SIST

# SLOVENSKI STANDARD
# oSIST prEN ISO 13885-1:2021

**01-maj-2021**

---

**Gelska permeacijska kromatografija (GPC) - 1. del: Tetrahidrofuran (THF) kot izpiralna tekočina (eluent) (ISO 13885-1:2020)**

Gel permeation chromatography (GPC) - Part 1: Tetrahydrofuran (THF) as eluent (ISO 13885-1:2020)

Gelpermeationschromatographie (GPC) - Teil 1: Tetrahydrofuran (THF) als Elutionsmittel (ISO 13885-1:2020)

Chromatographie par perméation de gel (GPC) - Partie 1: Utilisation de tétrahydrofurane (THF) comme éluant (ISO 13885-1:2020)

**Ta slovenski standard je istoveten z: prEN ISO 13885-1**

---

**ICS:**

| | | |
|---|---|---|
| 87.060.20 | Veziva | Binders |

**oSIST prEN ISO 13885-1:2021 en,fr,de**

# INTERNATIONAL STANDARD

# ISO 13885-1

Third edition
2020-07

# Gel permeation chromatography (GPC) —

## Part 1: Tetrahydrofuran (THF) as eluent

*Chromatographie par perméation de gel (GPC) —*

*Partie 1: Utilisation de tétrahydrofurane (THF) comme éluant*

Reference number
ISO 13885-1:2020(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

# Contents

# 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 35, *Paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 13885-1:2008), which has been technically revised. The main changes compared to the previous edition are as follows:

- this document has been adapted to the actual state of the art, especially with regards to software engineering;
- the scope has been revised;
- the definition for gel-permeation chromatography has been revised;
- the text has been revised editorially.

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.

**INTERNATIONAL STANDARD** **ISO 13885-1:2020(E)**

# Gel permeation chromatography (GPC) —

## Part 1:
## Tetrahydrofuran (THF) 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 tetrahydrofuran (THF) by gel permeation chromatography (GPC).

NOTE Also known as size exclusion chromatography (SEC).

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.

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 of polymers with elution limits outside the calibration range (see 7.6 and Annex C).

This document includes no correction method (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 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 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
- 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 the size and/or shape of molecules (size exclusion chromatography) or in charge (ion exclusion chromatography)

### 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.

## 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 the components which come into contact with the eluent or the sample solution shall be resistant and shall not exhibit adsorption or memory effects in any form. The individual components of the GPC apparatus, which in this case uses THF as eluent, shall be connected to capillary tubes made of high-quality steel or titanium.

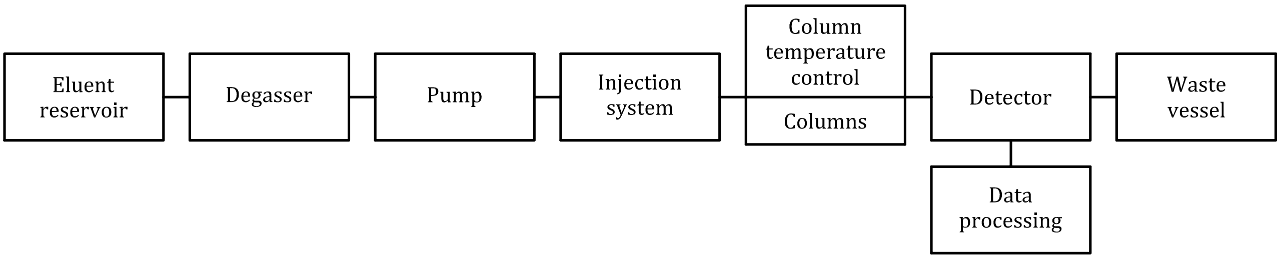


**Figure 1 — Block diagram of a GPC apparatus**

### 5.1 Eluent supply

The eluent reservoir shall adequately protect the eluent against external influences such as the atmosphere and light, if necessary by means of a blanket of inert gas above the liquid level.

The eluent reservoir shall contain a sufficient quantity of the eluent to bring the apparatus to equilibrium and to carry out several repeat analyses.

The eluent shall be degassed, either before it is introduced into the reservoir or by use of a device fitted between the reservoir and the pump, to prevent malfunctions of the pump or the formation of bubbles in the detector. The method of degassing used (e.g. bubble trap, online purging with helium, or vacuum degassing) is open to choice.

## 5.2 Pump

The pump shall ensure that the eluent flow through the separation column is as smooth and pulse free as possible. The flow rate shall be 1 ml/min (see Annex A). To fulfil these requirements, the pump shall operate at optimum efficiency at this flow rate.

The flow rate of the pump used shall have a variation of max. 0,1 %.

