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

Part 3:
Low-temperature method

Plastiques — Détermination de la masse moléculaire moyenne et de la distribution des masses moléculaires de polymères par chromatographie d'exclusion stérique —

Partie 3: Mesurage aux basses températures

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso .org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 16014-3:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— publication dates of references have been removed;

— molecular mass has been changed to molecular weight according to IUPAC rule.

A list of all parts in the ISO 16014 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

Part 3:
Low-temperature method

1 Scope

This document specifies a method for determining the average molecular weight and the molecular weight distribution of polymers by size-exclusion chromatography (SEC) using an organic eluent at a temperature lower than 60 °C (see Annex A). The average molecular weight and the molecular weight distribution are calculated from a calibration curve prepared using polymer standards. Therefore, this test method is classified as a relative method (see ISO 16014-1).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

According to ISO 16014-1.

5   Reagents

5.1   Eluent.

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

For examples of eluents used for SEC measurements at temperatures < 60 °C, see Annex B.

NOTE   Water is often used for SEC measurements on water-soluble polymers at temperatures < 60 °C, but it is not suitable for use in this method.

5.2   Reagent for column evaluation, according to ISO 16014-1.

There are several low molecular weight compounds that can be used, for example ethylbenzene when tetrahydrofuran is used as eluent or diethylene glycol when N,N-dimethylformamide is used as eluent (see Annex B).

5.3   Molecular weight standards, according to ISO 16014-1.

Some examples of commercially available molecular weight standards are given in ISO 16014-1:2019, Annex B.

5.4   Reagent for flow rate marker (internal standard), according to ISO 16014-1.

It is often very difficult to find a low molecular weight compound suitable for use as a flow rate marker because it should not co-elute with the polymer peak, the system peak or the solvent peak.

Examples of compounds suitable for use as a flow rate marker are sulfur when tetrahydrofuran is used as eluent and ethylbenzene when N,N-dimethylformamide is used as eluent.

5.5   Additives.

LiBr or LiCl, for example, is used as an additive in N,N-dimethylformamide to avoid aggregation of polyacrylonitrile, and sodium trifluoroacetate is added to 1,1,1,3,3,3-hexafluoroisopropanol for SEC measurements on polyamide.

6   Apparatus

6.1   General

A schematic diagram of an SEC system is shown in ISO 16014-1.

Either commercially available or assembled SEC systems may be used, provided they meet the component requirements specified and have the capability to maintain a constant column temperature < 60 °C.

6.2   Eluent reservoir, according to ISO 16014-1.

It is not necessary to keep the reservoir at the same temperature as the columns.

6.3   Pumping system, according to ISO 16014-1.

In order to maintain the flow rate accurate to within ±0,3 %, the pumping system shall be kept at a controlled temperature. It is, however, not necessary to keep the pumping system at the same temperature as the columns.

6.4   Injector, according to ISO 16014-1.

In order to maintain an accurately known flow rate, the injector temperature-control equipment shall be capable of keeping the injector at within ±1 K of the temperature set. It is not necessary to keep the injector at the same temperature as the columns.

6.5   Columns, according to ISO 16014-1.

Organic or inorganic packing materials may be used. There are no limitations on particle size or shape.

The set of columns used shall have a total theoretical plate number > 15 000, and the resolution factor *R* shall be > 1,5 close to the polymer peak. The asymmetry factor shall be within the range 1,00 ± 0,15. The set of columns used should preferably cover the whole range of molecular weights being determined, and the calibration curve shall be as linear as possible (the correlation factor shall be very close to 1). Determination of the theoretical plate number, the resolution factor and the asymmetry factor of the columns shall be carried out as described in ISO 16014-1.

The column temperature-control equipment shall be capable of keeping the columns within ± 0,5 K of the temperature set, to ensure adequate reproducibility of the results.

