Designation: D6144 – 22

Standard Test Method for
Analysis of AMS (α-Methylstyrene) by Capillary Gas Chromatography[1]

This standard is issued under the fixed designation D6144; 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 the purity of AMS (α-methylstyrene) by gas chromatography. Calibration of the gas chromatography system is done by the external standard calibration technique.

1.2 This test method has been found applicable to the measurement of impurities such as cumene, 3-methyl-2-cyclopentene-1-one, *n*-propylbenzene, *tert*-butylbenzene, *sec*-butylbenzene, *cis*-2-phenyl-2-butene, acetophenone, 1-phenyl-1-butene, 2-phenyl-2-propanol, *trans*-2-phenyl-2-butene, *m*-cymene, *p*-cymene, and phenol, which are common to the manufacturing process of AMS. The method has also been found applicable for the determination of para-tertiary-butylcatechol (TBC or PTBC) typically added as a stabilizer to AMS. The impurities in AMS can be analyzed over a range of 5 to 800 mg/kg by this method. (See Table 2.) Based on the results in ASTM Research Report RR:D16-1022, summarized in Table 2, the limit of quantitation for these impurities averages 4 mg/kg, while the limit of detection averages 1.2 mg/kg.

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 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 hazard statements, 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]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

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

E355 Practice for Gas Chromatography Terms and Relationships

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

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

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[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 D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

Current edition approved Jan. 1, 2022. Published January 2022. Originally approved in 1997. Last previous edition approved in 2017 as D6144 – 17. DOI: 10.1520/D6144-22.

[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**

4. Summary of Test Method

4.1 AMS (α-methylstyrene) is analyzed by a gas chromatograph (GC) equipped with a flame ionization detector (FID). A precisely repeatable volume of the sample to be analyzed is injected onto the gas chromatograph. The peak areas of the impurities are measured and converted to concentrations via an external standard methodology. Purity by GC (the AMS content) is calculated by subtracting the sum of the impurities from 100.00. Purity results are reported in mass percent.

5. Significance and Use

5.1 This test method is suitable for setting specifications on the materials referenced in 1.2 and for use as an internal quality control tool where AMS is produced or is used in a manufacturing process. It may also be used in development or research work involving AMS.

5.2 This test method is useful in determining the purity of AMS with normal impurities present. If extremely high boiling or unusual impurities are present in the AMS, this test method would not necessarily detect them and the purity calculation would be erroneous.

6. Apparatus

6.1 *Gas Chromatograph*—Any instrument having a flame ionization detector that can be operated at the conditions given in Table 1. The system should have sufficient sensitivity to obtain a minimum peak height response for 10 mg⁄kg acetophenone of twice the height of the signal background noise.

6.2 *Columns*—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from AMS. The column described in Table 1 has been used successfully and shall be used as a referee in cases of dispute.

6.3 *Recorder*—Chromatographic data systems are preferred but electronic integration may be used if the user can demonstrate that the results are consistent with the precision statement. Recorders are not considered adequate for meeting the precision requirements of this standard.

TABLE 1 Recommended Operating Conditions

Detector	flame ionization
Injection Port	capillary splitter
Column A:	
Tubing	fused silica
Stationary phase	crosslinked methylsilicone
Film thickness, ÷µm	1.0
Length, m	60
Diameter, mm	0.32
Temperatures:	
Injector, °C	250
Detector, °C	300
Oven, °C	85 hold for 13 min Ramp 1 = 6 °C⁄min to 125 °C, hold for 2 min Ramp 2 = 30 °C⁄min to 250 °C, hold for 7.5 min
Carrier gas	helium
Flow rate, mL/min	3
Split ratio	60:1
Sample size, ÷µL	1.0

6.4 *Injector*—The specimen must be precisely and repeatably injected into the gas chromatograph. An automatic sample injection device is highly recommended. Manual injection can be employed if the precision stated in Table 2 can be reliably and consistently satisfied.

7. Reagents and Materials

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

7.2 *Carrier Gas, Makeup, and Detector Gases*—Helium, hydrogen, nitrogen, or other carrier, makeup and detector gases 99.999 % minimum purity. Oxygen in carrier gas less than 1 ppm, less than 0.5 ppm is preferred. Purify carrier, makeup, and detector gases to remove oxygen, water, and hydrocarbons. **Warning—**Helium carrier, makeup, and detector gas was used to develop this standard. Use of other gases requires different conditions. The user must conduct the necessary evaluation to determine that equivalent results are obtained.

7.3 *Compressed Air*—Purify air to remove water and hydrocarbons. Air for a FID should contain less than 0.1 ppm THC (total hydrocarbon content).

7.4 *Equipment Setup Check Sample:*

7.4.1 For GC standards, a setup check sample should be included to:

7.4.1.1 Determine retention times for the components measured in GC standards,

7.4.1.2 Verify there is adequate resolution to measure the components of interest in GC standards, and

7.4.1.3 Determine that the equipment has the sensitivity specified in the scope of the standard.

7.4.2 For GC standards and standards that determine trace levels, the equipment setup check sample should contain a component with a concentration that is approximately two times the LOD stated in the scope of the standard. When the equipment setup check sample is analyzed, an acceptable result for the trace component is ±50 % of the expected concentration.

7.4.2.1 For GC standards where the primary material cannot be purified so that no impurities are detected, the following is suggested:

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[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.

(1) Add an impurity that is not present in the primary material. Determine that the impurity has the following properties:

(a) The impurity is essentially inert and unreactive in the primary material;

(b) The retention time is sufficiently separated from other impurities so that there will be no mistake in identification;

(c) The impurity is completely vaporized in the injection port;

(d) The impurity is well behaved on the column, that is, no fronting or tailing;

(e) The response factor is known and not significantly different from the components of interest; and

(f) A straight chain hydrocarbon will work for most materials. Undecane has been used as the internal standard to determine the purity of *p*-xylene.

