
Tekstilje in tekstilni izdelki - Per- in polifluoroalkilne snovi (PFAS) - 1. del: Analiza alkalnega ekstrakta s tekočinsko kromatografijo in tandemsko masno spektrometrijo

Textiles and textile products - Per- and polyfluoroalkyl substances (PFAS) - Part 1: Analysis of an alkaline extract using liquid chromatography and tandem mass spectrometry

Textilien und textile Erzeugnisse - Per- und polyfluorierte Alkylverbindungen (PFAS) - Teil 1: Untersuchung eines alkalischen Extraktes mittels Flüssigkeitschromatographie und Tandem-Massenspektrometrie

Textiles et produits textiles - Substances perfluoroalkylées et polyfluoroalkylées (PFAS) - Partie 1 : Analyse d'un extrait alcalin par chromatographie en phase liquide et spectrométrie de masse en tandem

<https://standards.iteh.ai/catalog/standards/sist/22dec5fa-72be-4bc1-897b-bcfa46a0a81f/osist-pren-17681-1-2024>

Ta slovenski standard je istoveten z: prEN 17681-1

ICS:

59.080.01	Tekstilije na splošno	Textiles in general
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

oSIST prEN 17681-1:2024

en,fr,de

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

DRAFT
prEN 17681-1

November 2023

ICS 59.080.01

Will supersede EN 17681-1:2022

English Version

**Textiles and textile products - Per- and polyfluoroalkyl
substances (PFAS) - Part 1: Analysis of an alkaline extract
using liquid chromatography and tandem mass
spectrometry**

Textiles et produits textiles - Fluor organique - Partie 1
: Détermination de composés non volatils par une
méthode d'extraction utilisant la chromatographie en
phase liquide

Textilien und textile Erzeugnisse - Per- und
Polyfluorierte Alkylverbindungen (PFAS) - Teil 1:
Untersuchung eines alkalischen Extraktes mittels
Flüssigkeitschromatographie und Tandem-
Massenspektrometrie

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 248.

If this draft becomes a European Standard, 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.

This draft European Standard was established by CEN 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, Türkiye and United Kingdom.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents	Page
European foreword.....	4
Introduction	6
1 Scope	16
2 Normative references	16
3 Terms and definitions	16
4 Principle	16
5 Reagents	17
6 Equipment	17
7 Sampling	18
7.1 General.....	18
7.2 Sampling based on area	18
7.3 Sampling based on mass.....	18
7.4 Preparation of test specimen.....	18
8 Procedure	19
8.1 Preparation of stock solutions	19
8.2 Preparation of the check solution.....	19
8.3 Preparation of calibration solutions.....	19
8.4 Extraction.....	19
8.5 Analysis.....	19
9 Expression of results	20
9.1 Calibration	20
9.2 Calculation of the results of individual PFAS	20
9.3 Calculation of the sums of certain PFAS.....	21
9.4 Precision of the test method	21
10 Test report	21
Annex A (normative) Usable ions and reachable quantification limits for PFAS analysis of sample matrix by LC-MS/MS	22
Annex B (informative) Interferences with LC-MS/MS	25
Annex C (informative) Non-regulated PFAS	26
Annex D (informative) Chromatographic conditions	27
D.1 General.....	27
D.2 Chromatographic conditions	27
D.3 MS/MS conditions	28
Annex E (informative) Reliability of the test method	29
Annex F (informative) Extraction with methanol	33
F.1 General.....	33
F.2 Procedure.....	33
Annex G (informative) Analysis with GC-MSD	34

G.1	General	34
G.2	Reagents	34
G.3	Equipment	34
G.4	Procedure	34
G.4.1	Preparation of stock solutions	34
G.4.2	Preparation of internal standard solution	34
G.4.3	Preparation of calibration solutions	35
G.4.4	Analysis	35
G.5	Expression of results	35
G.5.1	Calibration	35
G.5.2	Calculation of the results	35
G.5.3	Reliability of the method	36
G.6	Test report	36
G.7	Usable ions and LOQ for PFAS analysis by GC	37
G.7.1	Using GC-MS/MS	37
G.7.2	Using GC-MS/PCI	38
G.7.3	Using GC-MS/EI	39
G.8	Interferences with GC	40
G.9	Instrumental conditions	40
G.9.1	General	40
G.9.2	GC-PCI/MS	41
G.9.3	GC-MS/EI	42
G.10	Reliability of the method	42
	Bibliography	43

prEN 17681-1:2023 (E)

