



Designation: ~~D7620 – 10~~ **D7620 – 10 (Reapproved 2015)**

Standard Test Method for Determination of Total Sulfur in Liquid Hydrocarbon Based Fuels by Continuous Injection, Air Oxidation and Ultraviolet Fluorescence Detection¹

This standard is issued under the fixed designation D7620; 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 total sulfur in liquid hydrocarbon based fuel with a final boiling point of up to ~~450°C~~ **450 °C**. It is applicable to analysis of natural, processed and final product materials containing sulfur in the range of ~~4.0~~ **4.0 mg/kg to 830** ~~830 mg/kg~~ (see **Note 1**).

NOTE 1—For liquid hydrocarbons containing less than ~~4.0~~ **4.0 mg/kg** total sulfur or more than ~~830~~ **830 mg/kg** total sulfur, Test Method **D5453** may be more appropriate.

1.2 This test method is applicable for total sulfur determination in liquid hydrocarbons containing less than ~~0.35% (m/m)~~ **0.35 % (m/m)** halogen(s).

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 specific hazard statements, see **4.1**, **8.3**, and Section **9**.

2. Referenced Documents

2.1 *ASTM Standards:*²

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

3. Terminology

3.1 *Definitions:*

3.1.1 See Terminology **D4175** for definitions of other terms used in this test method.

3.1.2 *oxidative pyrolysis, n*—process in which a sample undergoes complete combustion in an appropriate oxygen containing environment at a sufficiently elevated temperature.

¹ This test method is under the jurisdiction of ASTM Committee **D02** on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee **D02.03** on Elemental Analysis.

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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.

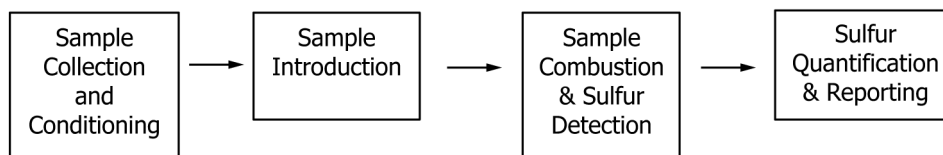


FIG. 1 Basic Block Diagram Describing Sulfur Determination

3.1.2.1 Discussion—

Organic compounds pyrolytically oxidize to carbon dioxide and water and oxides of other elements that are in the sample.

4. Summary of Test Method

4.1 A small, very controlled flow of hydrocarbon sample is continuously injected during measurement. It is introduced via a syringe into a high temperature combustion tube containing air where sulfur is oxidized to sulfur dioxide (SO₂). Water produced during the sample combustion is removed, as required, and the sample combustion gases are next exposed to ultraviolet (UV) light. The SO₂ absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO₂*). Fluorescence emitted from the excited SO₂* as it returns to a stable state SO₂ is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample. (**Warning**—Exposure to excessive quantities of ultraviolet light is injurious to health. The operator shall avoid exposing their body, especially their eyes, not only to direct UV light but also to secondary or scattered radiation that is present.)

4.2 Fig. 1 illustrates a basic block diagram describing sulfur determination. Sample collection and conditioning, sample introduction, detection system and data handling are depicted.

5. Significance and Use

5.1 Some process catalysts used in refining can be poisoned when trace amounts of sulfur bearing materials are contained in the feedstocks. There are also government regulations as to how much sulfur is permitted to be present in commercial transportation fuels. This test method can be used to determine sulfur in process and downstream distribution streams. It can also be used for purposes of screening and quality control of finished hydrocarbon fuel products.

6. Interferences

6.1 Halogens above 0.35 % (mass/mass) will interfere with accurate sulfur determination.

6.2 Bound nitrogen at concentration greater than ~~150 mg~~ 150 mg N/kg can cause a ~~1 mg~~ 1 mg S/kg positive bias.

6.3 Excessive moisture produced during the combustion step will interfere if not removed prior to the detector.

7. Apparatus

7.1 **Furnace**—An electric furnace held at a temperature sufficient to pyrolyze the entire sample (typically ~~1050~~ 1050 °C ± 25°C) and oxidize sulfur to SO₂.

7.2 **Combustion Tube**—A quartz combustion tube constructed to allow the direct injection of a continuous flow of sample into the heated oxidation zone of the furnace. The oxidation section shall be large enough to ensure complete combustion of the sample. Fig. 2 illustrates a typical combustion tube (Note 2).

