



**SLOVENSKI STANDARD**  
**oSIST prEN 17681-1:2021**  
**01-junij-2021**

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**Tekstil in tekstilni izdelki - Organski fluor - 1. del: Določevanje nehlapnih spojin z ekstrakcijsko metodo s tekočinsko kromatografijo**

Textiles and textile products - Organic fluorine - Part 1: Determination of non-volatile compounds by extraction method using liquid chromatography

Textilien und textile Erzeugnisse - Organisches Fluor - Teil 1: Bestimmung des Gehaltes an nichtflüchtigen Verbindungen durch Extraktionsverfahren mittels Flüssigkeitschromatographie

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**ICS:**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**DRAFT**  
**prEN 17681-1**

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ICS 59.080.01

English Version

**Textiles and textile products - Organic fluorine - Part 1:  
Determination of non-volatile compounds by extraction  
method using liquid chromatography**

Textilien und textile Erzeugnisse - Organisches Fluor -  
Teil 1: Bestimmung des Gehaltes an nichtflüchtigen  
Verbindungen durch Extraktionsverfahren mittels  
Flüssigkeitschromatographie

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.

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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.

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

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

<b>Contents</b>	<b>Page</b>
European foreword.....	3
Introduction .....	4
1 Scope.....	9
2 Normative references.....	9
3 Terms and definitions .....	9
4 Principle of method.....	9
5 Reagents .....	9
5.4 Selected PFAS listed in Table 2 and Table C.1.....	10
6 Equipment .....	10
7 Sampling.....	10
7.1 General.....	10
7.2 Sampling based on area .....	10
7.3 Sampling based on mass.....	11
7.4 Preparation .....	11
7.4.1 Preparation of test specimen.....	11
8 Procedure.....	11
8.1 Preparation of stock solutions.....	11
8.2 Preparation of the check solution.....	11
8.3 Preparation of calibration solutions.....	11
8.4 Extraction.....	11
8.5 Analysis.....	12
9 Expression of results.....	12
9.1 Calibration .....	12
9.2 Calculation of the results.....	12
9.3 Calculation of the results of a sum .....	13
9.4 Limit of detection (LoD) and limit of quantification (LoQ).....	13
10 Test report.....	13
Annex A (normative) Usable ions and LOQ for PFAS analysis by LC/MS/MS.....	14
Annex B (informative) Interferences with LC/MS/MS.....	16
Annex C (informative) Non-regulated PFAS .....	17
Annex D (informative) Chromatographic conditions.....	19
D.1 General.....	19
D.2 Chromatographic conditions .....	19
D.3 LC/MS/MS conditions .....	19
Bibliography .....	20

## European foreword

This document (prEN 17681-1:2021) 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.

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**prEN 17681-1:2021 (E)****Introduction**

In the European Union, according to Regulation (EU) 2019/1021 persistent organic pollutants (POP), Article 3, Clause 1, in connection with Annex I, amended by COMMISSION DELEGATED REGULATION (EU) 2020/784, the manufacturing, placing on the market and use of perfluorooctanoic acid (PFOA), its salts and PFOA-related compounds, whether on their own, in mixtures or in articles, shall be prohibited. This is in addition to the existing prohibition of perfluorooctane sulfonic acid and its derivatives (PFOS).

According to Article 4 Clause 1. (b) this shall 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 % by weight 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 µg/m<sup>2</sup> of the coated material. PFOS compounds have the formula C<sub>8</sub>F<sub>17</sub>SO<sub>2</sub>X where X = OH, Metal salt (O-M<sup>+</sup>), halide, amide and other derivatives, including polymers.

Annex I also states (point 5) that once standards are adopted by the European Committee for Standardization (CEN) they shall be used as the analytical test methods for demonstrating the conformity of articles to point 2.

In 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 % by weight) where they are present in substances, mixtures or articles. In addition, (Point 2) Article 4(1) shall apply 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 % by weight) 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 (C<sub>7</sub>F<sub>15</sub>)C as one of the structural elements.

