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Standard Test Method for Bromine Index of Petroleum Hydrocarbons by Electrometric Titration[1]

This standard is issued under the fixed designation D2710; 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 the determination of the amount of bromine-reactive material in petroleum hydrocarbons and is thus a measure of trace amounts of unsaturates in these materials. It is applicable to materials having bromine indexes below 1000.

1.2 This test method is applicable only to essentially olefin-free hydrocarbons or mixtures that are substantially free from material lighter than isobutane and have a distillation end point under 288°C (550°F).

NOTE 1—This procedure has been cooperatively tested on materials with bromine indexes in the range from 100 to 1000. These materials include petroleum distillates such as straight-run and hydrocracked naphtha, reformer feed, kerosine, and aviation turbine fuel.

NOTE 2—Materials with bromine index greater than 1000 should be tested for bromine number using Test Method D1159/IP 130.

NOTE 3—Bromine index of industrial aromatic hydrocarbons should be determined using Test Method D1492 or D5776. The subcommittee is currently examining ways to achieve more consistent end point values.

1.3 The values stated in SI units are to be regarded as standard. The values stated in inch-pound units are for information only.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1159 Test Method for Bromine Numbers of Petroleum Distillates and Commercial Aliphatic Olefins by Electrometric Titration

D1492 Test Method for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration

D5776 Test Method for Bromine Index of Aromatic Hydrocarbons by Electrometric Titration

3. Terminology

3.1 *Definitions:*

3.1.1 *bromine index, n*—the number of milligrams of bromine that will react with 100 g of sample under the conditions of the test.

4. Summary of Test Method

4.1 A known mass of the sample dissolved in a specified solvent is titrated with standard bromide-bromate solution. The end point is indicated by a dead stop electrometric titration apparatus when the presence of free bromine causes a sudden change in the electrical conductivity of the system.

5. Significance and Use

5.1 This test method provides a measure of trace amounts of unsaturated hydrocarbons in petroleum distillates boiling up to 288°C (550°F). An estimate of the quantity of these materials is useful in assessing the suitability of the lighter fractions for use as reaction solvents.

6. Apparatus

6.1 *Electrometric End Point Titration Apparatus*—Any apparatus designed to perform titrations to pre-set end points (see Note 4) may be used in conjunction with a high-resistance

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

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

[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.

polarizing current supply capable of maintaining approximately 0.8 V across two platinum electrodes and with a sensitivity such that a voltage change of approximately 50 mV at these electrodes is sufficient to indicate the end point. Other types of commercially available electric titrimeters, including certain pH meters, have also been found to be suitable.

NOTE 4—Pre-set end point indicated with polarized electrodes provides a detection technique similar to the dead stop technique specified in previous versions of this test method.

6.2 *Titration Vessel*—A jacketed glass vessel of approximately 150-mL capacity of such a form that can be conveniently maintained at 0 to 5°C (32 to 41°F). A pair of platinum electrodes spaced not more than 5 mm apart shall be mounted to extend well below the liquid level. Stirring shall be by a mechanical or electromagnetic stirrer and shall be rapid, but not so vigorous as to draw air bubbles down to the electrodes.

6.3 *Burets,* 10 and 50-mL capacity.

6.4 *Iodine Number Flasks,* glass-stoppered, 500-mL capacity.

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 American Chemical Society where such specifications are available.[3] Other grades may be used, providing 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 distilled water, or water of equivalent purity.

7.3 *Preparation and Standardization :*

7.3.1 *Bromide-Bromate Standard Solution* (*0.05 N*)—Dissolve 5.1 g of potassium bromide (KBr) and 1.4 g potassium bromate ($KBrO_3$) in water and dilute to 1 L. Standardize to four significant figures as follows: Place 50 mL of glacial acetic acid (**Warning** —Poison. Combustible. May be fatal if swallowed. Causes severe burns. Harmful if inhaled) and 1 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) (**Warning**—Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled.) in a 500-mL iodine number flask. Chill the solution in an ice bath for approximately 10 min, and with constant swirling of the flask, add from a 50-mL buret 40 to 45 mL of bromide-bromate solution, estimated to the nearest 0.01 mL, at a rate such that the addition takes between 90 and 120 s. Stopper the flask immediately, shake the contents, place it again in the ice bath, and add 5 mL of potassium iodide (KI) solution in the lip of the flask. After 5 min, remove the flask from the ice bath and allow the KI solution to flow into the flask by slowly removing the stopper. Shake vigorously, add 100 mL of water in such a manner as to rinse the stopper, lip, and walls of the flask, and titrate promptly with the standard sodium thiosulfate ($Na_2S_2O_3$) solution. Near the end of the titration, add 1 mL of starch indicator solution and titrate slowly to the disappearance of the blue color. Calculate the normality of the bromide-bromate solution as follows:

$$N_1 = A_2 N_2 / A_1 \tag{1}$$

where:

N_1 = normality of the bromide-bromate solution,
A_1 = millilitres of the bromide-bromate solution,
N_2 = normality of the $Na_2S_2O_3$ solution, and
A_2 = millilitres of the $Na_2S_2O_3$ solution required for titration of the bromide-bromate solution.

7.3.2 *Potassium Iodide Solution (150 g/L)*—Dissolve 150 g of KI in water and dilute to 1 L.

7.3.3 *Sodium Thiosulfate, Standard Solution* (0.05 *N)*—Dissolve 12.5 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) in water and add 0.01 g of sodium carbonate (Na_2CO_3) to stabilize the solution. Dilute to 1 L and mix thoroughly by shaking. Standardize by any accepted procedure that determines the normality with an error not greater than ±0.0002. Restandardize at intervals frequent enough to detect changes of 0.0005 in normality.

7.3.4 *Starch Indicator Solution*—Mix 5 g of soluble starch with approximately 3 to 5 mL of water. If desired, add about 0.65 g salicylic acid as preservative. Add the slurry to 500 mL of boiling water and continue boiling for 5 to 10 min. Allow to cool and decant the supernatant liquid into glass bottles and seal well. Starch solutions (some preserved with salicylic acid) are also commercially available and may be substituted.

7.3.5 *Sulfuric Acid* (*1+5*)—Carefully add 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 5 volumes of water and thoroughly mix. (**Warning** —Poison. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be fatal if swallowed.)

7.3.6 *Titration Solvent*—Prepare 1 L of titration solvent by mixing the following volumes of materials: 714 mL of glacial acetic acid, 134 mL of 1,1,1-trichloroethane or dichloromethane, 134 mL of methanol, and 18 mL of H_2SO_4 (1+5) (see 7.3.5).

7.4 *Solvents:*

7.4.1 *Acetic Acid,* glacial. (**Warning**—Poison. Combustible. May be fatal if swallowed. Causes severe burns. Harmful if inhaled.)

7.4.2 *Methanol* (**Warning**—Flammable. Vapor harmful. May be fatal if swallowed. Causes severe burns. Harmful if inhaled.)

7.4.3 *1,1,1-Trichloroethane* (**Warning**—Harmful if inhaled. High concentrations may cause unconsciousness or death. Contact may cause skin irritation and dermatitis.)

7.4.4 *Dichloromethane* (**Warning**—Harmful if inhaled. High concentrations may cause unconsciousness or death. Contact may cause skin irritation and dermatitis.)

NOTE 5—The replacement of 1,1,1-trichloroethane, an ozone-depleting chemical, is necessary because its manufacture and import has been discontinued. Dichloromethane is temporarily being allowed as an alternative to 1,1,1-trichloroethane until a permanent replacement can be

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[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.