European foreword

This document (prEN 17681-1:2023) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 17681-1:2022.

prEN 17681-1:2023 includes the following significant technical changes with respect to EN 17681-1:2022:

- Title has been changed;
- Introduction has been amended;
- Table 2 has been reordered and updated with regard to new EU regulations. The substance names have largely been aligned with prEN 17892:2022 [10];
- Clause 1: The scope has been changed considering extraction by alkaline hydrolysis now;
- Clause 2: EN 17681-2 has been deleted;
- Terms and definitions 3.1 and 3.2 have been added;
- Clause 4 has been changed, describing extraction by alkaline hydrolysis now;
- Clause 5 has been amended and reorganised;
- Clause 6 has been amended by subclause 6.12;
- Subclause 7.1: The note has been deleted;
- Subclause 8.4 has been technically revised, describing extraction by alkaline hydrolysis now;
- Subclause 8.4 has been amended and technically revised;
- Subclause 9.3 has been rephrased and amended;
- Subclause 9.4: The title and the content have been revised;
- Subclause 9.5: The subclause has been deleted, statistical data on the reliability are presented in Annex E now;
- Clause 10 has been rephrased;
- Table A.1: The substance names have largely been aligned with prEN 17892:2022 [10];
- Table C.1: Table 2 has been reordered and updated with regard to new EU regulations. The substance names have largely been aligned with prEN 17892:2022 [10];
- Annex E has been added;
- Annex F describing the methanol extraction according to EN 17681-1:2022 [13] has been added;

- Annex G describing the analysis of certain PAFS with gas chromatography with mass selective detector (GC-MSD) according to EN 17681-2:2022 has been added;
- Bibliography: Further references have been added.

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[oSIST prEN 17681-1:2024](https://standards.iteh.ai/catalog/standards/sist/22dec5fa-72be-4bc1-897b-bcfa46a0a81f/osist-pren-17681-1-2024)

<https://standards.iteh.ai/catalog/standards/sist/22dec5fa-72be-4bc1-897b-bcfa46a0a81f/osist-pren-17681-1-2024>

prEN 17681-1:2023 (E)

Introduction

In the European Union, according to Regulation (EU) 2019/1021 on persistent organic pollutants (POP) [1], Article 3, Clause 1, in connection with Annex I, amended by Commission Delegated Regulation (EU) 2020/784 [9] and Commission Delegated Regulation (EU) 2023/1608 [12] the manufacturing, placing on the market and use of the following per- and polyfluoroalkyl substances (PFAS) whether on their own, in mixtures or in articles, is prohibited:

- Perfluorooctane sulfonic acid and its derivatives (PFOS);
- Perfluorooctanoic acid (PFOA), its salts and PFOA-related compounds;
- Perfluorohexane sulfonic acid (PFHxS), its salts and PFHxS-related compounds.

According to Article 4 Clause 1. (b) this does not apply in the case of a substance present as an unintentional trace contaminant, as specified in the relevant entries of Annex I in substances, mixtures or articles.

Annex I Part A, describing perfluorooctane sulfonic acid and its derivatives (PFOS), contains the specific exemption (Point 2) to concentrations of PFOS in semi-finished products or articles, or parts thereof, if the concentration of PFOS is lower than 0,1 % mass fraction calculated with reference to the mass of structurally or micro-structurally distinct parts that contain PFOS or, for textiles or other coated materials, if the amount of PFOS is lower than $1 \mu\text{g}/\text{m}^2$ of the coated material. PFOS compounds have the formula $\text{C}_8\text{F}_{17}\text{SO}_2\text{X}$ where $\text{X} = \text{OH}$, Metal salt (O-M^+), halide, amide and other derivatives, including polymers.

