Designation: D6474 – 12

Standard Test Method for Determining Molecular Weight Distribution and Molecular Weight Averages of Polyolefins by High Temperature Gel Permeation Chromatography[1]

This standard is issued under the fixed designation D6474; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of molecular weight distributions and molecular weight averages of linear polyolefins by high temperature gel permeation chromatography (GPC). This test method uses commercially available polystyrene standards and equipment and is applicable to polyethylenes (excluding high pressure low density polyethylene–LDPE) and polypropylenes soluble in 1,2,4-trichlorobenzene (TCB) at 140°C. This test method is not absolute and requires calibration.

NOTE 1—Size exclusion chromatography (SEC) often is used as an alternative name for gel permeation chromatography (GPC).

NOTE 2—Specific methods and capabilities of users may vary with differences in columns, instrumentation, applications software, and practices between laboratories.

NOTE 3—One general method is outlined herein; alternative analytical practices can be followed and are attached in notes where appropriate.

NOTE 4—There is no known ISO equivalent to this standard.

1.2 The values stated in SI units, based on IEEE/ASTM S1-10, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics
D3016 Practice for Use of Liquid Exclusion Chromatography Terms and Relationships
D5296 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography
E685 Practice for Testing Fixed-Wavelength Photometric Detectors Used in Liquid Chromatography
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
IEEE/ASTM S1-10 Standard for Use of the International System of Units (SI): The Modern System (replaces ASTM E 380 and ANSI/IEEE Standard 268-1992)

3. Terminology

3.1 *Definitions*—Definitions of terms applying to plastics appear in Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *polyolefin, n—used in this context*, refers to PE (except LDPE) and PP thermoplastics.

4. Summary of Test Method

4.1 In this test method, a polyolefin sample is dissolved in a solvent and injected onto a chromatographic column(s) packed with a solid substrate, which separates the molecules according to their size in solution. The separated molecules are detected and recorded as they elute from the column according to concentration. Through calibration, retention times are converted to molecular weights. Average molecular weight parameters and molecular weight distribution are determined from the molecular weight concentration data.

5. Significance and Use

5.1 This test method measures the molecular weight distribution and molecular weight averages of polyethylene (except LDPE) and polypropylene resins. Differences in molecular weight and molecular weight distribution significantly affect physical properties, such as morphology, strength, melt flow etc., and as a result, the final properties of products made from these resins.

6. Interferences

6.1 A major interference is the presence of insoluble, highly entangled, high molecular weight material that may be linear or cross-linked. A successful outcome of the test requires that the

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

Current edition approved Dec. 15, 2012. Published December 2012. Originally approved in 1999. Last previous edition approved in 2006 as D6474–99(2006). DOI: 10.1520/D6474-XX.

[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.

***A Summary of Changes section appears at the end of this standard**

sample be dissolved completely prior to the chromatographic separation. The presence of the above-described material often precludes the necessary dissolution step.

6.2 A mismatch in the antioxidant level in the dissolved sample and that of the TCB eluent (see 8.1).

6.3 The accuracy of the molecular weight results decreases, that is, they become increasingly underestimated, as the α-olefin comonomer content increases in linear low density polyethylene (LLDPE). For example, the results for an octene copolymer containing 30 branches per 1000 carbon atoms will be about 6 % low.

7. Apparatus

7.1 *Essential Components*—The essential components of the instrumentation are a solvent reservoir, a pump, a solvent degasser, a sample injection system, packed columns, and a solute mass detector.

NOTE 5—Complete high temperature GPC units with a maximum operating temperature of 210°C are commercially available.

7.2 *Solvent Reservoir*—The solvent reservoir shall hold sufficient TCB to ensure consistency of composition for a number of runs. The TCB is protected from exposure to water in the air and the reservoir material shall be inert to the solvent.

7.3 *Pump*—The principal requirement of the pump is production of a relatively constant flow, with minimum pulsations, of solvent through the columns. In general, the rate is adjustable between 0.1 and 5.0 cm^3/min and back pressures are not to exceed limits specified by the column manufacturer. Flow rate precision shall be at least ±0.3 % as measured under the conditions and time interval for a typical analysis.

