National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material 1484a

Linear Polyethylene

(Narrow Molecular Weight Distribution)

This Standard Reference Material (SRM) is intended for the calibration and evaluation of instruments used in polymer technology and science for the determination of molecular weight and molecular weight distribution, and for use as a characterized sample for measurements of other physical properties of linear polyethylene. This SRM is supplied as a granular polyethylene in a 0.3-g unit.

Property	Value	Sample standard deviation of value, %	Number of degrees of freedom	Expected limit of systematic error, %
Number-average molecular weight, M_n, g/mol[a]	100,500	3.7	34	4[b]
Weight-average molecular weight, M_w, g/mol[c]	119,600	1.8	5	11[b,d]
Limiting viscosity number, ml/g:				
at 130°C in 1,2,4-trichloro-benzene	197.9	0.30	22	1
at 130°C in 1-chloro-naphthalene	169.4	0.35	22	1

[a]Determined by membrane osmometry in 1-chloronaphthalene at 130 °C.

[b]The expected limits of systematic error for the number-and-weight-average molecular weights are based on analyses of the osmometry and light-scattering determinations, respectively, without taking into account the necessity of M_w exceeding M_n.

[c]Determined by light scattering in 1-chloronaphthalene at 135 °C based on a value of 17.8×10^{-6} cm^{-1} for the Rayleigh ratio for the vertically polarized scattering of vertically polarized light, of wavelength 546 nm in vacuum, from benzene at 23 °C. This value was derived from published values of the unpolarized Rayleigh ratio and the depolarization ratio for unpolarized light [D J. Coumou, J. Colloid Sci. **15**, 408 (1960)]. The differential refractive index of this polyethylene in 1-chloronaphthalene at 135 °C, also required for the calculation of molecular weight, was found to be -0.191 ml/g at wavelength 546 nm in vacuum, based on the value of 0.1429 ml/g for the differential refractive index of sucrose in aqueous solution at 25 °C [Norberg and Sundelöf, Makromol. Chem. **77**, 77 (1964)].

[d]The expected limit of systematic error from all sources except the vertically polarized Rayleigh ratio for benzene is 4 %.

Gaithersburg, MD 20899
October 2, 1992
(Revision of certificate dated 9-25-92)

William P. Reed, Chief
Standard Reference Materials Program

(over)

SAMPLE PREPARATION

This sample of linear polyethylene was prepared by fractional recrystallization from 1,2,4-trichlorobenzene and subsequent blending of fractions produced by large scale gel permeation chromatography, from a linear polyethylene equivalent to SRM 1475 (Linear Polyethylene, Whole Polymer). The fractions as received, contained several % by weight of polyethylene components with molecular weights in the range 1,000 - 4,000. The amount of this material remaining after the fractional recrystallization is less than 0.5 %. Both total volatiles, estimated gravimetrically, and residual 1,2,4-trichlorobenzene content, estimated spectrophotometrically, do not exceed
0.1 %.

The maximum rate of shear in the Ubbelohde capillary viscometers employed for the determination of limiting viscosity numbers was 3,000 sec^{-1} for 1,2,4-trichlorobenzene and 2,000 sec^{-1} for 1-chloronaphthalene. The maximum specific viscosities were 0.4 in both solvents.

Measurements leading to the certification of this SRM were performed by J.E. Brown, R.G. Christensen, C.C. Han, J.R. Maurey, P.H. Verdier, and H.L. Wagner in the Polymers Division, NIST Institute for Materials Research. A report describing the investigations required for this and related polyethylene SRMs are described in reference [1].

The technical and support aspects involved in the revision, update, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.C. Colbert.

STORAGE: SRM 1484a should be stored in its tightly closed, original bottle under normal laboratory conditions.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: The certified values for this SRM will be valid for five years from date of shipment.

REFERENCE

[1] Verdier, P.H., and Wagner, H.L., Standard Reference Materials: The Characterization of Linear Polyethylene (SRMs 1482, 1483, 1484), NBS Special Publication 260-61.