7.5 *Pure Compounds for Calibration*—The purity of all reagents should be 99.9 % or greater. If the purity is less than 99 %, 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.

8. Hazards

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

9. Sampling and Handling

9.1 Sample the material in accordance with Practice D3437.

10. Preparation of Apparatus

10.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1 allowing sufficient time for the equipment to reach equilibrium. See Practice E1510 for more information on column installation. See Practice E355 for additional information on gas chromatography practices and terminology.

11. Calibration

11.1 Prepare a synthetic mixture of high purity AMS containing impurities at concentrations representative of those expected in the samples to be analyzed in accordance with Practice D4307. The weight of each hydrocarbon impurity must be measured to the nearest 0.1 mg. Because the availability of stock AMS with a purity higher than 99.80 % is problematic, the method of standard additions may be required for impurities such as *tert*-butylbenzene and *n*-propylbenzene, as well as for a number of the other impurities listed in 1.2 that are commonly present. In addition, nearly all commercially available AMS is stabilized with 10 to 30 mg/kg of para-tertiary-butylcatechol (commonly abbreviated PTBC or TBC), requiring a standard addition methodology for this component as well unless AMS can be obtained safely and directly from the point of manufacture.

11.2 Inject the resulting solution from 11.1 into the gas chromatograph, collect and process the data. A typical chromatogram is illustrated in Fig. 1 based on the conditions listed in Table 1.

11.3 Determine the response factor for each impurity in the calibration mixture as follows:

$$Rfi = Ci/Ai \tag{1}$$

where:

Rfi = response factor for impurity i,
Ci = concentration of impurity i in the calibration mixture, and
Ai = peak area of impurity i.

11.4 Initially analyze the calibration solution a minimum of three times and calculate an average Rfi. Subsequent calibrations may be a single analysis as long as the Rfis for all components of interest are within ±5 % of the initial validation Rfis. A "rolling" average as defined by most modern chromatographic software may also be used. The Rfi for *tert*-butylbenzene is used for the quantification of unknown impurities.

12. Procedure

12.1 Inject into the gas chromatograph an appropriate amount of sample sufficient to satisfy the sensitivity conditions detailed in 6.1 and start the analysis.

12.2 Obtain a chromatogram and peak integration report. Fig. 1 illustrates a typical analysis of AMS using the column and conditions outlined in Table 1.

13. Calculations

13.1 Of the impurities identified in AMS, only the butenylbenzene isomers are not available commercially. However, pure samples of these isomers can be prepared, and testing has shown that all three isomers have the same response factor ±5 %, and that it is equivalent to that for *tert*-butylbenzene ±5 %.[5]

13.2 Calculate the concentration of each impurity as follows:

$$C_i = (Ai)\,(Rfi) \tag{2}$$

where:

C_i = concentration of component i, in mg/kg,
Ai = peak area of component i, and
Rfi = response factor for component i.

13.3 Calculate the total concentration of all impurities in mass % as follows:

$$C_t = \Sigma C_i/10000 \tag{3}$$

where:

C_t = total concentration of all impurities.

13.4 Calculate the purity of AMS as follows:

$$\text{AMS, mass percent} = 100.00 - C_t \tag{4}$$

14. Report

14.1 Report the individual impurities to the nearest 0.1 mg/kg.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1022. Contact ASTM Customer Service at service@astm.org.

