Designation: D3160 – 16

# Standard Test Method for Phenol Content of Cumene (Isopropylbenzene) or AMS (α–Methylstyrene)[1]

This standard is issued under the fixed designation D3160; 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 phenol in refined cumene (isopropylbenzene) or AMS (α–methylstyrene).

1.2 This test method has been found applicable in the range from 5 to 50 mg/kg of phenol in refined cumene (isopropylbenzene) or AMS (α–methylstyrene).

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 limitations prior to use.* For specific hazard statements, see Section 7.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D3437 Practice for Sampling and Handling Liquid Cyclic Products

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 1910.000 and 1910, 1200 [3]

## 3. Summary of Test Method

3.1 The phenol content of cumene or AMS is determined by the color development of phenol with 4-aminoantipyrine. The sample absorbance is compared to phenol standards at 472 nm on a spectrophotometer.

## 4. Significance and Use

4.1 This test method is useful in determining phenol in the range from 5 to 50 mg/kg in commercially available cumene or AMS.

4.2 Phenol will inhibit certain reactions involving cumene or AMS.

## 5. Apparatus

5.1 *Balance*—Any balance capable of measuring weights to the nearest 0.001 g.

5.2 *Spectrophotometer*—Any spectrophotometer that can measure 0 to 2 absorbance units at 472 nm with a wavelength repeatability of 5 nm.

5.3 *Spectrophotometer Cells,* 2 cm.

5.4 *Filter Paper.*

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used. 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

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

Current edition approved June 1, 2016. Published June 2016. Originally approved in 1991. Last previous edition approved in 2012 as D3160 – 12. DOI: 10.1520/D3160-16.

[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**

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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D1193. Type I is the highest purity with a maximum conductivity of 0.056 µS/cm. Type II water requires distillation. Type III water has a maximum conductivity of 0.25 µS/cm and there are significant quantities of other impurities. Type IV water has a maximum conductivity of 5 µS/cm and unlimited quantities of TOC and silica.

6.3 *Cumene or AMS*—Wash 1 L of cumene or AMS with 5 % aqueous sodium hydroxide in a 2 L separatory funnel. Discard the aqueous sodium hydroxide phase and filter the hydrocarbon through dry filter paper. Store the hydrocarbon under a nitrogen blanket. The previous steps are taken to ensure that the hydrocarbon will not contain phenol or peroxides.

6.4 *Solution of 4-Amino-Antipyrine*—Dissolve 3.00 g of amino-antipyrine in distilled water and dilute to volume in a dark amber 100-mL volumetric flask. This should be stable for two weeks. Alternate volumes of solutions may be prepared so long as the preparation meets the concentration specified.

6.5 *Ammonium Persulfate Solution*—Dissolve 2.00 g of ammonium persulfate in distilled water and dilute to volume in a 100-mL volumetric flask. A fresh solution should be made up weekly. Alternate volumes of solutions may be prepared so long as the preparation meets the concentration specified.

6.6 *Ammonium Hydroxide,* 0.880 specific gravity.

6.7 *Isopropyl Alcohol,* reagent grade.

6.8 *Sodium Hydroxide,* 5 % weight in distilled water.

## 7. Hazards

7.1 Some materials used in this test method are toxic or flammable, or both.

7.2 If cumene has been exposed to air, cumene hydroperoxide may be in the sample. Exercise suitable precautions for handling cumene that may contain cumene hydroperoxide.

7.3 All glassware and equipment must be clean and free of acid contamination.

7.4 Cumene peroxides will decompose violently when in contact with strong acids.

7.5 Sodium hydroxide is corrosive to the skin and eyes.

7.6 Phenol is corrosive and toxic. Wear rubber gloves and chemical-type safety goggles, as a minimum.

7.7 Isopropyl alcohol is flammable. Keep away from ignition sources.

7.8 Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this test method.

## 8. Sampling

8.1 Sample the material in accordance with Practice D3437.

8.2 Cumene will form peroxides when contacted with air. Sample and store cumene in air-tight containers.

## 9. Preparation and Calibration of Standards

9.1 Transfer approximately 0.1 g of phenol to a 250-mL tared volumetric flask and record the weight. Bring the total net weight to approximately 100 g with phenol-free hydrocarbon as prepared in 6.3. **Warning**—See 7.6. Mix well to dissolve. This is stock Standard Solution A whose phenol concentration should be expressed as mg/kg by weight based on the actual amounts of phenol and phenol-free hydrocarbon used.

