National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1473c

Low Density Polyethylene Resin

This Standard Reference Material (SRM) is intended for use in calibration and performance evaluation of instruments used in polymer technology and science for the determination of the melt flow rate using ASTM D1238-13, Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer [1] Standard Test Condition 190/2.16. A unit of SRM 1473c consists of approximately 60 g of white polyethylene pellets in an amber glass bottle.

Certified Values and Uncertainties: This material is certified for melt flow rate using ASTM D1238-13 [1]. The flow rate of the melt was determined at 190.0 ºC ± 0.1 ºC and a load of 2.16 kg by procedure A of the ASTM method. A manually operated extrusion plastometer was used. Under these conditions, the certified melt flow rate for this material is as follows:

Melt Flow Rate (FR): 1.16 g/10 min ± 0.097 g/10 min

The measurand is the flow rate. Metrological traceability is to the SI units for mass and time (expressed as grams per ten minutes). Fifteen units of the SRM were measured in duplicate by NIST according to the procedures of ASTM D1238-13 [1]. The certified melt flow rate is the weighted mean of the NIST measurements estimated using a Gaussian random effects model [2–4] and the DerSimonian Laird procedure [5,6]. The associated measurement uncertainty was evaluated by an application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [7-10]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of the within unit measurement uncertainty (0.0025 g/10 min), unit-to-unit variability (0.0055 g/10 min), uncertainty in the temperature measurement within the plastometer cylinder (0.02 g/10 min), and the uncertainty due to laboratory-to-laboratory variability (calculated based on Table 4 of ASTM D 1238-13, 0.0449 g/10 min). The expansion factor, $k = 1.96$, corresponds to an approximately 95 % confidence level.

Expiration of Certification: The certification of **SRM 1473c** is valid, within the measurement uncertainty specified, until **01 January 2021**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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 the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to certification of this SRM was provided by K. Migler of the NIST Materials Science and Engineering Division.

Technical measurement and data interpretation were provided by W.G. McDonough and C.M. Guttman of the NIST Materials Science and Engineering Division.

Statistical analysis was provided by D.D. Leber of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Materials Science and Engineering Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 September 2016

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE

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

Homogeneity: The homogeneity of SRM 1473c was tested by melt flow rate measurements using ASTM D1238-13 [1]. The characterization of this polymer is described in reference 11.

REFERENCES

[1] ASTM International. ASTM D1238-13 *Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer*. West Conshohocken, PA: ASTM International (2013).

[2] Searle, S.R.; Casella, G. and McCulloch, C.E.; *Variance Components*, John Wiley & Sons, Hoboken, NJ (2006).

[3] Pinheiro, J.C.; Bates, D.M.; *Mixed Effects Models in S and S-Plus*; Springer, New York, NY (2000).

[4] Toman, B.; Possolo, A.; *Laboratory Effects Models for Interlaboratory Comparisons.* Accredit. Qual. Assur., Vol. 14, pp. 553–563 (2009), see also Toman, B.; Possolo, A.; *Erratum to: Laboratory Effects Models for Interlaboratory Comparisons*; Accredit. Qual. Assur.; Vol. 15, pp. 653–654 (2010).

[5] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).

[6] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

[7] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2016); 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://www.nist.gov/pml/pubs/index.cfm (accessed Sep 2016).

[8] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (JCGM) (2008); available at www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Sep 2016).

[9] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).

[10] Davison, A.C.; Hinkley, D.V.; *Bootstrap Methods and their Application*; Cambridge University Press, New York (1997).

[11] Maurey, J.R.; Schultheisz, C.; Blair, W.R.; Guttman, C.M.; *Certification of Standard Reference Material 1473b, Low Density Polyethylene Resin*; NIST Special Publication SP 260-144; U.S. Government Printing Office, Washington, DC (2002); available at http://www.nist.gov/srm/publications.cfm (accessed Sep 2016).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.