

BS EN ISO 16014-5:2012



BSI Standards Publication

Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography

Part 5: Method using light-scattering detection (ISO 16014-5:2012)

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 65053 6

ICS 83.080.01

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2012.

Amendments issued since publication

Date	Text affected
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English Version

Plastics - Determination of average molecular mass and
molecular mass distribution of polymers using size-exclusion
chromatography - Part 5: Method using light-scattering detection
(ISO 16014-5:2012)

Plastiques - Détermination de la masse moléculaire
moyenne et de la distribution des masses moléculaires de
polymères par chromatographie d'exclusion stérique -
Partie 5: Méthode utilisant la détection par diffusion
lumineuse (ISO 16014-5:2012)

Kunststoffe - Bestimmung der durchschnittlichen Molmasse
und der Molmassenverteilung von Polymeren mittels
Gelpermeationschromatographie - Teil 5:
Lichtstreuendetektionsverfahren (ISO 16014-5:2012)

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Foreword

This document (EN ISO 16014-5:2012) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by January 2013.

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The text of ISO 16014-5:2012 has been approved by CEN as a EN ISO 16014-5:2012 without any modification.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16014-5 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 16014 consists of the following parts, under the general title *Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography*:

- *Part 1: General principles*
- *Part 2: Universal calibration method*
- *Part 3: Low-temperature method*
- *Part 4: High-temperature method*
- *Part 5: Method using light-scattering detection*

Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography —

Part 5: Method using light-scattering detection

1 Scope

This part of ISO 16014 specifies a general method for determining the average molecular mass and the molecular mass distribution of polymers using SEC-LS, i.e. size-exclusion chromatography coupled with light-scattering detection. The average molecular mass and the molecular mass distribution are calculated from molecular mass data and mass concentrations determined continuously with elution time. The molecular mass at each elution time is determined absolutely by combining a light-scattering detector with a concentration-sensitive detector. Therefore, SEC-LS is classified as an absolute method.

For the applicability of the method, see ISO 16014-1:2012, Clause A.1.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 16014-1:2012, *Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography — Part 1: General principles*

ISO 16014-2, *Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography — Part 2: Universal calibration method*

ISO 16014-3:2012, *Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography — Part 3: Low-temperature method*

ISO 16014-4:2012, *Plastics — Determination of average molecular mass and molecular mass distribution of polymers using size-exclusion chromatography — Part 4: High-temperature method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 16014-1 and the following apply.

3.1

light-scattering detection

LS detection

a technique for determining the mass or size of polymer molecules in solution by measuring the light scattered by the polymer molecules

3.2 refractive index increment

dn/dc

rate of change of the refractive index n of a polymer solution as a function of the mass concentration c

NOTE 1 It is also called the “specific refractive index increment” in the literature.

NOTE 2 The limiting value of dn/dc at zero concentration is commonly used in light scattering.

3.3 L-point

measured data point of a low molecular mass compound on the graph of molecular mass vs. elution time used for justification of the polynomial fit of the calibration curve and/or construction of the calibration curve.

NOTE In the lower molecular mass region, the LS signal is too low to calculate molecular mass. Therefore the measurement of the L-point is needed for justification or construction of the molecular mass calibration curve for the whole range of molecular mass. The L-point is determined by measuring an oligomer of the polymer or an organic compound with a similar chemical structure to the oligomer.

4 Symbols

R_g	radius of gyration of a polymer molecule in solution	nm
A_2	second virial coefficient for a polymer molecule in solution	$\text{cm}^3 \cdot \text{mol} \cdot \text{g}^{-2}$
c	mass concentration of polymer in solution	$\text{g} \cdot \text{cm}^{-3}$
dn/dc	refractive index increment	$\text{cm} \cdot \text{g}^{-1}$
H_i	excess signal intensity of a concentration detector at the i th elution time	
$I_{LS,i}$	excess signal intensity of scattered light at the i th elution time	
V_e	volume eluted during data acquisition time (interval)	cm^3

5 Principle

5.1 SEC

For a discussion of size-exclusion chromatography in general, see ISO 16014-1:2012, Clause 4.

5.2 Light-scattering SEC

In SEC-LS, polymer molecules eluted from the SEC columns are irradiated by a beam of monochromatic visible light. The light scattered by the molecules is continuously detected by a light-scattering detector. Since the eluate is a dilute polymer solution, the intensity of the scattered light is approximately proportional to the product of the molecular mass and the mass concentration of the polymer molecules. The scattered-light intensity divided by the concentration therefore gives the molecular mass at a particular elution time. The values of the molecular mass and the mass concentration or mass fraction at each elution time are used to calculate the molecular mass distribution and the average molecular mass of the polymer.

6 Reagents

6.1 Eluent

For a general discussion of eluents, see ISO 16014-1:2012, 5.1.

For examples of eluents used for SEC measurements at temperatures below and above 60 °C, see Annex B of ISO 16014-3:2012 and Annex B of ISO 16014-4:2012, respectively.

6.2 Reagent for column evaluation

For examples of low molecular mass compounds used for column evaluation, see ISO 16014-3:2012, 5.2, for measurements at temperatures below 60 °C and ISO 16014-4:2012, 5.2, for those above 60 °C.

6.3 Calibration standards

Since the Rayleigh ratios of toluene and benzene are well-known, these solvents are recommended for determining the calibration constant of the light-scattering detector (see Annex B, Clause B.2).

Aqueous solutions of potassium chloride (KCl) or sodium chloride (NaCl) are used for determining the calibration constant of a refractive index detector. The concentration dependence of the differential refractive index of the solutions is used to calculate the constant.

A low molecular mass, monodisperse polymer is used to determine the delay volume between the light-scattering and concentration-sensitive detectors. This polymer may also be used to calibrate the angular dependence of the detector sensitivity of a multiple-angle light-scattering detector. The radius of gyration R_g of the polymer molecule used to calibrate the detector sensitivity, should preferably be less than 10 nm. A radius of gyration less than 5 nm is desirable. Other compounds with a well-known R_g value may also be used.