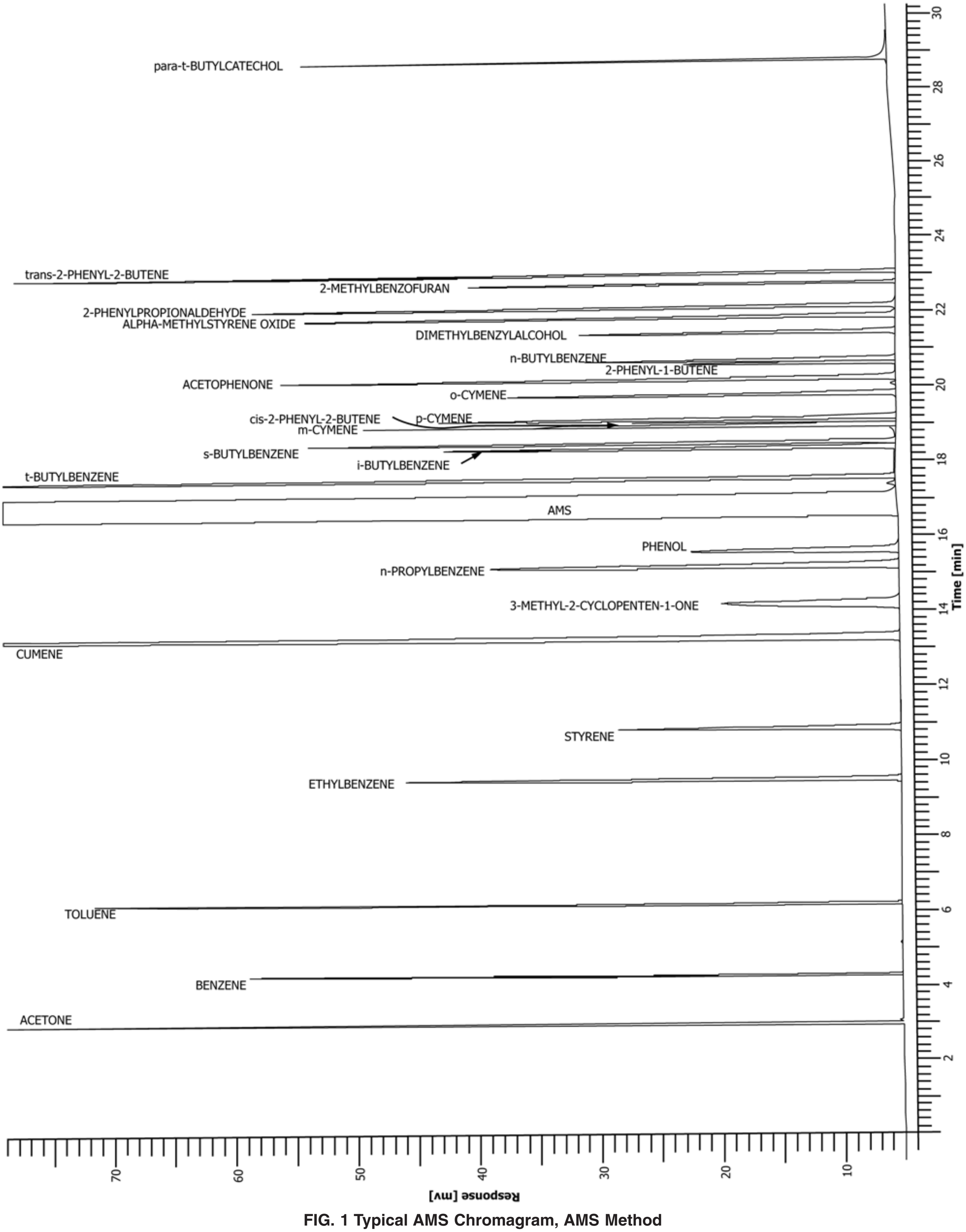

FIG. 1 Typical AMS Chromagram, AMS Method

14.2 Report numbers below the LOD as <1 mg/kg.

14.3 Report the purity of AMS to the nearest 0.01 mass %.

15. Precision and Bias[5]

15.1 The following criteria should be used to judge the acceptability of results obtained by this test method (95 % confidence level). The precision criteria were derived from an ILS that was conducted using the conditions listed in Table 2 which included five laboratories analyzing six samples in triplicate by the same operator on the same day. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1022.

15.1.1 Helium carrier gas was used to develop this standard. In cases of dispute, the carrier gas used to develop this standard must be used.

15.2 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ by more than the amounts calculated from the appropriate equations in Table 2. On the basis of test error alone, the difference between two results obtained in the same laboratory on the same material will be expected to exceed this value only 5 % of the time

15.3 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the amounts calculated from the appropriate equations in Table 2. On the basis of test error alone, the difference between two results obtained in different laboratories on the same material will be expected to exceed this value only 5 % of the time.

15.4 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method for measuring these impurities, bias has not been determined.

16. Quality Guidelines

16.1 Laboratories shall have a quality control system in place.

16.1.1 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.

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

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

16.1.4 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.

16.2 *Interlaboratory Testing:*

16.2.1 A program that includes multiple laboratories analyzing the same samples is strongly encouraged. This program should allow labs to compare their results with other laboratories. This is particularly important when a plant is selling the product to customers or the laboratory is analyzing the product for acceptance. Producers and customers need to have confidence that results from different producers are comparable. ASTM currently has a Proficiency Testing Program that sends a sample of mixed xylenes, *p*-xylene, and benzene to multiple labs twice a year for testing. Other programs are acceptable.

17. Keywords

17.1 acetophenone; alpha methylstyrene; AMS; analysis by gas chromatography; benzene; butenylbenzene; butylbenzene; catechol; cumene; cymene; phenol; propylbenzene; toluene

TABLE 2 Summary of Precision Data (mg/kg)

Compound	Repeatability (*r*)	Reproducibility (*R*)	Range Studied
Acetone	1.61 + 0.035*Acetone	0.47 + 1.10*Acetone	0.5 – 26
Cumene	–0.46 + 0.031*Cumene	7.88 + 0.19*Cumene	45 – 290
n-Propylbenzene (NPB)	2.11 + 0.03*NPB	–7.81 + 0.37*NPB	55 – 195
Phenol	1.84	3.65 + 0.58*Phenol	1 – 40
tert-Butylbenzene (TBB)	–1.22 + 0.035*TBB	3.63 + 0.087*TBB	150 – 650
sec-Butylbenzene (SBB)	4.23 + 0.019*SBB	21.60 + 0.25*SBB	200 – 765
m-Cymene	0.31 + 0.035*mCymene	2.34 + 0.35*mCymene	2 – 50
o-Cymene	1.63	8.00	29 – 31
p-Cymene	5.12	15.79	10 – 18
cis-2-Phenyl-2-Butene (CPB)	0.17 + 0.030*CPB	5.39 + 0.11*CPB	50 – 225
trans-2-Phenyl-2-Butene (TPB)	1.57	0.54 + 0.20*TPB	19 – 70
1-Phenyl-1-Butene (PB)	4.00 + 0.018*PB	0.17 + 0.19*PB	0.2 – 735
Acetophenone (AP)	–1.09 + 0.15*AP	1.17 + 0.63*AP	15 – 115
para-tert-Butylcatechol (PTBC)	2.21	17.82	10 – 19
2-Methylbenzofuran (MBF)	0.33 + 0.76*MBF	0.75 + 0.60*MBF	1 – 2
2-Phenylpropion aldehyde (PPA)	0.59 + 0.11*PPA	0.29 + 0.23*PPA	1.5 – 15
α-Methylstyrene Oxide (AMSO)	4.61	6.51 + 0.23*AMSO	13 – 32
DimethylBenzyl Alcohol (DMBA)	0.38	0.55 + 2.03*DMBA	0.1 – 1

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D6144 – 17) that may impact the use of this standard. (Approved January 1, 2022.)

