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Rubber, raw synthetic — Determination of the molecular-mass distribution of solution polymers by gel permeation chromatography

Caoutchouc synthétique brut — Détermination de la répartition de la masse moléculaire pour les caoutchoucs polymérisés en solution par chromatographie par perméation de gel

Reference number
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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 11344:2004), which has been technically revised by replacing the hazardous *o*-dichlorobenzene with BHT (butylated hydroxy toluene) in the procedure. It also incorporates the Technical Corrigendum ISO 11344:2004/Cor.1:2008.

Rubber, raw synthetic — Determination of the molecular-mass distribution of solution polymers by gel permeation chromatography

WARNING 1 — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

WARNING 2 — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard describes a method for the determination of the molecular mass, expressed as polystyrene, and the molecular-mass distribution of polymers produced in solution which are completely soluble in tetrahydrofuran (THF) and which have a molecular-mass range from 5×10^3 to 1×10^6.

It is not the purpose of this International Standard to explain the theory of gel permeation chromatography.

2 Principle

The molecular components of a polymer are separated on the basis of macromolecule size on a gel permeation column. A known quantity of a dilute solution of the polymer is injected into a stream of solvent, which carries it through the column at a constant rate. The concentration of the separated molecular components in the solvent stream is measured by a suitable detector. Through the use of a calibration curve, both the number-average molecular mass (M_n) and mass-average molecular mass (M_w) of the material analysed can be determined from the retention time and the corresponding concentration.

3 General

3.1 Gel permeation chromatography (GPC), which is also known as size exclusion chromatography (SEC), is a particular type of liquid chromatography which allows the separation of the various components of a polymer based on molecular size.

3.2 The molecules of a polymer do not all have the same mass, but comprise a range of different masses. For this reason, the usual concept of molecular mass is not applicable to polymeric materials. Instead, different average molecular masses are determined as shown in Table 1.

Table 1 — Definitions of various kinds of molecular mass

Mass-average molecular mass M_w	$= \Sigma(N_i M_i^2)/\Sigma(N_i M_i)$ $= \Sigma(A_i M_i)/\Sigma A_i$
Number-average molecular mass M_n	$= \Sigma(M_i N_i)/\Sigma N_i$ $= \Sigma A_i/\Sigma(A_i/M_i)$
z-Average molecular mass M_z	$= \Sigma(N_i M_i^3)/\Sigma(N_i M_i^2)$ $= \Sigma(A_i M_i^2)/\Sigma(A_i M_i)$
Peak molecular mass M_p	Molecular mass at peak maximum
where N_i is the number of molecules having a molecular mass of M_i; A_i is the area of the time-slice that corresponds to molecular mass M_i.	

The molecular-mass distribution is an important parameter in determining the properties of the polymer. It may be represented by the polydispersity D given by

$$D = M_w / M_n$$

NOTE Polymers invariably consist of macromolecules with a range of molecular sizes. Even the so-called monodisperse polystyrenes have a polydispersity of 1,1 compared to a value of 1,0 for a pure compound with a single molecular mass. As the range of molecular sizes present within the polymer increases, so does the polydispersity.

4 Reagents and materials

4.1 Tetrahydrofuran (THF), with or without 2,6-di-*tert*-butyl-4-methylphenol (BHT), solvent for the mobile phase, analytical grade.

4.2 THF containing 2,6-di-*tert*-butyl-4-methylphenol, solvent for sample dissolution, analytical grade (THF containing BHT solution).

The solution of 2,6-di-*tert*-butyl-4-methylphenol (also known as BHT, butylated hydroxytoluene) in THF is commercially available. For the purpose of this International Standard, the solution is called THF containing BHT.

When it is difficult to find this solution in the market, the alternative can be obtained by adding 100 mg to 500 mg of BHT to 1 l of THF. Preparation of this solution is also effective when a noticeable peak is not obtained for BHT.