6.6   Detector, according to ISO 16014-1.

The detector temperature-control equipment shall be capable of keeping the detector within ± 1 K of the temperature set, in order to meet the requirements for flow rate and baseline stability (sensitivity). It is recommended that the columns and detector be kept at the same temperature.

6.7   Tubing, according to ISO 16014-1.

The temperature of the tubing shall be kept constant to ensure that the column performance requirements are met, but it is not necessary to keep the tubing at the same temperature as the column.

6.8   Temperature control.

Refer to 6.4 for the injector temperature-control equipment. And refer to 6.5 and 6.6 for the columns and detector.

One of the important factors in SEC is that all components need to be kept at a constant temperature.

6.9   Recorder and plotter, according to ISO 16014-1.

6.10   Data-processing system, according to ISO 16014-1.

6.11   Other components.

In addition to the components described above, a column guard filter, pressure monitor, pulse damper or related components may be used, if necessary.

7   Procedure

7.1   Preparation of solutions of molecular weight standards

The molecular weight standards used to prepare the calibration curve should preferably be selected so as to cover the range of molecular weights of the polymer being analysed and so that there are at least two standards in each molecular weight decade. Solutions may be prepared which contain more than one narrow molecular weight distribution standard, but only when the standards are perfectly separated from each other on the chromatogram.

A solution of mixed molecular weight standards containing standards of high molecular weight (>1 000 000) might give peaks which are retarded and/or deformed because of the high viscosity of the solution. In such cases, the solutions of high molecular weight standards shall be prepared separately.

If molecular weight standards having the same chemical structure as the polymer being analysed are not available, the calibration curve may be prepared using standards consisting of a different type of polymer, and a universal calibration curve prepared for this different type of polymer (according to ISO 16014-2).

If gentle shaking and/or stirring or heating is required to accelerate dissolution, the time shall be as short as possible to avoid any rupture of the polymer chains.

Filtration of the solutions is recommended to protect the column from clogging. In such cases, membrane filters or sintered-metal filters with a pore size between 0,2 μm and 1 μm shall be used. If solid material is observed on the filter, indicating incomplete dissolution, repeat the dissolution process. If a membrane filter is used, the membrane and backing shall be inert to the solvent being used.

In general, use solutions within 48 h of preparation. However, longer storage times are allowed if the solution is kept in a cool, dark place to prevent polymer degradation and solvent evaporation.

Recommended concentrations for solutions of molecular weight standards are as follows:

$M_p < 5 \times 10^4$ 0,4 mg/cm^3

$5 \times 10^4 \leq M_p < 10^6$ 0,2 mg/cm^3

$10^6 \leq M_p$ 0,1 mg/cm^3

If a viscometric detector is used, higher molecular weight standard concentrations are required in the lower molecular weight region. Sample elution times should preferably be measured at lower concentrations, however.

7.2 Preparation of sample solutions

Prepare sample solutions by weighing accurately 10 mg to 250 mg of sample into a 10 cm^3 to 50 cm^3 flask. Add eluent and, if necessary, an internal standard and dissolve, in the same way as for the molecular weight standard solutions (see 7.1), within 30 min. In general, however, samples with molecular weights > 10^5 have a slow rate of dissolution, and it might be necessary to continue beyond 30 min to ensure complete dissolution. Filtration of solutions is recommended to avoid clogging of the column.

Sample solution concentrations shall not exceed the following limits:

$M_w < 1 \times 10^5$ 5,0 mg/cm^3

$1 \times 10^5 \leq M_w < 10^6$ 2,0 mg/cm^3

$10^6 \leq M_w$ 0,5 mg/cm^3

7.3 Preparation of solutions for column performance evaluation

Prepare a 10 mg/cm^3 solution of a suitable low molecular weight compound to determine the theoretical plate number, asymmetry factor and resolution factor of the set of columns.

7.4 Setting up the apparatus

Place the amount of eluent required for the SEC measurements in the reservoir and degas. Flush all the SEC components, except for the columns, with fresh eluent. Connect the set of columns into the system. Inspect all connections for leakage under the test conditions.

