Designation: D8126 – 26

Standard Test Method for Efficient Basicity Determination by Potentiometric Hydrochloric Acid Titration[1]

This standard is issued under the fixed designation D8126; 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.

1. Scope*

1.1 This test method covers a procedure for the determination of the efficient basicity (EBas) in new marine cylinder lubricants that lubricate the upper part, that is, piston-ring-cylinder area, of two-stroke marine engines.

1.2 This test method has been developed for marine cylinder lubricants, having base number (BN) (measured by Test Method D2896) from 20 mg KOH/g to 100 mg KOH/g, and an EBas from 10 mg KOH/g to 36 mg KOH/g.

1.3 In this test method, only the efficient basicity is determined, corresponding to the components or part of components that have a major role in neutralization of the acidic species formed in the combustion of the fuel in the marine engine. It differentiates between the fastest reacting species and the last one to react like overbasing compounds in detergent (classically mineral calcium carbonate, $CaCO_3$). The values obtained, however, are intended to be compared with the other values obtained by this test method only; base numbers obtained by this test method are not intended to be equal to values by other test methods.

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

1.5 *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. Some specific hazards statements are given in Sections 6 and 7.*

1.6 *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

D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4739 Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *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.

3.1.3 *efficient basicity, EBas, n*—the quantity of hydrochloric acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide per gram of sample, required to neutralize the basic components, which are organic species, of the tested lubricant in a specified solvent to a specified buffer endpoint using a specified detection system.

4. Summary of Test Method

4.1 The sample is dissolved in a mixture of toluene, propan-2-ol (isopropyl alcohol), chloroform, and a small amount of water and titrated potentiometrically with alcoholic

[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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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. 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**

hydrochloric acid solution. The test results of this procedure are obtained by titration mode of fixed increment and fixed time additions of the titrant. An endpoint is selected from a titration curve according to the criteria given in Section 12 and used to calculate the EBas.

5. Significance and Use

5.1 Lubricants can contain basic constituents that are present as additives. The relative amount of these materials can be determined by titration with acids. The base number is a measurement of the amount of basic substances in the oil under the conditions of the testing procedure.

5.2 A primary objective of a marine cylinder lubricant is the neutralization of sulfuric acid, produced during fuel combustion, to protect the engine from corrosion. The EBas method characterizes the more efficient basic species of the lubricant reacting with acids from the beginning of the neutralization (when the lubricant milieu is basic) until an equilibrium state where the lubricant becomes slightly acidic. The use of hydrochloric acid (HCl) allows differentiating basicities of various strengths during titration.

5.3 In marine lubricants, the constituents that can be considered to have basic properties are primarily organic and inorganic bases coming from the detergent. Basicity can also be brought to the lubricant by other components including dispersants, amino compounds, or any organic basic components. This test method uses the same titration system as that of Test Method D4739, however the evaluation and interpretation of the titration result is different. The hydrochloric acid is used as the titrant in this test method and Test Method D4739, whereas Test Method D2896 uses a stronger acid, perchloric acid. While all three methods can be considered as complementary to evaluate the basicity of lubricants, this test method is only used for marine cylinder lubricants.

5.4 This test method measures only the efficient basicity of the lubricant, which represents only the part of the total basicity, until the point of which the lubricant becomes slightly acidic. It will depend on the components of the formulation itself. In that respect, the EBas cannot be compared to base number given by Test Methods D4739 or D2896. The EBas is then complementary information to the base number, giving performance in efficiency of neutralization of acidic species.

6. Apparatus

6.1 *Potentiometric Titration,* automatic or manual, with capability of adding fixed increments of titrant at fixed time intervals (see Annex A1).

6.1.1 The titrator must automatically (or manually) control the rate of addition of titrant as follows: Delivery of titrant will be incremental; after delivery of precisely a 0.100 mL increment (see 6.1.2), the delivery is stopped and a fixed time period of 90 s is allowed to pass before another 0.100 mL increment of titrant is delivered. This procedure is repeated until the titration is completed.

6.1.2 The precision of addition of the 0.100 mL increments of titrant must be ±0.001 mL for automatic titrators. For manual buret, it should be ±0.005 mL. A higher incremental precision is required for an automatic buret, because the total volume to the end point is summed from the individual increments; it is read from a scale with a manual buret.

6.2 *Sensing Electrode,* standard pH with glass membrane, suitable for non-aqueous titrations.

6.3 *Reference Electrode,* Silver/Silver Chloride (Ag/AgCl) reference electrode with sleeve junction, filled with 1 M to 3 M lithium chloride (LiCl) in ethanol.

6.3.1 *Combination Electrode*—Sensing electrodes may have the silver/silver chloride (Ag/AgCl) reference electrode built into the same electrode body, which offers the convenience of working with and maintaining only one electrode. The combination electrode shall have a sleeve junction type of reference and shall use an inert ethanol electrolyte, for example, 1 M to 3 M LiCl in ethanol.

6.3.1.1 In the reference compartment, the sensing electrode part shall use a glass membrane designed for nonaqueous titrations. These combination electrodes shall have the same response or better response than a dual electrode system. They shall have removable sleeves for easy rinsing and addition of electrolyte. When a movable sleeve is part of the electrode system, ensure that the sleeve is unimpaired before every titration.

6.4 *Stirrer, Buret, Stand, Titration Vessel,* as specified in Annex A1, are required.

7. Reagents

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 *Buffer, Aqueous Acid*—Commercial pH 5 buffer solution with a tolerance of ±0.02 pH units at 25 °C. This solution shall be replaced at regular intervals consistent with its stability or when contamination is suspected. Information related to the stability should be obtained from the manufacturer.

7.4 *Chloroform*—Reagent grade. (**Warning—**Toxic and suspected carcinogen.)

7.5 *Hydrochloric Acid Solution, Standard Alcoholic (0.1 M)*—Mix 9 mL of reagent grade hydrochloric acid (HCl, sp gr 1.19) (**Warning—**Toxic and corrosive), with 1 L of anhydrous isopropyl alcohol. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration. of

[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.