(1) Tweaks to the verbiage in Sections 1, 4, 7, 14, and 15 based on current editorial guidelines.

(2) Tables 1 and 2 renumbered and moved.

(3) Addition of 16.2 to the Quality Guidelines.

Designation: D1209 − 05 (Reapproved 2019)

Standard Test Method for
Color of Clear Liquids (Platinum-Cobalt Scale)[1]

This standard is issued under the fixed designation D1209; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method describes a procedure for the visual measurement of the color of essentially light colored liquids (Note 1). It is applicable only to materials in which the color-producing bodies present have light absorption characteristics nearly identical with those of the platinum-cobalt color standards used.

NOTE 1—A procedure for estimating color of darker liquids, described for soluble nitrocellulose base solutions, is given in Guide D365.

1.2 For purposes of determining conformance of an observed or a calculated value using this test method to relevant specifications, test result(s) 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 E29.

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 For specific hazard information, see the Material Safety Data Sheet.

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 specific hazard statements see Section 6.

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]

D156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
D365 Guide for Soluble Nitrocellulose Base Solutions
D1193 Specification for Reagent Water
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols
E346 Test Methods for Analysis of Methanol (Withdrawn 2017)[3]

3. Significance and Use

3.1 The property of color of a solvent varies in importance with the application for which it is intended, the amount of color that can be tolerated being dependent on the color characteristics of the material in which it is used. The paint, varnish, and lacquer solvents, or diluents commercially available on today's market normally have little or no color. The presence or absence of color in such material is an indication of the degree of refinement to which the solvent has been subjected or of the cleanliness of the shipping or storage container in which it is handled, or both.

3.2 For a number of years the term "water-white" was considered sufficient as a measurement of solvent color. Several expressions for defining "water-white" gradually appeared and it became evident that a more precise color standard was needed. This was accomplished in 1952 with the adoption of Test Method D1209 using the platinum-cobalt scale. This test method is similar to the description given in *Standard Methods for the Examination of Water and Waste Water*[4] and is referred to by many as "APHA Color." The preparation of these platinum-cobalt color standards was originally described by A.

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved April 1, 2019. Published April 2019. Originally approved in 1952. Last previous edition approved in 2011 as D1209 – 05 (2011). DOI: 10.1520/D1209-05R19.

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] *Standard Methods for the Examination of Water and Waste Water*, M. Franson, Ed., American Public Health Assoc., 14th ed., 1975, p. 65.

TABLE 1 Absorbance Tolerance Limits For No. 500 Platinum-Cobalt Stock Solution

Wavelength, nm	Absorbance
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

TABLE 2 Platinum-Cobalt Color Standards

Color Standard Number	Stock Solution, mL	Color Standard Number	Stock Solution, mL
5	1	70	14
10	2	100	20
15	3	150	30
20	4	200	40
25	5	250	50
30	6	300	60
35	7	350	70
40	8	400	80
50	10	450	90
60	12	500	100[A]

[A] This is platinum-cobalt color No. 10 in Guide D365.

TABLE 3 Platinum-Cobalt Color Standards for Very Light Colors

Color Standard Number	Stock Solution, mL	Color Standard Number	Stock Solution, mL
1	0.20	9	1.80
2	0.40	10	2.00
3	0.60	11	2.20
4	0.80	12	2.40
5	1.00	13	2.60
6	1.20	14	2.80
7	1.40	15	3.00
8	1.60		

Hazen in the *American Chemical Journal*[5] in which he assigned the number 5 (parts per ten thousand) to his platinum-cobalt stock solution. Subsequently, in their first edition (1905) of Standard Methods for the Examination of Water, the American Public Health Association, using exactly the same concentration of reagents, assigned the color designation 500 (parts per million) which is the same ratio. The parts per million nomenclature is not used since color is not referred directly to a weight relationship. It is therefore recommended that the incorrect term "Hazen Color" should not be used. Also, because it refers primarily to water, the term "APHA Color" is undesirable. The recommended nomenclature for referring to the color of organic liquids is "Platinum-Cobalt Color, Test Method D1209."

3.3 The petroleum industry uses the Saybolt colorimeter Test Method D156 for measuring and defining the color of hydrocarbon solvents; however, this system of color measurement is not commonly employed outside of the petroleum industry. It has been reported by various sources that a Saybolt color of +25 is equivalent to 25 in the platinum-cobalt system or to colors produced by masses of potassium dichromate ranging between 4.8 and 5.6 mg dissolved in 1 L of distilled water. Because of the differences in the spectral characteristics of the several color systems being compared and the subjective manner in which the measurements are made, exact equivalencies are difficult to obtain.

4. Apparatus

4.1 *Spectrophotometer,* equipped for liquid samples and for measurements in the visible region.

NOTE 2—The spectrophotometer used must be clean and in first-class operating condition. The instrument should be calibrated in accordance with the instructions given in the Standards for Checking the Calibration of Spectrophotometers (200 to 1000 nm).[6]

4.2 *Spectrophotometer Cells,* matched having a 10-mm light path.

4.3 *Color Comparison Tubes*—Matched 100-mL, tall-form Nessler tubes, provided with ground-on, optically clear, glass caps. Tubes should be selected so that the height of the 100-mL graduation mark is 275 to 295 mm above the bottom of the tube.

