



Designation: D808 – 11

Standard Test Method for Chlorine in New and Used Petroleum Products (High Pressure Decomposition Device Method)¹

This standard is issued under the fixed designation D808; 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 Department of Defense.

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 m% chlorine. The procedure assumes that compounds containing halogens other than chlorine will not be present.

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

1.2.1 The preferred units are mass percent.

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.* Attention is called to specific warning statements incorporated in the test method.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a high pressure decomposition device containing oxygen under pressure. (**Warning**—Strict adherence to all of the provisions prescribed

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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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.

hereinafter 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 ($\frac{1}{2}$ -in.) thick, or equivalent protection be provided against unforeseeable contingencies.) 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.

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.

4.2 This test method can also serve as a qualitative tool for the presence or non-detection of chlorine in petroleum products. In light of the efforts in the industry to prepare chlorine free products, this test method would provide information regarding the chlorine levels, if any, in such products.

5. Apparatus

5.1 *High Pressure Decomposition Device*, 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 readily achieved. 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 chlorine content of the liquid in the high pressure decomposition device.

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, No. 26 B & S gage 0.41 (16 thou), 27 SWG or equivalent.

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

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

5.4.1 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.³ 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.

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 **D1193**.

6.3 *Nitric Acid (1 + 1)*—Mix equal volumes of concentrated nitric acid (HNO₃, sp gr 1.42) and water.

6.4 *Oxygen*, free of combustible material and halogen compounds, available at a pressure of 41 kgf/cm² (40 atmos). (**Warning**—Oxygen vigorously accelerates combustion.)

6.5 *Silver Nitrate Solution (50 g AgNO₃/L)*—Dissolve 50 g of silver nitrate (AgNO₃) in water and dilute to 1 L.

6.6 *Sodium Carbonate Solution (50 g Na₂CO₃/L)*—Dissolve 50 g of anhydrous Na₂CO₃, 58.5 g of Na₂CO₃·H₂O, or 135 g of Na₂CO₃·10 H₂O in water and dilute to 1 L.

6.7 *White Oil*, refined.

6.8 *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**.

7. Sampling

7.1 Take samples in accordance with the instructions in Practice **D4057**.

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 High Pressure Decomposition Device and Sample*—Cut a piece of firing wire approximately 100 mm

³ *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.

TABLE 1 Quantities of Sample and White Oil

Chlorine Content, m%	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

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 Na₂CO₃ solution in the high pressure decomposition device and by means of a rubber policeman, wet the interior surface of the high pressure decomposition device, including the head, as thoroughly as possible. Introduce into the sample cup the quantities of sample and white oil (**Note 1**). (**Warning**—Do not use more than 1 g total of sample and white oil or other chlorine free combustible material) specified in **Table 1**. Do not add oxygen or ignite the sample if the high pressure decomposition device has been jarred, dropped, or tilted), 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.

8.1.1 After repeated use of the high pressure decomposition device for chlorine determination, 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 with Grit No. 2/0 or equivalent paper coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide⁴ and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before using the high pressure decomposition device wash it with soap and water to remove oil or paste left from the polishing operation. high pressure decomposition devices with porous or pitted surfaces should never be used because of the tendency to retain chlorine from sample to sample.

8.1.2 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. (**Warning**—Do not use more than 1 g total of sample and white oil or other chlorine-free combustible material.)

NOTE 1—The practice of running alternately high and low samples in chlorine content shall be avoided whenever possible. It is difficult to rinse the last traces of chlorine from the walls of the high pressure decomposition device and the tendency for residual chlorine to carry over from

⁴ The sole source of supply of the chromic oxide known to the committee at this time is J. T. Baker & Co., 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.