SLOVENSKI STANDARD
SIST-TS CEN ISO/TS 23973:2022

01-marec-2022

Tekočinska kromatografija pri kritičnih pogojih (LCCC) - Kemijska heterogenost polietilen oksidov (ISO/TS 23973:2020)

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)

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71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

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TECHNICAL SPECIFICATION

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TECHNISCHE SPEZIFIKATION

CEN ISO/TS 23973

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)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

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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

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Contents

Page

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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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.

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.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

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

3.1
liquid chromatography at critical conditions
LCCC
special form of liquid chromatography of polymers at the point of adsorption, where chemically and structurally identical polymers with a certain repeat unit elute independently of the molar mass at the same retention time

Note 1 to entry: The individual monomer units do not contribute to the retention. Under these determined system parameters (defined combination of separation column/eluent mixture/temperature), a separation of polymer mixtures of the same repeat unit takes place based on chemical heterogeneity. Chemical heterogeneities can take the form of different functional groups, end groups, differences in the microstructure (e.g. copolymers and their composition) as well as topological differences (e.g. branching).

4 Principle

In the following, the method for the separation of polyethylene oxides at critical conditions of adsorption is described for the ethylene oxide repeat unit.

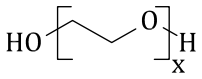
Three polyethylene glycols (PEG) with different molar masses (1 mg/ml to 2 mg/ml dissolved in the relevant eluent mixture (see 7.1) are measured, starting with a high proportion of the thermodynamically good solvent B (in this case acetonitrile). The eluent mixture is successively changed by increasing the proportion of component A (in this case water). This is followed by measurements in different compositions until all three standards elute independently of the molar mass at the same retention time. The determined critical solvent composition (csc) corresponds to the critical conditions.

Afterwards, the unknown mixtures are dissolved in this eluent mixture and measured.

From the peaks of the resulting chromatograms, the number of species with different functionalities and their relative content (taking into account the detection properties) can be determined.

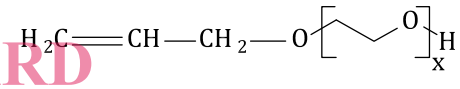
Species contained in the mixtures:

(1) Polyethylene glycol



(2) Methylene polyethylene glycol

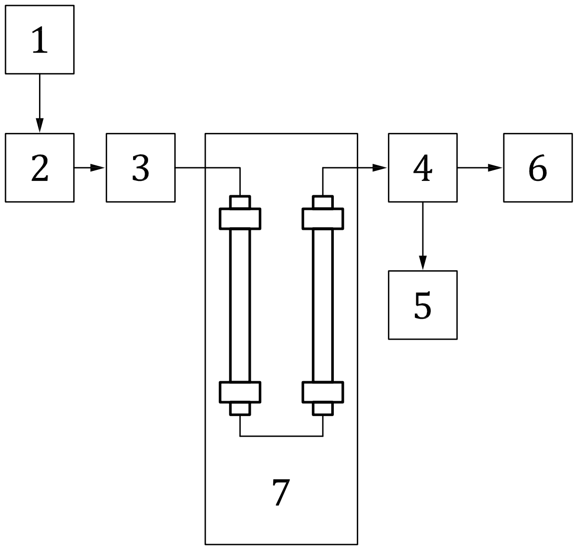
(3) Polyethylene glycol monoallyl ether



5 Apparatus

5.1 General

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



Key

1 LCCC mobile phase
2 pump, 0,1 ml/min to 2 ml/min
3 injection valve, autosampler
4 detectors: RI, ELSD, corona
5 data processing
6 waste
7 separation columns/column temperature control

Figure 1 — LCCC apparatus

All components shall come into contact with the eluent or the sample solution are resistant to them and do not exhibit any adsorption or memory effects. The individual modules should be generally connected with steel capillary tubes for polymer analytics.

5.2 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, but shall be stated in the test report.

For polymer mixtures that contain chromophoric groups, other detectors may be used, e.g. UV or IR detectors.

5.3 Pump

The pump ensures that the eluent flow through the separation columns is as smooth and pulse-free as possible. The flow rate shall be 0,5 ml/min to 1 ml/min, depending on the dimensions of the columns used. To fulfil these requirements, the pump shall operate at optimum efficiency at this flow rate and at the counterpressure established in the process. The variation in the flow rate of the pump used may have a variation of max. 0,1 %.