Polymer reference materials are used for molecular mass calibration ranges from 20 000 to 50 000.

Low molecular mass organic compounds or oligomers of the polymer in the sample under investigation are used for determining the “L-point”.

6.4 Reagent for flow rate marker

See ISO 16014-1:2012, 5.4.

For examples of compounds suitable for use as a flow rate marker, see ISO 16014-3:2012, 5.4, for measurements at temperatures below 60 °C and ISO 16014-4:2012, 5.4, for those above 60 °C.

6.5 Additives

See ISO 16014-1:2012, 5.5.

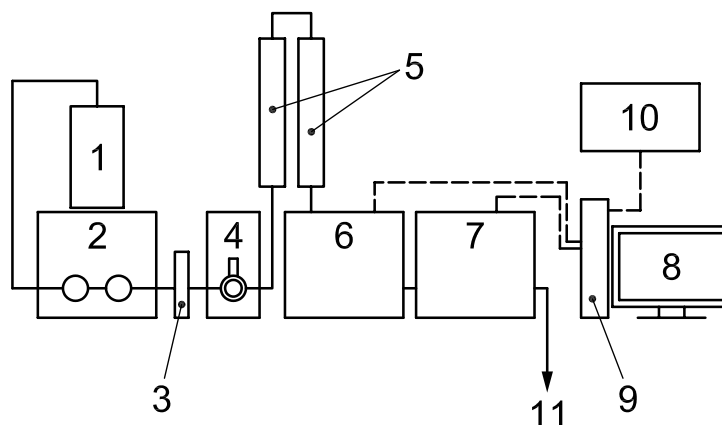
Some examples of additives are given in ISO 16014-3:2012, 5.5, for measurements below 60 °C and ISO 16014-4:2012, 5.5, for those above 60 °C.

7 Apparatus

7.1 General

A typical schematic diagram of an SEC-LS system is shown in Figure 1, which is similar to that shown in ISO 16014-1:2012, Figure 1. The main difference is that a light-scattering detector is connected in series with the concentration-sensitive detector. The light-scattering detector and concentration-sensitive detector may also be connected in parallel. Any component that meets the performance requirements specified for this method may be used.

Either commercially available SEC-LS systems or SEC-LS systems assembled in the laboratory may be used for this method, provided they meet the levels of performance required.



Key

1 eluent reservoir	5 columns	9 computer
2 pump	6 light-scattering detector	10 printer
3 in-line filter	7 concentration-sensitive detector	11 to waste
4 injector	8 display	

Figure 1 — Schematic diagram of a typical SEC-LS system

7.2 Eluent reservoir

See ISO 16014-1:2012, 6.2, and ISO 16014-3:2012, 6.2.

7.3 Pumping system

See ISO 16014-1:2012, 6.3, and ISO 16014-3:2012, 6.3.

7.4 Injector

See ISO 16014-1:2012, 6.4, and ISO 16014-3:2012, 6.4.

7.5 Columns

7.5.1 General

See ISO 16014-1:2012, 6.5.1, ISO 16014-3:2012, 6.5, and ISO 16014-4:2012, 6.5.

7.5.2 Determination of theoretical plate number

See ISO 16014-1:2012, 6.5.2.

7.5.3 Determination of resolution factor

See ISO 16014-1:2012, 6.5.3.

7.5.4 Determination of asymmetry factor

See ISO 16014-1:2012, 6.5.4.

7.6 Detector

7.6.1 Concentration-sensitive detector

See ISO 16014-1:2012, 6.6.

7.6.2 Light-scattering detector

This detector shall continuously monitor the intensity of the light scattered by the eluent coming off the columns. Commercially available light-scattering detectors that may be used include single detectors set at a very low angle and detectors which can be set up at two or more angles.

To avoid band-broadening of the chromatogram, the volume of the flow cell shall be as small as possible.

7.7 Tubing

See ISO 16014-1:2012, 6.7.

7.8 Temperature control

See ISO 16014-1:2012, 6.8.

7.9 Recorder and plotter

See ISO 16014-1:2012, 6.9.

7.10 Data-processing system

See ISO 16014-1:2012, 6.10.

7.11 Other components

See ISO 16014-1:2012, 6.11.

An in-line filter is necessary to remove any particulates which might cause noise (spikes) in the output of the light-scattering detector.

8 Procedure

8.1 Preparation of calibration solutions

Prepare solutions of a monodisperse polymer for determining the delay volume between the two detectors. The concentration of the solutions shall be such that the light-scattering detector and concentration-sensitive detector provide a signal intensity sufficient for data handling. A typical concentration of the polymer is 5 mg/ml to 10 mg/ml for low molecular mass polymers.

These polymer solutions may also be used for correcting or normalizing the sensitivity of the light-scattering detector.

8.2 Preparation of a solution for determining the L-point

A solution for determining the L-point may be prepared, if required, by dissolving appropriate oligomers or other low molecular mass compounds in a suitable solvent. Typically, the concentration of this solution is 1 mg/ml to 5 mg/ml.

8.3 Preparation of sample solutions

See ISO 16014-3:2012, 7.2, for measurements below 60 °C and ISO 16014-4:2012, 7.2, for those above 60 °C.

8.4 Preparation of solutions for column performance evaluation

See ISO 16014-3:2012, 7.3.

8.5 Setting up the apparatus

See ISO 16014-3:2012, 7.4.

8.6 Operating parameters

8.6.1 Flow rate

See ISO 16014-3:2012, 7.5.1.

8.6.2 Injection masses and injection volumes

See ISO 16014-3:2012, 7.5.2.

8.6.3 Column temperature

See ISO 16014-3:2012, 7.5.3.

