Designation: D1492 – 13

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

This standard is issued under the fixed designation D1492; 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 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 D1159, D1491, D2710, and D5776.

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 E29.

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 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:*[2]

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

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

D1193 Specification for Reagent Water

D1491 Test Method for Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (Withdrawn 1985)[3]

D2710 Test Method for Bromine Index of Petroleum Hydrocarbons by Electrometric Titration

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D3505 Test Method for Density or Relative Density of Pure Liquid Chemicals

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

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

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *bromine index (B), n*—the number of milligrams (mg) of bromine consumed by 100 g of sample under given conditions (mg-Br/100 g).

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.

[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 D16.04 on Instrumental Analysis.

Current edition approved July 15, 2013. Published September 2013. Originally approved in 1957. Last previous edition approved in 2008 as D1492 – 08ε1. DOI: 10.1520/D1492-13.

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

***A Summary of Changes section appears at the end of this standard**

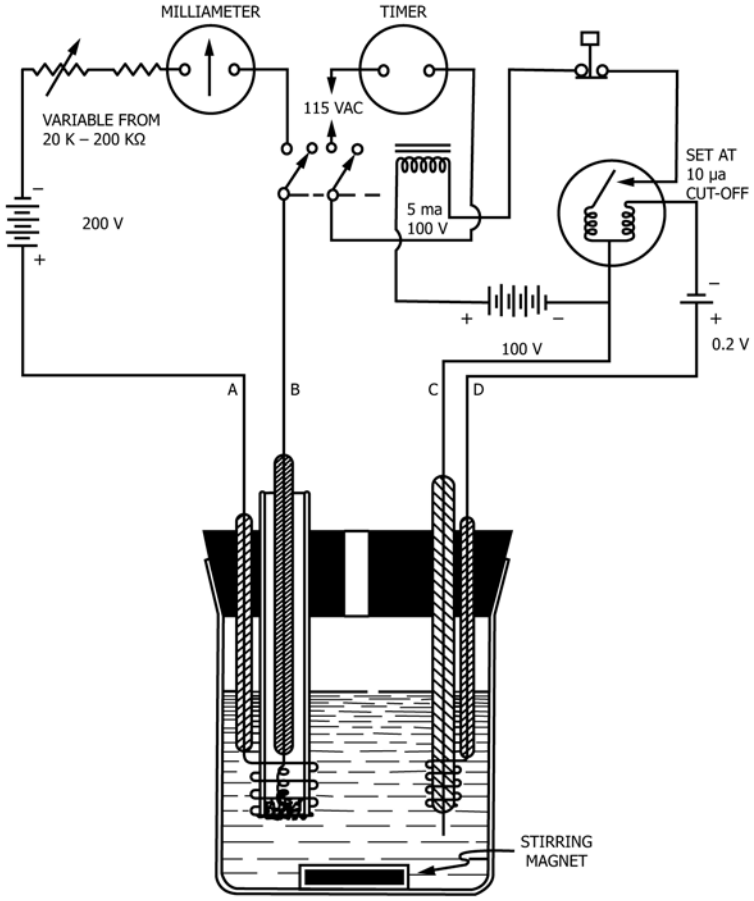

FIG. 1 Automatic Amperometric-Coulometric Titrator Circuit

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.

6. Apparatus

6.1 *Amperometric-Coulometric Apparatus,* automatic, suitable for bromine index titrations with variable generator current and timer. A typical circuit diagram of suitable equipment is shown in Fig. 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.[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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

7.3 *Electrolyte:*

7.3.1 *Preparation Cell Electrolyte*—To make 1 L, mix 600 mL of glacial acetic acid, 260 mL of absolute methanol, and 140 mL of KBr solution (119g/L).

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

[5] *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 *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.

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 D3437.

10. Procedure

10.1 Place 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.

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

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 D891, D3505, or D4052 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 (mg-Br/100 g)*, as follows:

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

where:

T = titration time, s
I = generation current, mA
W = weight of specimen, g
79.9 = MW bromine (grams/mole), and
965 = Faraday's constant (96 500 coulomb/mole)/100

12. Report

12.1 Report the following information:

12.1.1 Report bromine index to the nearest unit.

13. Precision and Bias

13.1 *Precision*—A ruggedness study was performed by analyzing three samples 20 times and one sample five times each over the shortest practical time. The calculated repeatability is shown in Table 2.

TABLE 2 Repeatability[A]

Sample Name	Replicates (n)	Conc. mg-Br/100 g	r
997.5 mg-Br/100g Std	20	1012	39.1
Toluene-Pit EPX	20	31.2	2.9
Xylene-Plant Feed	20	10.7	2.3
Reformate-Feed	5	670	53.9

[A] Repeatability = SD * 2.8.

13.2 *Repeatability*—See Table 2.

13.3 *Reproducibility*—To be determined.

14. Quality Guidelines

14.1 Refer to Guide D6809 for suggested QA/QC activities that can be used as a part of this test method. It is recommended that the operator of this test method select and perform relevant QA/QC activities like the ones in Guide D6809 to help ensure the quality of data generated by this test method.

15. Keywords

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

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D1492-08$^{\varepsilon 1}$) that may impact the use of this standard. (Approved July 15, 2013.)

(1) Deleted Section 1.5.
(2) Deleted Section 7.3.
(3) Changed Section 7.3.2 to Section 7.3.1.
(4) Deleted Section 13.1 through 13.1.3.
(5) Changed Section 13.2.1 to Section 13.1.
(6) Changed Section 13.2.2 to Section 13.2.
(7) Changed Section 13.2.3 to Section 13.3.