4.3 Set of certified polystyrene reference standards (minimum 10), with molecular masses in the range 5×10^2 to 1×10^7 (depending on the sample molecular-mass range) and a very narrow molecular-mass distribution ($D < 1{,}10$) (see Table 2 for an example of such a set, available from various chemical suppliers).

Table 2 — Set of polystyrene standards

Standard No.	Actual molecular mass M_i	D (= M_w/M_n)
1	1 030 000	1,05
2	770 000	1,04
3	336 000	1,03
4	210 000	1,03
5	156 000	1,03
6	66 000	1,03
7	30 300	1,03
8	22 000	1,03
9	11 600	1,03
10	7 000	1,04
11	5 050	1,05

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Gel permeation chromatograph, consisting of the components specified in 5.1.1 to 5.1.8.

5.1.1 Solvent reservoir, of sufficient capacity to complete the analysis without refilling.

NOTE A large stock of THF is needed to avoid frequent refills. Changes in the quantity of dissolved air or impurities due to addition of fresh solvent cause significant variations in the refractive index and can also affect the retention time. Air bubbles at the pump head reduce the quantity of solvent pumped (leading to errors in retention volumes and times) and can block the pump if the volume of the air bubbles reach excessive levels. After adding fresh solvent, it takes 2 h to 3 h to obtain a stable baseline.

5.1.2 Automatic online degassing system or **helium sparging of solvent reservoir**, to stabilize the solvent flow, mainly to prevent formation of bubbles in the solvent.

5.1.3 Pump, to ensure that the THF solvent flows at a constant rate, programmable over the range 0,1 ml/min to 2,0 ml/min with a high degree of precision.

5.1.4 Injector or **automatic sampler**, with a 100 mm^3 (100 µl) injection loop.

5.1.5 Columns, packed with regular, rigid, porous spheres. The pore size on the column packing material is expressed either in Angström units (1 Å = 10^{-10} m), molecular weight range or size exclusion limit molecular weight. The packing spheres are made of cross-linked polystyrene, obtained by polymerization of styrene with divinylbenzene. The spheres shall have a nominal diameter in the range 3 µm to 10 µm. The columns are generally 150 mm to 300 mm long. The pore size is selected depending on the range of molecular masses to be analysed.

NOTE 1 Four columns with pore sizes 10^3 Å, 10^4 Å, 10^4 Å and 10^5 Å were used when the repeatability and reproducibility of the method described in this International Standard were determined. The solvent first enters the column with the lowest porosity and exits from the column with the highest porosity. Other suitable columns can be used. These types of column are available from many suppliers.

NOTE 2 The recommended column characteristics are:

— linear range: 1 000 to 400 000 000;

— guaranteed column efficiency: > 2 400 plates for 150 mm long columns and 4 800 plates for 300 mm long columns; this is also known as a number of theoretical plates, N, as shown in Figure 1. The following formula is used to calculate the theoretical plate number:

$$N = 5,54 \times (V_e / W_{1/2})^2$$

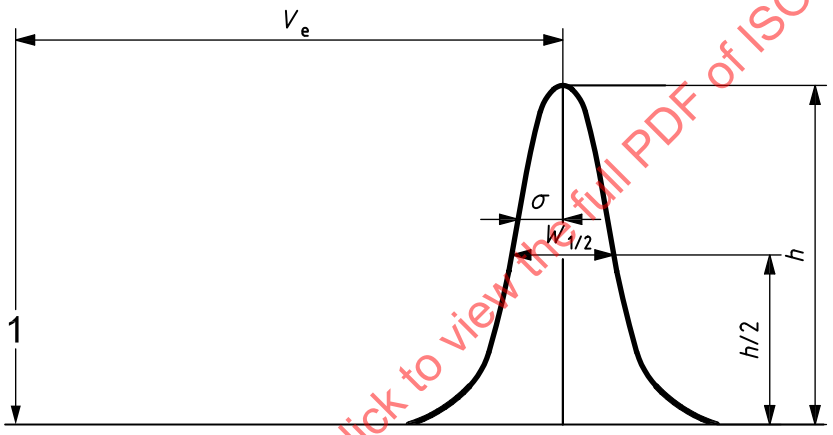
where

V_e is the retention volume to the peak maximum;

$W_{1/2}$ is the peak width at half height — using the same units for V_e and W.