8.6.4 Detector sensitivity

The signal intensity depends on the amount of sample injected, on the specific refractive index increment dn/dc for a refractive index detector, on the absorbance per unit mass concentration for a UV detector, and on the average molecular mass of the sample for a light-scattering detector. The detector sensitivity shall be set to obtain a strong peak signal for the sample, in order to ensure accurate data handling.

The linear relationship between solute concentration and peak height shall be maintained by keeping the sensitivity at the same setting. Recommended sensitivities are 1×10^{-5} to 9×10^{-4} RI units at full scale for a refractive index detector and around 0,1 to 0,9 absorbance units at full scale for a UV detector.

8.7 Number of determinations

See ISO 16014-3:2012, 7.6.

9 Calibration

9.1 Calibration of concentration-sensitive detector and light-scattering detector

9.1.1 General

Since SEC-LS is an absolute method, the concentration-sensitive and light-scattering detectors shall be properly calibrated so as to give the correct Rayleigh ratio and mass concentration, respectively, at each elution time. When using a refractive index detector as the concentration-sensitive detector, the calibration constants of the refractive index detector and the light-scattering detector shall be determined by one of the three calibration methods given in 9.1.2, 9.1.3 and 9.1.4. If another type of concentration-sensitive detector is being used, such as an ultraviolet/visible detector or an infrared detector, the calibration constants of the concentration-sensitive detector and the light-scattering detector shall be determined by the method given in 9.1.3 or that given in 9.1.4. It should be noted that the relative uncertainty of the calibration constant is directly proportional to that of the molecular mass at each elution time and to that of the average molecular mass.

9.1.2 Calibration method A

In this method, the calibration constant k_{RI} of a refractive index detector is determined by measuring the output I_{RI} of the detector for standard solution(s), such as an aqueous solution of NaCl with known dn/dc and known concentration c , and calculating the constant from the following equation:

$$k_{\text{RI}} = \frac{dn}{dc} \times (c/I_{\text{RI}}) \quad (1)$$

The calibration constant for the light-scattering detector is determined from the ratio of the detector output produced by a calibration sample to the Rayleigh ratio of the calibration sample. Pure filtered toluene is often used as the calibration sample, and is recommended because its Rayleigh ratio is well-known and because it provides a strong scattered-light signal.

Once the constant k_{RI} has been determined, the mass concentration c_i at the i th elution time can be calculated from the following equation:

$$c_i = \frac{k_{\text{RI}}}{(dn/dc)} H_i \quad (2)$$

where H_i is the intensity of the signal from the refractive index detector.

9.1.3 Calibration method B

In this method, the calibration constant for the concentration-sensitive detector is determined from the SEC chromatogram produced by a total injected mass m_{Tot} of a polymer sample of known dn/dc , such as a solution of polystyrene in THF, using the following equation:

$$k_{\text{RI}} = \frac{m_{\text{Tot}}}{V_e} (dn/dc) \frac{1}{\sum_i H_i} \quad (3)$$

where

H_i is the intensity of the signal from the concentration-sensitive detector;

V_e is the volume eluted.

Care shall be taken to ensure that the flow rate remains constant throughout the calibration and subsequent sample measurements. In this method, the mass of polymer injected shall be completely eluted from the columns.

The calibration constant for the light-scattering detector is determined by the method described in 9.1.2 (calibration method A).

9.1.4 Calibration method C

In this method, both light-scattering and concentration SEC chromatograms are produced for a standard polymer solution of known M_w and dn/dc . A combined calibration constant k_c is then calculated from the following equation:

$$k_c = \left(\frac{dn}{dc} \right) M_w \frac{\sum_i H_i}{\sum_i I_{\text{LS},i}} \quad (4)$$

where $I_{\text{LS},i}$ is the intensity of the signal produced from the scattered light observed by the light-scattering detector.

The mass-average molecular mass M_i at the i th elution time can be calculated directly from this constant using the following equation:

$$M_i = \frac{k_c}{(dn/dc)} \frac{I_{LS,i}}{H_i} \quad (5)$$

In the case of a two-angle light-scattering detector, it is possible to correct for the angular dependence of the signal when determining the molecular mass. In such cases, the calibration constant k_c of the concentration-sensitive detector can be calculated from the following equation:

$$k_c = \frac{A_p V_e}{(dn/dc) c V_i} \quad (6)$$

where

- A_p is the total peak area;
- c is the mass concentration of the sample solution injected;
- V_i is the volume injected;
- V_e is the volume eluted.

9.2 Determination of delay volume

Determine the interdetector delay volume by aligning the apex of the peak in the light-scattering chromatogram with that in the concentration chromatogram. If the volume of the tubing forming the delay volume is changed, determine the delay volume again.

9.3 Normalization of detector sensitivity

For a multiple-angle light-scattering detector, the detector sensitivity at different angles shall be determined from the output signal produced at each angle in order to ensure that the same value of the Rayleigh ratio is given for identical scattered-light intensities. This is done by injecting a polymer solution of the kind described in 6.3 and recording the output signal from each detector. The output signal from each detector is then normalized with respect to a standard detector. A detector set at 90° is often chosen for this.

9.4 Determination of refractive index increment

In SEC-LS measurements using light-scattering and refractive index detectors, the value of the refractive index increment dn/dc is required to determine the absolute molecular mass of the polymer sample. The value of dn/dc can be obtained by measurement or from the literature (see Annex B, Clause B.3).

The experimental parameters such as the value of dn/dc , the type of refractive index detector used, the wavelength used, the eluent used, the temperature of the eluate and the method used to calculate dn/dc shall be reported. If the value of dn/dc is determined for a polymer sample by using a reference material with a known value of dn/dc , the method used to calculate the molecular mass of the polymer sample shall also be reported.

10 Data acquisition and processing

10.1 Data acquisition

See ISO 16014-1:2012, 8.1.

10.2 Evaluation of data and correction of chromatograms

See ISO 16014-1:2012, 8.2.