4.4 *Color Comparator*—A color comparator constructed to permit visual comparison of light transmitted through tall-form, 100-mL Nessler tubes in the direction of their longitudinal axes. The comparator should be constructed so that white light is passed through or reflected off a white glass plate and directed with equal intensity through the tubes, and should be shielded so that no light enters the tubes from the side.[7]

5. Reagents

5.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. 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 such specifications are available.[8] 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

5.3 *Cobalt Chloride* ($CoCl_2 \cdot 6H_2O$).

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.5 *Potassium Chloroplatinate* (K_2PtCl_6).

6. Platinum-Cobalt Reference Standards

6.1 *Platinum-Cobalt Stock Solution*—Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6) and 1.00 g of cobalt

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[5] Hazen, A., "New Color Standard for Natural Waters," *American Chemical Journal*, Vol XIV, 1892, p. 300–310.

[6] See NIST Letter Circular LC-1017.

[7] The sole source of supply of the unit known to the committee at this time is Scientific Glass and Instruments, Inc., P.O. Box 6, Houston, TX 77001. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[8] *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.

chloride ($CoCl_2 \cdot 6H_2O$) in water. Carefully add 100 mL of hydrochloric acid (HCl, sp gr 1.19) and dilute to 1 L with water. The absorbance of the 500 platinum-cobalt stock solution in a cell having a 10-mm light path, with reagent water in a matched cell as the reference solution,[9] must fall within the limits given in Table 1.

NOTE 3—This stock solution is commercially available from reputable chemical suppliers.

6.2 *Platinum-Cobalt Standards*—From the stock solution, prepare color standards in accordance with Table 2 by diluting the required volumes to 100 mL with water in the Nessler tubes. Cap the tubes and seal the caps with shellac or a waterproof cement. When properly sealed and stored, these standards are stable for at least one year and do not degrade markedly for two years.[10]

6.2.1 For a more precise measurement of light colors below 15 platinum-cobalt, prepare color standards from the stock solution in accordance with Table 3 by diluting the required volumes to 100 mL with water in the Nessler tubes. Use a semi-microburet for measuring the required amount of stock solution.

7. Procedure

7.1 Introduce 100 mL of specimen into a Nessler tube, passing the specimen through a filter if it has any visible turbidity. Cap the tube, place in the comparator, and compare with the standards.

8. Report

8.1 Report as the color the number of the standard that most nearly matches the specimen. In the event that the color lies midway between two standards, report the darker of the two.

8.2 If, owing to differences in hue between the specimen and the standards, a definite match cannot be obtained, report the range over which an apparent match is obtained, and report the material as "off-hue."

9. Precision

9.1 *Color Samples With Pt-Co Color Greater Than 25:*[11]

9.1.1 These precision statements are based upon an interlaboratory study in which six platinum-cobalt standards having values of 25, 75, 170, 265, 385, and 475 were prepared in accordance with the instructions given in Section 6 of this test method and were given coded labels. These solutions were tested by one analyst in each of ten different laboratories making a single observation on one day and then repeating the observation on a second day. The analysts were requested to estimate the color to the nearest one unit for solutions below 40 platinum-cobalt, to the nearest five units for solutions between 40 and 100 platinum-cobalt and to the nearest ten units for solutions above 100 platinum-cobalt. Based on the results of this interlaboratory study, the following criteria, calculated according to RR:D02-1007, should be used for judging the acceptability of results at the 95 % confidence level when the results are obtained under optimum conditions where the hue of the sample matches exactly the hue of the standards. Poor precision will be obtained in varying degrees as the hue of the sample departs from that of the standards.

9.1.1.1 *Repeatability*—Two results obtained by the same analyst should be considered suspect if they differ by more than:

$$r = 0.027\ (X + 92)\ \text{platinum-cobalt units}$$

where X is the average of the two results.

9.1.1.2 *Reproducibility*—Two results, obtained by analysts in different laboratories, should be considered suspect if they differ by more than:

$$R = 0.087\ (X + 92)\ \text{platinum-cobalt units}$$

where X is the average of the two results.

9.1.1.3 Table 4 shows precision values for samples with Pt-Co Color of greater than 25.

TABLE 4 Precision Values for Greater than 25 Pt-Co Color

Color (Pt-Co units)	Repeatability, *r*	Reproducibility, *R*
25	3	10
75	5	15
165	7	22
265	10	31
385	13	41
475	16	49

9.2 *Color Samples With Pt-Co Color Less Than 25:*[12]

9.2.1 The results of two interlaboratory studies were pooled to give precision values calculated according to RR:D02-1007. One study of glycols included four samples and seven laboratories; the other study included five samples and six laboratories. Based on the pooled repeatability and reproducibility standard deviations, the following criteria should be used for judging, at the 95 % confidence level, the acceptability of results obtained on samples with less than 25 Pt-Co Color.

9.2.1.1 *Reapeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than two platinum-colbalt units.

9.2.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories, should be considered suspect of they differ by more than seven platinum-cobalt units.

9.3 *Bias*—The test procedure has no bias because the value of the test result is defined only in terms of the test method.

10. Keywords

10.1 clear liquids; color; platinum-cobalt color scale

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[9] See the manufacturer's instruction manual for complete details for operating the spectrophotometer.

[10] Scharf, W. W., Ferber, K. H., and White, R. G., "Stability of Platinum-Cobalt Color Standards," *Materials Research and Standards*, Vol 6, No. 6, June 1966, pp. 302–304.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D01-1024 and RR:D02-1007. Contact ASTM Customer Service at service@astm.org.

[12] These precision statements are based on interlaboratory studies conducted by Committee E15 on Industrial Chemicals on samples of ethylene glycol and methanol as reported in Test Methods E202, E346, and Research Reports RR:E15-0028 and RR:D01-1108. Research reports are available from ASTM International.

