

<b>STN P</b>	<b>Kvapalinová chromatografia pri kritických podmienkach (LCCC) Chemická heterogenita polyetylénoxidov (ISO/TS 23973: 2020)</b>	<b>STN P CEN ISO/TS 23973</b>  67 3059
------------------	---	--

Liquid chromatography at critical conditions (LCCC) - Chemical heterogeneity of polyethylene oxides (ISO/TS 23973:2020)

Táto norma obsahuje anglickú verziu európskej normy.  
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 03/22

Táto predbežná STN je určená na overenie. Pripomienky zasielajte ÚNMS SR najneskôr do 31. 12. 2023.

Obsahuje: CEN ISO/TS 23973:2021, ISO/TS 23973:2020

TECHNICAL SPECIFICATION

**CEN ISO/TS 23973**

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

December 2021

ICS 71.040.50

English Version

## Liquid chromatography at critical conditions (LCCC) - Chemical heterogeneity of polyethylene oxides (ISO/TS 23973:2020)

Chromatographie liquide aux conditions critiques -  
Hétérogénéité chimique des oxydes de polyéthylène  
(ISO/TS 23973:2020)

This Technical Specification (CEN/TS) was approved by CEN on 5 December 2021 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

**CEN ISO/TS 23973:2021 (E)**

<b>Contents</b>	<b>Page</b>
<b>European foreword.....</b>	<b>3</b>

## **European foreword**

The text of ISO/TS 23973:2020 has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TS 23973:2021 by Technical Committee CEN/TC 139 "Paints and varnishes" the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## **Endorsement notice**

The text of ISO/TS 23973:2020 has been approved by CEN as CEN ISO/TS 23973:2021 without any modification.

# TECHNICAL SPECIFICATION

# ISO/TS 23973

First edition  
2020-08

---

---

## Liquid chromatography at critical conditions (LCCC) — Chemical heterogeneity of polyethylene oxides

*Chromatographie liquide aux conditions critiques — Hétérogénéité  
chimique des oxydes de polyéthylène*



Reference number  
ISO/TS 23973:2020(E)

© ISO 2020

**ISO/TS 23973:2020(E)****COPYRIGHT PROTECTED DOCUMENT**

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Apparatus</b> .....	<b>2</b>
5.1 General.....	2
5.2 Eluent supply.....	3
5.3 Pump.....	3
5.4 Injection system.....	4
5.5 Separation columns.....	4
5.6 Column temperature control.....	5
5.7 Detectors.....	5
5.8 Eluent.....	5
5.9 Data acquisition.....	5
<b>6 Sample preparation</b> .....	<b>5</b>
<b>7 Performance of the measurements</b> .....	<b>6</b>
7.1 Determination of the critical conditions.....	6
7.2 Analysis of the validation kit.....	7
<b>8 Test report</b> .....	<b>7</b>
<b>Annex A (informative) Error sources</b> .....	<b>9</b>
<b>Annex B (informative) Evaluation of the interlaboratory testing</b> .....	<b>10</b>
<b>Annex C (informative) Elugrams of the participants (excerpts)</b> .....	<b>17</b>
<b>Annex D (informative) Investigations of the long-term stability of the test mixture</b> .....	<b>43</b>
<b>Bibliography</b> .....	<b>49</b>

## ISO/TS 23973:2020(E)

### 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](http://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](http://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](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

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](http://www.iso.org/members.html).



## Introduction

Since the first description of liquid chromatography at critical conditions (LCCC) in 1986 (see Reference [1]), the method has been continuously refined and has proved itself to be indispensable for polymer characterisation. Separation is required not only for the quantitative analysis of the individual species. It also offers the preconditions for qualitative characterisation of the fractions by means of spectroscopic and spectrometric techniques. The key factor here is the reduction of the polydispersity/chemical heterogeneity within a fraction, which represents a large problem for mass-spectrometric investigations.

The method has been described extensively in professional circles over the last two decades for different polymer systems, see References [2] to [9].

Within the framework of the Technical Committee, the extent that the method supplies consistent results for a simple, chemically heterogeneous polymer mixture was clarified as part of interlaboratory testing.

At this time, necessary experience relating to the selection of the system (interaction between the polarities separation phase/eluent/sample) was not expected of any of the participating laboratories.

The interlaboratory testing has shown that, even with a well-characterized system and with specification of all pertinent system parameters, it has to date not been possible to classify the process as a routine method in laboratories with experience in polymer analytics.

The idea presents itself of offering a validation kit (polymer mixture with the expecting separation result).



# Liquid chromatography at critical conditions (LCCC) — Chemical heterogeneity of polyethylene oxides

## 1 Scope

This document establishes a valid method for separation of chemically heterogeneous polyethylene oxide (PEO) mixtures and for the determination the number and content of the chemically heterogeneous species in the overall sample.

The method presented in this document serves as a technical guideline and enables laboratories to learn the principle of “critical chromatography” on a validated system.

This method presented in this document with its stated system parameters is not applicable for other polymer classes, due to the diversity of the interactions between the polymer/mobile phase/stationary phase and the number of separation systems that are therefore available.

The evaluation of the interlaboratory testing has shown that many error sources relate to the technique of liquid chromatography in general. Possible error sources are described in [Annex A](#).

Details on the evaluation of the interlaboratory testing are given in [Annex B](#).

Elugrams of the participants (excerpts) are given in [Annex C](#).

Investigations of the long-term stability of the test mixture are given in [Annex D](#).

## 2 Normative references

There are no normative references in this document.

**koniec náhľadu – text ďalej pokračuje v platenej verzii STN**