10.3 Data processing

10.3.1 Baseline determination

For the concentration chromatogram, see ISO 16014-1:2012, 8.3.1. The baseline of the LS chromatogram shall be assumed to be a straight line from just before the beginning of the peak to just after the end of the peak.

10.3.2 Determination of calculation range

For the concentration chromatogram, see ISO 16014-1:2012, 8.3.2. The calculation range for the LS chromatogram shall be the same as that specified in 10.3.1 above.

10.3.3 Calculation of signal intensity

Following determination of the baseline and the calculation range (see 10.3.1 and 10.3.2), calculate the signal intensity H_i from the concentration chromatogram and the signal intensity $I_{LS,i}$ from the LS chromatogram at the i th polymer sample elution time.

10.3.4 Calculation of molecular mass

Calculate the molecular mass M_i at the i th elution time using H_i , $I_{LS,i}$, the sample concentration, the injection volume, the flow rate, the instrument calibration constant, the refractive index increment dn/dc , the refractive index of the eluent, etc.

If calibration method A or B was used (see 9.1.2 and 9.1.3), M_i may be calculated from the following equation:

$$M_i = Kc\Delta R_\theta \quad (7)$$

where

ΔR_θ is the excess Rayleigh ratio (see Annex B), which is calculated from $I_{LS,i}$ and the calibration constant for the light-scattering detector;

K is an optical constant defined as follows:

$$K = \frac{4\pi^2 n^2 (dn/dc)^2}{\lambda_0^4 N_A} \quad (8)$$

where

n is the refractive index of the eluent;

λ_0 is the wavelength of the incident light in a vacuum;

N_A is the Avogadro constant.

In the case of high-temperature determinations, all values, such as the sample concentration, the injection volume, the flow rate, the detector calibration constant, the refractive index increment dn/dc and the refractive index of the eluent, shall be corrected or determined at the experimental temperature because of the change in density or volume of the eluent.

10.3.5 Second virial coefficient A_2

To determine the molecular mass of the sample polymer at each elution time, the second virial coefficient A_2 should preferably be taken into account (see Annex B, Clause B.1). However, since the correction for A_2 is often negligible, the calculation using A_2 is not required. If A_2 is used to calculate the molecular mass, however, the value of A_2 shall be reported.

11 Expression of results

11.1 Calibration curve

11.1.1 General

Choose one of the following two methods for constructing the calibration curve or the molecular mass vs. elution time relationship for calculating the average molecular mass and the molecular mass distribution. The first is a simple calculation method and the second is a more detailed and more accurate calculation method.

11.1.2 Method A

Calculate the molecular mass M_i at the i th elution time from the concentration and light-scattering chromatograms. If desirable or required, construct a calibration curve for molecular mass as a function of elution time using a least-squares calculation (see ISO 16014-1:2012, 9.1). Using the calibration curve and concentration chromatogram, calculate the number-average molecular mass M_n and the mass-average molecular mass M_w as described in ISO 16014-1:2012, 9.2. If the polydispersity index M_w/M_n is less than or equal to 1,2, calculate and report the mass-average molecular mass M_w only. If M_w/M_n is larger than 1,2, calculate and report average molecular masses and the molecular mass distribution and also include in the test report the calibration curve used and the concentration and LS chromatograms.

11.1.3 Method B

Calculate the molecular mass M_i at the i th elution time from the concentration and light-scattering chromatograms. Construct a calibration curve for molecular mass as a function of elution time using a least-squares calculation. If the polydispersity index M_w/M_n is less than or equal to 1,2, calculate and report the mass-average molecular mass M_w only.

If M_w/M_n is larger than 1,2, check whether the L-point is in the range covered by the calibration curve or not. If the L-point is in the range covered by the calibration curve, calculate and report average molecular masses, the molecular mass distribution and the distance separating the L-point from the calibration curve and also include in the test report the calibration curve used and the concentration and LS chromatograms.

NOTE The L-point is determined by measuring oligomers of the polymer sample or an organic compound with a similar chemical structure.

If the L-point is not in the range covered by the calibration curve, construct a universal calibration curve using molecular mass standards as described in ISO 16014-2. If the L-point is in the range covered by the universal calibration curve, calculate and report average molecular masses, the molecular mass distribution and the distance separating the L-point from the calibration curve and also include in the test report the calibration curve used and the concentration and LS chromatograms.

If the L-point is not in the range covered by the universal calibration curve, construct another calibration curve which does include the L-point. Using this calibration curve, calculate and report average molecular masses, the molecular mass distribution and the distance separating the L-point from the calibration curve and also include in the test report the calibration curve used and the concentration and LS chromatograms.

NOTE In this case, the universal calibration curve can be constructed without using the Mark-Houwink-Sakurada equation because the molecular masses of the sample polymer and the molecular mass standards at the same elution time are known.

These two options are described as a flow chart in Figures 2 and 3, respectively.

