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Plastics — Determination of average molecular weight and molecular weight 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 de polymères par chromatographie d'exclusion stérique —

Partie 4: Mesurage aux températures élevées

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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 16014-4:2012), 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;

— the operating temperature of the columns has been changed into 60 °C to 220 °C.

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.

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

Part 4: High-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 temperatures between 60 °C and 220 °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 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.

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.

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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.

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

There are several low molecular weight 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 weight standards, according to ISO 16014-1.

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

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

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. When an IR detector is used as concentration detector, a low molecular weight aliphatic, like heptane, can be used.

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.

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 220 °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, according to ISO 16014-1.

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, 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 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, according to ISO 16014-1.

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 K, as the columns. As manual injection is impossible at such temperatures, an automatic injection system shall be used.

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