9.2 Transfer 0, 1, 2, 3, and 5 mL of Stock Solution A to a 100-mL volumetric flask and dilute to volume with phenol-free hydrocarbon as prepared in 6.3 to yield approximately 0, 10, 20, 30, and 50-mg/kg solutions to be named Solutions B, C, D, E, and F respectively. Calculate the actual mg/kg phenol concentrations based on the weights in 9.1.

9.3 Use the following procedure for Solutions B, C, D, E, and F:

9.3.1 Accurately weigh and record 3.00 g of standard solution in a 25-mL volumetric flask and add 5 mL of distilled water and two drops of ammonium hydroxide. Mix well.

9.3.2 Add 0.5 mL of 4-amino-antipyrine solution followed by 0.5 mL of ammonium persulfate solution. Mix well and let stand for 10 min.

9.3.3 Dilute to volume with isopropyl alcohol and mix well. **Warning**—See 7.7.

9.3.4 Measure the absorbance of this solution at 472 nm using a 2-cm cell against a blank, using Solution B.

9.3.5 Plot a curve of absorbance versus milligram per kilogram phenol (sample size of 3.00 g).

## 10. Procedure

10.1 Use the procedure described in 9.3.1 – 9.3.4 using approximately 3 g of sample instead of a standard solution.

10.2 Obtain the milligram per kilogram phenol in the sample from the curve prepared in 9.3.5.

NOTE 1—This curve assumes a sample size of 3.00 g as described in 9.3.5. The adjusted, actual, mg/kg is calculated as described in Section 11.

## 11. Calculation

11.1 Calculate the phenol concentration in the sample adjusted for the actual sample weight versus the 3.00 standard curve with the following equation:

$$\text{milligram per kilogram phenol} = \frac{C \times 3.00\ g}{W} \quad (1)$$

where:

$C$ = the concentration of phenol for a 3.00 g sample (from the curve in 10.2) and

[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.

$W$ = the sample weight, g.

11.2 To accommodate phenol concentrations greater than 50 mg/kg, adjust the original sample size to obtain a proper absorbance reading on the curve prepared in 9.3.5 and generate the actual phenol concentration using Eq 1 where $W$ will be less than 3 g. Do not further dilute the final solution as this may cause turbidity.

## 12. Report

12.1 Report the concentration of phenol to the nearest mg/kg.

## 13. Precision and Bias[5]

13.1 *Precision*—The following criteria should be used to judge the acceptability of the 95 % probability level of the results obtained by this test method. The criteria were derived from an interlaboratory study between three laboratories. The data were obtained over four days using different operators. The study was conducted at 5, 10, and 20 mg/kg of phenol. The details are given in Research Report RR:D16-1014. It should be noted that the ILS did not meet the minimum requirements of Practice E691. Reproducibility and intermediate precision may change at higher concentrations.

13.1.1 *Intermediate Precision (formerly called Repeatability)*—Results in the same laboratory should not be considered suspect unless they differ by more than the following. Results differing by less than "*r*" have a 95 % probability of being correct:

| Level, mg/kg | Intermediate Precision (r), mg/kg |
|---|---|
| 5 | 0.8 |
| 10 | 1.1 |
| 20 | 2.6 |

13.1.2 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the following. Results differing by less than "*R*" have a 95 % probability of being correct:

| Level, mg/kg | Reproducibility (R), mg/kg |
|---|---|
| 5 | 1.1 |
| 10 | 2.7 |
| 20 | 5.3 |

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.

## 14. Quality Guidelines

14.1 Laboratories shall have a quality control system in place.

14.1.1 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.

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

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

14.1.4 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.

## 15. Keywords

15.1 α–methylstyrene; AMS; cumene; isopropylbenzene; phenol content

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1014. Contact ASTM Customer Service at service@astm.org.

## SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D3160–12) that may impact the use of this standard. (Approved June 1, 2016.)

*(1)* Section 2.1: Added reference to E691.
*(2)* Section 7: Changed MSDS to SDS.
*(3)* Section 13: Tweaked verbiage to align better with Editorial Guidelines.