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Designation: D 1492 – 02

Standard Test Method for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration[1]

This standard is issued under the fixed designation D 1492; 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] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.0~~E~~4 on Instrumental Analysis.

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1. Scope

1.1 This test method covers the determination of the amount of bromine-reactive material in aromatic hydrocarbons. It is usually applied to materials having bromine indexes below 500.

NOTE 1—Other test methods for determining bromine-reactive material are Test Methods D 1159, D 1491, D 2710, and D 5776.

1.2 This test method has been found applicable to aromatic hydrocarbons containing no more than trace amounts of olefins and that are substantially free from material lighter than isobutane and have a distillation end point under 288°C.

1.3 The following applies to all specified limits in this test method: For purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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.* For a specific hazard statement see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[2]

D 1159 Test Method for Bromine Number of Petroleum Distillates and Commercial Aliphatic Olefins by Electrometric Titration[3]

D 1193 Specification for Reagent Water[4]

D 1491 Test Method for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration[5]

D 2710 Test Method for Bromine Index of Petroleum Hydrocarbons by Electrometric Titration[6]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[7]

D 3505 Test Method for Density or Relative Density of Pure Liquid Chemicals[7]

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[6]

D 5776 Test Method for Bromine Index of Aromatic Hydrocarbons by Electrometric Titration[7]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[8]

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[9]

3. Terminology

3.1 *Definition:*

[2] *Annual Book of ASTM Standards*, Vol 15.05.
[3] *Annual Book of ASTM Standards*, Vol 05.01.
[4] *Annual Book of ASTM Standards*, Vol 11.01.
[5] Discontinued; see *1985 Annual Book of ASTM Standards*, Vol 06.03.
[6] *Annual Book of ASTM Standards*, Vol 05.02.
[7] *Annual Book of ASTM Standards*, Vol 06.04.
[8] *Annual Book of ASTM Standards*, Vol 14.02.
[9] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

3.1.1 *bromine index*—the number of milligrams of bromine consumed by 100 g of sample under given conditions.

4. Summary of Test Method

4.1 The specimen is added to a solvent and titrated with electrolytically generated bromine at room temperature. The end point is determined by a dead-stop method. The time of titration is proportional to the bromine added to the specimen.

5. Significance and Use

5.1 This test method is useful for setting specification, for use as an internal quality control tool, and for use in development or research work on industrial aromatic hydrocarbons and related materials. This test method gives a broad indication of olefinic content. It will not differentiate between the types of aliphatic unsaturation.

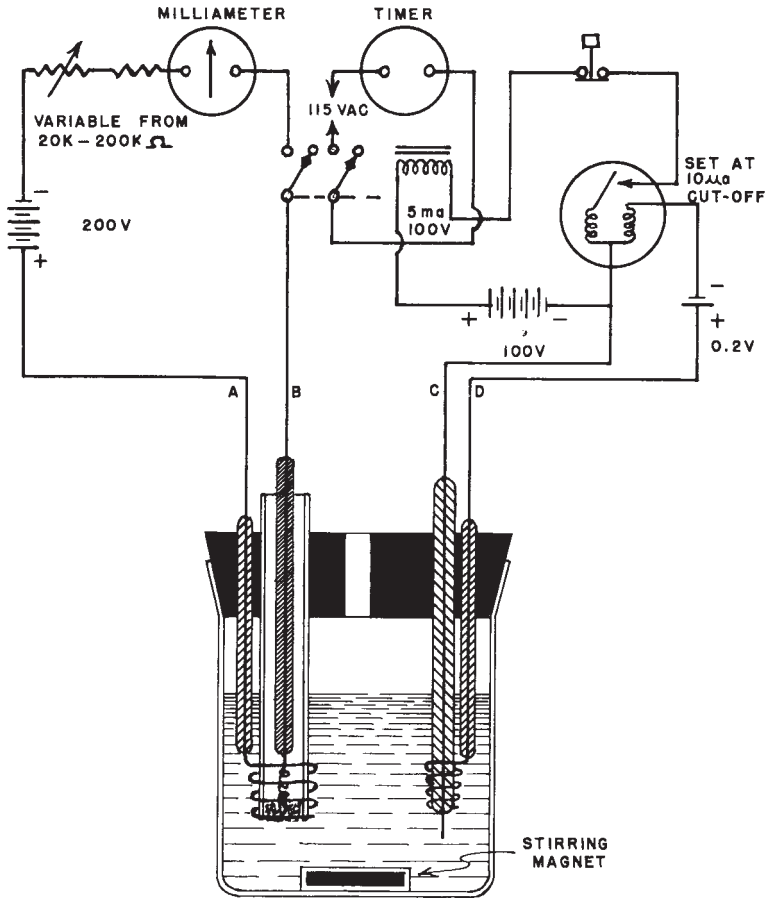

FIG. 1 Automatic Amperometric-Coulometric Titrator Circuit

6. Apparatus

6.1 *Amperometric-Coulometric Apparatus*, automatic, suitable for bromine index titration[2]s with variable generator current and timer. A typical circuit diagram of suitable equipment is shown in Fig. ~~1.~~[10] 1.

