Designation: D8266 – 19

Standard Test Method for Analysis of Bisphenol A (4,4'-Isopropylidenediphenol) by High Performance Liquid Chromatography[1]

This standard is issued under the fixed designation D8266; 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 trace impurities in bisphenol A (4,4'-Isopropylidenediphenol) by reverse-phase gradient high performance liquid chromatography (HPLC). It is generally meant for the analysis of bisphenol A of 99.5 % or greater.

1.2 This method is applicable to bisphenol A samples containing impurity concentration between 2 and 400 mg/kg. Users of this method believe it is linear over a wider range.

1.3 In determining the conformance of the test results using this method to applicable specification, 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 document:*[3]

OSHA Regulations 29 CFR paragraphs 1910.1000 and 1910.1200

3. Summary of Test Method

3.1 A known amount of an internal standard is added to a sample of bisphenol A. The prepared sample is mixed and analyzed by a reverse-phase gradient liquid chromatography (HPLC) equipped with an ultraviolet detector. The peak area of each impurity and the internal standard is measured. The amount of each impurity is calculated from the ratio of the peak area of the internal standard versus the peak area of the impurity.

4. Significance and Use

4.1 Bisphenol A is used for production of polycarbonate or epoxy resin. The presence of impurities in bisphenol A is undesirable because they may slow down the polymerization and be impurities in the final product.

4.2 Determination of the trace impurities, such as isomer of bisphenol A and the unreacted raw material, is often required. This test method is suitable for setting specifications and for using as an internal quality control where these products are produced or used.

4.3 Purity is commonly reported by subtracting the determined expected impurities and water from 100 %. However, a HPLC analysis cannot determine absolute purity if unknown components are contained within the material being examined.

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee 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, Washington, DC 20401-0001, http://www.access.gpo.gov.

5. Interferences

5.1 The internal standard chosen must be sufficiently resolved from any impurity and the bisphenol A peak.

5.2 Any solvent used must also be sufficiently resolved from any impurity, the internal standard, and the bisphenol A peak.

6. Apparatus

6.1 *High Performance Liquid Chromatograph (HPLC)*—Any HPLC capable of pumping the mobile phase at flow rates between 0.1 and 2.0 mL/min. The system should have sufficient sensitivity to obtain a minimum peak height response for a 2 mg/kg impurity twice the height of the signal background noise.

6.2 *Sample Injection System*—Capable of injecting 20 µL of testing solution with a repeatability of no more than 1 %.

6.3 *Detector*—Variable wavelength ultraviolet photometric detector (VWD), multi-wavelength detector, or *photometric diode array detector (PDA)*—capable of operating at 280 nm.

6.4 *Column Oven*—Any suitable HPLC column oven (block heating or air circulating) capable of maintaining a constant temperature within the range of 20 to 70.

6.5 *Chromatography Data System.*

6.6 *HPLC Columns:*

6.6.1 A stainless steel HPLC column packed with an octadecylsilane (C18) chemically bonded silica stationary phase is suitable. See Table 1 for recommended operating conditions.

6.6.2 A C18 column with different dimensions (inner diameter, length, particle size, etc.) that provides adequate resolution to impurities in a bisphenol A sample can also be used.

6.7 *Analytical Balance*—Readable to ±0.0001 g.

6.8 *Sample Filter*—A disposable syringe filter made of cellulose acetate, with a pore size between 0.22 and 0.45 µm, and is chemically inert to aqueous solutions, is recommended for the removal of particulate matter from the sample solution.

7. Reagents and Materials

7.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the reagent grade specification for 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 performance or accuracy of the determination. Reagent chemicals shall be used for all tests.

NOTE 1—Calibration and detection limits of this test method can be biased by the purity of the reagents.

7.2 *Bisphenol A Standard for Calibration*—A certified bisphenol A calibration standard should be 99.9 % purity or high, and must also be sufficiently resolved from any impurity in the samples.

7.3 *Purity of Water*—HPLC grade.

7.4 *Methanol*—HPLC grade.

7.5 *Acetonitrile*—HPLC grade.

7.6 *Pure Compounds for Calibration*—Shall include phenol (CAS No. 108-95-2), indane bisphenol (CAS No. 10527-11-4), 2,4-isopropylidenebisphenol (CAS No. 837-08-1, the isomer of bisphenol A), and chromanone component (I) (CAS No. 472-41-3, 4- (2,2,4-trimethylbenzopyran-4-yl) phenol). The purity of all reagents should be 95.0 % or greater. The concentration and identification of impurities must be known so that the composition of the standard can be adjusted for the presence of the impurities.

7.7 *Internal Standard*—*o*-cresol is one possible internal standard. However, other compounds may be found acceptable provided they meet the criteria as defined in Section 5 and 7.6.

7.8 *Internal Standard Solution*—30 mg/kg. Weigh 0.03 g *o*-cresol in 7.7 and 50 g acetonitrile, to the nearest 0.0001 g, into a 100-mL Erlenmeyer flask, cap the flask and mix well. Weigh 2 g of the above solution and 40 g acetonitrile, to the nearest 0.0001 g, into a 100-mL Erlenmeyer flask, cap the flask and mix well. Calculate the mass fraction of *o*-cresol in the internal standard solution.

7.9 *Mobile Phase:*

7.9.1 Solution A is acetonitrile:methanol = 9:1 (volume ratio), Solution B is water.

7.9.2 *Gradient*—The starting composition of the mobile phase is Solution A:Solution B = 30:70 (volume ratio), followed by a gradual increase in the concentration of Solution A, and the composition of mobile phase eventually reaches Solution A:Solution B = 85:15 (volume ratio) in 30 minutes.

TABLE 1 Recommended Operating Conditions

Column	C18
Stationary phase	Octadecylsilane chemically bonded silica
Particle size	5 µm
Material of column	Stainless steel
Length of column	250 mm
Inner diameter	4.6 mm
Mobile phase and gradient	As shown in 7.9
Flow rate	1 mL/min
UV detector	280 nm
Injection amount	20 µL
Column temperature	40°C

8. Hazards

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

9. Sampling and Test Specimens

9.1 Sample bisphenol A in accordance with Practice D4297.

[4] *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 Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.