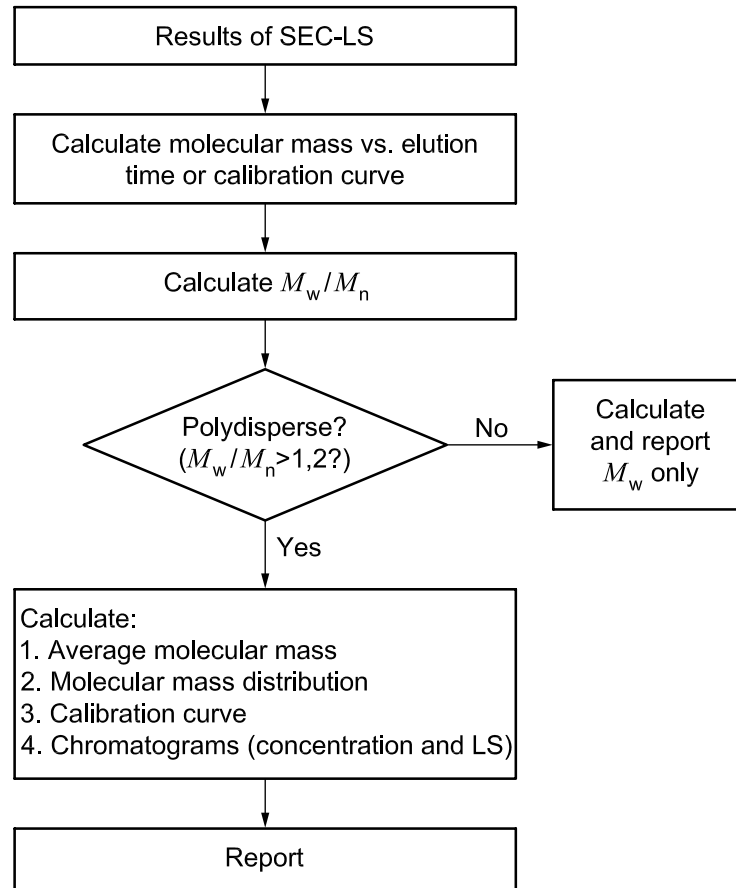


Figure 2 — Flow chart for method A

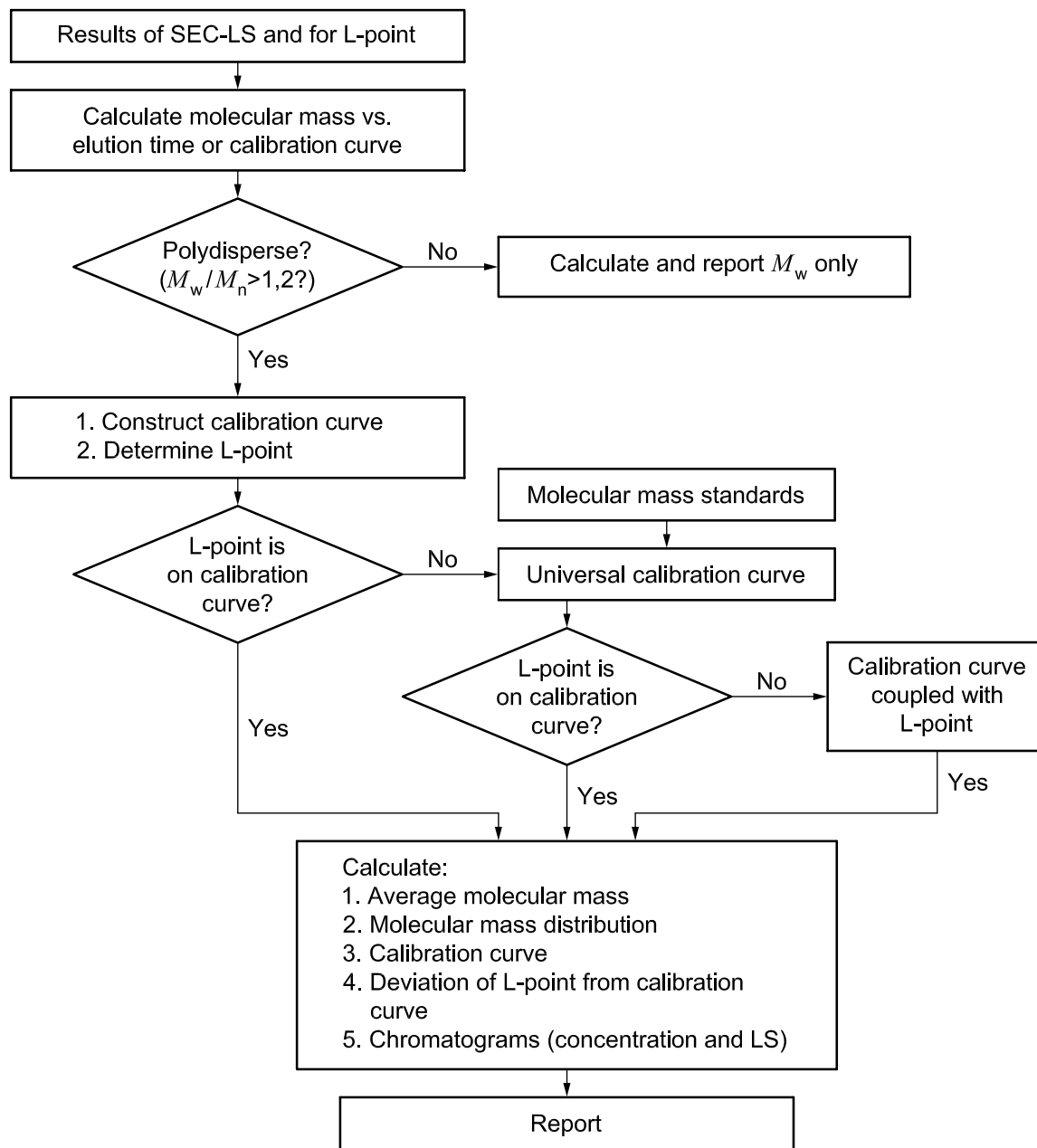


Figure 3 — Flow chart for method B

11.2 Calculation of average molecular mass

See ISO 16014-1:2012, 9.2.

11.3 Differential molecular mass distribution curve

See ISO 16014-1:2012, 9.3.

11.4 Cumulative molecular mass distribution curve

See ISO 16014-1:2012, 9.4.

12 Precision

The precision of this method was determined in a round-robin test using polystyrene samples. Details of the round-robin are given in Annex A.

13 Test report

13.1 General

See ISO 16014-1:2012, 11.1.

