

BS ISO 16014-4:2012



BSI Standards Publication

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

Part 4: High-temperature method

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National foreword

This British Standard is the UK implementation of ISO 16014-4:2012. It supersedes BS ISO 16014-4:2003 which is withdrawn.

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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Amendments issued since publication

Date	Text affected
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**Plastics — Determination of average
molecular mass and molecular mass
distribution of polymers using size-
exclusion chromatography —**

**Part 4:
High-temperature method**

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

Partie 4: Mesurage aux températures élevées





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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	1
5.1 Eluent	1
5.2 Reagent for column evaluation	1
5.3 Molecular mass standards	2
5.4 Reagent for flow rate marker (internal standard)	2
5.5 Additives	2
6 Apparatus	2
6.1 General	2
6.2 Eluent reservoir	2
6.3 Pumping system	2
6.4 Injector	2
6.5 Columns	2
6.6 Detector	3
6.7 Tubing	3
6.8 Temperature-control unit	3
6.9 Recorder and plotter	3
6.10 Data-processing system	3
6.11 Other components	3
7 Procedure	3
7.1 Preparation of solutions of molecular mass standards	3
7.2 Preparation of sample solutions	4
7.3 Preparation of solutions for column performance evaluation	4
7.4 Setting up the apparatus	5
7.5 Operating parameters	5
7.6 Number of determinations	5
8 Data acquisition and processing	5
9 Expression of results	6
10 Precision	6
10.1 General	6
10.2 Experimental conditions	6
10.3 Results of round-robin test	7
11 Test report	7
Annex A (informative) Further information on applicability of method	8
Annex B (informative) Further information on reagents	9
Annex C (informative) Further information on the round-robin test	10
Bibliography	14

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.

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.

ISO 16014-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 16014-4:2003), which has been technically revised. The main changes are as follows:

- a) the normative references have been updated;
- b) Subclause 6.1 (concerning the general requirements for the apparatus) has been revised;
- c) further information on the round-robin test carried out in 1999 has been added (see Annex C).

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 4: High-temperature method

1 Scope

This part of ISO 16014 specifies a method for determining the average molecular mass and the molecular mass distribution of polymers by size-exclusion chromatography (SEC) using an organic eluent at temperatures between 60 °C and 180 °C. The average molecular mass and the molecular mass distribution are calculated from a calibration curve prepared using polymer standards. Therefore, this test method is classified as a relative method (see ISO 16014-1:2012, Annex A).

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*

3 Terms and definitions

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

4 Principle

See ISO 16014-1:2012, Clause 4.

5 Reagents

5.1 Eluent

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

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

5.2 Reagent for column evaluation

See ISO 16014-1:2012, 5.2.

There are several low molecular mass compounds that can be used, for example diphenylmethane when 1,2-dichlorobenzene is used as eluent or ethylbenzene when 1,2,4-trichlorobenzene is used as eluent.

5.3 Molecular mass standards

See ISO 16014-1:2012, 5.3.

Some examples of commercially available molecular mass standards are given in Annex B.

5.4 Reagent for flow rate marker (internal standard)

See ISO 16014-1:2012, 5.4.

An example of a compound suitable for use as a flow rate marker is 2,6-di-*tert*-butyl-4-methylphenol when 1,2-dichlorobenzene or 1,2,4-trichlorobenzene is used as eluent.

5.5 Additives

Phenol-type antioxidants such as 2,6-di-*tert*-butyl-4-methylphenol should preferably be added to avoid degradation of the polymer sample. For further examples of antioxidants, see Annex B.

6 Apparatus

6.1 General

A schematic diagram of an SEC system is shown in ISO 16014-1:2012, Figure 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 between 60 °C and 180 °C. However, components connected upstream of the injector, such as the eluent reservoir, the pumping system and tubing, does not need to be kept at the same temperature as the columns.

6.2 Eluent reservoir

See ISO 16014-1:2012, 6.2.

As mentioned in 6.1, it is not necessary to keep the eluent reservoir at the same temperature as the columns.

6.3 Pumping system

See ISO 16014-1:2012, 6.3.

In order to maintain the flow rate accurate to within $\pm 0,3$ %, the pumping system shall be kept at a constant temperature. As mentioned in 6.1, it is not, however, necessary to keep the pumping system at the same temperature as the columns.

6.4 Injector

See ISO 16014-1:2012, 6.4.

In order to ensure that the polymer solution remains clear, without any precipitation, the injector temperature-control equipment shall be capable of keeping the injector at the same temperature, to within ± 1 °C, as the columns. As manual injection is impossible at such temperatures, an automatic injection system shall be used.

6.5 Columns

See ISO 16014-1:2012, 6.5.