Express the result as the number of theoretical plates of total column length.

— Column arrangement: two to four columns (150 mm to 300 mm long and 4,6 mm to 8,0 mm ID).



Key

1 injection

$$N = \left(\frac{V_e}{\sigma} \right)^2$$

$$= 5,54 \times \left(\frac{V_e}{W_{1/2}} \right)^2$$

Figure 1 — Determination of the number of theoretical plates N by the half-height method

5.1.6 Detector.

Various types of detectors may be used, such as differential refractometer or UV.

5.1.7 Integrator, capable of integrating at least 150 time-slices during the elution of the polymer being analysed.

5.1.8 Personal computer and software, to avoid long and difficult manual calculations.

5.2 PTFE filters, having a pore size of 0,50 µm or 0,45 µm.

5.3 10 cm³ (10 ml) and 250 mm³ (250 µl) syringes.

5.4 Autocollector (optional), with glass vials.

5.5 Mixer.

6 Analytical conditions

Flow rate: 0,2 ml/min to 1,0 ml/min.

Injection volume: 100 mm^3 (100 µl) of solution, or a quantity suitable for the volume of the column used.

The injection volume shall be matched to the set of columns used. The total injection volume shall not exceed 250 µl. The concentration of the sample solution injected shall be 0,1 g/l to 5,0 g/l.

Column temperature: 40 °C - 45 °C.

7 Procedure

7.1 Solvent degassing

Degass 1 dm^3 of solvent under vacuum and/or in an ultrasonic bath for about 30 min.

To obtain a constant baseline, degassing should preferably be done 12 h before use. From time to time, the columns should be flushed, for a period of 8 h, with THF solvent, degassed as specified in this subclause, to remove any peroxides left in the column.

If an automatic online degassing system is available, the degassing operation given in this subclause can be omitted.

7.2 Calibration

7.2.1 Use polystyrene standards (4.3) dissolved in THF containing BHT solution (4.2) for calibration purposes. To ensure constant peak size, weigh out a different amount of each individual standard as a function of its molecular mass, for example 1 g/l [0,025 g in 25 cm^3 of solution (4.2)] for molecular masses around 1 000 000, 5 g/l [0,125 g in 25 cm^3 of solution (4.2)] for molecular masses lower than 30 000. The calibration plot shall cover the entire range of molecular masses present in the polymer being analysed.

7.2.2 Shake the solutions gently.

7.2.3 Filter each solution through a PTFE filter (5.2) attached to a 10 cm^3 syringe.

NOTE The reference standard solutions can be kept in a refrigerator at 6 °C to 7 °C for a maximum of 3 months.

7.2.4 The calibration procedure described in 7.2.4.1 to 7.2.4.5 is given by way of example.

7.2.4.1 Prepare 11 solutions of polystyrene in accordance with Table 3.

Table 3 — Solutions of polystyrene reference standards

Solution No.	Concentration g in 25 cm^3 of BHT solution (4.2)	Actual molecular mass M
1	0,025	1 030 000
2	0,025	770 000
3	0,030	336 000
4	0,050	210 000
5	0,050	156 000
6	0,075	66 000
7	0,125	30 300
8	0,125	22 000
9	0,125	11 600
10	0,125	7 000
11	0,125	5 050

7.2.4.2 When using manual injection, draw off 250 mm^3 (250 µl) from each vial, flush the injection loop and then inject 100 µl. Read off the retention time corresponding to the peak for each standard. With an automatic sampler, follow the manufacturer's instructions. Repeat calibration if necessary.

