



Designation: D 4590 – 95a

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Colorimetric Determination of *p*-*tert*-Butylcatechol In Styrene Monomer or AMS (α -Methylstyrene) by Spectrophotometry¹

This standard is issued under the fixed designation D 4590; 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 is applicable to the determination of residual 4-*tert*-butylcatechol (TBC) in styrene monomer or AMS in the 1 to 100 ppm range. Any other compound known to produce color when contacted with aqueous sodium hydroxide solution will interfere. It may be compensated for by including it in the preparation of the standard solutions, if its identity and concentration in the sample are known.

1.2 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, 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.

1.3 *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 statements on hazards, see Section 7.

2. Referenced Documents

- 2.1 *ASTM Standards*:
D 1193 Specification for Reagent Water²
D 3437 Practice for Sampling and Handling 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 1910.1000 and 1910.1200⁵

3. Summary of Method

3.1 Color is developed in the specimen by the addition of caustic in a methanol-octanol solvent. The pink color intensity is measured with a spectrophotometer and compared to a calibration curve for quantitation.

4. Significance and Use

4.1 This test method is suitable for determining the quantity of TBC inhibitor, both for the protection against polymerization while in transit and storage, and for internal quality control.

5. Apparatus

5.1 *Visible Range Spectrophotometer*, equipped with absorption cells providing light paths from 1 to 5 cm for use at approximately 490 nm.

5.2 *Volumetric Pipets*, 5 mL.

5.3 *Pipetors*, 0.1 and 0.2 mL.

6. Reagents and Materials

6.1 *Purity of Reagents*—Where unspecified, chemicals shall be reagent grade and conform to recognized specifications if such exist.⁶ If found to have no adverse effect on accuracy, other grades may be used.

6.2 *4-Tertiary-Butylcatechol*, Mp 52-55°C.

6.3 *Toluene*, ACS reagent grade.

6.4 *Methanol*, reagent grade.

6.5 *n-Octanol*, reagent grade.

6.6 *Sodium Hydroxide Pellets*, reagent grade.

6.7 *Alcoholic Sodium Hydroxide*, approximately 0.15 N. Dissolve 0.3 g NaOH in 25 mL methanol. Add 25 mL of *n*-octanol and 100 μ L of water. Let this reagent mature for two days before use. This solution is stable for two weeks.

6.8 *TBC Stock Standard*—Weigh 0.5 g of TBC into 500 g of toluene. This solution will contain 1000 ppm TBC. This standard should have a shelf life of one year or better if stored in a refrigerator or freezer. Storing standards in amber bottles

¹ This test method is under the jurisdiction of ASTM Committee D-16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.0H on Styrene, Ethylbenzene, and C₉ and C₁₀ Aromatic Hydrocarbons.

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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 the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

⁶ *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.