



Designation: D2896 – 21

Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration¹

This standard is issued under the fixed designation D2896; 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 basic constituents in petroleum products by titration with perchloric acid in glacial acetic acid.

1.2 Procedures A and B use different titration solvent volumes and sample weights.

NOTE 1—A round robin on a series of new and used oils and additive concentrates has shown that the two procedures give statistically equivalent results.

1.3 **Appendix X2** provides the use of an alternative solvent system which eliminates the use of chlorobenzene in this test method. The use of the alternative solvent gives statistically equivalent results; however, the precision is worse. Paragraph **X2.5.5** provides guidance when comparing results using the two different solvents.

1.4 The constituents that may be considered to have basic characteristics include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, and salts of heavy metals.

NOTE 2—This test method is applicable to both fresh oils and used oils as described in Sections 16, 17, and 19 and **Appendix X1**.

1.5 This test method can be used to determine base number >300 mg KOH/g. However, the precision statement in Section 19 has been obtained only on base number ≤ 300 mg KOH/g.

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

1.7 *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 deter-*

*mine the applicability of regulatory limitations prior to use. For specific warning statements, see Section 7, Section 10, and **X2.2**.*

1.8 *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

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions:*

3.1.1 *base number, n* —the quantity of a specified acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide per gram of sample, required to titrate a sample in a specified solvent to a specified endpoint using a specified detection system.

4. Summary of Test Method

4.1 The sample is dissolved in an essentially anhydrous mixture of chlorobenzene and glacial acetic acid and titrated with a solution of perchloric acid in glacial acetic acid using potentiometric titrimeter. A glass indicating electrode and a reference electrode are used, the latter being connected with the sample solution by means of a salt bridge. The meter readings are plotted against the respective volumes of titrating solution, and the end point is taken at the inflection in the resulting curve.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Liquid Fuels and Lubricants.

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This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

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

4.2 Procedure A uses 120 mL of titration solvent. Procedure B uses 60 mL of titration solvent. In addition, the two procedures use different equations for the calculation of appropriate sample weights. Since many portions of the test method are identical for Procedures A and B, only the unique sections will be described separately for the two versions of the test method.

4.3 Occasionally certain used oils give no inflection in the forward titration mode, in which case a back titration modification with sodium acetate titrant is employed.

5. Significance and Use

5.1 New and used petroleum products can contain basic constituents that are present as additives. The relative amounts of these materials can be determined by titration with acids. The base number is a measure of the amount of basic substance in the oil, always under the conditions of the test. It is sometimes used as a measure of lubricant degradation in service; however, any condemning limits must be empirically established.

6. Apparatus

6.1 *Potentiometric Titrimeters*, either automatic recording or manual.

6.2 *Glass Electrode*, pH 0 to 11, general-purpose type.

6.3 *Reference Electrode*, silver/silver chloride (Ag/AgCl) reference electrode with a nonaqueous bridge as described in Section 10. (See also 19.1.)

NOTE 3—Some reference electrodes with fritted or fiber diaphragms and some combined glass plus reference electrodes systems are commercially available, such as the single-rod glass plus silver/silver chloride electrode assembly. During the development of this test method, the use of electrodes of these types gave problems in some laboratories, but not in others. Accordingly, these electrodes are permitted in this test method, provided that the sodium perchlorate bridge is used; however, when stability or other problems arise with their use, the sleeve-type electrode should be used.

6.4 *Stirrer*, either mechanical or electrical, with variable speeds and with propeller or paddle of chemically inert material. When an electrical stirrer is used, it must be grounded so that disconnecting or connecting the power to the motor will not produce a permanent change in meter reading during the course of a titration. A magnetic stirrer with stirring bar can be used provided it meets these conditions.

6.5 *Buret*, 10 mL or 20 mL, graduated in 0.05 mL divisions and calibrated with an accuracy of ± 0.02 mL, or an automatic buret of similar accuracy.

6.6 *Titration Beaker*, made of borosilicate glass or other suitable titration beaker, tall form recommended.

6.6.1 For Procedure A, use a beaker of 250 mL or 300 mL capacity. For Procedure B, use a beaker of about 150 mL capacity such that 60 mL of titration solvent will cover the electrodes.

NOTE 4—Other beakers of suitable size capacity may be used.

6.7 *Titration Stand*, suitable to support the beaker, electrodes, stirrer, and buret. An arrangement that allows for

the removal of the beaker without disturbing the electrodes, buret, and stirrer is desirable.

NOTE 5—Some apparatus may be sensitive to interference by static electricity, shown by erratic movements of recorder pen or meter indicator, when the titration assembly (beaker and electrodes) is approached by the operator. In this case surround the beaker closely with a cylinder of copper gauze that is electrically grounded.

7. Reagents and Materials

7.1 *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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets the requirement of either Type I, II, or III of Specification D1193.

7.3 *Acetic Acid*, glacial (**Warning**—Toxic and irritant).

7.4 *Acetic Anhydride* (**Warning**—Toxic and irritant).

7.5 Chlorobenzene (**Warning**—Toxic and irritant).

7.6 *Perchloric Acid, Standard Solution in Acetic Acid (0.1 N)*⁴ (**Warning**—Powerful oxidant when dry or heated. Great care should be taken to avoid contact with organic matter under conditions that may result in subsequent drying or heating, and spills should be washed immediately and thoroughly with water)—Mix 8.5 mL of 70 % to 72 % perchloric acid (HClO₄, 70 % to 72 %) (or 10.2 mL of 60 % to 62 % HClO₄ solution) with 500 mL of glacial acetic and 30 mL (or 35 mL if the 60 % to 62 % HClO₄ solution is used) of acetic anhydride. Dilute to 1 L with glacial acetic acid. Allow the solution to stand for 24 h.

NOTE 6—Excess acetic anhydride should be avoided to prevent acetylation of any primary or secondary amines that may be present.

7.7 *Potassium Hydrogen Phthalate*—(KHC₈H₄O₄).

7.8 *Sodium Perchlorate Electrolyte*—(**Warning**—Sodium perchlorate is toxic and an irritant. It is also a powerful oxidizing agent when heated. Great care should be taken to avoid contact with organic matter under conditions that may result in subsequent drying or heating, and spills should be washed immediately and thoroughly with water.) Prepare a saturated solution of sodium perchlorate (NaClO₄) in glacial acetic acid. An excess of undissolved NaClO₄ shall always be present at the bottom of the solution.

7.9 *Titration Solvent*—Add one volume of glacial acetic acid to two volumes of chlorobenzene.

³ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

⁴ Available commercially for purchase already prepared.