



## Standard Test Method for Iron Content of Bisphenol A (4,4' - Isopropylidenediphenol)<sup>1</sup>

This standard is issued under the fixed designation D 6143; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method describes 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 20 mg/kg of iron in bisphenol A. If the iron content is higher, it will be necessary to dilute the sample.

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 limits prior to use.* For a specific hazard statement, see Section 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 4297 Practice for Sampling and Handling Bisphenol A (4,4' - Isopropylidenediphenol)<sup>3</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>4</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine Precision of a Test Method<sup>4</sup>

#### 2.2 Other Documents:

OSHA Regulations 29CFR paragraphs 1910.1000 and 1910.1200.<sup>5</sup>

### 3. Summary of Test Method

3.1 Bisphenol A is dissolved in methanol, then acidified

with hydrochloric acid to convert any ferric iron present to ferrous iron. The ferrous iron is then complexed with FerroZine to form a red/pink 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 2 mg/kg of iron.

### 5. Interferences

5.1 Acetone and ethanol cause slow color development and should not be used as solvents for this test method.

### 6. Apparatus

6.1 Vis. Spectrophotometer.

6.2 Glass cell, 10 cm.

6.3 Volumetric Flasks, 100 mL and 1000 mL.

NOTE 1—All glassware used in this test method should be dedicated and thoroughly cleaned prior to use, by rinsing with concentrated hydrochloric acid solution followed by ferrozine solution, prepared as a blank in Section 7.

6.4 pH Meter.

### 7. Reagents and Materials

7.1 Hydrochloric Acid, concentrated, American Chemical Society (ACS) reagent grade.

7.2 Methanol, ACS reagent grade. Do not use methanol stored in steel containers.

7.3 Sodium Acetate, 2 M solution, ACS reagent grade.

7.3.1 This solution may be prepared by dissolving 272.0 g  $\pm$  0.1g of ACS reagent grade sodium acetate trihydrate or 164.0  $\pm$  0.1g of ACS reagent grade sodium acetate in 1000 mL distilled water.

7.3.2 The sodium acetate trihydrate or sodium acetate may contain iron and should be purified as follows: To a separatory funnel, add 10 mL of bathophenanthroline solution (prepared by dissolving 0.25 g bathophenanthroline in 250 mL methanol), and 100 mL of 2 M solution of sodium acetate, and mix well. Add 15 mL of chloroform and shake the solution for 30 s. Allow the layers to separate. Draw off and discard the bottom red layer. Repeat the extraction with 15-mL portions of chloroform until the bottom layer is colorless.

<sup>1</sup> 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.

Current edition approved June 10, 1997. Published August 1997.

<sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 06.04

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>5</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

7.4 *Distilled Water*, conforming to Type IV of Specification D 1193.

7.5 *Standard Iron Solution*, 1000 mg/L.

7.5.1 The iron solution is prepared by digesting 1.00 g of ACS reagent grade iron with 4 mL of concentrated ACS reagent grade hydrochloric acid in a 100-mL beaker until a clear solution is obtained. This solution is quantitatively transferred into a 1000-mL volumetric flask, with careful rinsing, containing about 500 mL of distilled water. The flask is then diluted to the mark with distilled water.

7.6 *Sodium Hydroxide*, 0.5 normal, ACS Reagent grade.

7.7 *Ferrozine*, 0.1 % in methanol.

7.8 *Bathophenanthroline*, 0.1 % in methanol, ACS reagent grade.

7.9 *Chloroform*, ACS reagent grade.

7.10 *Hydroxylamine Hydrochloride*, 10 % aqueous, ACS reagent grade.

NOTE 2—The hydroxylamine hydrochloride may contain iron and should be purified as follows: To a separatory funnel, add 10 mL of bathophenanthroline solution (prepared by dissolving 0.25 g of bathophenanthroline in 250 mL of methanol), and 100 mL of hydroxylamine hydrochloride; mix well. Add 15 mL of chloroform and shake the solution for 30 s. Allow the layers to separate. Draw off and discard the bottom red layer. Repeat the extraction with 15 mL portions of chloroform until the bottom layer is colorless.