Keep the system at the test conditions (e.g. flow rate, detection sensitivity and temperature) until a flat baseline is obtained, with no drift or noise.

7.5 Operating parameters

7.5.1 Flow rate

A flow rate of approximately 1 cm³/min is recommended for a series of two or three high-performance columns of approximately 30 cm in length and 8 mm in diameter. For high molecular weight and/or shear-sensitive polymers, the flow rate should preferably be reduced so that no chain rupture will occur during elution of the polymer.

7.5.2 Injection masses and injection volumes

The mass of polymer sample and volume of sample solution injected depend on the column dimensions and the detector sensitivity. The optimum sample injection mass has been found experimentally to be approximately 0,005 mg/cm³ of empty column (without packing). The maximum mass injected shall be less than 0,05 mg/cm³ of empty column.

The optimum sample solution injection volume has been found experimentally to be approximately 0,005 cm³/cm³ of empty column. The maximum injection volume shall be < 0,01 cm³/cm³ of empty column.

The injection volumes of the solutions of molecular weight standards shall be the same as for the sample solution.

The injection volume of the solution of low molecular weight compound shall be < 0,005 cm³/cm³ of empty column.

7.5.3 Column temperature

The column temperature should preferably be selected based on the solubility of the sample, the viscosity and boiling point of the eluent, and the ambient temperature.

7.5.4 Detector sensitivity

The signal intensity depends on the amount of sample injected and on the specific refractive index increment d_n/d_c for an RI detector and the absorbance per unit mass concentration for a UV detector. The detector sensitivity should preferably be set to obtain a strong peak signal for the sample, 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 approximately $1 \cdot 10^{-5}$ to $9 \cdot 10^{-4}$ RI units at full scale for a refractive index detector and approximately 0,1 to 0,9 absorbance units at full scale for a UV detector.

7.6 Number of determinations

Carry out at least two sample runs to demonstrate the repeatability of the positions and shapes of the peaks in the chromatogram. If the deviation in the flow rate between the two runs is > 0,3 %, the deviation in M_n > 3 % and the deviation in M_w > 2 %, the measurements shall be repeated.

8 Data acquisition and processing

According to ISO 16014-1.

9 Expression of results

According to ISO 16014-1.

10 Precision

10.1 General

The precision of this method has been determined in several interlaboratory tests carried out between 1995 and 1998 in accordance with ISO 5725-1 and ISO 5725-2.

10.2 Experimental conditions

The test samples, which included three types of polystyrene, one type of poly(methyl methacrylate) and one type of polyacrylonitrile, and the calibration standards of narrow molecular weight distribution were distributed to the participating laboratories by the organizer. The details of the interlaboratory tests are as follows:

1st round-robin (1995)

Polymer samples (three samples)	Polystyrene PS-1
	Polystyrene PS-2
	Polystyrene PS-3
Calibration	14 polystyrene standards
Column packing material	Polystyrene gel
Eluent	Tetrahydrofuran
Column temperature	40 °C
Number of laboratories	13

2nd round-robin (1996)

Polymer sample	Poly(methyl methacrylate) (PMMA)
Calibration	14 polystyrene standards
Column packing material	Polystyrene gel
Eluent	Tetrahydrofuran
Column temperature	40 °C
Number of laboratories	14

3rd round-robin (1997–1998)

Polymer sample		Polyacrylonitrile (PAN)
Calibration	a)	14 polystyrene standards
	b)	14 poly(ethylene glycol) and poly(ethylene oxide) standards

Column packing materials	a) Polystyrene gel
Eluent	b) Poly(vinyl alcohol) gel
Eluent	*N*,*N*-dimethylformamide (additive: 20 mM of LiBr)
Column temperature	40 °C
Number of laboratories	a) 8 for polystyrene gel column
	b) 10 for poly(vinyl alcohol) gel column

10.3 Results of interlaboratory tests

The results, expressed as repeatability and reproducibility, are summarized in Table 1. The raw data are shown in Annex C.

