 10.2.1, or in a gas or electrically heated muffle furnace as described in 10.2.2. The use of artificial gas for heating the sample and Eschka mixture is permissible only when the crucibles are heated in a muffle furnace (see Note 3).

10.2.1 Heat the crucible, placed in a slanting position partially covered on a triangle, over a very low flame. This prevents rapid expulsion of the volatile matter and affords more complete oxidation of the sulfur. After 30 min of low flame heating, gradually increase the temperature and occasionally stir the mixture until all black particles have disappeared, which is an indication of complete combustion.

10.2.2 Place the crucible in a cold muffle furnace and gradually raise the temperature to $800 \pm 25^\circ\text{C}$ in about 1 h. Maintain this maximum temperature until upon stirring all black particles have disappeared (about $1\frac{1}{2}$ h).

10.3 *Subsequent Treatment:*

10.3.1 Remove the crucible, cool, and empty the contents quantitatively into a 200-mL beaker. Digest with 100 mL of hot water for $\frac{1}{2}$ to $\frac{3}{4}$ h with occasional stirring.

10.3.2 Decant the supernatant liquid through a filter into a 600-mL beaker. Wash the insoluble matter with hot water several times using 25 mL of water at each washing and filter the washings through filter paper into a 600-mL beaker. After washing, transfer the insoluble matter to the filter and wash five times with hot water, keeping the mixture well agitated, collecting the washings in the 600-mL beaker.

10.3.3 Treat the filtrate with 10 to 20 mL of saturated bromine water. Make slightly acid with HCl and boil to expel the liberated bromine.

10.3.4 Neutralize using methyl orange indicator with NaOH or Na_2CO_3 solution; then add 1 mL of HCl solution (1 + 9).

10.3.5 Boil again and then, while stirring constantly, add slowly from a pipet 10 mL or more of BaCl_2 solution.

NOTE 2—Barium chloride solution must be added in excess. If more than 10 mL of BaCl_2 solution is required, reduce the weight of sample to about 0.5 g and repeat the ignition and digestion.

10.3.6 Continue boiling for 15 min and allow to stand for at least 2 h, or preferably overnight, at a temperature just below boiling.

NOTE 3—When standing overnight at a temperature slightly less than boiling, cover the flask with a watchglass to prevent the solution from evaporating to dryness and to protect it from external contamination.

10.3.7 Filter the solution and the precipitate of barium sulfate (BaSO_4) through ashless paper and wash the BaSO_4 residue with hot water until 1 drop of silver nitrate (AgNO_3) solution produces no more than a slight opalescence when added to 8 to 10 mL of filtrate.

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.”