7.2.4.3 In the case of repeat, average the replicates retention times of BHT averaged over all the runs.

7.2.4.4 Plot the average retention time, in minutes, against the corresponding value of $\log(M_i)$ for each standard and calculate the best-fit line (see Figure 2).

7.2.4.5 The correlation coefficient shall be higher than 0,999 5. If not, repeat the calibration procedure for the standards that are causing imperfect alignment, found by computing the difference between the certified (actual) molecular masses and the molecular masses calculated (see Table 4) using the third-degree polynomial representing the best-fit line in Figure 2.

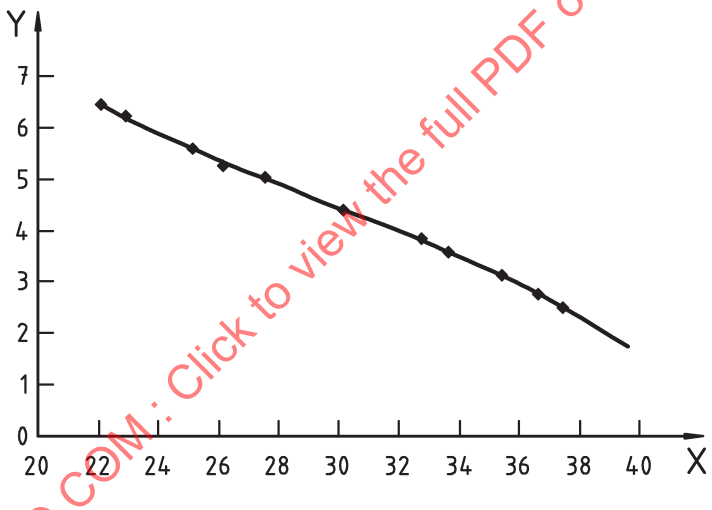
For the data plotted in Figure 2, the best-fit line is given by the following third-degree polynomial:

$$\log(M_i) = 17{,}569\,426\,28 - 1{,}027\,363\,146\,t_i + 0{,}030\,450\,485\,t_i^2 - 0{,}000\,344\,616\,t_i^3$$

For these data, the correlation coefficient is 0,999 53.

Table 4 — Calibration data corresponding to plot in Figure 2

Actual molecular mass M_i	Retention time t_i min	Calculated molecular mass
1 030 000	22,08	1 049 591
770 000	22,89	749 228
336 000	25,15	323 397
210 000	26,15	231 316
156 000	27,58	147 045
66 000	30,18	66 955
30 300	32,76	29 978
22 000	33,68	22 039
11 600	35,46	11 542
7 000	36,64	7 168
5 050	37,47	4 979



Key

X retention time (min)

Y $\log(M_i)$

Figure 2 — Calibration plot

7.3 Preparation of test solution

7.3.1 The test solution concentration specified in 7.3.2 is suitable for most circumstances, but may be varied depending on the actual polymer being tested, the molecular-mass range expected, the volumes of the columns, the type of detector and the volume of solution injected.

7.3.2 Place 0,075 g of the sample in a 50 cm^3 graduated flask and add roughly 35 cm^3 of filtered (see 7.2.3) THF containing BHT solution (4.2).

7.3.3 Agitate the solution gently on a shaker to ensure the polymer has dissolved completely and then make up to 50 cm^3 with filtered THF containing BHT solution.

Shake the solution at room temperature to ensure complete dissolution and homogenization; in the case of samples with a mean molar mass of less than 700 000 g/mol, a magnetic stirrer may be used. The use

of ultrasound is not permitted because of the risk of degradation. The use of heat should preferably also be avoided. Exceptions, e.g. for PVC, shall be justified in the test report.

Remove insoluble foreign matter from the injection solution by suitable methods, e.g. ultracentrifugation, filtration or membrane filtration. Even if the solution appears clear to the eye, filtration through membrane filters is always recommended. If the sample contains insoluble polymer particles, e.g. microgel, the test report shall expressly point out that the GPC results refer only to the soluble components. The appearance of such samples shall be described.