7.4 *Sample Injection System*—The purpose of the injection system is to introduce the solution containing the sample into the flow stream as a sharply defined zone. Either a six-port valve with an attached sample loop or a variable volume injector can be used for this purpose in conjunction with an autosampler. Requirements include minimal contribution to band spreading, injector ability to operate at the back pressure generated by the columns, repeatability of injection volume, and no carryover.

7.5 *Columns*—Stainless steel columns with uniform and highly polished inside walls are recommended for high temperature GPC. Columns with lengths ranging from 20 to 50 cm with fittings, frits, and connectors designed to minimize dead volume and mixing are recommended. Generally, the packing materials, typically styrene divinylbenzene copolymers, have narrow particle size distributions in the 3 to 20 µm range. Packing materials are available in a variety of shapes and pore sizes. Columns are packed with particles of relatively uniform pore size or with a "mixed bed" of particles to produce a broad range of pore sizes. If a set of columns with uniform pore size is used, it is recommended that the columns be connected in order of increasing pore size towards the low pressure detector side.

NOTE 6—Packed high temperature GPC columns are available from a number of manufacturers.

7.6 *Detector*—The detector provides a continuous measure of the concentration of solute eluting from the column(s). The detector shall be sufficiently sensitive and respond linearly to the solute concentration, independently of molecular weight, and shall be of low internal volume so as not to distort the concentration gradient during elution. For this test method, the detector cell volume should be 30 µL or less. The most commonly used concentration detectors for high temperature GPC are refractive index or infrared. The former has moderate sensitivity and general utility. When testing detector performance, follow the recommendations of the instrument manufacturer.

NOTE 7—For polyolefins, the refractive index (dn/dc) increment is essentially constant above molecular weights of 5 000 g/mol. The response of the components below 5 000 g/mol should be corrected prior to the molecular weight calculations using a pre-established dn/dc molecular weight calibration. The principal disadvantage of the differential refractometer is that the temperature within the detector cell shall be controlled to within 0.0001°C.

7.7 *Tubing and Fittings*—All tubing between the sample injector and the detector must be no greater than 0.25 mm (0.01 in.) internal diameter and rated for pressures up to 42 MPa. Connecting column tubing must be kept as short as possible and all fittings and connectors shall have low dead volumes to prevent mixing.

7.8 *Data Acquisition/Handling System*—Means shall be provided for determining chromatographic peak heights or integrated area segments at prescribed time intervals and for handling and reporting data. This is best accomplished using a computer with appropriate software.

NOTE 8—Data acquisition and handling systems for high temperature GPC have not been standardized. However, a number of different manufacturers provide GPC specific computer software.

8. Reagents and Materials

8.1 *Solvent*—1,2,4-trichlorobenzene (TCB) is recommended as the solvent for this test method. With a refractive index detector, the solvent shall have a refractive index different than that of the polyolefins analyzed. Solvent purity and consistency shall be considered when choosing a solvent. For example, unless freshly distilled, and subsequently, kept in a glass container under an inert gas, TCB will react with water to form hydrochloric acid that will attack tubing walls and degrade column packing. The TCB reservoir must be protected against exposure to moisture, or be replaced frequently with fresh solvent, or both. An antioxidant, such as 2,6-di-tert-butyl-4-methylphenol (BHT) must be added to the solvent reservoir at the same concentration, that is, about 250 mg/L, as in the solvent used to dissolve the polymer to minimize any interference due to an antioxidant mismatch peak.

NOTE 9—Several laboratories are successfully recycling TCB using partial vacuum distillation.

NOTE 10—Any solvent that has a boiling point higher than the operating temperature, is considered a good solvent for polyolefins, and is compatible with the GPC components, can be used

8.2 *Polymer Standards*—Narrow MWD (M_W/M_n < 1.1) polystyrene standards of known molecular weight (available from several suppliers) are used for calibration.

8.3 *Other Chemicals*—Low MW compounds, such as toluene or hexadecane, that are used for determining plate count, shall be of high purity.

9. Hazards

9.1 Solvents used in this test method are toxic, or highly flammable, or both. The user is advised to consult literature and follow recommended procedures pertaining to the safe handling of solvents.

