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Standard Test Method for Iron Content of Bisphenol A (4,4′ - Isopropylidenediphenol)[1]

This standard is issued under the fixed designation D6143; 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 procedure to determine the iron content of bisphenol A (4,4′-isopropylidenediphenol).

1.2 This test method has a lower detection limit of 0.1 mg/kg, and an upper limit of 10 mg/kg of iron in bisphenol A. If the iron content is higher, it may be necessary to dilute the sample. A longer path length cell can also be used for better accuracy at lower Fe levels, as well as calibration within the range expected (for example, 0 to 1 mg/kg versus 0 to 10 mg/kg for samples expected to be in the 0 to 1 mg/kg range.

~~1.3In~~1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off 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 limits prior to use.* For a specific hazard statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D4297 Practice for Sampling and Handling Bisphenol A (4,4 -Isopropylidinediphenol)

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 Documents:*

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

3. Summary of Test Method

3.1 Bisphenol A is dissolved in methanol, then treated with hydroxylamine hydrochloride to convert any ferric iron present to ferrous iron. The ferrous iron is then complexed with FerroZine to form a purple/maroon chromophore that is quantified by visible spectrophotometry at 560 nm.

4. Significance and Use

4.1 Iron may increase the color of bisphenol A and affect other properties of end-use products.

4.2 High purity bisphenol A typically has less than 1 mg/kg of iron.

5. Interferences

5.1 No direct interferences have been observed in the use of this method.

6. Apparatus

6.1 *Visible Spectrophotometer*, capable of measuring absorbance at 560 nm.

6.2 *Analytical Balance*, capable of weighing 100 g to the nearest 0.01 g.

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[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of D16.02 on Oxygenated Aromatics.

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[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.

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

6.3 *Glassware*, 100 mL and 1 L volumetric flasks, 500 mL graduated cylinders, 10 mL volumetric pipettes, 1 cm square quartz cuvettes.

6.4 *All Glassware* used in this test method should be dedicated and thoroughly cleaned prior to use.

7. Reagents and Materials

7.1 *Methanol*, ACS reagent grade, ≥ 99.8 % purity.

7.2 *Sodium Acetate*, pH 5.5.

7.2.1 This solution may be prepared by dissolving 272.0 g ± 0.1g of reagent grade sodium acetate in 1000 mL distilled water and adjusting to pH 5.5 with concentrated hydrochloric acid.

7.3 *Distilled Water*, conforming to Type IV of Specification D1193.

7.4 *Standard Iron Solution*, Titrisol, 100 mg/L (ca. 98 mg/kg) iron chloride in 15 % hydrochloric acid or equivalent.

7.5 *Sodium Hydroxide*, 0.5 normal, ACS reagent grade.

7.6 *Hydroxylamine Hydrochloride* , 99.999 % min.

7.7 *Hydroxylamine Hydrochloride Solution*—Make up a 10 % by weight aqueous solution.

7.8 *Ferrozine Iron Reagent*, 3–(2–Pyridyl)-5,6–diphenyl-1,2,4–triazine-p,p'-disulfonic acid, or equivalent.

7.9 *Ferrozine Solution*:

7.9.1 In a 1 L volumetric flask, dissolve 1.00 ± 0.01 g of ferrozine iron reagent in 20 mL of 0.5 N NaOH and dilute to 1 L with methanol.

8. Hazards

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

9. Sampling and Handling

9.1 Sample bisphenol A (BPA) in accordance with Practice D4297.

10. Calibration

10.1 Weigh into separate 100 mL volumetric flasks, to the nearest 0.01 g, the following amounts of the standard iron solution in 7.4: 0.2, 0.5, 1.0, 2.0, 4.0, 8.0, and 10.0. Dilute to volume with distilled water, stopper, and shake to mix. This will give solutions with nominal concentrations of 0.2, 0.5, 1.0, 2.0, 4.0, 8.0, and 10 mg/kg, respectively.

10.2 Prepare and analyze two 10.0 ± 0.1 g aliquots of each of the above calibration standard solutions and two blanks in accordance with the instructions given in Section 11.

10.3 Calibrate the instrument in accordance with manufacturer's instructions.

10.4 If manual calibration is required, calculate the average of the replicate measurements of each of the calibration and blank solutions.

10.5 Plot the nominal concentrations versus the average absorbance measurement for each of the calibration standards.

10.6 Using linear regression, determine the slope and intercept of the calibration curve according to Eq 1:

$$C = mA + b \quad (1)$$

where:

C = concentration of iron in the solution in ~~mg/kg,~~ mg/kg

m = slope of the calibration curve, mg/kg per absorbency ~~units,~~ units

A = absorbance of the solution in absorbency ~~units, and~~ units

b = intercept of the calibration curve in ~~mg/kg.~~mg/kg

11. Procedure

11.1 Weigh 10.0 g of the BPA sample to be analyzed to the nearest 0.1 g into a 100–mL volumetric flask.

11.2 Label a second, clean, empty, 100 mL volumetric flask as a blank.

11.3 Add 40 mL of methanol to each of the above flasks.

TABLE 1 Summary of Precision Data

Precision, characterized by repeatability, Sr, r, and reproducibility, SR, R for analysis of Fe in BPA					
Sample #	mg/kg Average	Sr	SR	r	R
1	0.29	0.09	0.10	0.26	0.27
2	0.02	0.00	0.04	0.00	0.11
3	7.30	0.32	0.65	0.90	1.83
4	0.51	0.15	0.17	0.41	0.49
5	3.30	0.15	0.91	0.42	2.55