Designation: D6143 – 13

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.

1.3 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 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

3.1 See Terminology D4790 for definitions of terms used in this standard.

4. Summary of Test Method

4.1 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.

5. Significance and Use

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

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

6. Interferences

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

7. Apparatus

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

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

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

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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**

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

8. Reagents and Materials

8.1 *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 specification 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.

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

8.3 *Sodium Acetate,* pH 5.5.

8.3.1 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.

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

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

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

8.7 *Hydroxylamine Hydrochloride,* 99.999 % min.

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

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

8.10 *Ferrozine Solution:*

8.10.1 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.

9. Hazards

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

10. Sampling and Handling

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

11. Calibration

11.1 Weigh into separate 100 mL volumetric flasks, to the nearest 0.01 g, the following amounts of the standard iron solution in 8.5: 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.

11.2 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 12.

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

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

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

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

$$C = mA + b \tag{1}$$

where:

C = concentration of iron in the solution in mg/kg
m = slope of the calibration curve, mg/kg per absorbency units
A = absorbance of the solution in absorbency units
b = intercept of the calibration curve in mg/kg

12. Procedure

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

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

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

12.4 Gently swirl until all of the sample is dissolved.

12.5 Add 30 mL of the sodium acetate solution to each of the flasks.

12.6 Gently swirl then allow to stand for approximately 10 min.

12.7 Add 2 mL of the 10 % aqueous hydroxylamine hydrochloride solution.

12.8 Gently swirl then allow to stand for approximately 10 min.

12.9 Add 4 mL of ferrozine solution to each of the flasks.

12.10 Dilute the contents of each flask to the mark with methanol, cap and shake the flasks to thoroughly mix the contents.

12.11 Set a timer for 10 min to allow the color complex to become stable.

NOTE 1—The color is somewhat unstable and care should be taken to adhere to this development time as closely as possible.

12.12 Transfer a portion of each of the above solutions to a 1 cm quartz cuvette and read the absorbance using the spectrophotometer set to 560 nm.

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[4] *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.

NOTE 2—It is crucial, especially for low levels of iron, that the windows of the cuvette be absolutely clean. Carefully wiping the windows with a paper towel wetted with water followed by one wetted with methanol and then drying with a clean paper towel after the sample has been added to the cuvette is strongly recommended.

13. Calculation and Report

13.1 If manual calculations are required, calculate the concentration of iron in each of the samples as follows:

$$C_1 = m(A_S - A_B) + b \qquad (2)$$

where:

C_1 = concentration of iron in the sample in mg/kg,
m = slope of the calibration curve from Section 11, mg/kg per absorbency units,
A_S = absorbance of the sample solution in absorbency units,
A_B = absorbance of the blank solution in absorbency units,
b = intercept of the calibration curve from Section 11, in mg/kg.

13.2 Report iron in the bisphenol A to the nearest 0.1 mg/kg.

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

14. Precision and Bias[5]

14.1 The criteria in Table 1 should be used to judge the acceptability at the 95 % probability level of the results obtained by this test method (95 % confidence level). The criteria were derived from an interlaboratory study of five samples in triplicate between three laboratories. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1034.

14.2 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ by more than the amounts listed in Table 1. Results differing by less than "*r*" have a 95 % probability of being correct.

14.3 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the amounts listed in Table 1. Results differing by less than "*R*" have a 95 % probability of being correct

14.4 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method for measuring these impurities, bias has not been determined.

15. Quality Guidelines

15.1 Laboratories shall have a quality control system in place.

15.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

15.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

15.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

15.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

16. Keywords

16.1 bisphenol A; iron

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D6143–09) that may impact the use of this standard. (Approved June 1, 2013.)

(1) Brought all Sections up-to-date with current D16 Editorial Guidelines.

(2) Section 3 added and all subsequent sections re-numbered.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1034.

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