
**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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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.

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 $\pm 0,3$ %, 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 $1,00 \pm 0,15$. 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 $\pm 0,5$ 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.