NOTE   Both the repeatability and the reproducibility of this method are sufficient for it to be the standard method except when measurements are carried out using non-ideal SEC conditions as shown in the 3rd round-robin using polystyrene gel columns and *N*,*N*-dimethylformamide, with 20 mM of LiBr as additive, as eluent. In this round-robin, large deviations in s_R were caused by interactions between the polystyrene gel column and the polymer sample and/or the standards used for calibration. Therefore, this test method cannot be used for polymers that exibit appreciable secondary effects, such as adsorption of the polymer molecules on the column packing material or repulsion between the polymer molecules and the packing material.

Table 1 — Results of interlaboratory tests

Polymer	Average values of M_n and M_w[a]	Repeatability, s_r[a] %	Reproducibility, s_R[a] %	Round-robin
PS-1	M_n = 137 000	3,1	8,0	1st
	M_w = 373 000	1,3	8,1	
PS-2	M_n = 70 400	3,1	5,6	1st
	M_w = 226 000	2,1	6,1	
PS-3	M_n = 38 000	2,8	6,9	1st
	M_w = 157 000	2,4	4,9	
PMMA[b]	M_n = 163 000	2,1	9,0	2nd
	M_w = 617 000	1,7	7,8	
PMMA[c]	M_n = 215 000	2,4	5,7	2nd
	M_w = 834 000	1,4	8,4	
PAN[d]	M_n = 202 000	2,3	28,2	3rd
	M_w = 467 000	0,7	19,8	

[a]   Outliers were eliminated by Grubbs' and Cochran's methods[1].

[b]   Calibration: polystyrene standards.

[c]   Calibration: poly(methyl methacrylate) standards.

[d]   Column: polystyrene gel; calibration: polystyrene standards.

[e]   Column: polystyrene gel; calibration: poly(ethylene glycol) and poly(ethylene oxide) standards.

[f]   Column: poly(vinyl alcohol) gel; calibration: polystyrene standards.

[g]   Column: poly(vinyl alcohol) gel; calibration: poly(ethylene glycol) and poly(ethylene oxide) standards.

Table 1 *(continued)*

Polymer	Average values of M_n and M_w[a]	Repeatability, s_r[a] %	Reproducibility, s_R[a] %	Round-robin
PAN[e]	M_n = 79 200	3,2	18,7	3rd
	M_w = 208 000	1,1	8,2	
PAN[f]	M_n = 126 000	2,3	5,8	3rd
	M_w = 418 000	0,4	5,1	
PAN[g]	M_n = 77 800	1,9	6,4	3rd
	M_w = 468 000	0,7	6,6	

[a]   Outliers were eliminated by Grubbs' and Cochran's methods[1].

[b]   Calibration: polystyrene standards.

[c]   Calibration: poly(methyl methacrylate) standards.

[d]   Column: polystyrene gel; calibration: polystyrene standards.

[e]   Column: polystyrene gel; calibration: poly(ethylene glycol) and poly(ethylene oxide) standards.

[f]   Column: poly(vinyl alcohol) gel; calibration: polystyrene standards.

[g]   Column: poly(vinyl alcohol) gel; calibration: poly(ethylene glycol) and poly(ethylene oxide) standards.

11 Test report

According to ISO 16014-1.

Annex A
(informative)

Further information on applicability of method

The method described in this document concerns SEC measurements at temperatures < 60 °C and assumes the sample is a linear homopolymer. However, because it is a relative method, it is also applicable to nonlinear homopolymers, such as branched, star-shaped, comb-like, stereo-regular and stereo-irregular polymers, and to other types of polymer, such as random, block, graft and heterophasic copolymers. The method is applicable to molecular weights ranging from that of the monomer to 3 000 000, but is not applicable to samples that contain > 30 % of components having a molecular weight < 1 000.

