~~Designation:D129–00 (Reapproved 2005)~~ Designation: D129 – 11

British Standard 4454

Designation: 61/99

Standard Test Method for ~~Sulfur in Petroleum Products (General Bomb Method)~~Sulfur in Petroleum Products (General High Pressure Decomposition Device Method)[1]

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 (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~~

This standard has been approved for use by agencies of the 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 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.~~

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

D1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)

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 Bombs

3. Summary of Test Method

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

---

[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.

~~Current edition approved May 1, 2005. Published May 2005. Originally approved in 1922. Last previous edition approved in 2000 as D129–00.~~

Current edition approved Oct. 1, 2011. Published October 2011. Originally approved in 1922. Last previous edition approved in 2005 as D129–00(2005). DOI: 10.1520/D0129-11.

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. ~~DOI: 10.1520/D0129-00R05.~~

[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.

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

~~3.2~~**~~Warning—~~** 3.2 (**Warning—**Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the ~~bomb,~~ high pressure decomposition device, or a blow-out, provided the ~~bomb~~ high pressure decomposition device is of proper design and construction and in good mechanical condition. It is desirable, however, that the ~~bomb~~ 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.)

4. Apparatus and Materials

4.1 ~~*Bomb,* 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.~~ High Pressure Decomposition Device (see Note 2),[3] 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 *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 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.[4] 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/litre)~~*(85 g/L)—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in distilled water and dilute to 1 ~~litre.~~ 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^2 (40 atm).

5.7 *Sodium Carbonate Solution ~~(50 g/litre)~~*(50 g/L)—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.~~ 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 ~~*Preparation of Bomb and Sample*~~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 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 (~~solution in the high pressure decomposition device (Note 3~~and~~) and rotate 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).

NOTE ~~2—After~~ 3—After repeated use of the ~~bomb~~ high pressure decomposition device for sulfur determinations, a film may be noticed on the inner surface. This dullness can be removed by periodic polishing of the ~~bomb.~~ high pressure decomposition device. A satisfactory method for doing this is

~~[3] Criteria for judging the acceptability of new and used oxygen combustion bombs are described in Practice E144.~~

[3] A high pressure decomposition device conforming to the test specifications in IP Standard IP 12 is suitable.

~~[4] A bomb conforming to the test specifications in IP Standard IP 12 is suitable.~~

[4] *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.