## 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. Procedure for Calibration

9.1 Prepare a reagent blank concurrently.

9.2 Using the 1000-mg/L standard solution, prepare Solution A by diluting 10 mL of the standard solution to 100 mL with distilled water. Prepare Solution B by dilution 10 mL of Solution A to 100 mL with distilled water. Each mL of Solution B contains 10 µg of iron.

9.3 Prepare calibration standards in 100-mL volumetric flasks as follows:

9.3.1 Add 50 mL of methanol.

9.3.2 Add 2.5 mL of concentrated hydrochloric acid and mix.

9.3.3 Add 2.0 mL of hydroxylamine hydrochloride solution.

9.3.4 Add 20 mL of sodium acetate solution and shake each flask. The pH should be in the range of 4 to 9. Adjust the amount of hydrochloric acid added to the sample to adjust the pH.

9.3.5 Add 1.0 mL of Solution B.

9.3.6 Add 5 mL of Ferrozine solution. Shake and set for 5 min. The color is now stable for 1 h.

9.3.7 Dilute all three calibration standards of Solution B to 100-mL mark with methanol.

9.3.8 Repeat 9.3.1 to 9.3.7, except at 9.3.5 add aliquots of 5.0 mL respectively of Solution B. Calibration concentrations are tabulated as follows:

Concentration of Calibration Standards	
Solution B added, mL	Concentration mg/L in 100-mL flask
1.00	0.1

5.00	0.5
10.00	1.0

9.4 Zero the vis-spectrophotometer at 560 nm with the blank.

9.5 Measure the absorbance at 560 nm of the three prepared calibration solutions.

9.6 Plot a standard curve of absorbance versus iron concentration. The range covered is 0.1 to 1.0 mg/kg.

9.7 Calculate an absorbance factor ( $F$ ) as the inverse of the slope of a line drawn through data plotted in 9.6 as follows:

$$F = [1 / (d(Ab) / d(\text{mg/L}))] \quad (1)$$

where:

$d(Ab)$  = difference in absorbance measured for the calibration range 0.1 mg/L to 1.0 mg/L. For the above calibration range, ( $Ad$  (mg/L)) would be (1.0 mg/L - 0.1 mg/L) = 0.9.

## 10.

## 11. Sample Analysis

11.1 Sample bisphenol A (BPA) in accordance with Practice D 4297. Weigh 5 g of sample to nearest 0.1 mg into a 100-mL volumetric flask.

11.2 Add 50 mL of methanol and swirl the flask to dissolve the bisphenol A.

11.3 Add 2.5 mL of concentrated hydrochloric acid and shake the flask. Let set for 30 mins.

11.4 Add 2.0 mL of hydroxylamine hydrochloride solution and shake the flask. Let set for 5 mins.

11.5 Add 20 mL of sodium acetate solution and shake the flask.

11.6 Check pH in accordance with 9.3.4.

11.7 Add 5 mL of ferrozine solution and let set for 5 mins. for full color development.

11.8 Add methanol to 100-mL volumetric mark and shake the flask well.

11.9 Zero the vis-spectrophotometer at 560 nm with a blank.

11.10 Measure absorbance of the bisphenol A sample at 560 nm.

11.11 Calculate the dilution factor ( $D$ ) by dividing 100 by the sample weight in grams.

## 12. Calculation and Report

12.1 Calculate ppm iron in bisphenol A as follows:

$$\text{absorbance at 560 nm} \times (D) \times (F) \quad (2)$$

12.2 Report iron in the bisphenol A to the nearest 0.1 µg/g.

## 13. Precision and Bias

13.1 *Precision*—The following criteria should be used to judge the acceptability at the 95 % probability level of the results obtained by this test method. The criteria were derived from intermediate precision data for three calibration solutions, with at least 25 individual data points per each calibration level. The data were obtained by using multiple analysts within one laboratory. The results were analyzed in accordance with Practice E 691.

13.1.1 Results for intermediate precision should not be

considered suspect unless they differ by more than the amount shown in Table 1.

13.1.2 *Reproducibility*—Data will be obtained at a later date.

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

**TABLE 1 Intermediate Precision of Iron Standards**

Iron Level, µg/mL	Average Calculated, µg/mL	Standard Deviation, µg/mL at 95 % confidence
0.1	0.10	± 0.019
0.5	0.48	± 0.049
1.0	0.99	± 0.068

## 14. Keywords

14.1 bisphenol A; iron

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