The method cannot be used with water as eluent, i.e. for water-soluble polymers, or at column temperatures > 60 °C (e.g. for polyethylene) or with polymers that exhibit appreciable secondary effects such as adsorption of the polymer molecules on the column packing material or repulsion between the polymer molecules and the packing material.

Annex B
(informative)

Further information on reagents

B.1 Examples of suitable eluents

The following are examples of combinations of eluents and polymers for SEC measurements at temperatures < 60 °C:

a) tetrahydrofuran for polystyrene, poly(methyl methacrylate), polycarbonate, poly(vinyl acetate), etc.;

b) chloroform for aliphatic polyesters, etc.;

c) toluene for poly(dimethylsiloxane), etc.;

d) N,N-dimethylformamide for polyacrylonitrile, polyurethanes, etc.;

e) 1,1,1,3,3,3-hexafluoroisopropanol for polyamides, polyesters, etc.

B.2 Examples of column evaluation reagents

Examples of organic reagents recommended for the evaluation of polystyrene gel columns are given in Table B.1. Reagents having ionic groups, such as carboxyl or amino groups, cannot be used because of their potential to interact with the column packing material.

Table B.1 — Examples of column evaluation reagents

Eluent	Reagent
Tetrahydrofuran	Ethylbenzene
Chloroform	Ethylbenzene
Toluene	Ethylbenzene
N,N-Dimethylformamide	Ethylene glycol
1,1,1,3,3,3-Hexafluoroisopropanol	Ethyl acetate

B.3 Narrow molecular weight distribution standards

According to ISO 16014-1.

Polymers soluble only at high temperatures, such as polyethylene and polypropylene, cannot be used.

Annex C
(informative)

Further information on interlaboratory tests

C.1   Data from 1st round-robin test (polystyrene samples)

Tables C.1 to C.3 present the raw data (molecular weight averages and variances) obtained in the 1st round-robin test.

Table C.1 — Molecular weight averages and variances obtained for polystyrene (PS-1)

Laboratory	M_n ($\times 10^{-4}$)		M_w ($\times 10^{-4}$)	
	Average	Variance	Average	Variance
A1	14,6	0,281	35,9	0,013
B1	14,6	0,051	38,9	0,031
B2	13,9	0,249	37,2	0,823
B3	13,8	0,059	37,8	0,178
C1	13,0	0,666	37,1	0,002
C2	13,3	0,446	37,6	0,020
C3	13,6	0,095	38,4	0,162
D1	13,1	0,135	41,9	0,062
D2	13,1	0,135	41,9	0,062
D3	13,8	0,140	42,2	0,002
E1	13,5	0,000	38,3	0,000
E2	13,3	0,012	38,0	0,007
F1	14,2	0,007	40,8	0,018
F2	14,2	0,002	40,4	0,005
F3	14,3	0,017	40,2	0,000
G1	15,0	0,012	37,6	0,009
G2	13,6	0,038	35,8	0,037
H1	13,3	0,001	38,0	0,011
H2	13,5	0,035	38,2	0,021
I1	16,3	0,436	40,2	0,203
I2	16,6	0,247	39,4	0,859
J1	11,9	0,187	31,8	0,815
J2	11,6	0,086	31,8	0,482
J3	12,5	0,016	32,4	0,002
K1	13,8	0,009	37,5	0,007
K2	14,5	0,036	38,1	0,016
L1	13,9	0,024	37,0	0,158
L2	12,6	0,000	35,2	0,260
M1	12,6	0,062	33,3	0,140
M2	13,1	0,107	32,2	0,002
M3	12,0	0,109	32,1	0,362

11

Three sample runs (see 7.6) were carried out for each determination (i.e. *n* = 3). Some of the laboratories carried out two or three separate determinations. For example, laboratory B carried out three determinations, shown as B1, B2 and B3. Such data were processed as being from quasi-laboratories.