Designation: D4590 – 18

Standard Test Method for
Colorimetric Determination of *p-tert*-Butylcatechol In Styrene Monomer or AMS (α–Methylstyrene) by Spectrophotometry[1]

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 1910.1200 [3]

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.

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.

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

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[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 D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

Current edition approved June 1, 2018. Published June 2018. Originally approved in 1986. Last previous edition approved in 2013 as D4590 – 13. DOI: 10.1520/D4590-18.

[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.

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

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.

Sample Pipet, mL	0.15 NaOH Pipetor, µL	Methanol Pipetor, µL
15	300	600
5	100	200

7. Reagents and Materials

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

7.2 *4-Tertiary-Butylcatechol,* Mp 52 to 55°C.

7.3 *Toluene,* ACS reagent grade.

7.4 *Methanol,* reagent grade.

7.5 *n-Octanol,* reagent grade.

7.6 *Aqueous Sodium Hydroxide, 10 Normal (10N),* reagent grade.

7.7 *Alcoholic Sodium Hydroxide,* approximately 0.15 N: Mix 0.75 mL of 10N Aqueous Sodium Hydroxide with 25 mL methanol. With stirring, add 25 mL of *n*-octanol and then 0.75 mL of water. Store the reagent in an amber glass bottle. This reagent can be used immediately after preparation and is stable for at least 2 months. To reduce exposure of the reagent to the atmosphere, transfer enough for several samples to a small clean vial.

7.8 *TBC Stock Standard*—This standard may be purchased if desired. Prepare a stock solution by weighing 0.500 ± 0.001 g of TBC to the nearest 0.0001 g into 500 ± 1 g of toluene (weighed to the nearest 0.1 g). This solution will have a concentration of approximately 1000 mg/kg TBC in toluene and the exact concentration can be calculated using Eq 1. This standard should have a shelf life of one year or better if stored in a refrigerator or freezer. Storing standards in amber bottles significantly reduces degradation of standards with time.

$$mg\ TBC/kg\ toluene = \left[(g\ of\ TBC) \times (Purity\ of\ TBC) \times (10^6)\right]/g\ of\ toluene \quad (1)$$

7.9 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean any reagent conforming to type I as defined in Specification D1193.

8. Hazards

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

8.2 *Handling Precautions—p-tert*-butylcatechol, particularly when molten or in concentrated solution, is very corrosive to the skin. It is also a systemic poison when taken orally or absorbed in quantity through the skin.

8.3 *Flammable Hazards*—Styrene monomer is flammable and can polymerize exothermally under a variety of conditions, most especially in the presence of peroxides, mineral acids, and Lewis acids such as aluminum chloride.

9. Sampling and Handling

9.1 Sample the material in accordance with Practice D3437.

10. Preparation of Calibration Curve

10.1 Prepare standards of approximately 5, 10, 20, 30, 40, 50, 70, and 100 mg/kg TBC in toluene by diluting 0.5, 1, 2, 3, 4, 5, 7, and 10 mL of TBC stock standard to 100 mL with toluene. Record the actual concentrations generated to the nearest 0.5 mg/kg.

NOTE 1—Both the stock solution and toluene diluent should be at room temperature. Temperature differences will cause dilution errors due to variations in density.

10.2 Determine the absorbance of each standard solution and one reagent blank in accordance with Section 11.

10.3 Plot absorbance versus concentration on standard graph paper.

NOTE 2—The plot should be close to a straight line. The maximum absorbance should be within the absorbance range of the spectrometer. If not, repeat the calibration with a spectrometer cell that has less path length.

11. Procedure

11.1 Zero the spectrometer with the specimen to be analyzed.

11.2 Add 15 mL of specimen to a clean round container.

11.3 Verify that the alcoholic NaOH reagent and pipetor are free of particulates.

NOTE 3—The particulates formed in the sodium hydroxide reagent from exposure to carbon dioxide in the atmosphere will scatter light and may cause an error in the determination.

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[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.

11.4 Add 300 µL of alcoholic NaOH reagent to the container and mix vigorously with a vortex mixer for 30 s.

NOTE 4—If a vortex mixer is not available, continuous vigorous shaking for 30 s is required. Rapid reaction completion depends on the formulation of an emulsion of aqueous NaOH in the sample mixture.

11.5 Add 600 µL of methanol to the container and shake for about 15 s, creating a clear solution from the reaction emulsion.

11.6 Measure the absorbance at 490 nm as soon as possible and within 5 min.

11.7 Read the concentration in mg/kg TBC from the graph. Calculate the inhibitor content using Eq 2 for density correction:

$$\text{Inhibitor Content, mg/kg} = (\text{Value from curve}) \times [(\text{toluene density})/(\text{sample density})] \quad (2)$$

12. Report

12.1 Report the inhibitor content as mg/kg (or ppm) of *p-tert*-butylcatechol to the nearest 0.1 mg/kg..

12.2 Results less than the LOD should be reported as <0.2 mg/kg.

13. Precision and Bias[5]

13.1 The following criteria should be used to judge the acceptability of results obtained by this test method (95 % confidence level). The precision criteria were derived from an ILS that was conducted using six laboratories analyzing three sample of unknown concentration of TBC with two determinations for each sample. Each determination was the result of one measurement as specified in this standard. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1009.

13.2 *Intermediate Precision (formerly called Repeatability)*—Results in the same laboratory should not be considered suspect unless they differ by more than the values shown in Table 1. Results differing by less than "*r*" have a 95 % probability of being correct.

13.3 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the values shown in Table 1. Results differing by less than "*R*" have a 95 % probability of being correct.