7.4 Analysis

7.4.1 Pass solvent through the columns (flow rate 0,2 ml/min to 1,0 ml/min) until the baseline stabilizes.

NOTE With some detectors and column sets, this might take up to 7 h.

7.4.2 When the baseline has stabilized, run the analysis as described below, under the conditions given in Clause 6.

7.4.2.1 Using a syringe, draw off 10 cm^3 of the test solution prepared in 7.3.

7.4.2.2 Filter it through a PTFE filter (5.2) directly into a vial.

7.4.2.3 When using manual injection, inject 250 mm^3 (250 µl) to flush the injection loop and then inject 100 µl to start the analysis. Repeat measurement if necessary.

The retention volume is very sensitive to the quantity of polymer injected. If anomalous peak shapes are observed with a particular sample, the concentration of the injection solution should be changed (increased or decreased) until the variation in the calculated M_w-value has been reduced to below 5 %.

7.4.2.4 With an automatic sampler, follow the manufacturer's instructions. Repeat the procedure if necessary.

7.4.2.5 The molecular parameters will normally be computed by an integrator (5.1.7), using the data stored from the calibration procedure.

NOTE The calibration curve is used by the instrument software to calculate the sample parameters to be determined.

8 Expression of results

8.1 Results are acceptable if the retention time of the BHT marker is within ± 30 s of the value obtained during the calibration stage (see 7.2). If this is not achieved, the columns shall be cleaned by flushing fresh solvent through them for at least 3 h and then the retention time of the BHT marker is determined again.

8.2 Should the anomalous retention time be confirmed, the system needs recalibration using the polystyrene standards (see 7.2).

8.3 The instrument software allows calculation of a great deal of information about the molecular-mass distribution (see Annex A).

8.4 The following information shall be reported:

a) the mass-average molecular mass M_w;

b) the number-average molecular mass M_n;

c) the z-average molecular mass M_z;

d) the polydispersity D (= M_w/M_n);

e) the peak molecular mass M_p;

f) the percentage peak area corresponding to the molecular fractions present.

8.5 When suitable software is unavailable, the results can be obtained by using the procedure given in Annex B (manual procedure).

8.6 Comparison of the results obtained by the automatic procedure (software) and the manual procedure is shown in Annex C.

9 Precision

See Annex D.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 11344;

b) all details necessary for the identification of the sample analysed;

c) the type and number of columns used;

d) the type of detector used;

e) the set of polystyrene standards used;

f) the molecular-mass results obtained, particularly:

   1) the mass-average molecular mass M_w,

   2) the number-average molecular mass M_n,

   3) the polydispersity D (= M_w/M_n);

g) any deviations from the procedure specified;

h) any operation not included in this International Standard or regarded as optional;

i) the date of the analysis;

j) the procedure used (software or manual).

Annex A
(informative)

Molecular-mass parameters determined by instrumental software

A.1 General

The instrument software allows calculation of molecular-mass parameters that characterize a polymer from its GPC chromatogram and the use of a specific calibration curve obtained in accordance with the procedure outlined in 7.2.

A.2 Chromatogram acquisition

Set the chromatogram acquisition parameters and start the analysis. During the analysis, the GPC curve is shown in real time on the computer screen (see Figure A.1).

A.3 Chromatogram analysis

A.3.1 At the end of the run (run time 55,0 min), the molecular parameters are computed by the software, after setting the following parameters:

a) A baseline time-frame that includes all the peaks in the chromatogram.

NOTE 1 In the example in Figure A.2, the baseline time-frame is from 22,0 min to 29,5 min.

b) An appropriate number of data points around the baseline time-frame extremes that allow the software to draw a straight line from one extreme to the other to eliminate noise.

c) The retention time of the peak of BHT, with the maximum variation range (window) allowed to consider the chromatogram acceptable.

