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Gel permeation chromatography (GPC) —

Part 3: **Water as eluent**

Chromatographie par perméation de gel (GPC) —

Partie 3: Eluant à l'eau

Reference number
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Contents

Page

Foreword ... **v**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Principle ... **2**

5 Apparatus ... **2**
5.1 Eluent supply ... 3
5.2 Pump ... 3
5.3 Injection system ... 3
5.4 Separation columns ... 3
5.5 Column temperature control ... 5
5.6 Detector ... 6

6 Reagents ... **6**

7 Calibration of the apparatus ... **6**
7.1 General ... 6
7.2 Specification for the calibration standard ... 6
7.3 Preparation of the calibration solutions for injection ... 7
7.4 Conditions for calibration runs ... 7
7.5 Measurement of elution volume ... 7
7.6 Plotting the calibration curve ... 7

8 Sampling ... **8**

9 Preparation for the test ... **8**
9.1 Preparation of the injection solution ... 8
9.2 Preparation of the apparatus ... 9

10 Analytical parameters ... **9**

11 Data acquisition and evaluation ... **9**
11.1 General ... 9
11.2 Calculation of the net chromatogram from the raw data ... 10
11.2.1 Determination of the baseline ... 10
11.2.2 Correction of the measured values and of the net chromatogram ... 10
11.2.3 Evaluation limits ... 10
11.3 Calculation of the average values ... 10
11.4 Calculation of the distribution curves ... 11

12 Precision ... **12**
12.1 General ... 12
12.2 Repeatability ... 12
12.3 Reproducibility ... 12

13 Test report ... **13**
13.1 General ... 13
13.2 General data on the equipment and settings ... 13
13.2.1 Data on the equipment used ... 13
13.2.2 Calibration ... 13
13.2.3 Evaluation ... 14
13.3 Special data on the sample ... 14

Annex A (informative) **Conversion of experimental parameters for variant column sizes** ... **15**

Annex B (informative) **Example of a data sheet for a polymer standard** ... **16**

Annex C (informative) **Explanations** ... **18**

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Bibliography **23**

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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).

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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 35, *Paints and varnishes*.

A list of all parts in the ISO 13885 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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Gel permeation chromatography (GPC) —

Part 3:
Water as eluent

WARNING — This document can involve hazardous materials, operations or equipment. It does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This document specifies the determination of the molar-mass distribution and the average molar mass values M_n (number average) and M_w (weight average) of polymers that are soluble in water by gel permeation chromatography (GPC).

NOTE Also known as size exclusion chromatography (SEC).

This method is applicable to neutral polymers and polyanions (e.g. polycarboxylates, polysaccharides, fully hydrolyzed polyvinyl alcohols and high-molecular polyethylene oxides). It is not applicable to polycations [e.g. polyvinylpyrrolidone, polyvinylpyridine, salts of poly(diallyl-N,N-dimethyl-azacyclopentane), chitosan].

Despite good solubility in the mobile phase and even though the chromatograms obtained show good repeatability, it is possible that this method cannot be used with certain polymer types because of specific interactions (e.g. adsorption) within the sample/eluent/column system (see also Clause 12).

The conditions specified in this document are not applicable to the GPC analysis of polymer samples with M_w values greater than 10^6 g/mol and/or polymers with elution limits outside the calibration range (see 7.6 and Annex C).

This document includes no correction methods (e.g. for the elimination of peak broadening). If absolute molar mass values are required, an absolute method (e.g. membrane osmometry for M_n or light scattering for M_w) can be used.

2 Normative references

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

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following 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

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

3.1
gel permeation chromatography
GPC
separation of molecules, mainly based on exclusion effects such as differences in size and/or shape of molecules (size exclusion chromatography) or in charge (ion exclusion chromatography)

[SOURCE: ISO 13885-1:2020, 3.1]

3.2
system peak
signal peculiar to the *gel permeation chromatography* (3.1) using a refractive index detector

Note 1 to entry: These signals appear at the total penetration limit of the columns and are not part of the sample, but of the overall system.

[SOURCE: ISO 13885-1:2020, 3.2]

3.3
polycation
water-soluble polymer that carries permanent positive charges or forms temporary cationic structures due to its pK_B value under the measurement conditions

3.4
polyanion
water-soluble polymer that carries permanent negative charges or forms temporary anionic structures due to its pK_S value under the measurement conditions

3.5
neutral polymer
water-soluble polymer that carries no charges and forms o-charged groups or structures under the measurement conditions

4 Principle

The dissolved (molecularly disperse) molecules of a polymer sample are fractionated on a porous column material, with separation taking place according to the size of the molecule (or, more precisely, the polymer coil size which forms in this eluent). Small molecules diffuse into the pores of the column material more frequently and are therefore retarded more than large molecules. Thus, large molecules are eluted earlier, small molecules later. Under the test conditions given, the elution volume is solely a function of the coil size of the molecule.

The polymer content of a sample is determined, the sample is then diluted with eluent to give a concentration of less than 5 g/l and an aliquot of the diluted sample is injected into the GPC system. The concentration of the molecules eluted from the column is measured in order of decreasing coil size with a concentration-sensitive detector (typically a differential refractometer). With the aid of a calibration curve that has been determined for the particular GPC system, the relative molar-mass distribution, the relative quantities M_n and M_w and the heterogeneity or polydispersity M_w/M_n are calculated from the chromatogram obtained.

5 Apparatus

The apparatus shall consist of the components shown in Figure 1, which are described below.

All components which come into contact with the eluent or the sample solution shall not exhibit any adsorption and memory effects and shall not exhibit any expansion effects at the increased temperature.

The eluent described in Clause 6 can cause corrosion in the case of long-term use. For this reason, it is necessary that high-quality steel, titanium, polyetherketones or polytetrafluoroethylene are used for

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