13.4 *Bias*—There is a bias associated with running the test method that is statistically significant at the 95 % confidence level. The bias for the unknown samples is an average absolute value of 0.6 mg/kg with the observed results lower than the actual amounts added. However, at the 99 % confidence level, the bias is not statistically significant.

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 for 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 alpha-methylstyrene; AMS; para-tertiary butyl catechol; PTBC; styrene, inhibitor content; styrene, TBC content; TBC in styrene

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D4590–13) that may impact the use of this standard. (Approved June 1, 2018.)

(1) Minor changes to sections 1, 8, and 12.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1009. Contact ASTM Customer Service at service@astm.org.

Designation: D2121 – 16

Standard Test Methods for Polymer Content of Styrene Monomer[1]

This standard is issued under the fixed designation D2121; 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 These test methods cover the determination of the polymer content of styrene monomer. It should be noted, however, that dimers and trimers are not measured by these test methods.

1.2 *Test Method A,* which is based on the use of a spectrophotometer or photometer, is intended for the quantitative determination of the polymer content of styrene monomer in concentrations up to 15 mg/kg. Samples containing more than 15 mg/kg of polymer must be suitably diluted before measurement.

1.3 *Test Method B* is a rapid visual procedure that is intended for the approximate evaluation of polymer to a maximum concentration of 1.0 mass %. Samples having a polymer content of 1.0 mass % or greater should be suitably diluted prior to measurement.

1.4 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.

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

1.6 *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 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2827 Specification for Styrene Monomer
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, 29CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

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

TEST METHOD A—DETERMINATION OF POLYMER IN STYRENE MONOMER PHOTOMETER METHOD

4. Summary of Test Method

4.1 This test method utilizes the fact that polymers present in the monomers are insoluble in methanol. The polymer content of styrene monomer is determined by measurement of the degree of turbidity produced by the addition of dry methanol to the styrene sample.

5. Significance and Use

5.1 This test method can be used for determining polymer concentrations in styrene monomer.

5.2 This test method will not detect dimers and trimers.

5.3 This test method can be used for plant control and for specification analysis.

6. Interferences

6.1 Small changes in turbidity may occur with time. It is, therefore, important that the absorbance of calibration mixtures and samples be determined after standing the same length of time.

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

Current edition approved June 1, 2016. Published July 2016. Originally approved in 1962. Last previous edition approved in 2015 as D2121 – 15. DOI: 10.1520/D2121-16.

[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**

6.2 Hexane is used for two reasons:

6.2.1 To block out any color in the styrene, and

6.2.2 To indicate dissolved water in the styrene.

7. Apparatus

7.1 *Pipets,* 10 and 15-mL.

7.2 *Bottles or Flasks,* of suitable size equipped with glass stoppers.

7.3 *Spectrophotometer or Photometer Cells,* with 50 to 150-mm light path.

7.4 *Spectrophotometer or Photometer,* capable of absorbance measurements in wavelength region of 420 nm and sensitive to 1 mg polymer/kg monomer.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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 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.

8.2 *Hexane,* dry.

8.3 *Methanol,* dry.

8.4 *Polystyrene:*

8.4.1 Prepare polystyrene as follows: wash 50 mL of styrene monomer twice with equal volumes of 1 *N* NaOH solution and twice with equal volumes of water. After the second water wash, filter the styrene through two layers of rapid filtering ready folded filter paper. Pour about 20 mL of this styrene monomer into a test tube and heat in an oven at 100°C for 24 h to promote polymerization. At the end of this time, remove the polystyrene from the test tube by breaking the tube and discarding all glass. Grind the polymer plug to a fine powder in an agate mortar.

8.4.2 Commercially available high-purity polystyrene pellets can be used; however, high-molecular weight polystyrene (>150 000 molecular weight) should be specified.

8.5 *Styrene Monomer,* conforming to Specification D2827.

8.6 *Toluene,* dry.

9. Hazards

9.1 Styrene monomer is flammable and polymerizes exothermically on contact with peroxides, mineral acids and aluminum chloride.

9.2 Styrene monomer both in liquid and vapor state, when in sufficient concentrations, acts as an irritant to the eyes and respiratory tract.

9.3 Consult current OSHA regulations, local regulations, and suppliers' Safety Data Sheets for all materials used in these test methods.

10. Sampling and Handling

10.1 Sample the material in accordance with Practice D3437.

11. Calibration

11.1 *Apparatus*—Prepare and operate the spectrophotometer or photometer in accordance with the manufacturer's instructions.

11.2 *Reference Standards and Blanks:*

11.2.1 Dissolve 0.0905 g of polystyrene in 1000 mL of toluene measured at 25°C, which is equivalent to 100 mg/kg of polymer in monomer. This serves as the standard for polymer in styrene.

11.2.2 Make standard solutions containing 1, 3, 6, 9, 12, and 15 mg/kg of styrene polymer by diluting 1, 3, 6, 9, 12, and 15 mL of the 100 mg/kg standard solution to 100 mL with toluene in a volumetric flask at 25°C.

11.3 *Calibration Curves and Tables:*

11.3.1 Into each of a series of bottles equipped with glass stoppers pipet 15 mL of dry methanol and 10 mL of a polymer standard and mix thoroughly. Into another series of bottles pipet 15 mL of hexane and 10 mL of each polymer standard and mix thoroughly. Other volumes may be used, depending on the capacity of the spectrophotometer cell as long as the 3:2 proportion is maintained.

11.3.2 Allow the solutions to stand in the stoppered bottles for 15 min ± 1 min (Note 1). At the end of this time, pour the solutions into the spectrophotometer cells and measure the absorbance of each at a wavelength of 420 nm using the hexane/polymer standard as the blank (Note 2).