NOTE 2 In the example in Figure A.2, the retention time of the BHT marker peak is 45,41 min, with a time tolerance (window) of ± 30 s. The tolerance of ± 30 s is the limit imposed for the calculation. However, in practice a narrower tolerance of ± 20 s is used.

d) The start and end points for the integration of the chromatogram.

NOTE 3 In the example in Figure A.2, the start point is 23,54 min and the end point is 28,30 min.

e) The total number of chromatogram time-slices between the start and end points defined in d).

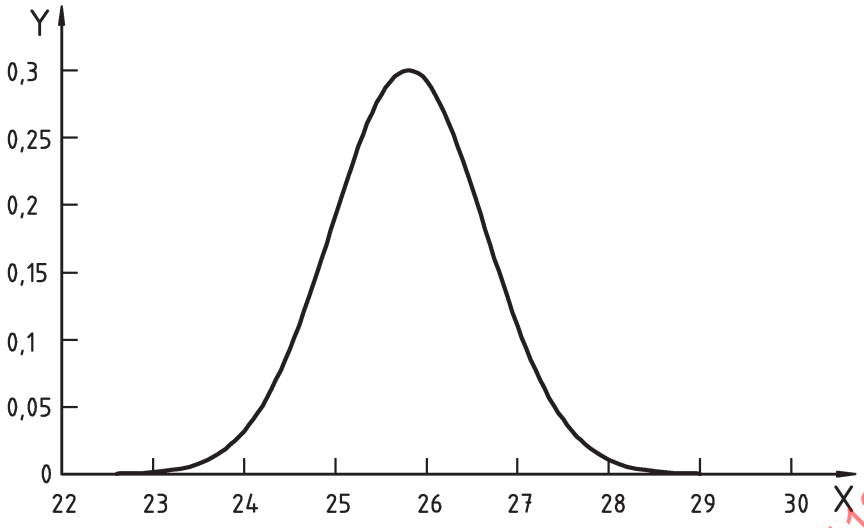
NOTE 4 In the example in Figure A.2, the number of time-slices is equal to 30; generally, the optimum number of time-slices is 150.

Figure A.2 shows the chromatogram after slicing. Table A.1 shows all the parameters of each time-slice making up the chromatogram.

Table A.1 — GPC chromatogram time-slices

Retention time t_i (at centre of time-slice) min	**Area of time-slice**	M_i [a]
23,621 9	4 306	562 357
23,780 5	4 043	529 530
23,939 1	3 862	498 958
24,097 7	4 875	470 462
24,256 3	15 380	443 878
24,414 9	72 628	419 057
24,573 5	268 561	395 863
24,732 1	708 384	374 171
24,890 7	1 395 968	353 868
25,049 3	2 135 893	334 849
25,207 9	2 553 702	317 020
25,366 4	2 316 451	300 303
25,525 0	1 578 818	284 597
25,683 6	859 802	269 839
25,842 2	420 762	255 962
26,000 8	210 947	242 904
26,159 4	119 798	230 607
26,318 0	78 122	219 019
26,476 6	55 340	208 092
26,635 2	40 511	197 780
26,793 8	30 408	188 043
26,952 4	23 412	178 842
27,111 0	18 926	170 142
27,269 5	15 512	161 916
27,428 1	12 895	154 123
27,586 7	10 632	146 741
27,745 3	8 459	139 744
27,903 9	6 722	133 107
28,062 5	5 232	126 809
28,221 1	3 764	120 829

[a] Determined from calibration curve (see Figure 2).

