SLOVENSKI STANDARD
SIST EN ISO 12010:2019

01-junij-2019

Nadomešča:
SIST EN ISO 12010:2014

Kakovost vode - Določevanje polikloriranih alkanov s kratko verigo (SCCP) v vodi - Metoda s plinsko kromatografijo z masno selektivnim detektorjem (GC-MS) in negativno kemijsko ionizacijo (NCI) (ISO 12010:2019)

Water quality - Determination of short-chain polychlorinated alkanes (SCCP) in water - Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI) (ISO 12010:2019)

Wasserbeschaffenheit - Bestimmung von kurzkettigen Chloralkanen (SCCP) in Wasser - Verfahren mittels Gaschromatographie-Massenspektrometrie (GC-MS) und negativer chemischer Ionisation (NCI) (ISO 12010:2019)

Qualité de l'eau - Détermination des alcanes polychlorés à chaîne courte (SCCP) dans l'eau - Méthode par chromatographie gazeuse-spectrométrie de masse (CG-SM) avec ionisation chimique négative (ICN) (ISO 12010:2019)

Ta slovenski standard je istoveten z: **EN ISO 12010:2019**

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

SIST EN ISO 12010:2019 **en,fr,de**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 12010

April 2019

ICS 13.060.50

Supersedes EN ISO 12010:2014

English Version

Water quality - Determination of short-chain polychlorinated alkanes (SCCP) in water - Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI) (ISO 12010:2019)

Qualité de l'eau - Détermination des alcanes polychlorés à chaîne courte (SCCP) dans l'eau - Méthode par chromatographie gazeuse-spectrométrie de masse (CG-SM) avec ionisation chimique négative (ICN) (ISO 12010:2019)

Wasserbeschaffenheit - Bestimmung von kurzkettigen Chloralkanen (SCCP) in Wasser - Verfahren mittels Gaschromatographie-Massenspektrometrie (GC-MS) und negativer chemischer Ionisation (NCI) (ISO 12010:2019)

This European Standard was approved by CEN on 18 February 2019.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, 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

Ref. No. EN ISO 12010:2019 E

Contents

Page

European foreword

This document (EN ISO 12010:2019) has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with Technical Committee CEN/TC 230 “Water analysis” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2019, and conflicting national standards shall be withdrawn at the latest by October 2019.

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.

This document supersedes EN ISO 12010:2014.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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

Endorsement notice

The text of ISO 12010:2019 has been approved by CEN as EN ISO 12010:2019 without any modification.

INTERNATIONAL STANDARD

ISO 12010

Second edition
2019-03

Water quality — Determination of short-chain polychlorinated alkanes (SCCP) in water — Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)

Qualité de l'eau — Détermination des alcanes polychlorés à chaîne courte (SCCP) dans l'eau — Méthode par chromatographie gazeuse-spectrométrie de masse (CG-SM) avec ionisation chimique négative (ICN)

Reference number
ISO 12010:2019(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

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.

This second edition cancels and replaces the first edition (ISO 12010:2012), which has been technically revised. The main changes compared to the previous edition are:

— the *m*/*z* values (mass/charge ratios) for quantification and identification;

— the calibration mixtures;

— the clean up procedure by gel chromatography;

— reduced interferences.

Introduction

The user should be aware that particular problems might require the specifications of additional marginal conditions.

This document achieves synergetic effects in the practical laboratory work. The following points partially allow a combination of water and sediment analysis:

1) same mass combination as for sediment analysis (see ISO 18635[2]);

2) same calibration mixtures as for sediment analysis (see ISO 18635);

3) same GPC-clean up as for sediment analysis (see ISO 18635).

Water quality — Determination of short-chain polychlorinated alkanes (SCCP) in water — Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance to this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the quantitative determination of the sum of short-chain polychlorinated *n*-alkanes also known as short-chain polychlorinated paraffins (SCCPs) in the carbon bond range n-C_{10} to n-C_{13} inclusive, in mixtures with chlorine mass fractions ("contents") between 50 % and 67 %, including approximately 6 000 of approximately 8 000 congeners.

This method is applicable to the determination of the sum of SCCPs in unfiltered surface water, ground water, drinking water and waste water using gas chromatography-mass spectrometry with electron capture negative ionization (GC-ECNI-MS).

Depending on the capability of the GC-ECNI-MS instrument, the concentration range of the method is from 0,1 µg/l or lower to 10 µg/l. Depending on the waste water matrix, the lowest detectable concentration is estimated to be > 0,1 µg/l. The data of the interlaboratory trial concerning this method are given in Annex I.

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 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO/TS 13530, *Water quality — Guidance on analytical quality control for chemical and physicochemical water analysis*

3 Terms and definitions

No terms and definitions are listed in this document.

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

Determination of the sum of SCCPs in the carbon bond range n-C_{10} to n-C_{13} inclusive, in technical and environmental transposed mixtures with chlorine mass fractions ("contents") between 50 % and 67 % (for example a mean of approximately 4 to 10 chlorine atoms per molecule) and independent of the C-number distribution pattern of the congeners. No recognition of the chlorine content is necessary.

The analysed sum of SCCPs includes the variety of SCCPs with their differing chlorine content and C-number distribution patterns as found in technical mixtures as well as compositions in the environment (References [5] to [9]).