13.2 Apparatus and measurement parameters

Include the following information:

- a) the type of SEC-LS apparatus, the model and the manufacturer;
- b) the type of column packing, its particle size and the manufacturer;
- c) the column temperature;
- d) the theoretical plate number, resolution factor and asymmetry factor of the set of columns used, and the low molecular mass standard and narrow molecular mass distribution standard used to determine them;
- e) the eluent, details of any additives, and the value of each flow rate used;
- f) the type of concentration detector, the model and the manufacturer and, if a refractive index detector was used, the wavelength of the light.
- g) the temperature of the concentration detector cell;
- h) the type of light-scattering detector, the model and the manufacturer;
- i) the wavelength of the incident light beam;
- j) the scattering angle(s) used for calculation purposes;
- k) the temperature of the light-scattering cell;
- l) the concentration and volume of the injected polymer sample solution;
- m) the type of data-processing system, the model and the manufacturer;
- n) the version number of the software used.

13.3 Calibration of the system

Include the following information:

- a) the calibration method used, i.e. method A, method B, or method C;
- b) the delay volume;
- c) the concentration and injected volume of the calibration solution;
- d) the properties, such as average molecular mass, of the calibration standards used;
- e) the Rayleigh ratio of the solvent used for calibrating the light-scattering detector;
- f) if used, the value of the second virial coefficient A_2 ;
- g) the value of dn/dc and its source;

- h) the radius of gyration R_g used for normalization of the light-scattering detector.

13.4 Calibration curve

Include the following information:

- a) details of the method used to fit the curve to the data, including the equation;
- b) a copy of the calibration curve itself.

13.5 Results

Include the following:

- a) the characteristic points on the chromatogram (t_a , t_b , t_c , t_d , $t_{1\ 000}$, as applicable);
- b) the calculated average molecular masses M_n , M_w , M_z , and the polydispersity M_w/M_n , indicating the calculation range used (see 10.3.2);
- c) the concentration chromatogram and the LS chromatogram and, in tabular or graphical form, the differential molecular mass distribution and cumulative molecular mass distribution.

Annex A (informative)

Round-robin test

A.1 General

The precision of this method was determined in a round-robin test carried out in 2004 in accordance with ISO 5725-1 [27] and 5725-2 [28].

A.2 Experimental conditions

Test samples, which included polydisperse polystyrene PS-1 and monodisperse polystyrene PS-2, and one monodisperse polystyrene for calibration, were distributed to the participating laboratories. The measurement results were then analysed statistically. The details of the round-robin were as follows:

Polymer samples	Polystyrene PS-1 (polydisperse) Polystyrene PS-1 (monodisperse)
Column size	7,8 mm or 8,0 mm I.D. × 30 cm (in most cases, two columns with a linear calibration curve)
Column packing material	Polystyrene gel
Mobile phase	Tetrahydrofuran
Flow rate	1 ml/min
Column temperature	40 °C
Sample size	In most cases, 1 mg/ml × 0,1 ml
Number of laboratories	20
Types of LS detector used	Multi-angle (15 laboratories) Two angles (2 laboratories) Low angle (3 laboratories)

A.3 Results of round-robin test

The results, expressed as repeatability and reproducibility, are summarized in Table A.1.

Table A.1 — Results of round-robin test for SEC-LS

Polymer	Average values of M_n and M_w ^a	Repeatability, s_r ^a %	Reproducibility, s_R ^a %
PS-1	$M_n = 179\ 000$ (0/20) ^b	2,9	13,2
	$M_w = 455\ 000$ (0/20)	0,8	4,6
PS-2	$M_n = 420\ 000$ (1/20)	0,6	5,7
	$M_w = 427\ 000$ (1/20)	0,5	4,6
^a Outliers were eliminated by Grubbs' and Cochran's methods [26],[28].			
^b Numbers in brackets indicate (outliers)/(total number of laboratories) [28].			

Annex B (informative)

Information on light scattering

B.1 Principle of light scattering [1]

When a beam of monochromatic visible light passes through polymer solution, the light is scattered in all directions by solvent and polymer molecules in the solution. For most polymer solutions, the light scattering is called Rayleigh scattering. Since the intensity of the light scattered by the solution is proportional to the intensity of the primary beam I_0 , the scattering volume V , and the reciprocal of the square of the sample/detector distance r , it is convenient to introduce a reduced intensity, which is classified as the Rayleigh ratio R_θ as follows:

$$R_\theta = \frac{I_\theta r^2}{I_0 V} \quad (\text{B.1})$$

where I_θ is the detected scattered-light intensity at the scattering angle θ . Since it is too difficult to determine R_θ from the geometry of the light-scattering detector, the value of R_θ for pure toluene is frequently used as the reference quantity.

The excess Rayleigh ratio ΔR_θ of a dilute polymer solution is often expressed by the Debye equation:

$$\frac{Kc}{\Delta R_\theta} = \frac{1}{M_w P(\theta)} + 2A_2c + \dots \quad (\text{B.2})$$

where θ is the scattering angle and K is an optical constant which is defined as follows (see 10.3.4):

$$K = \frac{4\pi^2 n^2 (dn/dc)^2}{\lambda_0^4 N_A} \quad (\text{B.3})$$

$P(\theta)$ is a particle-scattering factor describing the angular dependence of the scattered light. For small angles, $P(\theta)$ is given by the following power series:

$$\frac{1}{P(\theta)} = 1 + \frac{16\pi^2 n^2}{3I_0^2} R_g^2 \sin^2\left(\frac{\theta}{2}\right) + \dots \quad (\text{B.4})$$

It should be noted that Equation (B.2) assumes vertically polarized incident light and unpolarized scattered light.

Extrapolating $Kc/\Delta R_\theta$ obtained at different angles and concentrations to $\theta = 0$ and $c = 0$ gives M_w^{-1} . For most light-scattering detectors used in SEC-LS measurement, the extrapolation to $c = 0$ is not applied because the solution eluted from SEC columns is assumed to be sufficiently dilute. Other types of plot, such as the Berry plot, are often used, depending on the nature of the polymer.