10. Sampling

10.1 Whenever possible, grinding is used to ensure a representative sample is analyzed.

11. Preparation of Apparatus

11.1 *Flow Rate*—A flow rate of 1 ± 0.1 cm^3/min is suggested. It is recommended that the retention time of the air peak or that of an added low molecular weight flow rate marker, such as toluene, be used to ascertain a flow rate constant to within 0.3 %.

11.2 *Detector*—Detector performance must be checked regularly for any deterioration in signal-to-noise ratio. The calibration mixtures can be used for this purpose.

12. Preparation of Solutions

12.1 *Polymer Samples:*

12.1.1 Weigh the polymer samples directly into GPC autosampler vials or into larger heat resistant vials having a cap lined with solvent resistant material.

12.1.2 Add antioxidant containing, that is, about 250 mg /L solvent, preferably siphoned from the solvent reservoir, to give a polymer concentration of between 0.05 and 0.2 weight % (see 12.3).

12.1.3 Cap the vials and heat the solutions to about 150°C for 3 to 6 h to completely dissolve the samples.

NOTE 11—For polypropylene and some high MW polyethylenes, the samples may have to be heated to between 160 and 180°C for complete dissolution.

NOTE 12—Magnetic stirring, frequent manual agitation, or a slow rotational arrangement inside an oven is recommended to aid dissolution. Excessive temperatures, prolonged dissolution times, and ultrasonic devices may cause the polymer to degrade.

NOTE 13—Filtration of hot polymer solutions to remove or identify the presence of nonvisible gels or other undissolved material is not recommended due to the difficulty involved in filtering at 150°C. Unless performed very carefully, sample losses may occur due to a drop in temperature during the filtration. Also, most commercial instruments are or can easily be outfitted with an inline pre-column filter.

12.2 *Polymer Standards*—Prepare 3 to 5 "cocktails", that is, mixtures containing 3 to 4 baseline resolved standards, containing a total of at least 12 narrow MWD polystyrene standards, as well as some hydrocarbon and polyolefin standards, if available, to give individual concentrations of 0.01 to 0.03 weight % (the high molecular weight standards being the more dilute. The standards must be dissolved at room temperature (up to three days dissolution time) or at 150°C, as described in 12.1, for a few hours. Analyze the standards within a month of their preparation. Stabilized polystyrene solutions have been shown to be stable at room temperature for several months.

12.3 *Test for Sample Solution Suitability*—The mass of the polymer injected is typically between 0.05 and 0.5 mg depending on the expected breadth of the molecular weight distribution. Smaller samples are used when the molecular weight distribution is narrower, or the molecular weight is higher, or both. This test method assumes that the mass of the injected polymer is low enough for the hydrodynamic volume of the polymer and the chromatographic separation not to be mass dependent. If the injected sample mass is too high, the peak elution volume and the shape of the chromatogram may be affected and lead to erroneous MW values. If in doubt, it is advisable to rerun an unknown sample or standard at one half its original concentration to ensure that its elution profile is repeatable. When a change is observed, the analysis must be repeated with a lower sample concentration.

13. Performance Requirements

13.1 *Plate Count Number*—The plate count number (*N*) is a dimensionless quantity related to column efficiency and provides an indication of the extent of band broadening. Follow recommendations of the column manufacturer when initially evaluating columns. The plate count number is determined under the same conditions as those used in this test method. For example:

Solvent:	TCB
Temperature:	140°C
Flow rate:	1 cm^3/min
Test solute:	Hexadecane
Concentration:	~0.01 % w/v
Injection volume:	300 µL

For an approximately Guassian-shaped solute peak, the following expression is used to calculate the number of plates (*N*)/m:

$$N/L = 16 \times (1/L) \times (V_r/W)^2 \quad (1)$$

where:

L = total column length, m,
V_r = peak elution volume, mL, or time, min, and
W = peak width in units of volume (mL) or time (min) as determined by measuring the distance between the baseline intercepts of lines drawn tangent to the peak inflection points.

13.1.1 High temperature GPC columns are expected to exceed 10 000 plates/m. Plate counts are monitored regularly and column sets not meeting this performance requirement must be discarded.

