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Designation: D 808 - 87

An American National Standard

Standard Test Method for Chlorine in New and Used Petroleum Products (Bomb Method)¹

This standard is issued under the fixed designation D 808; 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 5651 of Federal Test Method Standard No. 791b.

Attention is called to Note 1 on Safety and to the specific precautionary directions incorporated in the test method.

1. Scope

1.1 This test method covers the determination of chlorine in lubricating oils and greases, including new and used lubricating oils and greases containing additives, and in additive concentrates. Its range of applicability is 0.1 to 50 % chlorine. The procedure assumes that compounds containing halogens other than chlorine will not be present.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.*

1.3 The preferred units are mass percent.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1317 Test Method for Chlorine in New and Used Lubricants (Sodium Alcoholate Method)³

D 4057 API MPMS 8.1 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure (**Caution**—See Note 1). The chlorine compounds thus liberated are absorbed in a sodium carbonate solution and the amount of chlorine present is determined gravimetrically by precipitation as silver chloride.

NOTE 1: (Caution)—Safety—Strict adherence to all of the provisions prescribed hereinafter insures 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 1/2 in. (12.7 mm) thick, or equivalent protection be provided against unforeseeable contingencies.

¹ This test method is under the jurisdiction of ASTM Committee D-2 on the Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D 02.03 on Elemental Analysis.

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In 1963, this test method is adopted as standard without revision.

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.

4. Significance and Use

4.1 This test method may be used to measure the level of chlorine-containing compounds in petroleum products. This knowledge can be used to predict performance or handling characteristics of the product in question.

5. Apparatus

5.1 **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 readily achieved. 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 chlorine content of the liquid in the bomb.

5.2 **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.

5.3 **Firing Wire**, platinum, approximately No. 26 B & S gage.

5.4 **Ignition Circuit** (Note 2), capable of supplying sufficient current to ignite the nylon thread or cotton wicking without melting the wire.

NOTE 2—Caution—The switch in the ignition circuit shall be of a type that remains open, except when held in closed position by the operator.

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

5.6 **Filter Crucible**, fritted-glass, 30-mL capacity, medium porosity.

6. Reagents and Materials

6.1 **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.⁵ Other grades may be used, provided it is first ascertained that the

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."



Documents

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TABLE 1 Quantities of Sample and White Oil

Chlorine Content, %	Weight of Sample, g	Weight of White Oil, g
2 and under	0.8	0.0
Above 2 to 5, incl	0.4	0.4
Above 5 to 10, incl	0.2	0.6
Above 10 to 20, incl	0.1	0.7
Above 20 to 50, incl	0.05	0.7

reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or III of Specification D 1193.

6.3 *Nitric Acid (1+1)*—Mix equal volumes of concentrated nitric acid (HNO_3 , sp gr 1.42) and water.

6.4 *Oxygen*, free of combustible material and halogen compounds, available at a pressure of 40 atmos. (Warning—See Note 3).

NOTE 3—Warning—Oxygen vigorously accelerates combustion.

6.5 *Silver Nitrate Solution (50 g AgNO_3/L)*—Dissolve 50 g of silver nitrate (AgNO_3) in water and dilute to 1 L.

6.6 *Sodium Carbonate Solution (50 g $\text{Na}_2\text{CO}_3/\text{L}$)*—Dissolve 50 g of anhydrous Na_2CO_3 , 58.5 g of $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$, or 135 g of $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$ in water and dilute to 1 L.

6.7 *White Oil*, refined.

7. Sampling

7.1 Take samples in accordance with the instructions in Practice D 4057.

7.2 Take care that the sample is thoroughly representative of the material to be tested and that the portion of the sample used for the test is thoroughly representative of the whole sample.

8. Procedure

8.1 *Preparation of Bomb and Sample*—Cut a piece of firing wire approximately 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 into the coil a nylon thread, or wisp of cotton, 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 4) and by means of a rubber policeman, wet the interior surface of the bomb, including the head, as thoroughly as possible. Introduce into the sample cup the quantities of sample and white oil (Notes 5 and 6) specified in Table 1 (Caution—Note 7), 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 4—After repeated use of the bomb for chlorine determination, a film may be noticed on the inner surface. This dullness should 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 Grit No. 2/00 or equivalent paper⁶ coated with a

⁶ Emery Polishing Paper Grit No. 2/00 may be purchased from the Behr-Manning Co., Troy, NY. Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, NJ.

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 using the bomb it should be washed with soap and water to remove oil or paste left from the polishing operation. Bombs with porous or pitted surfaces should never be used because of the tendency to retain chlorine from sample to sample.

NOTE 5—When the sample is not readily miscible with white oil, some other nonvolatile, chlorine-free combustible diluent may be employed in place of white oil. However, the combined weight of sample and nonvolatile diluent shall not exceed 1 g. Some solid additives are relatively insoluble, but may be satisfactorily burned when covered with a layer of white oil.

NOTE 6—The practice of running alternately samples high and low in chlorine content should be avoided whenever possible. It is difficult to rinse the last traces of chlorine from the walls of the bomb and the tendency for residual chlorine to carry over from sample to sample has been observed in a number of laboratories. When a sample high in chlorine has preceded one low in chlorine content, the test on the low-chlorine sample should be repeated and one or both of the low values thus obtained should be considered suspect if they do not agree within the limits of repeatability of this method.

NOTE 7—Caution—Do not use more than 1 g total of sample and white oil or other chlorine-free combustible material.

8.2 *Addition of Oxygen*—Place the sample cup in position and arrange the nylon thread, or wisp of cotton, so that the end dips into the sample. Assemble the bomb and tighten the cover securely. Admit oxygen (Caution—See Note 8) slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in Table 2.

NOTE 8—Caution—Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.

8.3 *Combustion*—Immerse the bomb in a cold water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. 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 the determination, and thoroughly clean the bomb before again putting it in use (Note 4).

8.4 *Collection of Chlorine Solution*—Rinse the interior of the bomb, the sample cup, and the inner surface of the bomb cover with a fine jet of water, and collect the washings in a 600-mL beaker. Scrub the interior of the bomb and the inner surface of the bomb cover with a rubber policeman. Wash the base of the terminals until the washings are neutral to the indicator methyl red. (The volume of the washings is normally in excess of 300 mL.) Take special care not to lose any wash water.

8.5 *Determination of Chlorine*—Acidify the solution by adding HNO_3 (1+1) drop by drop until acid to methyl red. Add an excess of 2 mL of the HNO_3 solution. Filter through a qualitative paper (if the solution is cloudy, the presence of

TABLE 2 Gage Pressures

Capacity of Bomb, mL	Minimum Gage Pressure, ^a atm	Maximum Gage Pressure, ^a atm
300 to 350	38	40
350 to 400	35	37
400 to 450	30	32
450 to 500	27	29

^a The minimum pressures are specified to provide sufficient oxygen for complete combustion, and the maximum pressures represent a safety requirement.