



Standard Test Method for Molecular Mass Averages and Molecular Mass Distribution of Atactic Polystyrene by Matrix Assisted Laser Desorption/ Ionization (MALDI)-Time of Flight (TOF) Mass Spectrometry (MS)¹

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1. Scope

1.1 This test method covers the determination of molecular mass (MM) averages and the distribution of molecular masses for linear atactic polystyrene of narrow molecular mass distribution (MMD) ranging in molecular masses from 2000 g/mol to 35 000 g/mol by matrix assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF-MS). This test method is not absolute and requires the use of biopolymers for the calibration of the mass axis. The relative calibration of the intensity axis is assumed to be constant for a narrow MMD. Generally, this is viewed as correct if the measured polydispersity is less than 1.2 for the molecular mass range given above.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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.*

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D883 Terminology Relating to Plastics](#)

[D1600 Terminology for Abbreviated Terms Relating to Plastics](#)

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

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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.

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

3. Terminology

3.1 *Definitions*—For definitions of technical terms pertaining to plastics used in this test method see Terminologies [D883](#) and [D1600](#).

4. Summary of Test Method

4.1 The MALDI process involves the ablation and the ionization of an analyte dispersed in an organic small molecule matrix, most commonly an organic acid. One way to cationize the analyte is to add a metal salt. The process is as follows: A polymer (biological or synthetic) is co-crystallized or co-mixed with the matrix molecule in the solid phase and deposited on the target often made of stainless steel (details of this process will be described later). A short duration UV or IR laser pulse is used to ablate the matrix and the analyte mixture. The ablation process involves UV or IR absorption by the matrix molecule. The laser energy excites the matrix molecule causing it to vaporize and decompose. Analyte and matrix leave the target surface in a plume. This ablation process involves the transfer of energy from electronic or vibration modes into translational modes of the matrix. The MALDI-TOF-MS method described in this test method uses a UV nitrogen laser operating at 337 nm. This laser has a pulse width of about 3 ns.

4.2 In the test method described below, the polystyrene polymer in the ablation plume gains an Ag cation and is accelerated by a high voltage, often about 20 keV. Following acceleration, the polymer species drifts down the field free flight tube and is detected at the end of the flight tube. The time-of-flight of the species is a measure of its mass. From the distribution of arrival times and the calibration of the arrival times with known mass standards, the mass distribution of the polymer is determined.