



Designation: D1072 – 06 (Reapproved 2012)

Standard Test Method for Total Sulfur in Fuel Gases by Combustion and Barium Chloride Titration¹

This standard is issued under the fixed designation D1072; 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 is for the determination of total sulfur in combustible fuel gases, when present in sulfur concentrations between approximately 25 and 700 mg/m³ (1 to 30 grains per 100 cubic feet). It is applicable to natural gases, manufactured gases, mixed gases, and other miscellaneous gaseous fuels.

1.2 The values stated in inch-pound units are to be regarded as standard.

1.3 *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.*

2. Referenced Documents

- 2.1 *ASTM Standards:*²
D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 A metered sample of gas is burned in a closed system in an atmosphere of sulfur-free air. The oxides of sulfur produced are absorbed in sodium carbonate solution, where they are oxidized to sulfate. The sulfate in the absorbent solution is determined by titration with standardized barium chloride solution, using tetra-hydroxy-quinone (THQ) as an indicator.

4. Interferences

4.1 There are no known interferences for the determination of total sulfur in fuel gases when combustion is followed by barium chloride titration. However, users employing barium chloride titration following collection of sulfur dioxide by

alternative procedures are cautioned that ammonia, amines, substances producing water soluble cations, and fluorides will interfere with the titration.

5. Apparatus

5.1 *Burner* (Fig. 1), as specified in the **Appendix X1**.

5.2 *Chimneys, Absorbers and Spray Traps*, (Fig. 2), as specified in the **Appendix X1**.

5.3 *Flow meter*—A calibrated capillary flow meter for predetermining and indicating the rate of flow of gas to the burner. The capillary selected should be of such size that at the required rate of flow the differential pressure is at least 20 cm of water. A scale divided into millimeters will then provide a reading precision of $\pm 0.5\%$. Other metering devices, including but not limited to rotameters or dry displacement meters, are suitable provided the reading precision is $\pm 0.5\%$ or better. A flow controlling valve is attached to the inlet connection of the flow meter.

5.4 *Vacuum System*—A vacuum manifold equipped with a vacuum regulating device, valves, and other necessary accessories. An example vacuum system capable of performing multiple test measurements is shown in Fig. 3. Other vacuum system configurations can be used to perform this test method. The vacuum system shall be connected to a vacuum pump capable of providing a steady gas flow of 3 L of air per minute through each absorber and capable of maintaining a constant manifold pressure of approximately 40 cm of water below atmospheric pressure.

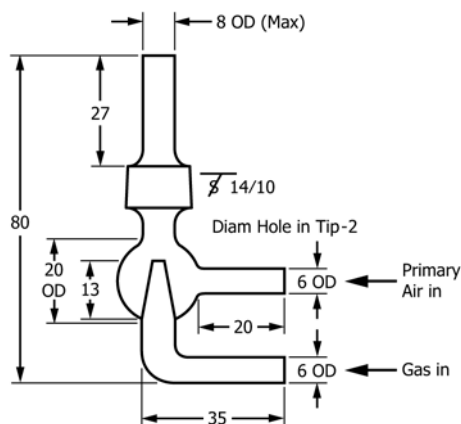
5.5 *Air-Purifying System*—A device supplying purified air to the burner manifold at a constant pressure of approximately 200 mm of water and to the chimney manifold at a pressure of 1 to 2 cm of water. An example system configuration for multiple tests is illustrated in Fig. 4; however, other air-purifying system configurations can be used to perform this test method. The tubing that connects the chimneys to the manifold shall be of an internal diameter not smaller than 0.63 cm in order to prevent unnecessary restriction of airflow.

5.6 *Manometer*—A water manometer for indicating the gas pressure at the point of volume measurement. It is connected between the flowmeter and the burner, with one leg open to the atmosphere.

¹ This test method is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of D03.05 on Determination of Special Constituents of Gaseous Fuels.

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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.



NOTE 1—All dimensions in millimetres.
FIG. 1 Gas Burner for Sulfur Determination

6. Reagents and Materials

6.1 *Reagents Purity*—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 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 adversely impacting the accuracy of the determination. **Warning**—Sodium hydroxide is corrosive and can cause severe damage to eyes and skin. Inhalation will irritate the nose, throat and lungs. It reacts exothermically with water.

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

6.3 *Alcohol*—Ethyl alcohol, denatured by Formula 30 or 3-A, or isopropyl alcohol.

