Designation: D7977 – 14

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-Methystyrene). 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. 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 8.

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

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.

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:

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 and 15-mL.

[1] These test methods are 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 Dec. 1, 2014. Published March 2015. DOI: 10.1520/D7977-14.

[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.

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

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

6.4 *Spectrophotometer of 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.

7.3 *Methanol,* dry.

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.

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.

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, 3, 6, 9, 12, and 15 mg/kg of polymer by diluting 1, 3, 6, 9, 12, 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 Pipe 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.

11.3 Add 10 mL of AMS to each bottle and mix thoroughly.

11.4 Proceed as described in 10.3.2 using the hexane mixture as the blank.

12. Calculation

12.1 Read the milligrams per kilogram of polymer directly from the calibration curve (Note 4).

NOTE 4—Milligrams per kilogram can be converted to weight percent by dividing by 10 000.

13. Report

13.1 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.

14. Precision and Bias[5]

14.1 A single sample containing 4.8 mg/kg polymer was analyzed 20 times by one person using one instrument over the shortest practical time. Details are given in Research Report RR:D16-1054.

[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.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1054. Contact ASTM Customer Service at service@astm.org.

14.2 *Repeatability:*

14.2.1 The average of the 20 results was 4.65. Repeatability "*r*" was 0.2. Results should not be suspect unless they differ by more than "*r*". Results that differ by less than "*r*" have a 95 % probability of being correct.

14.3 Reproducibility has not been determined and will be determined within five years.

14.4 *Bias:*

14.4.1 Since there is no accepted reference material suitable for determining the bias in this test method, bias has not been determined.

15. Quality Guidelines

15.1 Laboratories shall have a quality control system in place.

15.1.1 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.

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

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

15.1.4 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 α-METHYLSTYRENE

16. Summary of Test Method

16.1 This test method utilizes the fact that AMS polymers are insoluble in methanol. The polymer content of a sample of AMS 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 AMS in the incremental steps 0.001, 0.01, 0.1, and 1.0 weight % may readily be differentiated visually. For 0 % observe pure dry methanol.

17. Apparatus

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

17.2 *Class A Volumetric Pipets,* 2 and 10-mL.

TABLE 1 Relationship Between Polymer Content of AMS and Turbidity of Mixture of Two Parts by Volume AMS and Ten Parts by Volume Dry Methanol

Polymer Content of AMS mg/kg[A]	Description of Turbidity of Styrene-Methanol Mixture
10 000 or greater	milk-white opaque liquid with heavy white precipitate
1000	milk-white opaque liquid with no evidence of sedimentation
100	cloudiness readily visible, but mixture still transparent
10	faint trace of cloudiness; detectable only by comparison with pure dry methanol
<1	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.

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

18. Reagents

18.1 *Methanol,* dry.

18.2 *Poly-AMS,* uncolored, unfilled, unlubricated (see 7.4).

18.3 *Toluene,* dry.

19. Procedure

19.1 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.

19.2 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 1 or against known standards. If standards are desired, they may be prepared using poly-AMS and toluene.

20. Report

20.1 From Table 1 select the turbidity description that most nearly approximates that of the sample, and report the corresponding polymer content.

21. Keywords

21.1 α-methylstyrene; AMS; polymer; polymer content