NOTE 2—Other combustion tube configurations are acceptable if precision and accuracy are not degraded.

7.3 **Flow Control**—The apparatus shall be equipped with suitable flow control apparatus capable of maintaining a constant supply of air.

7.4 **Drier Tube**—The apparatus shall be equipped with a mechanism for the removal of excessive water vapor. The oxidation reaction produces water vapor which must be eliminated prior to measurement by the detector. This may be accomplished with a membrane drying tube, or a permeation dryer, that utilizes a selective capillary action for water removal.

7.5 **UV Fluorescence Detector**—A quantitative detector capable of measuring the energy emitted from the fluorescence of sulfur dioxide by UV light.

7.6 **Millilitre Syringe**—A disposable ~~1 mL~~ 1 mL syringe capable of accurately delivering a controlled and constant flow of calibration and sample materials. The syringe shall accommodate a disposable tip to aid the filling of the syringe and a disposable septum seal to accommodate penetration and sample flow.

7.7 **Sample Inlet System**—An automatic sample injection device that is compatible with a disposable ~~1 mL~~ 1 mL syringe is required. The injector shall allow the introduction of an appropriate continuous flow of sample into a combustion tube carrier stream, which directs the sample into the oxidation zone at a controlled and repeatable rate.

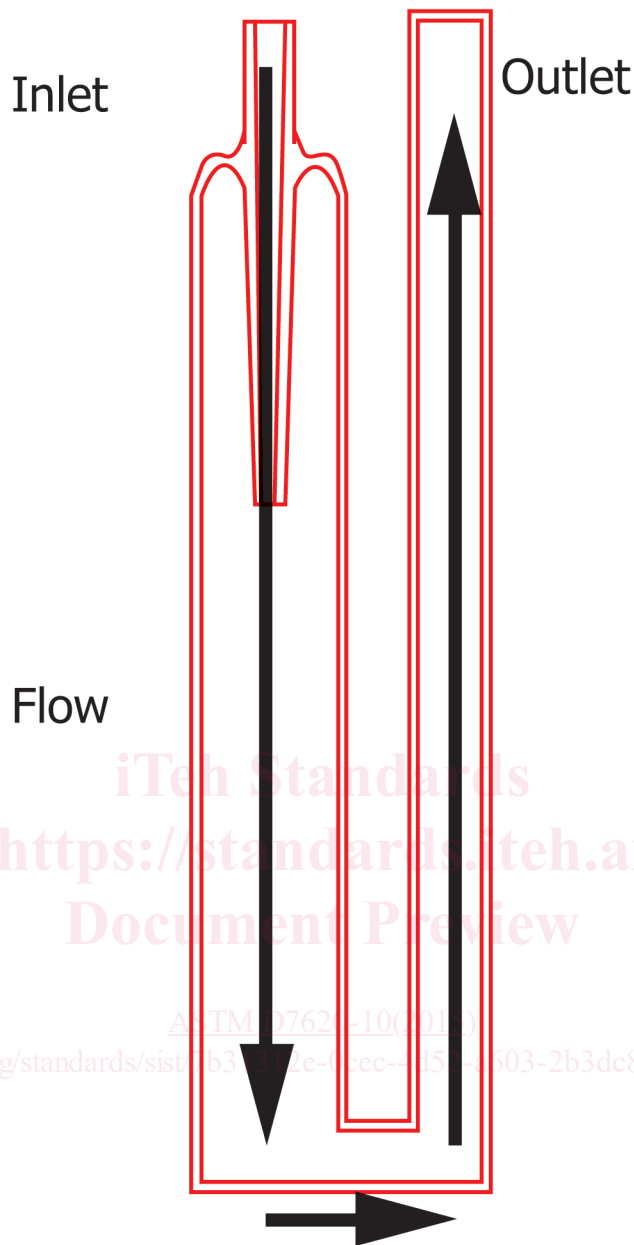


FIG. 2 Typical Combustion Tube

7.8 *Strip Chart Recorder*—Equivalent electronic data logger, integrator or recorder (optional).

7.9 *Balance*—With a precision of ± 0.01 mg ± 0.01 mg (optional).

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in 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 lessening the accuracy of the determination.

8.2 *Air*—Filtered (with a $2\text{-}\mu\text{m}$ $2\text{-}\mu\text{m}$ filter).

8.3 *Iso-octane, Toluene, Xylenes*—Reagent grade. (**Warning**—Organic solvents are flammable.)

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 *Annual 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.