Designation: D7977 – 23

Standard Test Methods for Polymer Content of AMS (α-Methylstyrene)[1]

This standard is issued under the fixed designation D7977; 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 polymer content of AMS (α-Methylstyrene). Dimers and trimers are not measured by these test methods.

1.2 This test method has been found applicable to determining the polymer content of AMS in concentrations up to 15 mg⁄kg with an LOQ of 0.6 mg/kg and an LOD of 0.2 mg/kg based on the data in Table 1. Samples containing more than 15 mg/kg of polymer must be suitably diluted before measurement.

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, 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

D6367 Specification for AMS (α-Methylstyrene)

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:*[3]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

TEST METHOD A—DETERMINATION OF POLYMER IN α-METHYLSTYRENE VIA SPECTROPHOTOMETER

3. Summary of Test Method

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

4. Significance and Use

4.1 This test method can be used for determining polymer concentrations in AMS monomer.

4.2 This test method will not detect dimers and trimers.

4.3 This test method can be used for plant control and for specification analysis. Information regarding plant control and specification analysis can be found in test method D6367.

5. Interferences

5.1 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.

5.2 Hexane is used for two reasons:

[1] These test methods are 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.

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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 Polymer Content (mg/kg)

Material	Average X	Repeatability Limit r	Reproducibility Limit R
Alpha methyl styrene	0.56	0.04	1.55
Alpha methyl styrene	1.12	0.16	0.51
Alpha methyl styrene	1.89	0.24	0.69
Alpha methyl styrene	3.77	0.10	0.61
Alpha methyl styrene	8.4	0.27	2.57
Alpha methyl styrene	15.14	0.62	1.65

5.2.1 To block out any color in the AMS, and

5.2.2 To indicate high levels of dissolved water in the AMS.

5.3 Water content was found to have a slight impact on the measurement of polymer in AMS solution. When the water concentration is below 260 ppm, its effect on the measurement of polymer in AMS solution is negligible.

6. Apparatus

6.1 *Class A Volumetric Pipets,* 10 mL and 15 mL.

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

6.3 *Spectrophotometer or Photometer Cells,* with 100 mm to 150 mm light path.

6.4 *Spectrophotometer or Photometer,* capable of absorbance measurements in wavelength region of 330 nm and sensitive to 1 mg polymer/kg monomer.

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 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.

7.2 *Hexane,* dry. (CAS 110-54-3.)

7.3 *Methanol,* dry. (CAS 57-56-1.)

7.4 *Poly-AMS:* Commercially available high-purity poly-AMS can be used; low-molecular weight poly-AMS (<150 000 molecular weight) was found to work and was used in the development of this standard. (CAS 25014-31-7.)

7.4.1 Be aware of possible error when the molecular weight of the polymer measured is different from that of the polymer used for calibration curves.

[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.

8. Hazards

8.1 AMS (α-Methylstyrene) monomer is flammable and polymerizes exothermically on contact with peroxides, mineral acids and aluminum chloride.

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

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

9. Sampling and Handling

9.1 Sample the material in accordance with Practice D3437.

10. Calibration

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

10.2 *Reference Standards and Blanks:*

10.2.1 Dissolve 0.0910 g of poly-AMS in 1000 mL of AMS monomer measured at 25 °C. This serves as the 100 mg⁄kg standard solution.

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

10.3 *Calibration Curves and Tables:*

10.3.1 Into each of a series of 25 mL volumetric flasks equipped with glass stoppers pipet 15 mL of dry methanol and 10 mL of a polymer standard and mix thoroughly. Into another series of flasks 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 proportions are maintained.

10.3.2 Allow the solutions to stand in the stoppered bottles for 15 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 330 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 AMS, and (*2*) to indicate high levels of dissolved water in the AMS.

10.3.3 Prepare a calibration curve by plotting the absorbance against the milligrams per kilogram of polymer (Note 3).

NOTE 3—Background levels of poly-AMS absorbance may exist depending on the quality of AMS monomer used. In such cases, prepare calibration curves using the method of standard additions, effectively subtracting out the background level.

11. Procedure

11.1 Pipet 15 mL of hexane into a 25 mL volumetric flask equipped with a glass stopper.

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