Designation: D 5296 – 97

Standard Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography[1]

This standard is issued under the fixed designation D 5296; 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 molecular weight (MW) averages and the distribution of molecular weights for linear, soluble polystyrene by liquid high-performance size-exclusion chromatography (HPSEC). This test method is not absolute and requires the use of commercially available narrow molecular weight distribution (MWD) polystyrene standards for calibration. This test method is applicable for samples containing molecular weight components that have elution volumes falling within the elution volume range defined by polystyrene standards (that is, molecular weights generally from 2000 to 2 000 000 $g \cdot mol^{-1}$).

1.2 The HPSEC is differentiated from traditional size-exclusion chromatography SEC (also referred to as gel permeation chromatography (GPC)) in that the number of theoretical plates per metre with an HPSEC system is about ten times greater than that for traditional SEC (see Terminology D 883 and Practice D 3016).[2] The HPSEC systems employ low-volume liquid chromatography components and columns packed with relatively small (generally 3 to 20 µm) microporous particles. High-performance liquid chromatography instrumentation and automated data handling systems for data acquisition and processing are required.

1.3 The values stated in SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 9.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[3]

D 2857 Test Method for Dilute Solution Viscosity of Polymers[4]

D 3016 Practice for Use of Liquid Exclusion Chromatography Terms and Relationships[4]

E 685 Practice for Testing Fixed-Wavelength Photometric Detectors Used in Liquid Chromatography[5]

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods[6]

3. Terminology

3.1 *Definitions*—For definitions of technical terms pertaining to plastics used in this test method see Terminology D 883.

4. Summary of Test Method

4.1 In this test method a dilute solution of a polystyrene sample is injected into a liquid mobile phase containing the same solvent used to prepare the polymer solution. The mobile phase transports the polymer into and through a chromatographic column (or set of columns connected in series) packed with a solid or semirigid, porous substrate which separates the polymer molecules according to their size in solution. Starting from injection, a detector continuously monitors the eluate as a function of elution volume (or time). Upon emerging from the column(s), the size-separated molecules are detected and recorded according to their concentration. Through calibration, the elution volumes (or times) are converted to molecular weights, and various molecular weight parameters for the sample are calculated from the molecular weight/concentration data.

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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[2] See also *AMD Bibliography and Bibliography Supplements AMD 40-S1, 40-S2, and 40-S3 on Size Exclusion Chromatography,* available from ASTM Headquarters.

[3] *Annual Book of ASTM Standards,* Vol 08.01.

[4] *Annual Book of ASTM Standards,* Vol 08.02.

[5] *Annual Book of ASTM Standards,* Vol 14.01.

[6] *Annual Book of ASTM Standards,* Vol 14.02.

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

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