SCCPs in whole water samples are fortified with an internal standard and extracted using liquid-liquid extraction with an organic solvent. The sample enrichment procedure is followed by a clean-up procedure to eliminate interfering compounds. Gas chromatography (GC) is undertaken using a short capillary column within a short retention time range. The detection of selected mass fragments is carried out using mass spectrometry (MS) in selected ion-monitoring mode using electron capture negative ionization mode (ECNI). The mass fragments and the compositions of the calibration solutions used in this document are essential for the analysis of the sum of SCCPs (see References [3] and [4]).

The selected ion chromatogram is integrated over the full retention time range of the SCCPs. The quantification of the sum of SCCPs is carried out after establishing a calibration by a multiple linear regression. The calibration requires the specified three differently composed standard mixtures fortified with an internal standard.

These standard mixtures mimick different mixtures found in the environment. In this method, only the multiple linear regression quantification with these specific mixtures enables the quantification of the variety of observed mixtures of SCCP in the environment and in technical compositions, described in Clause 1 and in References [3] and [4]. It is not possible to use only one reference mixture for that complex task.

The method allows for a quantification of the sum of SCCPs expected to be within an expanded measurement uncertainty of less than 50 %.

5 Interferences

Non-specific matrix interferences, as well as interferences from other environmental situations, are dealt with using the given clean-up procedure. A further reduction of matrix effects can be achieved by reducing the mass spectrometric resolution power to, for example, 0,4 u, which is often possible with a quadrupole mass spectrometer. The exact *m/z* values are 374,958 8; 410,916 9; 422,935 5; 448,810 6 (see Reference [8]).

Applying the entire procedure using the clean up procedure given in 9.3, a selection of chlorinated pollutants has been tested and found not to cause interferences below the concentrations given in Table 1.

Table 1 — Highest concentration level which causes no interferences higher than the limit of quantification of 0,1 µg/l

Potential interfering compounds	Highest concentration level which causes no interferences higher than the limit of quantification of 0,1 µg/l
Aroclor 1262[a]	1,25 µg/l
Aroclor 1242[a]	10 µg/l
Aroclor 1221[a]	10 µg/l

[a] Aroclor 1262, Aroclor 1242, Aroclor 1221, Halowax 1014 and Halowax 1051 are examples of suitable products available commercially. These examples are given for the convenience of users of this document and do not constitute an endorsement by ISO of these products.

Table 1 *(continued)*

Potential interfering compounds	Highest concentration level which causes no interferences higher than the limit of quantification of 0,1 µg/l
Campheclor (toxaphene)	1,75 µg/l
Halowax 1014[a]	10 µg/l
Halowax 1051[a]	0,4 µg/l
MCCP (medium-chain chlorinated *n*-alkanes C_{14}-C_{17}) 42 % chlorine	10 µg/l
MCCP (medium-chain chlorinated *n*-alkanes C_{14}-C_{17}) 52 % chlorine	6 µg/l
MCCP (medium-chain chlorinated *n*-alkanes C_{14}-C_{17}) 57 % chlorine	10 µg/l
[a] Aroclor 1262, Aroclor 1242, Aroclor 1221, Halowax 1014 and Halowax 1051 are examples of suitable products available commercially. These examples are given for the convenience of users of this document and do not constitute an endorsement by ISO of these products.	

6 Reagents and standards

Use solvents and reagents of sufficient purity, i.e. with negligibly low concentrations of SCCPs, e.g. lower than the limit of detection of the method. Check blanks regularly over the entire procedure to ensure they are suitable and establish proper analytical control.

6.1 Solvents for extraction and preparation of stock solutions

The solvent for extraction is *n*-heptane. Other non-polar solvents, e.g. *n*-hexane (C_6H_{14}), cyclohexane (C_6H_{12}), can be used if the extraction efficiency is comparable with those of *n*-heptane.

Use 2,2,4-trimethylpentane (C_8H_{18}, isooctane) for conditioning of the glass bottles (7.1).

For preparation of the stock solution and dilutions of the internal standard, use propanone (acetone), C_3H_6O.

For conditioning of the clean-up columns, use mixtures of *n*-heptane and propanone (acetone).

For the first elution step of the filtrated suspended matter, use methanol (CH_3OH).

6.2 Reference SCCP stock solutions

Use commercially available solutions, such as in cyclohexane or *n*-hexane, of the single mixtures of SCCP congeners with defined carbon chain length and with different defined chlorine contents (see Table 2, first two columns). Alternatively, use commercially available ready mixed solutions with the same composition.

Mixtures of synthetic solutions were used to simulate environmentally occurring SCCPs or technical products of SCCPs. For example, the synthetic mixed calibration stock solution “Lake Ontario water” is mixed to resemble a Lake Ontario water as reported in Reference [6]. Its characteristic is a relatively high content of C_{10} to C_{12}, especially C_{12} and a low chlorine content as partly reported in water samples too. The synthetic mixed calibration stock solution “Perch” simulates a C-number distribution found in a perch (see Reference [7]). The standard mixture “Sediment Drevnice” simulates a natural mixture reported about a sediment of the river Drevnice (see Reference [8]) with a high content of C_{13} and a higher chlorine content.

The compositions of the calibration mixtures as well as of the independent quality assurance solutions are mandatory to achieve the quantification of the variety of SCCP-mixtures.

Prepare the solutions “Lake Ontario water”, ”Perch”, and “Sediment Drevnice” according to Table 2.