13.2 *Resolution*—The resolution (*R*) provides an indication of the component separation and band broadening of a column set. A GPC specific resolution (R_s) of two standard polymers differing in molecular weight values by a factor of ten and having polydispersities of less than 1.1 is defined as:[3]

$$R_s = 2(V_{r2} - V_{r1})/(W_1 + W_2) \quad (2)$$

[3] "Modern Size Exclusion Chromatography—Practice of Gel Permeation and Gel Filtration Chromatography," W.W. Yau, J.J. Kirkland, and D.D. Bly, John Wiley and Sons, 1979.

where:

V_{r1}, V_{r2} = peak elution volume or time of Standards 1 and 2, and

W_1, W_2 = peak widths of Standards 1 and 2 determined as outlined in 13.1.

13.2.1 The two standards are analyzed at a concentration of ≤0.03 % w/v and an injection volume of ≤300 µL. This test method requires that the calculated R_s values equal or exceed 2.0.

NOTE 14—General information regarding plate number and resolution can be found in most textbooks on chromatography.

13.3 *Detector Response*—Practice E685 addresses determination of detector response in Sections 5 and 7. For this test method to be valid, the integrated peak area of the eluted polymer must be directly proportional to the mass of polymer injected. This can be ascertained by injection of different concentrations of the same polyolefin sample.

13.4 *Baseline Stability*—Practice E685 classifies deviations from a perfectly horizontal baseline for a photometric detector as drift and short-term and long-term noise. These deviations must be minimized while retaining a high sample response. Drift, defined as the average slope of the noise envelope over a period of 1 h, is a potential problem when the data handling software is unable to correct for it. Without correction, erroneous results are obtained when the drift exceeds 2 % of the maximum polymer peak signal. Short-term noise, defined as the maximum peak-to-peak amplitude for random variations of the detector signal with a frequency greater than 1 cycle/min, must not exceed 2 % of the maximum polymer signal. Long-term noise, defined as the maximum amplitude for all random variations of the detector signal of frequencies between 0.1 and 1 cycles/min, must not exceed 5 % of the maximum polymer signal.

13.5 *Flow Rate*—Small differences (>0.3 %) in the flow rate between the time of calibration and sample analysis will cause significant, systematic errors in MW values calculated. Users determine the average flow rate of their system by measuring the volume of solvent eluted over a specified time period. When flow rate variations in excess of 0.3 % are observed, replace or service the pump or correct for flow variations by addition of a flow measuring device or use a flow rate marker such as the air peak or toluene, that is, internal standard.

14. Calibration

14.1 *Selection of Polystyrene Standards*—Prepare solutions of polystyrene calibration standards as outlined in 12.2. A minimum number of three standards per decade of MW need to be used to adequately define the calibration curve over the MW range covered by the columns.

14.2 *Injection of Polystyrene Standards*—Make injections with the instrument autosampler at the same temperature as the column oven temperature, that is, typically 140°C. Add the internal standard, if used, to the solution prior to injection. The injection volumes of all standards must be identical regardless of concentration. The recommended volume is ≤300 µL for columns with internal diameters of 0.8 to 1.0 cm. For diameters of <0.8 cm, use a smaller volume.

14.3 *Data Acquisition*—Determine the elution peak maxima and the corresponding elution volumes (or times) for the various polystyrene standards (and internal standard). Usually, this is accomplished with a data acquisition software package.

14.4 *Generation of Calibration Curve*—Convert the polystyrene peak molecular weights to polyolefin molecular weights. This is accomplished using the following equation:

$$log_{10}M_2 = \{1/(1+\alpha_2)\} \times log_{10}(K_1/K_2) + \{(1+\alpha_1)/(1+\alpha_2)\} \times log_{10}M_1 \quad (3)$$

where:

M_2 = the molecular weight of the polyolefin,

α_2, K_2 and α_1, K_1 = Mark-Houwink constants for the polyolefin and polystyrene, respectively, and

M_1 = molecular weight of the polystyrene. The above equation is derived from the empirical Mark-Houwink equation:

$$[\eta] = K \times M^{\alpha} \quad (4)$$

where:

$[\eta]$ = intrinsic viscosity and the universal calibration concept[4] which states that the product of intrinsic viscosity and molecular weight for any polymer is proportional to its hydrodynamic volume, which in turn defines the elution volume of the polymer. Values for *K*'s and α's may be determined with an on-line viscometer or obtained from the literature. The following values are recommended for polystyrene (PS), polyethylene (PE), and polypropylene (PP) in TCB at 140°C:

$$K_{PS} = 19 \times 10^{-3}\ mL/g \quad \alpha_{PS} = 0.655 \quad (5)$$

$$K_{PE} = 39 \times 10^{-3}\ mL/g \quad \alpha_{PE} = 0.725$$

$$K_{PP} = 19 \times 10^{-3}\ mL/g \quad \alpha_{PP} = 0.725$$

14.4.1 Generate the polyolefin GPC calibration curve by plotting the logarithm of the peak molecular weight values versus the measured peak elution volumes (or times). The calibration curve generally assumes an *s*-shape that asymptotically approaches total permeation at low *MW* and total exclusion at high *MW*. It is recommended that a third or fifth order polynomial be used to fit the calibration data. A number of software packages are available to do this.

15. Procedure

15.1 *Preparation for Analysis*—Prepare polymer sample solutions as described in Section 12. An internal standard is added to each sample solution before injection. Prepare the apparatus and complete the performance requirements in Section 13.

NOTE 15—Alternatively, a "stock" solution containing an internal standard for monitoring eluent flow rate may be used to prepare the sample solutions.

15.2 *Injection of Sample Solutions*—Following guidelines described in 14.2, the injection volume shall be identical to that

[4] Z. Grubistic, R. Rempp, and H. Benoit, *J. Polym. Sci.*, Part B, Vol 5, 753 (1967).

selected for calibration. A sharp increase or "pulse" in back pressure upon injection indicates a serious problem in the GPC system that shall be remedied before continuing. When operated unattended, the system must possess the ability to shut down when a specified maximum pressure, for example 250 MPa, is reached.

15.3 *Baseline Determination*—Satisfy baseline criteria discussed in 13.4. The baseline is assumed to be linear. It is established by averaging the baseline noise before and after the chromatographic envelope and connecting the two with a straight line. If the actual baseline deviates from the generated line due to drift, shift of excessive noise, the analysis should be discarded.

15.4 *Integration Limits*—The establishment of the low elution volume, that is, high *MW* end of the chromatogram is usually straightforward. Here, the baseline is not affected by low *MW* impurities, and the slope of the polymer response tends to be relatively steep.

15.4.1 The establishment of the high elution volume, that is, low *MW* is generally more ambiguous and depends largely upon the presence of peaks from antioxidants and low *MW* impurities as well as the recovery of a stable baseline. Samples frequently exhibit tailing towards low *MW*'s and it is difficult to determine precisely where the chromatogram ends.

15.4.2 It is recommended that the integration limits not fall outside the elution volumes for the highest and lowest calibration standards. Integration below a *MW* of 500 g/mol is not recommended due to rapidly changing *d*n/*d*c values, poor definition of the calibration curve and frequent interference from additive peaks.

15.5 *Data Acquisition*—Data systems and computer software may handle data acquisition differently. Upon acquisition, data usually is handled in discrete rectangular area segments, A_i, or as digitized heights, H_i, by recording the vertical displacement between the chromatogram trace and the baseline at elution volumes, V_i, over designated intervals. A minimum of 40 area segments or heights are required.

15.6 *Flow Rate Correction*—If the GPC system does not contain a continuous flow rate monitor, then the flow rate shall be within ±0.3 % of its value measured at calibration or an internal standard must be used. When an internal standard is used, correct sample elution volumes, V_i', by the following relation:

$$\text{corrected } V_i = V_i' \times (V_{is})/(V_{is})' \quad (6)$$

where:

(V_{is}) and $(V_{is})'$ = the elution volumes of the internal standard measured at calibration and for the sample, respectively.

16. Calculation

16.1 *Tabulation of Data*—The data acquisition software will record either area slices or heights of the chromatogram at designated elution volume intervals. The appropriate values for the molecular weight at each elution volume are obtained from the calibration curve.

