



Designation: **D4590 – 13 D4590 – 18**

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 D4590; 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-tertiary-butylcatechol (TBC) in styrene monomer or AMS in the 1 to 100 mg/kg range. Any other compound producing color at 490 nm 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 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. The limit of detection is 0.2 mg/kg while the limit of quantitation is 0.8 mg/kg based on the ILS data in Table 1.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific statements on hazards, see Section 8.

1.5 *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

D1193 Specification for Reagent Water

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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 1910.1000 and 1910.1200³

3. Terminology

3.1 See Terminology D4790 for definition of terms used in this standard.

4. Summary of Method

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

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

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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, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

*A Summary of Changes section appears at the end of this standard

TABLE 1 Intermediate Precision and Reproducibility

TBC Observed Concentration	Repeatability	Reproducibility
4.7	0.23	0.94
15.2	0.47	5.08
24.5	1.55	4.91
94.6	3.98	9.94

5. Significance and Use

5.1 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.

6. Apparatus

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

6.2 *Volumetric Pipets and Pipetors*—The sample pipet volume should be sized to fill the spectrometer absorption cell. Pipetor volumes must be scaled to the sample volume. This procedure is written using a 15 mL sample volume. The following table shows how reagent pipetor volumes could be scaled to use a 5 mL sample volume.

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