6.2 *Syringe*, 2 mL with needle and rubber cap seal.

6.3 *Stirrer*, magnetic.

7. Reagents

7.1 *Purity of Reagent*—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.[10] 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 conforming to Type IV of Specification D 1193.

7.3 *Electrolyte*—To make 1 L, mix 600 mL of glacial acetic acid, 260 mL of absolute methanol, and 140 mL of KBr solution (119 g/L). Dissolve 2 g of Mercury II acetate in this mixture.

7.4 *Potassium Bromide Solution* (119 g/L)—Dissolve 119 g of potassium bromide (KBr) in water and dilute to 1 L.

8. Hazards

8.1 Consult current OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

9. Sampling

9.1 Sample the material in accordance with Practice D 3437.

10. Procedure

10.1 Place ~~50~~ 125 mL of electrolyte in a clean, dry titration cell, insert the electrodes, and begin stirring. Verify the volume of electrolyte is sufficient to ensure the electrodes are completely submerged and if necessary, increase the volume of electrolyte required. Apply the generation current in accordance with Table 1.

10.2 Before introducing the specimen and immediately before each determination, bring the coulometer to equilibrium.

10.3 Draw into the syringe the amount of sample prescribed in Table 1 corresponding to the estimated bromine index. Wipe the needle with a clean cloth, attach a rubber cap seal to the needle, and weigh on the analytical balance. Remove the seal, add the specimen to the electrolyte, and set the timer to zero. Replace the seal, reweigh the syringe, and calculate the specimen weight.

NOTE 2—If the density or specific gravity of the specimen is known (Test Methods D 891, D 3505, or D 4052 can be used), the specimen can be added by means of a pipet or microburet and the weight calculated.

10.4 Begin titration of the specimen. As the titration proceeds, keep the generation current at the selected value. The generation of bromine will continue as long as it is consumed by the sample. At the end point an incremental increase in bromine concentration causes the titration and timer to stop automatically. Forty seconds after the titration has shut off, continue the titration. If the titration cuts off, immediately, the end point has been reached and the titration may be considered complete. Otherwise, it may be necessary to continue the titration in steps, waiting about 40 s between steps, until the titration time increment is 4 s or less. Note the total titration time and generation current.

11. Calculation

11.1 Calculate the bromine index, *B*, as follows:

$$B = \frac{TI \times 79.9}{965W} \quad (1)$$

where:

T = titration time, s,
I = generation current, mA, and
W = weight of specimen, g.

12. Report

12.1 Report the following information:

[10] ~~The sole source of supply of~~ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the ~~apparatus known to the committee at this time is Refinery Supply Co., 6901 E12th St. Tulsa, OK 74112. If you are aware~~ testing of ~~alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of~~ reagents not listed by the ~~responsible technical committee,~~[1] ~~which you may attend.~~ 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.

TABLE 1 Specimen Size and Generation Current

Estimated Bromine Index	Specimen Weight, g	Generation Current, mA
0 to 20	1.000	1.0
20 to 200	0.600	5.0
200 to 2000	0.060	5.0

12.1.1 Report bromine index to the nearest unit.

13. Precision and Bias

13.1 Precision data were generated using titrators from Central Scientific Co. The precision obtained using titrators from other suppliers has not been determined.

13.2 *Precision*—The following data should be used for judging the acceptability of results (95 % probability) for bromine indexes from 0 to 50:

13.2.1 *Intermediate Precision (formerly called Repeatability)*—The standard deviation is 0.39. Duplicate results by the same operator should be considered suspect if results differ by more than 1.15.

NOTE 3—Number of data used, 91; number of degrees of freedom, 61; number of cooperating laboratories, 4.

13.2.2 *Reproducibility*—The standard deviation is 1.43. The results submitted by two laboratories should be considered suspect if they differ by more than 4.1.

NOTE 4—Number of data used, 41; number of degrees of freedom, 30; number of cooperating laboratories, 4.

13.3 *Bias*—The procedure in this test method has no bias because the value of bromine index can be defined only in terms of a test method.

14. Keywords

14.1 aromatic hydrocarbons; bromine index; bromine-reactive; coulometric titration; titration