

National Bureau of Standards Certificate

Standard Reference Material 1475

Linear Polyethylene (Whole Polymer)

C. A. J. Hoeve, H. L. Wagner, J. E. Brown,
 R. G. Christensen, L. J. Frolen, J. R. Maurey, G. S. Ross, and P. H. Verdier

This Standard Reference Material is intended for the calibration of instruments used in polymer science and technology for the determination of molecular weight and molecular weight distribution.

<u>Quantity</u>	<u>Average Values</u>	<u>Estimated Standard Deviation of Average</u>
Molecular Weight		
Weight-average molecular weight ^a	52,000	2,000
Number-average molecular weight	18,310	360
Weight-average molecular weight	53,070	620
Z-average molecular weight	138,000	3,700
Ratio of molecular weights $M_z:M_w:M_n$	7.54:2.90:1	
Molecular weight distribution		See Table 1
Limiting Viscosity Number (dl/g)		
In 1-chloronaphthalene at 130 °C	0.890	0.0032
In 1,2,4-trichlorobenzene at 130 °C	1.010	.0086
In decahydronaphthalene ^c at 130 °C	1.180	.0032
Melt-Flow Rate (g/10 min) ^d	2.07	.0062
Density (g/cm ³) ^e	0.97844	.00004

^aBy light scattering in 1-chloronaphthalene at 135 °C.

^bBy gel-permeation chromatography.

^c"Technical" grade, which assayed at approximately equal proportions of cis- and trans- decahydronaphthalenes.

^dBy a procedure similar to Procedure A, ASTM Method D1238-65T, Test Condition D, 190 °C, load 325 g.

^eBy ASTM Method D1505-67; sample prepared by Procedure A, ASTM Method D1928-68.

Measurements leading to the certification of this material were performed in the Molecular Properties and Characterization Section of the Polymer Division.

Washington, D.C. 20234
 November 6, 1969
 (Revised December 2, 1971)

J. Paul Cali, Chief
 Office of Standard Reference Materials

(over)

The methyl group content as determined by ASTM Method D2238-68 is 0.15 methyl groups per 100 carbon atoms. This shows the polyethylene to be essentially linear. A pellet to pellet coefficient of variation of 3 percent in the limiting viscosity number was found. All determinations should consequently be performed on samples containing at least 50 pellets or one gram of polymer (or material from a blend of one gram). This will reduce the expectation of the standard error due to pellet variability to less than 0.5 percent.

Table 1. Cumulative Molecular Weight Distribution by Gel-Permeation Chromatography

log M	Wt. %	log M	Wt. %	log M	Wt. %
2.800	0.0	4.014	15.2	5.065	90.7
2.865	0.005	4.070	18.1	5.113	92.2
2.929	0.020	4.126	21.5	5.161	93.7
2.992	0.052	4.182	25.2	5.209	94.8
3.056	0.105	4.237	29.3	5.256	95.8
3.119	0.185	4.292	33.7	5.303	96.6
3.181	0.343	4.346	38.5	5.349	97.3
3.243	0.475	4.400	43.4	5.395	97.9
3.305	0.706	4.454	48.5	5.440	98.4
3.366	0.999	4.507	53.5	5.485	98.7
3.427	1.38	4.560	58.3	5.530	99.1
3.488	1.88	4.612	62.9	5.574	99.3
3.548	2.51	4.664	67.3	5.618	99.5
3.607	3.30	4.715	71.4	5.662	99.7
3.667	4.28	4.766	75.1	5.705	99.8
3.725	5.46	4.817	78.5	5.789	99.9
3.784	6.87	4.868	81.6	5.87	100.0
3.842	8.56	4.918	84.4		
3.900	10.50	4.967	86.7		
3.957	12.7	5.016	88.9		

The sample of linear polyethylene was obtained from E. I. du Pont de Nemours and Company of Wilmington, Delaware.

This sample of linear polyethylene has an ash content of 0.002 percent. No volatiles were detected by a gas-chromatographic procedure capable of detecting 0.5 percent volatiles. The manufacturer added 111 ppm of the antioxidant, Irganox 1010 (Geigy), which is tetrakis [methylene-3-(3',5'-di-t-butyl-4'-hydroxyphenyl)propionate] methane.

The differential refractive index in 1-chloronaphthalene, required for the calculation of molecular weight by light scattering, was found to be -0.193 ml/g at 135°C and 546 nm.

The maximum rate of shear in the Ubbelohde viscometer was about 1500 sec⁻¹. All measurements were carried out at specific viscosities (0.1 or less) which were sufficiently low for negligible dependence on rate of shear.

A series of reports describing investigations required for this certificate will be published in the Journal of Research of the National Bureau of Standards, Volume 76 A, No. 2, 1972.