Table C.2 — Molecular weight averages and variances obtained for polystyrene (PS-2)

Laboratory	M_n (× 10^{-4})		M_w (× 10^{-4})	
	Average	Variance	Average	Variance
A1	7,24	0,026	22,6	0,006
B1	7,65	0,098	23,1	0,096
B2	7,54	0,038	22,4	(0,212)
B3	7,45	0,031	22,9	0,060
C1	6,70	0,149	21,9	0,007
C2	6,47	0,060	22,2	0,042
C3	6,95	0,009	22,7	0,002
D1	6,89	0,015	24,8	0,047
D2	7,00	0,003	24,7	0,002
D3	6,75	0,084	24,6	0,009
E1	7,19	0,000	23,0	0,000
E2	7,07	0,002	22,9	0,010
F1	7,51	0,002	24,1	0,009
F2	7,61	0,001	23,8	0,000
F3	7,65	0,002	23,6	0,004
G1	7,34	0,002	23,0	0,002
G2	7,09	0,000	22,4	0,010
H1	6,70	0,007	23,5	0,005
H2	6,88	0,002	23,2	0,019
I1	(9,13)	(5,707)	23,4	(2,628)
I2	(9,72)	(0,590)	24,9	(0,887)
J1	6,62	0,004	20,0	0,095
J2	6,73	0,001	20,1	0,042
J3	6,65	0,095	21,0	0,016
K1	7,07	0,007	22,9	0,007
K2	6,78	0,038	22,8	0,020
L1	6,52	0,136	21,7	0,016
L2	6,59	0,020	22,0	0,099
L3	6,74	0,013	20,6	0,050
M1	7,60	0,006	21,8	0,042
M2	7,66	0,084	21,4	0,047
M3	6,55	0,004	19,6	0,042
NOTE Values in brackets are outliers.				

Table C.3 — Molecular weight averages and variances obtained for polystyrene (PS-3)

Laboratory	M_n (× 10^{-4})		M_w (× 10^{-4})	
	Average	Variance	Average	Variance
A1	3,70	0,008	15,7	0,004
B1	4,12	0,006	16,4	0,059
B2	3,99	0,017	15,7	0,052
B3	4,05	0,005	16,0	0,014
C1	3,67	0,005	15,4	0,095
C2	3,55	0,001	15,3	0,020
C3	3,78	(0,038)	15,8	0,056
D1	3,84	0,001	16,8	0,020
D2	3,74	0,002	16,4	0,029
D3	3,76	0,014	16,6	0,007
E1	3,69	0,000	16,1	0,000
E2	3,60	0,000	16,0	0,000
F1	3,95	0,000	16,6	0,003
F2	4,00	0,001	16,3	0,001
F3	4,08	0,001	16,2	0,002
G1	3,99	0,000	16,2	0,001
G2	3,89	0,000	16,0	0,002
H1	3,50	0,001	16,8	0,020
H2	3,59	0,003	16,2	0,039
I1	4,04	0,005	15,3	(2,247)
J1	3,47	0,002	14,6	0,016
J2	3,50	0,002	14,5	0,002
J3	3,70	0,000	14,5	0,009
K1	3,80	0,000	16,1	0,000
K2	3,70	0,000	16,0	0,002
L1	3,75	0,004	15,8	0,012
L2	3,60	0,001	15,0	0,009
L3	3,50	0,002	14,4	0,031
M1	4,42	0,003	15,6	0,002
M2	4,50	(0,069)	15,4	0,016
M3	3,46	(0,038)	13,6	0,016
NOTE Values in brackets are outliers.				

C.2   Data from 2nd round-robin test [poly(methyl methacrylate samples)]

Table C.4 presents the raw data (molecular weight averages and variances) obtained in the 2nd round-robin test. Three sample runs (see 7.6) were carried out for each determination (i.e. *n* = 3).