Designation: ~~D1492 – 13~~ D1492 – 21

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.~~ Bromine Index in aromatic hydrocarbons, their derivatives, and related chemicals.

~~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.~~is applicable to samples with Bromine Index concentrations to 100 Bromine Index. The limit of detection (LOD) is for 1.5 Bromine Index and the limit of quantitation (LOQ) is 4.9 Bromine Index.

NOTE 1—LOD and LOQ were calculated using data from the D16 PTP program. This estimate of LOD includes within-lab variability.

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

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.*For specific hazard statements, see Section 8.

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.*

[1] This test method is under the jurisdiction of ASTM Committee D16 on ~~Aromatic Hydrocarbons~~ Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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

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
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

3.1 *Definitions:*

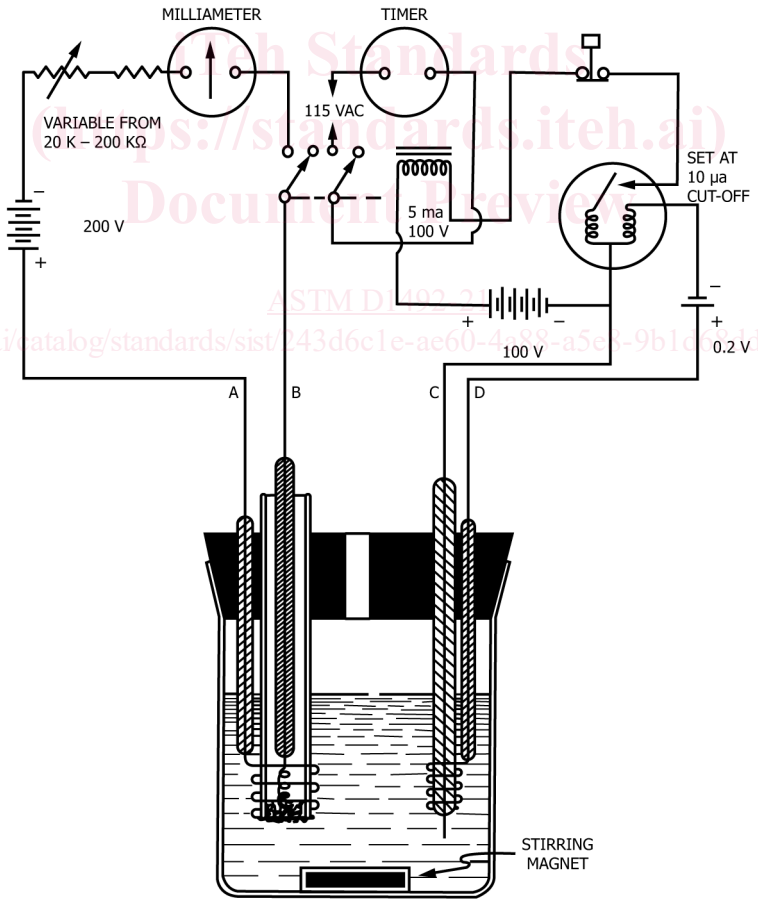

FIG. 1 Automatic Amperometric-Coulometric Titrator Circuit

[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] 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.

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.

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.[4] 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~~ I or Type II of Specification D1193.

7.3 *Electrolyte:*

7.3.1 *Preparation Cell Electrolyte*—To make 1 L, mix ~~600 mL~~ 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.

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

[4] ~~*Reagent Chemicals, American Chemical Society Specifications,*~~*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,~~BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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~~
20 to 100	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~~
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 0.1 unit.

13. Precision and Bias[5]

13.1 An ILS was conducted which included five laboratories analyzing two samples. Practice E691 was followed for the design and analysis of the data; the details are given is ASTM Research Report D16-2004. The ILS did not meet the minimum requirements of Practice E691 for six labs, four materials, and two replicates.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-2004. Contact ASTM Customer Service at service@astm.org.