



Designation: ~~D2121~~—16 D2121 – 23

## Standard Test Methods for Polymer Content of Styrene Monomer<sup>1</sup>

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 ( $\epsilon$ ) 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. The limit of detection (LOD) is 1.2 mg/kg and the limit of quantitation (LOQ) is 4.0 mg/kg. LOD and LOQ were calculated using the last sample in Table 1.

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, safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

1.7 *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:<sup>2</sup>

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

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons—Aromatic, Industrial, Specialty and Related Chemicals and are the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

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<sup>2</sup> 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.

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

**TABLE 1 Styrene (mg/kg)**

Material	Average <sup>A</sup>	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	r	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

**TABLE 1 Styrene (mg/kg)**

Material	Average <sup>A</sup>	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	r	R
Sample 1 — 2 mg/kg	2.6	0.3	0.7
Sample 2 — 5 mg/kg	4.1	0.7	1.3
Sample 3 — 6 mg/kg	7.7	1.1	3.4

<sup>A</sup> The average of the laboratories' calculated averages.

**TABLE 2 — % Recovery**

Material	Average <sup>A</sup> (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

<sup>A</sup> The average of the laboratories' calculated averages.

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<sup>3</sup>

### 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 PHOTOMETERSPECTROSCOPIC 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.

<sup>3</sup> 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 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~~ 10 mL 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~~ 50 mm 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.<sup>4</sup> 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~~ dry (CAS 110-54-3).

8.3 *Methanol*, ~~dry~~ dry (CAS 64-56-1).

8.4 ~~*Polystyrene*~~ *Polystyrene* (CAS 9003-53-6):

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~~ 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~~ (CAS 100-42-5).

8.6 *Toluene*, ~~dry~~ dry (CAS 108-88-3).

## 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.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications, 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.