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Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)[1]

This standard is issued under the fixed designation D 129; 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.

This test method has been adopted for use by government agencies to replace Method 5202 of Federal Test Method No. 791b

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, etc. 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 D 1552.

1.2 *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.* See 3.2 for specific precautionary directions incorporated in the test method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[3]

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[4]

E 144 Practice for Safe Use of Oxygen Combustion Bombs[5]

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb 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 bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforseeable contingencies.*

4. Apparatus and Materials

4.1 *Bomb,*[6,7] 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 bomb may be achieved readily. The inner surface of the bomb 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 bomb 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 bomb.

4.2 *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 to 11 g.

4.3 *Firing Wire*, platinum, No. 26 B & S gage, 0.41 mm (16 thou), 27 SWG, or equivalent. (**Warning—**The switch in the

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards,* Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Criteria for judging the acceptability of new and used oxygen combustion bombs are described in Practice E 144.

[7] A bomb conforming to the test specifications in IP Standard IP 12 is suitable.

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

ignition circuit shall be of a type which remains open, except when held in closed position by the operator.)

4.4 *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.

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

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.[8] 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 D 1193.

5.3 *Barium Chloride Solution* (85 g/litre)—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in distilled water and dilute to 1 liter.

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 ^{2} (40 atm).

5.7 *Sodium Carbonate Solution* (50 g/litre)—Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$) or its equivalent weight in distilled water and dilute to 1 litre.

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 *Preparation of Bomb 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 Na_2CO_3 solution in the bomb (Note 2) and rotate the bomb 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 3 and Note 4) 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).

NOTE 2—After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness can be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with emery polishing papers Grit No. 2/0 , or equivalent paper,[9] coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide[10] and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before the bomb is used it shall be washed with soap and water to remove oil or paste left from the polishing operation.

6.1.1 **Warning**—Do not use more than 1.0 g total of sample and white oil or other low sulfur combustible material or more than 0.8 g if the IP 12 bomb is used.

Sulfur Content percent	Weight of Sample, g	Weight of White Oil, g
5 or under	0.6 to 0.8	0.0
Over 5	0.3 to 0.4	0.3 to 0.4

NOTE 3—Use of sample weights containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 % chlorine, the sample weight be based on the chlorine content as given in the following table:

Chlorine Content percent	Weight of Sample, g	Weight of White Oil, g
2 to 5	0.4	0.4
Over 5 to 10	0.2	0.6
Over 10 to 20	0.1	0.7
Over 20 to 50	0.05	0.7

NOTE 4—If the sample is not readily miscible with white oil, some other low sulfur combustible diluent may be substituted. However, the combined weight of sample and nonvolatile diluent shall not exceed 1.0 g or more than 0.8 g if the IP 12 bomb is used.

6.2 *Addition of Oxygen*—Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely. (**Warning—**Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.) Admit oxygen slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure,[A] kgf/cm^2(atm)	Maximum Gage Pressure,[A] kgf/cm^2(atm)
300 to 350	39 (38)	41 (40)
350 to 400	36 (35)	38 (37)
400 to 450	31 (30)	33 (32)
450 to 500	28 (27)	30 (29)

[A] The minimum pressures are specified to provide sufficient oxygen for complete combustion and the *maximum pressures represent a safety requirement.*

6.3 *Combustion*—Immerse the bomb in a cold distilled-water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. (**Warning**—Do not go near the bomb until at least 20 s after firing.) Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard

[8] *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.

[9] Emery Polishing Paper Grit No. 2/0 can be purchased from Norton Co., Troy, N. Y.

[10] Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, N. J.