Annex I Part A, describing perfluorooctanoic acid (PFOA), its salts and PFOA-related compounds, contains the specific exemption (Point 1) to concentrations of PFOA or any of its salts equal to or below 0,025 mg/kg (0,000 002 5 % mass fraction), where they are present in substances, mixtures or articles. In addition, (Point 2) Article 4(1) applies to concentrations of any individual PFOA-related compound, or a combination of PFOA-related compounds equal to or below 1 mg/kg (0,000 1 % mass fraction) where they are present in substances, mixtures or articles.

PFOA, its salts and PFOA-related compounds means the following:

- i) perfluorooctanoic acid, including any of its branched isomers;
- ii) its salts;
- iii) PFOA-related compounds which, for the purposes of the Convention, are any substances that degrade to PFOA, including any substances (including salts and polymers) having a linear or branched perfluoroheptyl group with the moiety $(\text{C}_7\text{F}_{15})\text{C}$ as one of the structural elements.

The following compounds are not included as PFOA-related compounds:

- iv) $\text{C}_8\text{F}_{17}\text{-X}$, where $\text{X} = \text{F}, \text{Cl}, \text{Br}$;
- v) fluoropolymers that are covered by $\text{CF}_3[\text{CF}_2]_n\text{-R}'$, where $\text{R}' = \text{any group}$, $n > 16$;
- vi) perfluoroalkyl carboxylic acids (including their salts, esters, halides and anhydrides) with ≥ 8 perfluorinated carbons;
- vii) perfluoroalkane sulfonic acids and perfluoro phosphonic acids (including their salts, esters, halides and anhydrides) with ≥ 9 perfluorinated carbons;

viii) perfluorooctane sulfonic acid and its derivatives (PFOS), as listed in Annex I.

In Annex I Part A, describing perfluorohexane sulfonic acid (PFHxS), its salts and PFHxS-related compounds, contains the specific exemption (Point 1) to concentrations of PFHxS or any of its salts equal to or below 0,025 mg/kg (0,000 002 5 % mass fraction), where they are present in substances, mixtures or articles. In addition, (Point 2) Article 4(1) applies to concentrations of all PFHxS-related compounds equal to or below 1 mg/kg (0,000 1 % mass fraction) where they are present in substances, mixtures or articles.

PFHxS, its salts and PFHxS-related compounds means the following:

- i) perfluorohexane sulfonic acid, including any of its branched isomers;
- ii) its salts;
- iii) PFHxS-related compounds which, for the purposes of the Convention, are any substance that contains the chemical moiety $C_6F_{13}S-$ as one of its structural elements and that degrades to PFHxS.

Commission Regulation (EU) 2021/1297 [9] amending Regulation (EC) No 1907/2006 (REACH), Annex XVII [7] restricts perfluorocarboxylic acids containing 9 to 14 carbon atoms in the chain (C9-C14 PFCAs), their salts and C9-C14 PFCA-related substances from 25 February 2023.

This restriction concerns the following substances:

Linear and branched perfluorocarboxylic acids of the formula $C_nF_{2n+1}-C(=O)OH$ where $n = 8, 9, 10, 11, 12, \text{ or } 13$ (C9-C14 PFCAs), including their salts, and any combinations thereof;

Any C9-C14 PFCA-related substance having a perfluoro group with the formula $C_nF_{2n+1}-$ directly attached to another carbon atom, where $n = 8, 9, 10, 11, 12, \text{ or } 13$, including their salts and any combinations thereof;

Any C9-C14 PFCA-related substance having a perfluoro group with the formula $C_nF_{2n+1}-$ that it is not directly attached to another carbon atom, where $n = 9, 10, 11, 12, 13 \text{ or } 14$ as one of the structural elements, including their salts and any combinations thereof.

The following substances are excluded from this designation

$C_nF_{2n+1}-X$, where $X = F, Cl, \text{ or } Br$

where $n = 9, 10, 11, 12, 13 \text{ or } 14$, including any combinations thereof,

$C_nF_{2n+1}-C(=O)OX'$ where $n > 13$ and $X' = \text{any group, including salts.}$

Several per- and poly-fluorinated alkylated substances (PFAS), which are not restricted under the POP Regulation have been added as Substances of Very High Concern (SVHC) to the Candidate List according to Regulation (EC) No 1907/2006 (REACH), Article 59.

