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Designation: 118/84

Standard Test Method for Chlorine in New and Used Lubricants (Sodium Alcoholate Method)¹

This standard is issued under the fixed designation D 1317; 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 is also a standard of the Institute of Petroleum issued under the fixed designation IP 118. The final number indicates the year of last revision.

This test method was adopted as a joint ASTM - IP standard in 1965.

1. Scope

1.1 This test method covers determination of the chlorine content at levels of 0.1 mass % and higher in lubricating oils and greases with which chlorinated additives have been blended. It is equally applicable to new or used oils and greases and to the additive concentrate. The procedure assumes that compounds containing halogens other than chlorine will not be present.

NOTE 1—A method based on bomb combustion for analysis of the same type of material is described in Test Method D 808.

1.2 The values given in acceptable SI units are to be regarded as the standard.

1.3 *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. Specific precautionary statements appear in Notes 2, 3, 4, 5, 6, and 7.*

2. Referenced Documents

2.1 ASTM Standards:

- D 808 Test Method for Chlorine in New and Used Petroleum Products (Bomb Method)²
- D 1193 Specification for Reagent Water³
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

3. Summary of Test Method

3.1 The sample, dissolved in a low boiling hydrocarbon mixture, is boiled under reflux with metallic sodium and *n*-butyl alcohol. Under these conditions the chlorine is converted to sodium chloride which is then extracted with

water. The chloride in the extract is determined volumetrically by titration with silver in the presence of thiocyanate.

4. Significance and Use

4.1 This test method can 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 *Reaction Vessel*, consisting of a 300-mL round-bottom flask with standard-taper joint and surmounted with a water-cooled condenser. The condenser tube shall be wide enough to allow rapid reflux without hold-up.

6. Reagents

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 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 Types II or III of Specification D 1193.

6.3 *Benzyl Alcohol*—The benzyl alcohol used must not show a visible precipitate when tested with alcoholic silver nitrate (AgNO_3) solution.

6.4 1-Butanol

NOTE 2: Warning—Flammable. Liquid causes eye burns.

6.5 *Ferric Alum Indicator Solution*—Dissolve 350 g of ferric alum ($\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_3 \cdot \text{SO}_4 \cdot 24\text{H}_2\text{O}$) in 1 L of water and add 200 mL of aerated HNO_3 (1+2).

6.6 *Isoamyl Alcohol*—The isoamyl alcohol (Warning—See Note 3:) used must not show a visible precipitate when tested with alcoholic AgNO_3 solution.

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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In the IP, this test method is under the jurisdiction of the Standardization Committee.

In 1964, this test method was adopted as standard without revision.

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 05.03.

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."