NOTE 1—Small changes in turbidity may occur with time. It is, therefore, important that the absorbance of calibration mixtures and samples be determined after standing the same length of time.

NOTE 2—The hexane is used for two reasons: *(1)* to blank out any color in the styrene, and *(2)* to indicate dissolved water in the styrene.

11.3.3 Prepare a calibration curve by plotting the absorbance against the milligrams per kilogram of polymer.

12. Procedure

12.1 Pipet 15 mL of hexane into a bottle equipped with a glass stopper.

12.2 Into a second bottle, pipet 15 mL of dry methanol.

12.3 Add 10 mL of styrene monomer to each bottle and mix thoroughly.

12.4 Proceed as described in 11.3.2 using the hexane mixture as the blank.

13. Calculation

13.1 Read the milligrams per kilogram of polymer directly from the calibration curve.

NOTE 3—Milligrams per kilogram can be converted to mass percent by dividing by 10 000.

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[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.

TABLE 1 Styrene (*mg/kg*)

Material	Average[A] $\overline{X}$	Repeatability Limit r	Reproducibility Limit R
Sample 1 — 0.4 mg/kg	1.673	1.796	2.827
Sample 2 — 2 mg/kg	2.413	1.711	3.792
Sample 3 — 4 mg/kg	3.759	4.268	5.090
Sample 4 — 10 mg/kg	10.143	3.254	9.037

[A] The average of the laboratories' calculated averages.

TABLE 2 % Recovery

Material	Average[A] (mg/kg)	Average Recovery (%)
Sample 1 — 0.4 mg/kg	1.673	418.2
Sample 2 — 2 mg/kg	2.413	120.6
Sample 3 — 4 mg/kg	3.759	94.0
Sample 4 — 10 mg/kg	10.143	101.4

[A] The average of the laboratories' calculated averages.

14. Report

14.1 Report the polymer content of the sample as milligrams of polymer per kilogram of monomer to the nearest mg/kg. For results less than 1 mg/kg, report <1 mg/kg.

15. Precision and Bias[5]

15.1 An ILS was conducted which included ten laboratories analyzing four samples three times. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1053.

15.2 *Repeatability:*

15.2.1 Results should not be suspect unless they differ by more than shown in Table 1. Results differing by less than *r* have a 95 % probability of being correct.

15.3 Reproducibility results submitted by two labs should not be considered suspect unless they differ by more than shown in Table 1. Results differing by less than *R* have a 95 % probability of being correct.

15.4 Reproducibility limits are listed in Table 1.

15.5 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in these test methods, bias has not been determined.

16. Quality Guidelines

16.1 Laboratories shall have a quality control system in place.

16.1.1 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.

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

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

16.1.4 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.

TEST METHOD B—VISUAL EVALUATION OF POLYMER CONTENT OF STYRENE MONOMER

17. Summary of Test Method

17.1 This test method utilizes the fact that styrene polymers are insoluble in methanol. The polymer content of a sample of styrene monomer is evaluated by visual observation of the degree of turbidity produced by the addition of methanol to the sample. The order of magnitude of the polymer content of styrene monomer in the incremental steps 0.001, 0.01, 0.1, and 1.0 mass % may readily be differentiated visually. For 0 % observe pure dry methanol.

18. Apparatus

18.1 *Test Tube,* 25 by 150-mm.

18.2 *Pipets,* 2 and 10-mL.

18.3 *Daylight Fluorescent Tube,* equipped with curved reflector.

19. Reagents

19.1 *Methanol,* dry.

19.2 *Polystyrene,* uncolored, unfilled, unlubricated (see 8.4).

19.3 *Toluene,* dry.

20. Procedure

20.1 Pipet 2 mL of sample into a clean, dry test tube, add 10 mL of dry methanol by means of a pipet, stopper the test tube with a cork covered with aluminum foil, and shake vigorously for a few seconds.

20.2 After shaking the test tube, inspect the mixture visually by looking through it toward a source of artificial daylight. Compare the observed turbidity of the mixture with the descriptions of turbidity given in Table 3 or against known

TABLE 3 Relationship Between Polymer Content of Styrene Monomer and Turbidity of Mixture of Two Parts by Volume Styrene Monomer and Ten Parts by Volume Dry Methanol

Polymer Content of Styrene Monomer by Weight, %[A]	Description of Turbidity of Styrene-Methanol Mixture
1.0 or greater	milk-white opaque liquid with heavy white precipitate
0.1	milk-white opaque liquid with no evidence of sedimentation
0.01	cloudiness readily visible, but mixture still transparent
0.001	faint trace of cloudiness; detectable only by comparison with pure dry methanol
None	no cloudiness discernible by comparison with pure dry methanol

[A] It is suggested that the analyst initially perform the test using reference mixtures described in this table as a guide. An experienced analyst can estimate the polymer content reliably without the use of reference mixtures.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1053. Contact ASTM Customer Service at service@astm.org.

standards. If standards are desired, they may be prepared using polystyrene and toluene.

21. Report

21.1 From Table 3 select the turbidity description that most nearly approximates that of the sample, and report the corresponding polymer content.

22. Keywords

22.1 polymer; polymer content; styrene; styrene monomer

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D2121–15) that may impact the use of this standard. (Approved June 1, 2016.)

(1) Section 11.2.1 — The mass of polymer was revised.

Committee D16 has identified the location of selected changes to this standard since the last issue (D2121–07) that may impact the use of this standard. (Approved March 15, 2015.)

(1) All references to AMS removed.
(2) Section 3 Terminology added.
(3) Section 11.2 — mass for stock solution corrected.
(4) Section 15 — new precision statements.
(5) Section 16 — revised to match D16 Editorial Guidelines.