INTERNATIONAL STANDARD

ISO 18635

First edition
2016-04-01

Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in sediment, sewage sludge and suspended (particulate) matter — Method using gas chromatography-mass spectrometry (GC-MS) and electron capture negative ionization (ECNI)

Qualité de l'eau — Détermination des alcanes polychlorés à chaîne courte dans les sédiments et matières en supsension (particules) — Méthode par chromatographie en phase gazeuse-spectrométrie de masse (CPG-SM) et ionisation chimique négative (ICN)

Reference number
ISO 18635:2016(E)

© ISO 2016



COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, 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
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Principle ... **1**
4 Interferences ... **2**
5 Reagents ... **3**
6 Apparatus ... **6**
7 Sampling and sample pretreatment ... **7**
8 Procedure ... **7**
8.1 Extraction ... 7
8.2 Extract clean-up ... 7
8.3 Measurement and integration of the chromatogram ... 8
8.4 Calibration ... 8
8.4.1 General ... 8
8.4.2 Basic calibration ... 9
8.4.3 Identification and quantification with mass fragment combinations ... 10
8.4.4 Calculation of the results ... 10
8.4.5 Quality checks for internal standardization ... 11
9 Expression of results ... **11**
10 Test report ... **11**
Annex A (normative) **Programme for pressurized liquid extraction** ... **12**
Annex B (informative) **Explanation of the calibration of the sum of SCCPs with multiple linear regression** ... **13**
Annex C (informative) **Examples of typical GC-MS conditions** ... **18**
Annex D (normative) **Alternative clean-up with column chromatography** ... **22**
Annex E (informative) **Alternative clean-up with gel chromatography** ... **24**
Annex F (informative) **Typical chromatograms of standard solutions 1 µg/ml** ... **25**
Annex G (informative) **Presentation of goodness of fit of the calibration** ... **26**
Annex H (informative) **Example for recoveries of quality assurance solutions** ... **27**
Annex I (informative) **Chromatograms of real samples** ... **29**
Annex J (informative) **Performance data** ... **31**
Annex K (informative) **Quality control check solutions** ... **32**
Bibliography ... **33**

© ISO 2016 – All rights reserved

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

© ISO 2016 – All rights reserved

Introduction

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

© ISO 2016 – All rights reserved

Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in sediment, sewage sludge and suspended (particulate) matter — Method using gas chromatography-mass spectrometry (GC-MS) and electron capture negative ionization (ECNI)

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 and to ensure compliance with any national regulatory conditions.

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

1 Scope

This International Standard 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 sediment and suspended (particulate) matter, sewage sludge, and soil using gas chromatography-mass spectrometry with electron capture negative ionization (GC-ECNI-MS).

Depending on matrix and the detection capabilities of the GC-ECNI-MS, the method can be applied to samples containing, e.g. 0,03 µg/g to 3 µg/g sum of SCCPs.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5667-12, *Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments*

ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges*

ISO 5667-17, *Water quality — Sampling — Part 17: Guidance on sampling of bulk suspended solids*

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 12010, *Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in water — Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)*

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

3 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 %

© ISO 2016 – All rights reserved