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

- (i) C<sub>8</sub>F<sub>17</sub>-X, where X = F, Cl, Br;
- (ii) fluoropolymers that are covered by CF<sub>3</sub>[CF<sub>2</sub>]<sub>n</sub>-R', where R' = any group, n > 16;
- (iii) perfluoroalkyl carboxylic acids (including their salts, esters, halides and anhydrides) with ≥ 8 perfluorinated carbons;
- (iv) perfluoroalkane sulfonic acids and perfluoro phosphonic acids (including their salts, esters, halides and anhydrides) with ≥ 9 perfluorinated carbons;
- (v) perfluorooctane sulfonic acid and its derivatives (PFOS), as listed in Annex I.

As a further exemption in Annex I Part A (Point 5 c) the manufacturing, placing on the market and use of PFOA, its salts and PFOA-related compounds shall be allowed in textiles for oil and water repellency for the protection of workers from dangerous liquids that comprise risks to their health and safety, until 4 July 2023.

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 (e.g. from water sources). Categories of PFAS are shown in Table 1. Table 2 lists classes of regulated compounds (i.e. listed in regulation) including acids, telomers, sulfonates and sulfonamidalcohols. Compounds of concern (i.e. not listed yet in regulation) are shown in the informative Annex C.

**Table 1 — Categories of PFAS**

Type of PFAS	Sub-group	Applications		Category
		Use	Sources of contamination <sup>a</sup>	
PFAS salts	K <sup>+</sup> , Li <sup>+</sup> , 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/water repellence for carpets, fabric/upholstery, apparel, leather, metal/glass		H
	Copolymers, phosphate esters	Water repellence for carpet, fabric/upholstery, apparel, leather, metal/glass	Soil/oil/water repellence for plates, food containers, bags, wraps, folding cartons, containers, carbonless forms, masking papers	I

<sup>a</sup> These substances are not relevant in the manufacturing process of textiles but it is possible to find them as contaminants.

Table 2 — Regulated PFAS

No	Chemicals	CAS No.	LC	GC	law (§) <sup>a</sup>	PFAS category (Table 1)	
<b>Perfluorinated carboxylic acids</b>							
1	PFHxA	Perfluoro-n-hexanoic acid	307-24-4	x		under evaluation (REACH)	A and C
2 <sup>b</sup>	PFOA	Perfluoro-n-octanoic acid	335-67-1	x		POP	A and C
2.2 <sup>b</sup>	APFO Na-PFO K-PFO Ag-PFO F-PFO	Perfluoro-n-octanoic salts - Ammonium pentadecafluorooctanoate - Sodium perfluorooctanoate - Potassium perfluorooctanoate - Silver perfluorooctanoate - Perfluorooctanoyl fluoride	3825-26-1 335-95-5 2395-00-8 335-93-3 335-60-0	x		POP	C A A A A
3 <sup>b</sup>	8:2 FTS	1H,1H,2H,2H-Perfluorodecanesulfonic acid	39108-34-4	x		POP	A
4 <sup>b</sup>	Me-PFOA	Methyl perfluorooctanoate	376-27-2		x	POP	H
5 <sup>b</sup>	Et-PFOA	Ethyl perfluorooctanoate	3108-24-5		x	POP	H
6	PFNA	Perfluoro-n-nonanoic acid	375-95-1	x		Reach (SVHC)	
7	PFDA	Perfluoro-n-decanoic acid	335-76-2	x		Reach (SVHC)	
8	PFUnA	Perfluoroundecanoic acid	2058-94-8	x		Reach (SVHC)	
9	PFDoA	Perfluorododecanoic acid	307-55-1	x		Reach (SVHC)	
10	PFTrA	Perfluorotridecanoic acid	72629-94-8	x		Reach (SVHC)	
11	PFTeA	Perfluorotetradecanoic acid	376-06-7	x		Reach (SVHC)	
12	PF-3,7-DMOA	Perfluoro(3,7-dimethyloctanoic acid)	172155-07-6	x		POP	
13 <sup>b</sup>	4HPFUnA	2H,2H,3H,3H-Heptadecafluoroundecanoic Acid	34598-33-9	x		POP	



