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British Standard 4454

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## Standard Test Method for Sulfur in Petroleum Products (General High Pressure Decomposition Device Method)<sup>1</sup>

This standard is issued under the fixed designation D129; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

### 1. Scope\*

1.1 This test method covers the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases that cannot be burned completely in a wick lamp. The test method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0.1 % sulfur.

NOTE 1—This test method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead which are sometimes present in greases, lube oil additives, or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, and so forth. The test method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples that are excluded can be analyzed by Test Method D1552.

1.2 This test method is applicable to samples with the sulfur in the range 0.09 % to 5.5 % by mass.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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 Recom-*

*mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1193 Specification for Reagent Water

D1552 Test Method for Sulfur in Petroleum Products by High Temperature Combustion and Infrared (IR) Detection or Thermal Conductivity Detection (TCD)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

E144 Practice for Safe Use of Oxygen Combustion Vessels

### 3. Summary of Test Method

3.1 The sample is oxidized by combustion in a high pressure decomposition device containing oxygen under pressure. The sulfur, as sulfate in the high pressure decomposition device washings, is determined gravimetrically as barium sulfate.

3.2 (**Warning**—Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the high pressure decomposition device, or a blow-out, provided the high pressure decomposition device is of proper design and construction and in good mechanical condition. It is desirable, however, that the high pressure decomposition device be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforeseeable contingencies.)

3.3 (**Warning**—Initial testing and periodic examination of the pressure vessel is essential to ensure its fitness for service.

<sup>1</sup> 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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This test method was adopted as a joint ASTM-IP standard in 1964.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

<sup>2</sup> 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.

\*A Summary of Changes section appears at the end of this standard

This is particularly important if the pressure vessel has been dropped and has any obvious signs of physical damage.)

#### 4. Apparatus and Materials

4.1 *High Pressure Decomposition Device* (see *Note 2*),<sup>3</sup> having a capacity of not less than 300 mL, so constructed that it will not leak during the test and that quantitative recovery of the liquids from the high pressure decomposition device may be achieved readily. The inner surface of the high pressure decomposition device may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the high pressure decomposition device assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the high pressure decomposition device.

NOTE 2—Criteria for judging the acceptability of new and used oxygen combustion high pressure decomposition devices are described in Practice E144.

4.2 *Oxygen Charging Equipment*—The valves, gauges, filling tube, and fittings used in the oxygen charging system shall meet industry safety codes and be rated for use at input pressure up to 20 875 kPa and discharge pressure up to 5575 kPa. Separate gauges shall be provided to show the supply pressure and the pressure vessel pressure. The pressure vessel gauge shall not be less than 75 mm in diameter and preferably graduated from 0 kPa to 5575 kPa in 100 kPa subdivisions. Both gauges shall be absolutely oil-free and shall never be tested in a hydraulic system containing oil. The charging equipment shall include either a pressure reducing valve which will limit the discharge pressure to a maximum of 4055 kPa or a relief valve set to discharge at 4055 kPa in case the pressure vessel should accidentally be overcharged. Means shall also be provided for releasing the residual pressure in the filling tube after the pressure valve has been closed.

4.3 *Sample Cup*, platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 g to 11 g.

4.4 *Firing Wire*, platinum, No. 26 B & S gage, 0.41 mm (16 thou), 27 SWG, or equivalent. (**Warning**—The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.)

4.5 *Ignition Circuit*, capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire. The current shall be drawn from a step-down transformer or from a suitable battery. The current shall not be drawn from the power line, and the voltage shall not exceed 25 V. The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.

4.6 *Cotton Wicking or Nylon Sewing Thread*, white.

4.7 *Muffle Furnace*.

4.8 *Filter Paper*, “ashless,” 0.01 % by mass ash maximum.

<sup>3</sup> A high pressure decomposition device conforming to the test specifications in IP Standard IP 12 is suitable.

#### 5. Reagents and Materials

5.1 *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 Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup> 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean water as defined by Type II or III of Specification D1193.

5.3 *Barium Chloride Solution* (85 g/L)—Dissolve 100 g of barium chloride dihydrate ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ) in distilled water and dilute to 1 L.

5.4 *Bromine Water* (saturated).

5.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.6 *Oxygen*, free of combustible material and sulfur compounds, available at a pressure of 41 kgf/cm<sup>2</sup> (40 atm).

5.7 *Sodium Carbonate Solution* (50 g/L)—Dissolve 135 g of sodium carbonate decahydrate ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ) or its equivalent weight in distilled water and dilute to 1 L.

5.8 *White Oil, USP, or Liquid Paraffin, BP, or equivalent*.

5.9 *Quality Control (QC) Samples*, preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 10.

#### 6. Procedure

6.1 Take samples in accordance with the instructions in Practices D4057 or D4177.

6.2 *Preparation of High Pressure Decomposition Device and Sample*—Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 mL of  $\text{Na}_2\text{CO}_3$  solution in the high pressure decomposition device in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of sample and white oil (Note 4 and Note 5) specified in the following table, weighing the sample to the nearest 0.2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

<sup>4</sup> *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.