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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

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

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

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

This European Standard was approved by CEN on 24 July 2022.

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## European foreword

This document (EN 17681-1:2022) has been prepared by 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of an identical text or by endorsement, at the latest by March 2023, and conflicting national standards shall be withdrawn at the latest by March 2023.

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## EN 17681-1:2022 (E)

## Introduction

In the European Union, according to Regulation (EU) 2019/1021 on 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, is 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 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 ( $\text{O-M}^+$ ), halide, amide and other derivatives, including polymers.

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 % 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  $\geq 8$  perfluorinated carbons;
- vii) perfluoroalkane sulfonic acids and perfluoro phosphonic acids (including their salts, esters, halides and anhydrides) with  $\geq 9$  perfluorinated carbons;
- viii) 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 is 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.

Commission Regulation (EU) 2021/1297 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.

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 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/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.                                    | Substance                                  |  | CAS Registry Number® (CAS RN®) <sup>1</sup>                | Applicable test method |            | EU regulation <sup>a</sup> | PFAS category (Table 1) |
|--|--|--|--|------------------------|------------|----------------------------|-------------------------|
|  |  |  |  | EN 17681-1             | EN 17681-2 |                            |                         |
| <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 and REACH (SVHC)       | A and C                 |
| 2.2 <sup>b</sup>                       | APFO<br>Na-PFO<br>K-PFO<br>Ag-PFO<br>F-PFO | Perfluoro-n-octanoic acid salts<br>- Ammonium pentadecafluorooctanoate<br>- Sodium perfluorooctanoate<br>- Potassium perfluorooctanoate<br>- Silver perfluorooctanoate<br>- Perfluorooctanoyl fluoride | 3825-26-1<br>335-95-5<br>2395-00-8<br>335-93-3<br>335-66-0 | x                      |            | POP                        | C<br>A<br>A<br>A<br>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 Annex XVII and SVHC  |                         |
| 6.2                                    | NH <sub>4</sub> -PFN<br>Na-PFN             | Perfluoro-n-nonanoic acid salts<br>- Ammonium perfluorononanoate<br>- Sodium perfluorononanoate  | 4149-60-4<br>21049-39-8                                    | x                      |            | REACH Annex XVII and SVHC  |                         |

<sup>1</sup> CAS Registry Number® (CAS RN®) is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

## EN 17681-1:2022 (E)

| No.             | Substance                      |   | CAS Registry Number® (CAS RN®) <sup>1</sup> | Applicable test method |            | EU regulation <sup>a</sup> | PFAS category (Table 1) |
|-----------------|--------------------------------|---|---|------------------------|------------|----------------------------|-------------------------|
|                 |                                |   |   | EN 17681-1             | EN 17681-2 |                            |                         |
| 7               | PFDA                           | Perfluoro-n-decanoic acid   | 335-76-2                                    | x                      |            | REACH Annex XVII and SVHC  |                         |
| 7.2             | NH <sub>4</sub> -PFD<br>Na-PFD | Perfluoro-n-decanoic acid salts<br>- Ammonium perfluorodecanoate<br>- Sodium perfluorodecanoate | 3830-45-3<br>3108-42-7                      | x                      |            | REACH Annex XVII and SVHC  |                         |
| 8               | PFOA                           | Perfluoroundecanoic acid  | 2058-94-8                                   | x                      |            | REACH Annex XVII and SVHC  |                         |
| 9               | PFDoA                          | Perfluorododecanoic acid  | 307-55-1                                    | x                      |            | REACH Annex XVII and SVHC  |                         |
| 10              | PFTTrDA                        | Perfluorotridecanoic acid   | 72629-94-8                                  | x                      |            | REACH Annex XVII and SVHC  |                         |
| 11              | PFTeDA                         | Perfluorotetradecanoic acid   | 376-06-7                                    | x                      |            | REACH Annex XVII and SVHC  |                         |
| 12              | PF-3,7-DMOA                    | Perfluoro(3,7-dimethyloctanoic acid)  | 172155-07-6                                 | x                      |            | REACH Annex XVII           |                         |
| 13 <sup>b</sup> | 4HPFOA                         | 2H,2H,3H,3H-Heptadecafluoroundecanoic acid  | 34598-33-9                                  | x                      |            | POP                        |                         |

| No.   | Substance |   | CAS Registry Number® (CAS RN®) <sup>1</sup>                       | Applicable test method |            | EU regulation <sup>a</sup>    | PFAS category (Table 1) |
|---|-----------|---|---|------------------------|------------|-------------------------------|-------------------------|
|   |           |   |   | EN 17681-1             | EN 17681-2 |                               |                         |
| <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      | Perfluorooctanesulfonic 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<br>- Potassium perfluorooctane sulfonate<br>- Lithium perfluorooctane sulfonate<br>- Ammonium perfluorooctane sulfonate<br>- Bis2(hydroxyethyl)ammonium perfluorooctane sulfonate<br>- Tetraethyl ammonium heptadecafluorooctane sulfonate | 2795-39-3<br>29457-72-5<br>29081-56-9<br>70225-14-8<br>56773-42-3 | x                      |            | POP                           | A<br>A<br>A<br>A<br>A   |
| <b>Perfluoro-octanesulfonamides (FOSA)</b>        |           |   |   |                        |            |                               |                         |
| 17 <sup>c</sup>                                   | PFOSA     | Perfluorooctane sulfonamide   | 754-91-6  | x                      |            | POP                           | F                       |
| 18 <sup>c</sup>                                   | N-MeFOSA  | N-Methylperfluoro-1-octanesulfonamide   | 31506-32-8  | x                      |            | POP                           | F                       |
| 19 <sup>c</sup>                                   | N-EtFOSA  | N-Ethylperfluoro-1-octanesulfonamide  | 4151-50-2   | x                      |            | POP                           | F                       |
| <b>Perfluoro-octanesulfonamido ethanol (FOSE)</b> |           |   |   |                        |            |                               |                         |
| 20 <sup>c</sup>                                   | N-MeFOSE  | 2-(N-methylperfluoro-1-octanesulfonamideo)-ethanol  | 24448-09-7  | x                      | x          | POP                           | H                       |
| 21 <sup>c</sup>                                   | 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                       |
| 23  | 10:2 FTOH | 2-Perfluorodecylethanol   | 865-86-1  | x                      |            | REACH Annex XVII <sup>a</sup> | H                       |