Per- and poly-fluorinated compounds from C4 – C14 (PFAS) occur, for example, in soil and water repellent finishes within textiles or can be introduced as contaminants (for example from water sources). Categories of PFAS are shown in Table 1. Table 2 lists classes of regulated compounds (i.e., listed in a Regulation) including acids, telomers, sulfonates and sulfonamidalcohols. Compounds of concern (i.e., not listed yet in a Regulation) are shown in the informative Annex C.

Further EU regulation concerning additional PFAS are under development.

prEN 17681-1:2023 (E)

WARNING — The use of this document involves hazardous materials. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the document and fulfil statutory and regulatory requirements for this purpose.

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[oSIST prEN 17681-1:2024](https://standards.iteh.ai/catalog/standards/sist/22dec5fa-72be-4bc1-897b-bcfa46a0a81f/osist-pren-17681-1-2024)

<https://standards.iteh.ai/catalog/standards/sist/22dec5fa-72be-4bc1-897b-bcfa46a0a81f/osist-pren-17681-1-2024>

Table 1 — Categories of PFAS

Type of PFAS	Sub-group	Applications		Category
		Use	Sources of contamination ^a	
PFAS salts	K ⁺ , Li ⁺ , diethanolamine (DEA) salt analysed as acids	Surfactant for alkaline cleaners	Surfactant in fire-fighting foam, emulsifier in floor polish mist, suppressant for metal plating baths, surfactant for etching acids for circuit boards, pesticide active ingredient for ant bait traps	A
	Amines	-	Mist suppressant for metal plating baths	B
	Ammonium salts analysed as acids	-	Emulsifier for fluoropolymer production	C
	Amphoterics	Water/solvent repellence for leather/paper	-	D
PFAS substances	Carboxylates	-	Antistatic agent in photographic paper	E
	Amides	-	Pesticide active ingredient	F
	Oxazolidinones	-	Waterproofing casts (electronics)	G
PFAS polymers/oligomers	Alcohols, silanes, alkoxyates, fatty acid esters, adipates, urethanes, polyesters, acrylates	Soil and water repellence for carpets, fabrics, upholstery, apparel, leather, metal, glass	-	H
	Copolymers, phosphate esters	Water repellence for carpets, fabrics, upholstery, apparel, leather, metal, glass	Soil, oil and water repellence for plates, food containers, bags, wraps, folding cartons, containers, carbonless forms, masking papers	I

^a These substances are not relevant in the manufacturing process of textiles, but it is possible to find them as contaminants.

Table 2 —PFAS regulated in the EU ^a

No.	Compound		CAS Registry Number® (CAS RN®) ¹	Applicable chromatographic system		EU regulation ^a	PFAS category (Table 1)
				Liquid chromatography	Gas chromatography		
Perfluoro-<i>n</i>-hexanoic acid and salts ^b							
1	PFHxA	Perfluoro- <i>n</i> -hexanoic acid	307-24-4	x		Proposed for amending REACH annex XVII	A and C
1.1 ^b	APFHx	Perfluoro- <i>n</i> -hexanoic acid salts - Ammonium perfluoro- <i>n</i> -hexanoate	21615-47-4	x		Proposed for amending REACH annex XVII	A and C
Perfluoro-<i>n</i>-hexanoic acid related substances ^c							
2	6:2 FTOH	6:2 Fluorotelomer alcohol	647-42-7	x		Proposed for amending REACH annex XVII	H
3	6:2 FTA	6:2 Fluorotelomer acrylate	17527-29-6		x ^f	Proposed for amending REACH annex XVII	H
4	6:2 FTMA	6:2 Fluorotelomer methacrylate	2144-53-8		x ^f	Proposed for amending for REACH annex XVII	H
5	6:2 FTS	6:2 Fluorotelomer sulfonic acid	27619-97-2	x		Proposed for amending REACH annex XVII	A

¹ CAS Registry Number® (CAS RN®) is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named.