It should be noted that the extrapolation to $\theta = 0$ must be done at the small angle region called the Guinier region, i.e.

$$\sin\left(\frac{\theta}{2}\right) < \frac{\lambda_0}{4\pi n R_g} \quad (\text{B.5})$$

B.2 Rayleigh ratio of solvents

The Rayleigh ratio of a pure solvent such as toluene is used for calibrating light-scattering detectors. Table B.1 shows literature values of the Rayleigh ratio for some solvents frequently used. The Rayleigh ratios in the table are for the unpolarized scattered light from vertically polarized incident light of wavelength 632,8 nm.

Table B.1 — Rayleigh ratio of some important solvents ($\lambda_0 = 632,8 \text{ nm}$)

Solvent	Rayleigh ratio cm^{-1}	Temperature $^{\circ}\text{C}$	Reference
Benzene	$12,63 \times 10^{-6}$	23	[2]
	$11,84 \times 10^{-6}$	22	[3]
Toluene	$14,06 \times 10^{-6}$	23	[2]
	$13,59 \times 10^{-6}$	22	[3]
Methanol	$2,85 \times 10^{-6}$	23	[2]
	$2,71 \times 10^{-6}$	22	[3]
Water	$0,90 \times 10^{-6}$	23	[2]
	$0,92 \times 10^{-6}$	22	[3]

It should be noted that the values at the different temperatures are somewhat different from each other. The difference directly affects the results of average molecular masses.

B.3 Refractive index increment dn/dc

Since the intensity of the scattered light is proportional to the square of dn/dc , it should be noted that accurate results require an accurate dn/dc value. The dn/dc value used in SEC-LS measurements can be obtained by one of the following ways:

- 1) measurement of dn/dc using a refractive index detector in the batch mode;
- 2) measurement and calculation of dn/dc using a known injected mass and known calibration constant of the refractive index detector connected to an SEC-LS or ordinary SEC apparatus;
- 3) measurement and calculation of dn/dc using a known injected mass and known dn/dc of a reference material;
- 4) quotation from the literature.

The refractive index increment dn/dc is defined as the differential coefficient of refractive index of a polymer solution as a function of mass concentration c . It is also called the “specific refractive index increment” in the literature. The limiting value of dn/dc at zero concentration is commonly used in light-scattering measurements.

There are two equations for calculating dn/dc experimentally. In the first, the value of dn/dc is given as the limiting value of Δn at zero concentration:

$$dn/dc = \lim_{c \rightarrow 0} \frac{\Delta n}{c} \quad (\text{B.6})$$

where c is the mass concentration of a polymer solution and Δn is the difference in refractive index between the solution and solvent. In the other equation, dn/dc is expressed as follows:

$$dn/dc = \text{the slope of } \Delta n \text{ as a function of } c.$$

The values of dn/dc for some important polymers are shown in Table B.2.

Table B.2 — dn/dc values for some polymers ($\lambda_0 = 632,8$ nm)

Polymer	Solvent ^a	Temperature °C	dn/dc ml/g	Reference
Polyethylene	1-Chloronaphthalene	135	-0,183	[5], [10]
Polyethylene (PE-HD)	ODCB	145	-0,056 ^b	[4], [11]
Polyethylene (PE-HD)	TCB	135	-0,104	[4], [10]
Polypropylene	1-Chloronaphthalene	135	-0,177	[5], [16]
Polypropylene	TCB	135	-0,105	[5], [16]
Polybutadiene	THF	26	0,129 5	[4], [6]
Polyisoprene	THF	20	0,124	[5], [13]
Polystyrene	DMF	20	0,165	[5], [17]
Polystyrene	THF	25	0,186	[4], [18]
Polystyrene	Chloroform		0,155	[4], [19]
Poly(vinyl chloride)	THF	23	0,101	[4], [6]
Poly(vinyl acetate)	THF	25	0,051 7	[4], [21]
Poly(methyl methacrylate)	THF	25	0,086	[5], [14]
Poly(methyl methacrylate)	HFIP	25	0,19	[4], [15]
Nylon 6	HFIP	25	0,237 5	[4], [7]
Nylon 66	HFIP	25	0,241	[4], [8]
Bisphenol A polycarbonate	THF	23	0,177	[4], [6]
Poly(butylene terephthalate)	HFIP	25	0,236	[5], [9]
Poly(ethylene terephthalate)	HFIP	25	0,243	[5], [9]
Polyurethane	THF	25	0,086 3	[4], [20]
Epoxy resin	THF	23	0,187	[4], [6]
Poly(ethylene oxide)	Methanol	25	0,142	[5], [12]
Poly(acrylic acid)	THF	23	0,099 4	[4], [6]

^a THF: tetrahydrofuran, HFIP: 1,1,1,3,3,3-hexafluoroisopropanol, DMF: *N,N*-dimethylformamide, ODCB: 1,2-dichlorobenzene, TCB: 1,2,4-trichlorobenzene.

^b Value uncertain. Reference [10] gives -0,078.

B.4 Relation between radius of gyration R_g and molecular mass M

The radius of gyration of a polystyrene molecule is often used for normalizing a light-scattering detector. The value of R_g for polystyrene in tetrahydrofuran can be calculated from the following experimentally determined equation:

$$R_g = 2,45 \times 10^{-2} M^{0,546} \quad (\text{nm}) \quad (\text{B.7})$$

B.5 Refractive index of solvents

The values of the refractive indices of some important solvents are shown in Table B.3.

Table B.3 — Refractive indices of some important solvents

Solvent	Refractive index	Wavelength nm	Temperature °C	Reference
Chloroform	1,440 7	633	25	[23]
1-Chloronaphthalene	1,532 3	633	140	[23]
1,2-Dichlorobenzene	1,551	589	25	[5]
<i>N,N</i> -Dimethylformamide (DMF)	1,430 5	633	25	[23]
Dimethyl sulfoxide (DMSO)	1,477 3	633	25	[23]
1,1,1,3,3,3-Hexafluoroisopropanol (HFIP)	1,275 2	589	25	[1]
Methanol	1,372 4	633	25	[23]
1-Methyl-2-pyrrolidone (NMP)	1,467 5	589	25	[24]
Tetrahydrofuran (THF)	1,405	633	25	[23]
Toluene	1,489 8	633	25	[25]
1,2,4-Trichlorobenzene (TCB)	1,502	633	135	[23]
Water	1,332 3	633	25	[1]

B.6 Effect of the second virial coefficient A_2 on molecular mass

In calculating the molecular mass in SEC-LS, the correction for the second term in Equation (B.2) involving A_2 should be considered. However, since the concentration of the polymer in the eluent is very low, the second term can often be neglected, i.e. the term is expected to be much smaller than the first one, $1/M_w$.