## 5.3 Injection system

The injection system serves to introduce a given amount of the sample solution into the eluent stream in a rapid and smooth fashion. This introduction may be carried out either manually or automatically.

If the introduction is carried out manually, ensure that the sample loop is filled completely with solvent before loading with the sample.

Memory effects from the previous sample solution in the injection system shall be avoided by adequate flushing.

## 5.4 Separation columns

The apparatus shall have one or more columns connected in series and packed with spherical porous material, the diameter of the pores corresponding to the size of the polymer molecules being analysed.

The packing material typically consists of a styrene/divinylbenzene copolymer (S/DVB), produced by a special polymerization process, which swells only slightly in the solvent and therefore cannot deform under the pressure developed at the set flow rate.

In addition to these macroporous spherical S/DVB particles, packing materials based on other organic monomers or on silicon dioxide (silica) are also used. The criterion for their use is that no adsorptive interaction shall occur between their surface and the polymer molecules in the sample. Furthermore, the sample being analysed shall not be changed, either chemically or structurally, within the chromatographic system.

Certain polymers interact with the surface of the packing material (e.g. by adsorption) and other effects can sometimes interfere with the GPC separation mechanism. Details of such effects and notes on possible remedies are discussed in Annex C. If it is intended to compare analyses of such polymers by different laboratories, the laboratories shall agree on details of the test conditions that are not covered by this document.

For good repeatability of test results, it is necessary to adhere to the minimum requirements specified below with regard to peak broadening (expressed in terms of a number of theoretical plates) and separation efficiency.

a) Number of theoretical plates

   The number of theoretical plates, $N$, shall be determined, for the apparatus used per metre of column used, from the peak width at half height (see Figure 2). Inject up to 20 µl of ethylbenzene (mass concentration 1 g/l) on to the column (see Annex A) and evaluate the chromatogram obtained under the same conditions as are used for analysing polymers, using Formula (1):

$$N = 5{,}54 \times \left( \frac{V_e}{W_{1/2}} \right)^2 \times \frac{100}{L} \qquad (1)$$

where

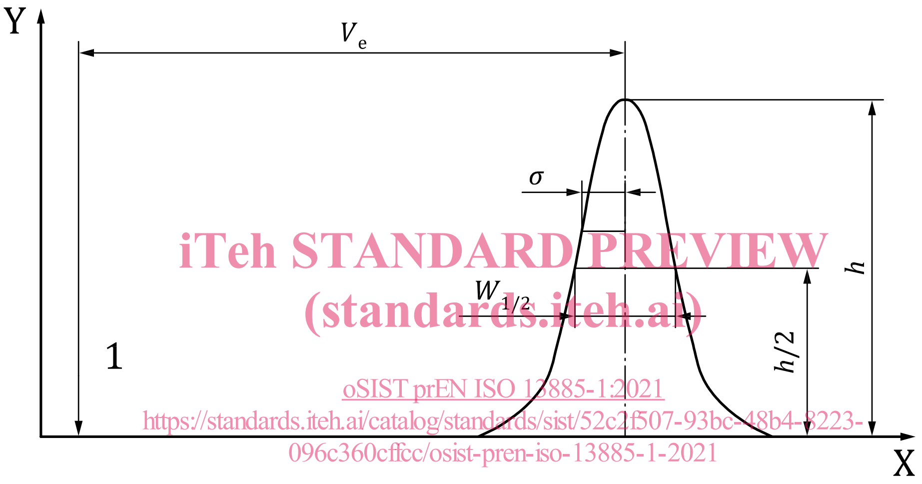
$V_e$ is the elution volume at the peak maximum;

$W_{1/2}$ is the peak width at half height (see Figure 2); the same units shall be used for $V_e$ and $W_{1/2}$;

$L$ is the length of the column (column combination), in centimetres.

Express as the result the number of theoretical plates per metre of column length. To conform to the requirements of this document, the column combination shall have at least 20 000 theoretical plates per metre.

NOTE See Annex C for tailing and fronting (asymmetry) of the peak used to calculate the plate count.



**Key**

X elution volume
Y peak intensity
1 injection
$V_e$ elution volume at the peak maximum
$W_{1/2}$ peak width at the half maximum height of the peak
$h$ maximum peak height
$\sigma$ standard deviation

Figure 2 — Determination of the number of theoretical plates by the half-height method

b) Separation efficiency

To ensure adequate resolution, the $\log_{10}M$ versus the elution volume, $V_e$, calibration curve for the column combination used shall not exceed a specified gradient. For the purposes of this document, the relation given in Formula (2) shall apply to the area of the peak maximum for the polymer sample under investigation:

$$\frac{V_{e,M_x} - V_{e,(10 \times M_x)}}{A_c} > 6{,}0 \qquad (2)$$

where