6.4 *Barium Chloride*, (CAS No: 10361-37-2), *Standard Solution* (1 mL = 1 mg S)—Dissolve 7.634 g of barium chloride (BaCl₂·2H₂O) in water and dilute to 1 L. The solution is standardized gravimetrically by precipitation as barium sulfate or by titration against sulfuric acid (see 6.12)

6.5 *Hydrochloric Acid* (CAS No 7647-01-0) (2.275-g HCl/L)—Titrated against Na₂CO₃ solution (see 6.15), using methyl orange indicator. Adjusted such that 1 mL of HCl solution is equivalent to 1 mL of Na₂CO₃ solution.

6.6 *Hydrogen Peroxide (30 %)* (H₂O₂; CAS No: 7722-84-1).

6.7 *iso-Propanol* (CAS No. 67630)

6.8 *Potassium Hydrogen Phthalate (KHP; CAS No 877-24-7)* —Dry use.

6.9 *Phenolphthalein* (CAS No 77-09-8)

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.

6.10 *Methyl Orange* (CAS No 547-58-0) *Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water.

6.11 *Silver Nitrate* (CAS No 7761-88-8) *Solution* (17-g AgNO₃/L)—Dissolve 1.7 g of silver nitrate (AgNO₃) in 100 mL of water. Store in a brown bottle.

6.12 *Sodium Carbonate* (CAS No 5968-11-6) *Solution* (3.306-g Na₂CO₃/L)—Dissolve 3.306 g of sodium carbonate (Na₂CO₃) in water and dilute to 1 L.

6.13 *Sodium Hydroxide Solution* (CAS No 1310-73-2) (100-g NaOH/L)—Dissolve 100 g of technical grade sodium hydroxide (NaOH) pellets in water and dilute to 1 L. Standardize against potassium hydrogen phthalate (See 6.1)

6.14 *Sulfuric Acid* (CAS No 7664-93-9) (1 + 16)—Mix 60 mL of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) with 960 mL of water.

6.15 *Tetrahydroxyquinone Indicator* (THQ CAS No. 5676-48-2), in powdered form.

6.16 *Thorin indicator*— (CAS No. 132-33-2)

7. Calibration and Standardization

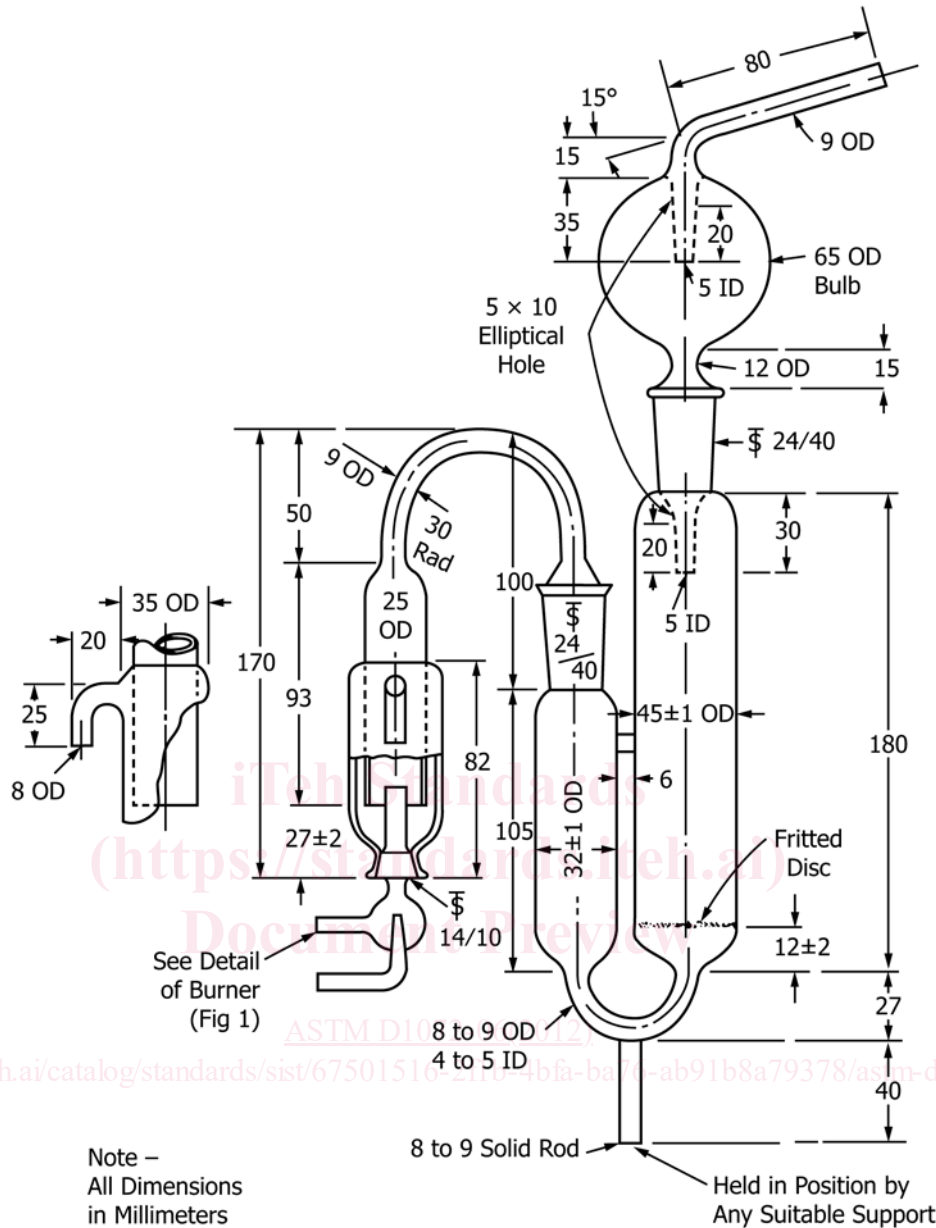
7.1 *Sodium Hydroxide Solution Standardization*— The following provides an example procedure for standardization; other quantities of reagents, as convenient, can be used. Dry and crushed potassium hydrogen phthalate (KHP) is heated in an oven at 105 °C for 2 hours and allowed to cool to room temperature in a desiccator. KHP (950 ± 50 mg weighed to the nearest 0.1 mg) is placed in an Erlenmeyer flask. Water (70 mL) and 2-4 drops of phenolphthalein are added. Titrate the KHP solution with the sodium hydroxide solution prepared under 6.13 to a faint pink color. Repeat the titration using a second portion of KHP. Titrate a 70 mL water blank containing 1-4 drops of phenolphthalein to a faint pink color using the sodium hydroxide solution prepared under 6.13. Repeat this procedure and average the results. For both the water blank and the KHP titration replicates should agree to 0.05 mL titrant. For each KHP trial, independently calculate the normality for the sodium hydroxide solution according to the following equation:

$$\text{Normality of NaOH} = \frac{\text{mg KHP}/204.23}{(\text{mL NaOH} - \text{avg. mL blank})} \quad (1)$$

Values for the two KHP trials should agree within ± 0.5 percent. If they do not, repeat the titrations or identify the cause for the excessive discrepancy, or both.

7.2 *Sulfuric Acid Standardization*— The following provides an example procedure for standardization; other quantities of reagents, as convenient, can be used. Titrate the sulfuric acid solution prepared under 6.14 against the sodium hydroxide standardized in 7.1 using 2-4 drops of phenolphthalein as the indicator. Repeat and average the result for the normality of the sulfuric acid. Values for the two trials should agree within ± 0.5 percent. If they do not, repeat the titrations or identify the cause for the excessive discrepancy, or both.

7.3 *Barium Chloride Solution Standardization*— Titrate the barium chloride solution against the previously standardized sulfuric acid solution (see 7.2). This can be conveniently accomplished by transferring 10.0 mL sulfuric acid to a flask



NOTE 1—In the case of those dimensions for which no specific tolerances are designated above, the permissible variation is $\pm 10\%$ to the nearest 1 mm, provided, however, that in no case shall the deviation be greater than 5 mm.

FIG. 2 Detailed Drawing of Combustion and Absorption Apparatus for Sulfur Determination

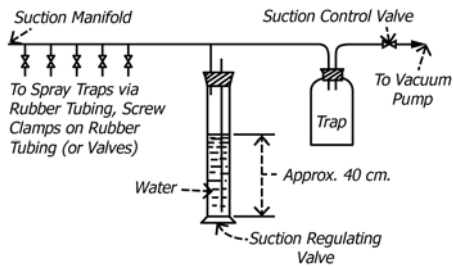


FIG. 3 Suction System for Sulfur Determination

added. This is titrated to a pink end point using the barium chloride solution. Repeat the titration and average the results. The replicate titrations should agree within 0.5 percent. If they do not, repeat the titrations or identify the cause for the excessive discrepancy, or both. Using this same procedure, perform duplicate blank titrations using water in place of sulfuric acid solution. The replicate titrations should agree within 0.5 percent. Calculate the normality of the barium chloride solution according to the following equation:

$$\text{Normality of Barium Chloride} = \frac{10.0 \text{ mL} \times N \text{ Sulfuric Acid}}{(\text{avg. mL} - \text{avg. blank})} \quad (2)$$

where 40 mL iso-propanol and 2-4 drops thordin indicator are