An example for this term is given below for polystyrene in THF. In this calculation, the following values are used: molecular mass of polystyrene, $1 \times 10^5 \text{ g}\cdot\text{mol}^{-1}$; second virial coefficient A_2 , $5,78 \times 10^{-4} \text{ cm}^3\cdot\text{g}^{-1}\cdot\text{mol}^{-1}$ [3]; sample concentration, 2 mg/ml; injected volume of sample solution, 0,2 ml, region of elution volume of polymer, 5 ml.

The result for the second term is as follows:

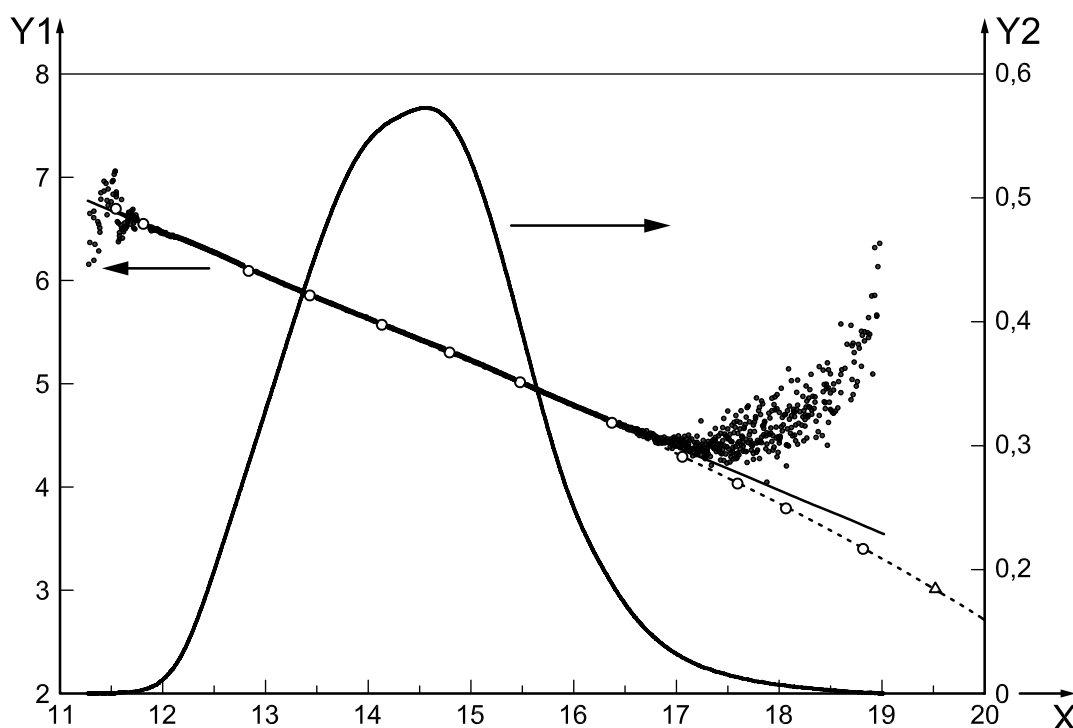
$$2A_2c = 2 \times (5,78 \times 10^{-4}) \times 0,002 \times (0,2/5) = 9,52 \times 10^{-8} \quad (\text{B.8})$$

which is much smaller than the value of the first term $1/M_w (= 1 \times 10^{-5})$.

Annex C (informative)

Calibration curve in low molecular mass range

As Figure C.1 shows, it is often observed that the curve of molecular mass vs. elution time and the calibration curve extrapolated from high molecular mass range deviate from the true calibration curve at low molecular masses. In Figure C.1, the true number-average molecular mass M_n is lower than the calculated M_n based on the observed molecular mass vs. elution time and extrapolated curve. To obtain correct M_n values, it is therefore necessary to construct molecular mass vs. elution time relationships by measuring low molecular mass standards or compounds. The concept including the L-point is introduced to construct more accurate calibration curves. It is also noted that a calibration curve extrapolated to high molecular masses deviates slightly from the true curve.



Key

X elution time (min)

Y1 $\log M$

Y2 concentration $c \times 10^4$ (g/ml)

The straight calibration line obtained by a first-order least-squares fit applied to the measured points (black dots) obtained for the polydisperse polymer sample is shown as a thin solid line. The third-order least-squares molecular mass vs. elution time curve obtained for monodisperse polystyrene samples (open circles) is shown as a thin dotted line. The L-point measured for justification of the polynomial fit to the measured points is indicated by an open triangle at bottom right.

Figure C.1 — Concentration chromatogram (solid curve) and measured molecular mass vs. elution time points (black dots) for the PS-1 sample used in the round-robin test.

During the round-robin test described in Annex A, the number-average molecular mass of the PS-1 sample was also measured by membrane osmometry. Table C.2 compares the values of M_n obtained in various ways.

Table C.1 — Effect of calibration curve at low molecular masses on observed number-average molecular mass M_n of the PS-1 sample used in the round-robin test

	Result of round-robin test (average of 20 measurements)	Calculated using a typical linear calibration curve ^a	Calculated using a third-order calibration curve ^b	By membrane osmometry
M_n	179 000	155 200	141 800	143 000
^a From the data from one of the participating laboratories. ^b Constructed using the molecular mass at peak apex elution time vs. elution time data for monodisperse polystyrene samples.				

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