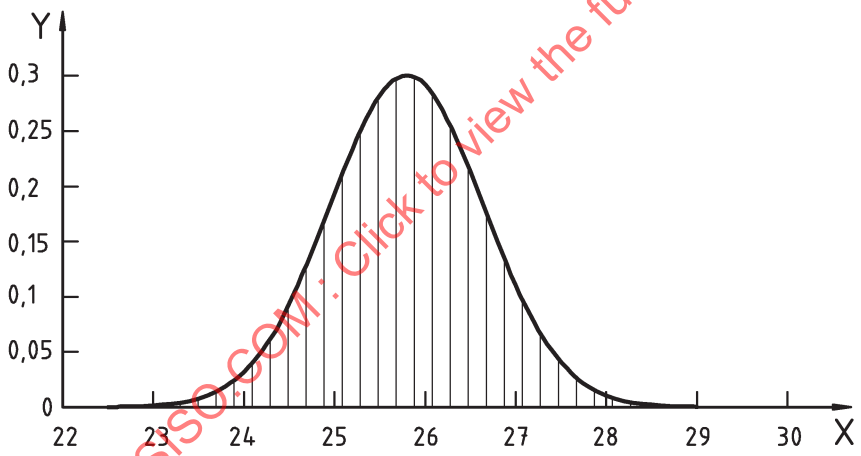


Key

X retention time (min)

Y detector response

Figure A.1 — GPC chromatogram acquisition



Key

X retention time (min)

Y detector response

Baseline:		Start: 22,00	End: 29,50
Number of data points in baseline region:		44	
Reference peak time:	45,41 min	Window: 30 s	
Reference peak found at:	45,42 min		
Processing:		Start: 23,54 min	End: 28,30 min
Number of slices:	30		

Figure A.2 — GPC chromatogram analysis

A.4 Results of GPC analysis

The final GPC analysis results are shown in Table A.2.

Table A.2 — Results of GPC analysis

Number-average molecular mass M_n	316 343	Intrinsic viscosity	1,146 0
Mass-average molecular mass M_w	322 380	Polydispersity M_w/M_n	1,019 1
z-Average molecular mass M_z	327 646	M_z/M_w	1,016 3
Peak molecular mass M_p	323 115		

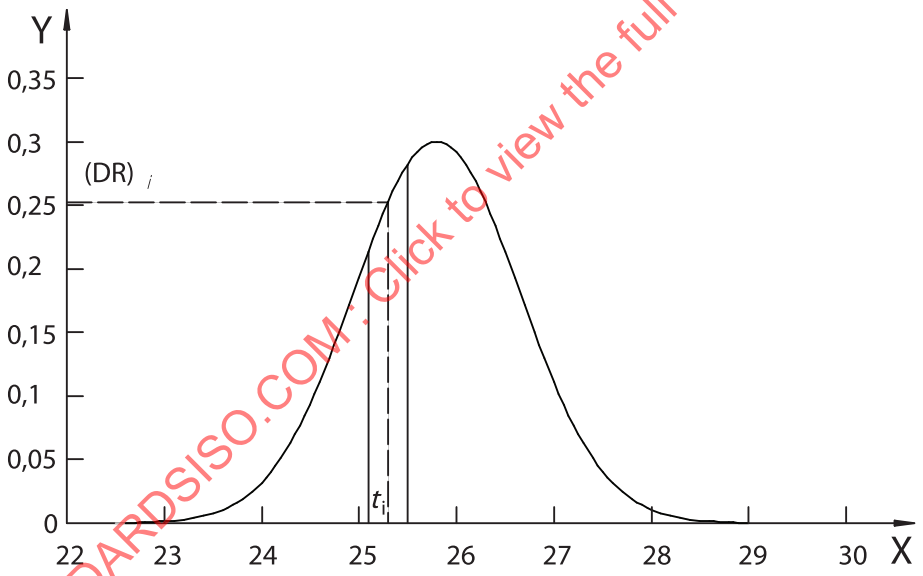
Annex B
(informative)

Calculation of molecular-mass parameters by manual procedure

B.1 When software is not available, the results may be obtained by using the procedure given in B.2 to B.8.

B.2 Divide the chromatogram shown in Figure A.1, which is a plot of detector response against retention time in minutes, into equal time-slices. In the example given below, the chromatogram has been divided into 16 slices (the difference between the retention times of adjacent slices being equal to 0,158 6 min, corresponding to 9 516 ms).

