Textiles - Determination of short-chain chlorinated paraffins (SCCP) and middle-chain chlorinated paraffins (MCCP) in textile products out of different matrices by use of gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS) (ISO 22818:2021)



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN ISO 22818:2021 sisaldab Euroopa standardi EN ISO 22818:2021 ingliskeelset teksti.

This Estonian standard EVS-EN ISO 22818:2021 consists of the English text of the European standard EN ISO 22818:2021.

Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.

This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation and Accreditation.

Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 31.03.2021.

Date of Availability of the European standard is 31.03.2021.

Standard on kättesaadav Eesti Standardimis-ja Akrediteerimiskeskusest.

The standard is available from the Estonian Centre for Standardisation and Accreditation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile <u>standardiosakond@evs.ee</u>.

ICS 59.060.01

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardimis- ja Akrediteerimiskeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardimis-ja Akrediteerimiskeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardimis-ja Akrediteerimiskeskusega: Koduleht www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation and Accreditation No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation and Accreditation.

 $If you have any questions about copyright, please contact \ Estonian \ Centre for \ Standard is at ion \ and \ Accreditation:$

Homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

EUROPEAN STANDARD

EN ISO 22818

NORME EUROPÉENNE EUROPÄISCHE NORM

March 2021

ICS 59.060.01

English Version

Textiles - Determination of short-chain chlorinated paraffins (SCCP) and middle-chain chlorinated paraffins (MCCP) in textile products out of different matrices by use of gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS) (ISO 22818:2021)

Textiles - Détermination de paraffines chlorées à chaîne courte (PCCC) et de paraffines chlorées à chaîne moyenne (PCCM) dans des produits textiles sur différentes matrices par chromatographie en phase gazeuse couplée à la spectrométrie de masse avec ionisation chimique négative (GC-NCI-MS) (ISO 22818:2021)

Textilien - Bestimmung von SCCP und MCCP in textilen Produkten aus verschiedener Matrices mittels GC-NCI-MS (ISO 22818:2021)

This European Standard was approved by CEN on 13 March 2021.

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, 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

European foreword

This document (EN ISO 22818:2021) has been prepared by Technical Committee ISO/TC 38 "Textiles" in collaboration with Technical Committee CEN/TC 248 "Textiles and textile products" the secretariat of which is held by BSI.

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 September 2021, and conflicting national standards shall be withdrawn at the latest by September 2021.

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.

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, 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 22818:2021 has been approved by CEN as EN ISO 22818:2021 without any modification.

Coı	ontents	Page
Fore	reword	iv
Intro	roduction	v
1	Scope	1
2	Normative references	1
3	Terms and definitions	1
4	Principle	
5	Reagents	
6	Apparatus	2
7	Preparation of test specimens	
8	Procedure 8.1 Extraction of test specimen 8.2 Sulfuric acid clean-up 8.3 Preparation of the calibration solutions 8.3.1 Preparation of SCCPs calibration solution (5 µg 8.3.2 Preparation of SCCPs calibration solution (50 µg 8.3.3 Preparation of SCCPs calibration solution (75 µg 8.3.4 Preparation of MCCPs calibration solution (5 µg 8.3.5 Preparation of MCCPs calibration solution (50 µg 8.3.6 Preparation of MCCPs calibration solution (75 µg 8.3.7 Daily calibration 8.4 GC-MS determination	3 (g/ml) with 59 % Cl 3 (g/ml) with 59 % Cl 3 (g/ml) with 59 % Cl 3 (g/ml) with 55 % Cl 3 (μg/ml) with 55 % Cl 4 (μg/ml) with 55 % Cl 4 (μg/ml) with 55 % Cl 4
9	Expression of results 9.1 Evaluation 9.2 Determination of the recovery rate of the IS 9.3 Interferences 9.3.1 Interferences of MCCPs masses 9.3.2 Interferences of SCCPs masses	
10	Test report	6
Ann	nex A (normative) Integration with peak shape evaluation (PSE	E)7
Anno	nex B (informative) Chromatographic parameters	12
Ann	nex C (informative) Reliability of method	14
Bibli	oliography	

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 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.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textiles and textile products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

Short-chain chlorinated paraffins (SCCPs) (C_{10} - C_{13} ; chlorine content > 48 %) are listed by the Stockholm Convention on Persistent Organic Pollutants.

In Europe, according to REGULATION (EU) 2019/1021^[1] of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutants alkanes C_{10} - C_{13} , chloro [short-chain chlorinated paraffins (SCCPs)] (CAS no. 85535-84-8) as constituents of articles are prohibited. Articles containing SCCPs in concentrations lower than 0,15 % by weight are allowed.

Furthermore, it became industrial practice to restrict alkanes C_{14} - C_{17} , known as medium-chain chlorinated paraffins (MCCPs) as well.

SCCPs and MCCPs are used as flame retardants in textiles, as plasticizers in polymers and as finishing agents in leather. SCCPs and MCCPs are an issue for textile manufacturers and retailers due to their use within fabrics, coated fabrics, plastisol prints, buttons, leather patches, etc.

The analysis of chlorinated paraffins is a great challenge. The technical compounds are always complex mixtures of substances with different chain lengths and different chlorination degrees. Gas chromatography (GC) separation of these mixtures show an overlapping part of chain length (between short and middle chained) and of chlorination degrees, too. The responses of the different chlorination degrees vary in a big range. This document describes a procedure to get comparable results for SCCPs and MCCPs with a defined calibration standard of the most typical used mixtures (59 % chlorination ree i raphy i. degree for SCCPs and 55 % chlorination degree for MCCPs) and using four ion traces for SCCPs and four ion traces for MCCPs with gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS).

Textiles — Determination of short-chain chlorinated paraffins (SCCP) and middle-chain chlorinated paraffins (MCCP) in textile products out of different matrices by use of gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS)

WARNING — This document calls for the use of substances/procedures that may be injurious to the health/environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.

1 Scope

This document specifies a chromatographic method to determine the amount of short-chain chlorinated paraffins (SCCPs: C_{10} - C_{13}) and middle-chain chlorinated paraffins (MCCPs: C_{14} - C_{17}) in textile articles, especially in polymer of the coated fabrics, prints made of polymer and buttons made of polymer (e.g. polyvinylchloride) by means of solvent extraction and gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS).

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 4787, Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use

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

The test specimens are extracted with toluene, followed by a sulfuric acid clean-up. The analysis is carried out using GC-NCI-MS with a defined calibration standard and eight typical ion traces.

It shall be emphasized that this method is a method of convention. All steps shall be performed as described. Variations in procedure induce deviant results.

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 *n***-Hexane**, CAS no. 110-54-3.