



Designation: D 3160 – 96a03

Standard Test Method for Phenol Content of Cumene (Isopropylbenzene) or AMS (α -Methylstyrene)¹

This standard is issued under the fixed designation D 3160; 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 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 Jan. 10 and March 10, 1996; 2003. Published May 1996. Originally published as D 3160 – 91; approved in 1991. Last previous edition approved in 1996 as D 3160 – 96a.

1. Scope

1.1 This test method covers the determination of phenol in the range from 0.25 to 50 mg/kg in refined cumene (isopropylbenzene) or AMS (α -methylstyrene).

~~1.2 The following applies to all specified limits in this test method: for purposes of~~

1.2 In determining the conformance with this of the test method, an observed value or a calculated value results using this method to applicable specifications, results 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.

1.3 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:*

D 1193 Specification for Reagent Water²

D 3437 Practice for Sampling and Handling of Liquid Cyclic Products³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

2.2 *Other Document:*

OSHA Regulations, 29 CFR, Paragraphs 1910.000 and 1910, 1200.⁵

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 0.25 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 *Separatory Funnel*, 2 L.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from Superintendent of Documents U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20402.

- 5.5 *Volumetric Flask*, 100 mL.
- 5.6 *Pipettes*, 1, 2, 3, and 5 mL.
- 5.7 *Filter Paper*.⁶

6. Reagents

- 6.1 *Purity of Reagents*—Reagent grade chemicals shall be used.
- 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 D 1193.
- 6.3 *Cumene or AMS*—Wash 1 L of cumene or AMS with 5 % aqueous sodium hydroxide in a separatory funnel. Discard the aqueous sodium hydroxide phase and filter the hydrocarbon through dry filter paper.⁶ 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.
- 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.
- 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' Material 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 D 3437.
- 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 Accurately weigh and transfer 0.100 g of phenol to a 250-mL tared volumetric flask and bring the total net weight to 100.00 g with phenol-free hydrocarbon as prepared in 6.3. **Precaution Warning**—See 7.6. Mix well to dissolve. This is stock Standard Solution A (1000 mg/kg by weight).
- 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 0, 10, 20, 30, and 50-mg/kg solutions to be named Solutions B, C, D, E, and F respectively.
- 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 3.00-g 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 g.

11. Calculation

- 11.1 Calculate the phenol concentration in the sample with the following equation:

⁶ Filter paper, IPS available from Whatman Inc., 9 Bridewell Place, Clifton, NJ 07014 or equivalent, have been found suitable for this purpose.

$$\text{milligram per kilogram phenol} = \frac{C \times 3}{W} \quad (1)$$

where:

C = the concentration of phenol for a 3 g sample (from the curve in 10.2), and

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. Do not further dilute the final solution as this may cause turbidity.

12. Precision and Bias ⁷

12.1 *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. Reproducibility and intermediate precision may change at higher concentrations.

12.1.1 *Intermediate Precision (formerly called Repeatability)*—Results in the same laboratory should not be considered suspect unless they differ by more than the following:

Level, mg/kg	Intermediate Precision, mg/kg
5	0.8
10	1.1
20	2.6

12.1.2 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the following:

Level, mg/kg	Reproducibility, mg/kg
5	1.1
10	2.7
20	5.3

12.2 *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.

13. Keywords

13.1 AMS; cumene; isopropylbenzene; α -methylstyrene; phenol content

⁷ Supporting data are available from ASTM International Headquarters. Request RR: D1 6 – 1014.

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