B.3 For each of the time-slices, draw a vertical line from the central point on the retention time axis to intersect the chromatogram curve. Determine the detector response at the point of intersection and multiply this value by 1 000 to give $(DR)_i$ (see Figure B.1 for an example).



Key

X retention time (min)

Y detector response

Figure B.1 — Example of manual chromatogram analysis

B.4 Record the time, in minutes, to the central point of each time-slice and convert the value into milliseconds.

B.5 For each of these times, calculate the value of $\log(M_i)$, using the formula, given in 7.2.4.5, of the calibration curve shown in Figure 1:

$$\log(M_i) = 17{,}569\,426\,28 - 1{,}027\,363\,146\,t_i + 0{,}030\,450\,485\,t_i^2 - 0{,}000\,344\,616\,t_i^3$$

B.6 Calculate the area A_i of each time-slice as follows:

$$A_i = \Delta(t_i) \times (\mathrm{DR})_i$$

where

$\Delta(t_i)$ is the time difference between slices (= 9 516 ms);

$(\mathrm{DR})_i$ is the detector response.

B.7 For each slice, determine the values of A_i/M_i and A_iM_i as shown in Table B.2.

B.8 By using the following formulae (see Table 1), calculate the number-average molecular mass, the mass-average molecular mass and the polydispersity, as follows:

$$M_\mathrm{n} = \frac{\sum A_i}{\sum \frac{A_i}{M_i}} = 318\ 085$$

$$M_\mathrm{w} = \frac{\sum (A_i M_i)}{\sum A_i} = 322\ 666$$

Polydispersity $D = M_\mathrm{w}/M_\mathrm{n} = 1{,}014$

Table B.1 — GPC chromatogram time-slices

Slice	**Retention time** t_i (at centre of time-slice) min	**Detector response** $(\mathrm{DR})_i$	**Area of time-slice** A_i	M_i (determined in B.5)
1	24,414 9	7,75	73 749	419 057
2	24,573 5	27,46	261 309	395 863
3	24,732 1	72,60	690 862	374 171
4	24,890 7	133,56	1 270 957	353 868
5	25,049 3	218,19	2 076 296	334 849
6	25,207 9	268,22	2 552 382	317 020
7	25,366 4	247,61	2 356 257	300 303
8	25,525 0	164,38	1 564 240	284 597
9	25,683 6	89,36	850 350	269 839
10	25,842 2	46,10	438 688	255 962
11	26,000 8	23,05	219 344	242 904
12	26,159 4	12,32	117 237	230 607
13	26,318 0	7,45	70 894	219 019
14	26,476 6	5,32	50 625	208 092
15	26,635 2	4,26	40 538	197 780
16	26,793 8	2,84	27 025	188 043
NOTE $\Delta(t_i)$ = 0,158 6 min = 9 516 ms.				

Table B.2 — Time-slice parameters

Slice	Area of time-slice A_i (from Table B.1)	M_i (from Table B.1)	A_i/M_i	$A_iM_i \times 10^{10}$
1	73 749	419 057	0,176 0	3,090 5
2	261 309	395 863	0,660 1	10,344 2
3	690 862	374 171	1,846 4	25,850 0
4	1 270 957	353 868	3,591 6	44,975 1
5	2 076 296	334 849	6,200 7	69,524 5
6	2 552 382	317 020	8,051 2	80,915 6
7	2 356 257	300 303	7,846 3	70,759 1
8	1 564 240	284 597	5,496 3	44,517 8
9	850 350	269 839	3,151 3	22,945 7
10	438 688	255 962	1,713 9	11,228 7
11	219 344	242 904	0,903 0	5,327 9
12	117 237	230 607	0,508 4	2,703 5
13	70 894	219 019	0,323 7	1,552 7
14	50 625	208 092	0,243 3	1,053 4
15	40 538	197 780	0,205 0	0,801 7
16	27 025	188 043	0,143 7	0,508 1
Totals	**12 660 753**	**—**	**41,060 8**	**396,099 1**