National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2888

Polystyrene

(M_w, 7 190 g/mol)

This Standard Reference Material (SRM) is intended primarily for use in calibration and performance evaluation of instruments used to determine the molar mass and molar mass distribution by size exclusion chromatography (SEC). A unit of SRM 2888 consists of approximately 0.3 g of polystyrene powder.

Certified Value

Mass-average molar mass* (M_w): 7.19×10^3 g/mol $\pm$ 0.57×10^3 g/mol

*Expressed as molar mass, previously expressed as weight average molecular weight [1].

Certified Uncertainties: The certified measurement uncertainty is expressed as a combined expanded uncertainty with a coverage factor $k = 2$, calculated in accordance with NIST procedure [2]. Type A and Type B contributions to the expanded uncertainty of the measured mass-average molar mass include the uncertainties in Rayleigh ratio of the scattering standard, optical alignment, and calibration of the differential refractometer.

Expiration of Certification: The certification of SRM 2888 is valid, within the measurement uncertainties specified, until **31 January 2010**, provided that the SRM is handled in accordance with the storage instructions given in this certificate. This certification is nullified if the SRM is modified or contaminated.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Technical coordination leading to certification of this SRM was provided by B.M. Fanconi of the NIST Polymers Division.

Technical measurement and data interpretation were provided by C.M. Guttman, W.R. Blair, B.M. Fanconi, R.J. Goldschmidt, W.E. Wallace, S.J. Wetzel, and D.L. Vanderhart of the NIST Polymers Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Eric J. Amis, Chief
Polymers Division

Gaithersburg, MD 20899
Certificate Issue Date: 09 May 2003

John Rumble, Jr., Chief
Measurement Services Division

Storage: The SRM should be stored in the original bottle with the lid tightly closed under normal laboratory conditions.

Homogeneity and Characterization: The homogeneity of SRM 2888 was tested by SEC analysis of solutions in tetrahydrofuran at 40 °C. The further characterization of this polymer is described in reference 3.

Supplemental Information: The number-average molar mass **(**M_n**)** was determined by nuclear magnetic resonance (NMR) analysis of the end groups and found to be 6.96×10^3 g/mol with an estimated uncertainty of 0.40×10^3 g/mol. Fourier transform infrared spectroscopy (FTIR) and matrix-assisted laser desorption ionization time of flight mass spectrometry (MALDI-TOF-MS) were used to analyze end groups on the polymer. Only one set of end groups was found on the polymer. This polystyrene was also used in an interlaboratory comparison for the measurement of molecular mass distribution by MALDI-TOF-MS. The MALDI-TOF-MS interlaboratory comparison yielded an M_n of 6.61×10^3 g/mol with a standard deviation of 0.12×10^3 g/mol and M_w of 6.74×10^3 g/mol with a standard deviation of 0.11×10^3 g/mol. Twenty-three laboratories took part in this study [4]. A representative MALDI-TOF-MS spectrum of SRM 2888 is given in Figure 1.

NIST Certification Method: The certified value for M_w was measured on SRM 2888 using static light scattering in toluene as solvent at 23 °C [3].

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, 1995 Ed. (April 1995).

[2] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO; Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office. Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[3] Guttman, C.M.; Blair,W.R.; Fanconi, B.M.; Goldschmidt, R.J.; Wallace, W.E.; Wetzel, S.J.; Vanderhart, D.L.; *Certification of a Polystyrene Synthetic Polymer, SRM 2888*; NIST Special Publication 260-152 (in press).

[4] Guttman, C.M.; Wetzel, S.J.; Blair, W.R.; Fanconi, B.M.; Girard, J.E.; Goldschmidt, R.J.; Wallace, W.E.; VanderHart, D.L.; *NIST-Sponsored Interlaboratory Comparison of Polystyrene Molecular Mass Distribution Obtained by Matrix-Assisted Laser Desorption/Ionization Time-of-Flight Mass Spectrometry*: *Statistical Analysis*; Analytical Chemistry, Vol. 73, pp. 1252-1262 (2001).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

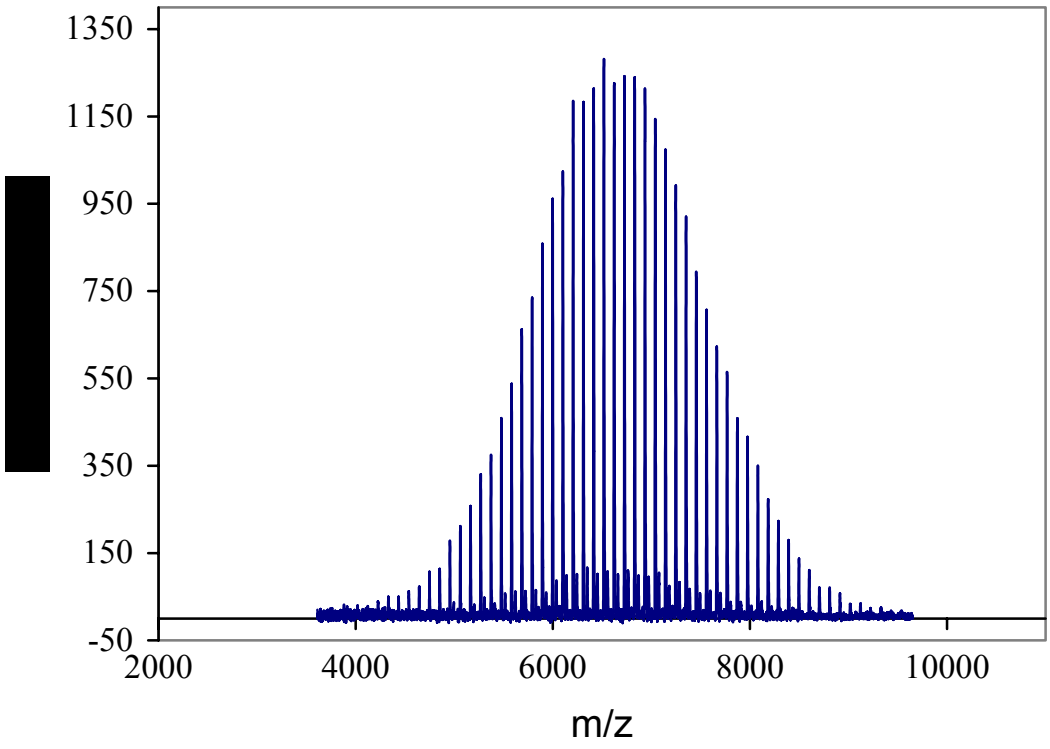

Figure 1. Mass spectrum of SRM 2888 measured by matrix assisted laser desorption/ionization mass spectrometry. Retinoic acid was used as the matrix and silver trifluoroacetate as the catonization salt.