16.2 *Calculation of Molecular Weight Averages*—The number-, weight-, and *z*-average molecular weights (M_n, M_w and M_z) are calculated using the recorded data and the following expressions:

$$M_n = \sum_{i=1}^{N} A_i / \sum (A_i / M_i) \quad (7)$$

$$M_w = \sum_{i=1}^{N} (A_i \times M_i) / \sum A_i \quad (8)$$

$$M_z = \sum_{i=1}^{N} (A_i \times M_i^2) / \sum (A_i \times M_i) \quad (9)$$

16.2.1 For a constant elution volume interval, ΔV_i, A_i, and M_i are the chromatographic peak slice area and polyolefin *MW*, respectively associated with the (corrected) elution volume, V_i, while *N* is equal to the number of data points obtained from the chromatogram between the integration limits (see 15.4). An example of this method of calculating the molecular weight averages is given in Test Method D5296. When *N* is sufficiently large, the use of area segments A_i or peak heights H_i will yield equivalent results.

16.3 *Molecular Weight Distributions/Cumulative Weight Fraction Distribution*—Calculate the cumulative distribution by integrating the chromatogram to different elution volumes, that is, molecular weights, using standard numerical integrating procedures, that is, rectangular approximation, and then dividing these areas by the total area under the chromatogram. This area ratio is the cumulative weight fraction, W_i, and equals the weight fraction of the polymer having retention volumes greater than V_i and molecular weights less than M_i.

16.4 *Molecular Weight Distributions/Differential Molecular Weight Distribution*—Determine the weight differential distribution by plotting $\Delta W/\Delta(\log_{10} M)$ versus $\log_{10} M$.[5] When determined correctly, plots of the differential distribution functions versus $\log_{10} M$ obtained using different GPC systems to analyze the same sample must be identical. Derivation of differential and cumulative molecular weight distribution functions can be found in Test Method D5296.[6]

17. Report

17.1 Report the following information:

17.1.1 *Apparatus:*

17.1.1.1 System type,

17.1.1.2 Column types, dimensions, and manufacturer,

17.1.1.3 Operating temperature,

17.1.1.4 Solvent (plus additives and treatment, if any),

17.1.1.5 Solvent flow rate,

17.1.1.6 Internal standard or flow monitor (if used), or both,

17.1.1.7 Injection volume, and

17.1.1.8 Polymer sample solution concentration (mg/mL).

17.1.2 *Plate Count and Resolution:*

17.1.2.1 Plate count, *N* (plates/m),

17.1.2.2 Test solute for plate count,

17.1.2.3 Resolution, R_s,

[5] W.W. Yau and S.W. Fleming, *J. Appl. Pol. Sci.*, Vol 12, 2111 (1968).

[6] These distribution functions can be found in "The Elements of Polymer Science and Engineering," Alfred Rudin, Academic Press, Inc., 1982, as well.

17.1.2.4 Standards used for resolution calculation, and
17.1.2.5 Equation used for resolution calculation.
17.1.3 *Calibration Standards:*
17.1.3.1 Polymer standards,
17.1.3.2 Molecular weight of polymer standards, and
17.1.3.3 Peak retention volumes, V_r.
17.1.4 *Calculated Parameters:*
17.1.4.1 Average molecular weights, and
17.1.4.2 Polydispersity, $D = M_w / M_n$.

18. Precision and Bias

18.1 Table 1 lists repeatability relative standard deviations for M_n and M_w for two linear polyethylene standards based on 30 analyses over a period of two months.

TABLE 1 Repeatability Relative Standard Deviations (RSD) for Linear Polyethylene

Sample	PD	RSD (M_n)	RSD (M_w)
NBS 1475	2.8	4.4	2.0
PE 105K	10.5	5.5	3.5

18.2 Table 2 lists repeatability relative standard deviations for M_n and M_w for two polypropylene materials based on 18 analyses over a period of three days.

TABLE 2 Repeatability Relative Standard Deviations (RSD) for Polypropylene

Sample	PD	RSD (M_n)	RSD (M_w)
Homopolymer PP	4.8	10.3	4.5
Copolymer PP	4.8	12.2	4.3

18.3 The reproducibility of this test method has not been determined. An interlaboratory study (ILS) has been initiated to address this issue, and the standard will be updated once that study is complete.

19. Keywords

19.1 gel permeation chromatography; molecular weight average; molecular weight distribution; polyolefin; size exclusion chromatography

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D6474 -99(2006)) that may impact the use of this standard. (December 15, 2012)

(1) Five-year review conducted with revisions where needed.
(2) Revised Section 18 to note an interlaboratory has been initiated.