



Designation: **D129—13 D129 – 18**

British Standard 4454



Designation: **61/99**

Standard Test Method for 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 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 mass %~~ 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 Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

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.

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

Current edition approved ~~June 15, 2013~~ April 1, 2018. Published ~~August 2013~~ April 2018. Originally approved in 1922. Last previous edition approved in ~~2014~~ 2013 as **D129—14**; **D129 – 13**. DOI: ~~10.1520/D0129-13~~ 10.1520/D0129-18.

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 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**—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. 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*),³ having a capacity of not less than ~~300 mL~~, 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~~ 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~~ 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~~ 4055 kPa or a relief valve set to discharge at ~~4055 kPa~~ 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~~ 24 mm in outside diameter at the bottom, ~~27 mm~~ 27 mm in outside diameter at the top, ~~12 mm~~ 12 mm in height outside, and weighing ~~10.10 g~~ to 11 g.

4.4 *Firing Wire*, platinum, No. 26 B & S gage, ~~0.41 mm~~ 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~~ 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 mass % ash~~ 0.01 % by mass ash maximum.

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

5.4 *Bromine Water* (~~saturated~~)—(~~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/cm²~~ 41 kgf/cm² (40 atm).

³ A high pressure decomposition device 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.

5.7 *Sodium Carbonate Solution (50 (50 g/g/L) L)*—Dissolve ~~135 g~~ 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~~ 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~~ 100 mm in length. Coil the middle section (about ~~20 mm~~) 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~~ 5 mL of Na_2CO_3 solution in the high pressure decomposition device (**Note 3**) 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~~ 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 3—After repeated use of the 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 high pressure decomposition device. A satisfactory method for doing this is to rotate the high pressure decomposition device in a lathe at about 300 rpm and polish the inside surface with emery polishing papers Grit No. 3/0, or equivalent paper,⁵ coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide⁶ and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before the high pressure decomposition device is used it shall be washed with soap and water to remove oil or paste left from the polishing operation.

6.2.1 (**Warning**—Do not use more than ~~1.0 g~~ 1.0 g total of sample and white oil or other low sulfur combustible material or more than ~~0.8 g~~ 0.8 g if the IP 12 high pressure decomposition device 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 4—Use of sample weights containing over ~~20 mg~~ 20 mg of chlorine may cause corrosion of the high pressure decomposition device. 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 5—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~~ 1.0 g or more than ~~0.8 g~~ 0.8 g if the IP 12 high pressure decomposition device is used.

6.3 *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 high pressure decomposition device and tighten the cover securely. (**Warning**—Do not add oxygen or ignite the sample if the high pressure decomposition device 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 High Pressure Decomposition Device, mL	Minimum Gauge Pressure, ^A kgf/cm ² (atm)	Maximum Gauge 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.

6.4 *Combustion*—Immerse the high pressure decomposition device 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 high pressure decomposition device

⁵ The sole source of supply of the apparatus known to the committee at this time is Emery Polishing Paper Grit No. 3/0, which can be purchased from Norton Co., 2600 10th Ave., Watervliet, NY 12189-1766. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁶ The sole source of supply of the apparatus known to the committee at this time is chromic oxide that may be purchased from J. T. Baker, Phillipsburg, NJ 08865. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.