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Responsible committee: TCI/100 Co-ordination of activities in textiles and clothing

Interested committees:

Title: BS EN 17681-1 Textiles and textile products - Per- and polyfluoroalkyl substances (PFAS)

Part 1: Determination of non-volatile compounds by extraction method using liquid chromatography

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- iii) requires additional national guidance or information

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/Figure/ Table/Note	Type of comment	Commend (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

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

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.

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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 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.

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.

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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.

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> -heptanoic acid							
6	PFHpA	Perfluoro- <i>n</i> -heptanoic acid	375–85–9	x		REACH (SVHC)	
Perfluoro- <i>n</i> -octanoic acid and salts ^b							
7	PFOA	Perfluoro- <i>n</i> -octanoic acid	335–67–1	x		POP and REACH (SVHC)	A and C
7.1 ^b	APFO Na-PFO K-PFO Ag-PFO F-PFO	Perfluoro- <i>n</i> -octanoic salts - Ammonium perfluorooctanoate - Sodium perfluoro- <i>n</i> -octanoate - Potassium perfluoro- <i>n</i> -octanoate - Silver perfluoro- <i>n</i> -octanoate - Perfluoro- <i>n</i> -octanoyl fluoride	3825–26–1 335–95–5 2395–00–8 335–93–3 335–66–0	x		POP	C A A A A
Perfluoro- <i>n</i> -octanoic acid related substances ^c							
8	Me-PFOA	Methyl perfluoro- <i>n</i> -octanoate	376–27–2		x ^e	POP	H
9	Et-PFOA	Ethyl perfluoro- <i>n</i> -octanoate	3108–24–5		x ^e	POP	H
10	8:2 FTOH	8:2 Fluorotelomer alcohol	678–39–7	x		POP	H
11	8:2 FTA	8:2 Fluorotelomer acrylate	27905–45–9		x ^f	POP	H
12	8:2 FTMA	8:2 Fluorotelomer methacrylate	1996–88–9		x ^f	POP	H
14	8:2 FTS	8:2 Fluorotelomer sulfonic acid	39108–34–4	x		POP	A

No.	Compound		CAS Registry Number® (CAS RN®) ¹	Applicable chromatographic system		EU regulation ^a	PFAS category (Table 1)
				Liquid chromatography	Gas chromatography		
C9-C14 Perfluorocarboxylic acids and salts ^c							
14	PFNA	Perfluoro- <i>n</i> -nonanoic acid	375-95-1	x		REACH Annex XVII and SVHC	A
14.1 ^b	NH ₄ -PFN Na-PFN	Perfluoro- <i>n</i> -nonanoic acid salts - Ammonium perfluoro- <i>n</i> -nonanoate - Sodium perfluoro- <i>n</i> -nonanoate	4149-60-4 21049-39-8	x		REACH Annex XVII and SVHC	A
15	PFDA	Perfluoro- <i>n</i> -decanoic acid	335-76-2	x		REACH Annex XVII and SVHC	A
15.1 ^b	NH ₄ -PFD Na-PFD	Perfluoro- <i>n</i> -decanoic acid salts - Ammonium perfluoro- <i>n</i> -decanoate - Sodium perfluoro- <i>n</i> -decanoate	3108-42-7 3830-45-3	x		REACH Annex XVII and SVHC	A and C
16	PFUnA	Perfluoro- <i>n</i> -undecanoic acid	2058-94-8	x		REACH Annex XVII and SVHC	A
17	PFDoA	Perfluoro- <i>n</i> -dodecanoic acid	307-55-1	x		REACH Annex XVII and SVHC	A
18	PFTTrDA	Perfluoro- <i>n</i> -tridecanoic acid	72629-94-8	x		REACH Annex XVII and SVHC	A
19	PFTeDA	Perfluoro- <i>n</i> -tetradecanoic acid	376-06-7	x		REACH Annex XVII and SVHC	A
20	PF-3,7-DMOA	Perfluoro(3,7-dimethyl- <i>n</i> -octanoic acid)	172155-07-6	x		REACH Annex XVII	A