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)





COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

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 Fax: +41 22 749 09 47 Email: copyright@iso.org Website: www.iso.org

Published in Switzerland

Con	tent	S		Page
Forew	ord			iv
Introd	luctio	n		v
1	Scope	e		1
2	Normative references			1
3	Terms and definitions			
4	Principle			
5	Interferences			
6	Reagents and standards			
	6.1 Solvents for extraction and preparation of stock solutions			3
	6.2		nce SCCP stock solutions	
	6.3 6.4		al standard stock solutions from individual congeners ation solutions	
	6.5		tion auxiliary and clean-up materials	
7				
8			d sample pretreatment	
9	Procedure			
	 9.1 Extraction with liquid-liquid extraction 9.2 Extraction with higher content of suspended matter 			
				7
		9.3 Extract clean-up		
	9.4	Measurement and integration of the chromatogram		
	9.5	Calibration		
		9.5.2	Basic calibration	
		9.5.3	Identification and quantification with mass fragment combinations	11
		9.5.4	Calculation of the results	11
		9.5.5	Quality checks for internal standardization	
10	_		f results	
11	Test	report		12
Annex	A (no	rmative)	Independent quality control check solutions	13
Annex	B (inf	formative	e) Explanation of the calibration of the sum of SCCPs with multiple	
_		U	sion	
			e) Typical GC-MS conditions	21
Annex	t D (in: checl	formative k solutio	e) Typical chromatograms of standard solutions and quality control ons 1 μg/ml	24
Annex			e) Presentation of goodness of fit	
	-		Alternative clean-up with column chromatography	
	-		e) Alternative clean-up with gel chromatography	
			e) Chromatograms of real SPM samples	
) Performance data	
Biblio	graph	y		42

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 μ g/l or lower to 10 μ g/l. Depending on the waste water matrix, the lowest detectable concentration is estimated to be > 0,1 μ g/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/