No	Chemicals		CAS No.	LC	GC	law (§) <sup>a</sup>	PFAS category (Table 1)
<b>Perfluorinated sulfonic acids</b>							
14	PFBS	Perfluorobutanesulfonic acid	375-73-5	x		Reach (SVHC)	A
15	PFHxS	Perfluorohexanesulfonic acid	355-46-4	x		Reach (SVHC)	H
16 <sup>c</sup>	PFOS	Perfluoro octanesulfonic acid	1763-23-1	x		POP	H
16.2 <sup>c</sup>	PFOS-X	Perfluorooctane sulfonic acid salts C <sub>8</sub> F <sub>17</sub> SO <sub>2</sub> X - potassium Perfluorooctane sulfonate - lithium Perfluorooctane sulfonate - ammonium Perfluorooctane sulfonate - bis2(hydroxyethyl)ammonium Perfluorooctane sulfonate - tetraethyl ammonium heptadecafluorooctane sulfonate	2795-39-3 29457-72-5 29081-56-9 70225-14-8 56773-42-3	x		POP	A A A A A
<b>Perfluoro-octanesulfonamides (FOSA)</b>							
17	PFOSA	Perfluorooctane sulfonamide	754-91-6	x		POP	F
18	N-MeFOSA	N-Methylperfluoro-1-octanesulfonamide	31506-32-8	x		POP	F
19	N-EtFOSA	N-Ethylperfluoro-1-octanesulfonamide	4151-50-2	x		POP	F
<b>Perfluoro-octanesulfonamido ethanol (FOSE)</b>							
20	N-MeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	x	x	POP	H
21	N-EtFOSE	2-(N-Ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	x	x	POP	H
<b>Fluorinated telomer alcohols (FTOH)</b>							
22 <sup>b</sup>	8:2 FTOH	2-Perfluorooctylethanol	678-39-7	x		POP	H
<b>Fluorinated telomer acrylate (FTA)</b>							
23 <sup>b</sup>	8:2 FTA	1H,1H,2H,2H-Perfluorodecyl acrylate	27905-45-9		x	POP	H
<b>Other</b>							
24 <sup>c</sup>	PFOSF	Heptadecafluorooctanesulfonyl fluoride	307-35-7	x		POP	
25 <sup>b</sup>	8:2 FTMA	1H,1H,2H,2H-heptadecafluorodecylmethacrylate	1996-88-9		x	POP	
26		1-decanaminium, N-decyl-N, N dimethyl-0,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-1-octanesulfonate	251099-16-8	x		POP	A
27	HPFO-DA	2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)propionic acid	13252-13-6	x		Reach (SVHC)	A and C

## prEN 17681-1:2021 (E)

No	Chemicals		CAS No.	LC	GC	law (§) <sup>a</sup>	PFAS category (Table 1)
27.2 <sup>c</sup>	HPFO-DA -X	2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)propionic acid salts and acyl halides X = F X = NH <sub>4</sub> X = K	21062-98-8 62037-80-3 67118-55-2	x x x		Reach (SVHC)	A and C
<sup>a</sup> Table 2 states the situation of the regulated PFAS on 2021-03-1. Readers should pay attention to update the information. <sup>b</sup> The results of PFOA related substances to be summed up. <sup>c</sup> For the halides a hydrolysis with water from the methanol extract is necessary.							

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## 1 Scope

This document specifies a test method (using liquid chromatography, LC) for detection and quantification of selected extractable perfluorinated and polyfluorinated substances in textile materials (fibres, yarns, fabrics) and coated fabrics.

NOTE CEN/TR 16741 defines which materials are applicable to this determination.

A test method (using gas chromatography, GC) for detection and quantification of selected extractable perfluorinated and polyfluorinated substances is specified in prEN 17681-2.

Classes of regulated compounds are listed in Table 2. Classes of other non-regulated compounds that can be determined by this document are defined in Annex C, Table C.1. This document is also applicable for further PFAS substances provided that the method is validated with the additional compounds.

## 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.

prEN 17681-2, *Textiles and textile products — Organic fluorine — Part 2: Determination of volatile compounds by extraction method using gas chromatography*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use (ISO 4787)*

EN ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing (ISO 5089)*

## 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:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

## 4 Principle of method

The selected compounds listed in Table 2 and in Table C.1 are extracted in an ultrasonic bath with methanol and the extract is analysed by liquid chromatography with a tandem mass spectrometric detector (LC-MS/MS).

## 5 Reagents

Unless otherwise specified, all reagents shall be of a recognized analytical grade.

**5.1 Methanol**, LC-MS grade, CAS number 67-56-1

**5.2 Check standards**

**5.2.1 Perfluoro-n-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]-octanoic-acid**