



# SLOVENSKI STANDARD

## SIST EN 17892:2024

01-oktober-2024

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**Kakovost vode - Določanje izbranih perfluoroalkilnih in polifluoroalkilnih snovi (PFAS) v pitni vodi - Metoda s tekočinsko kromatografijo s tandemsko masno spektrometrijo (LC-MS/MS)**

Water quality - Determination of selected per- and polyfluoroalkyl substances in drinking water - Method using liquid chromatography/tandem-mass spectrometry (LC-MS/MS)

Wasserbeschaffenheit - Bestimmung ausgewählter Per- und Polyfluoralkylsubstanzen in Trinkwasser - Verfahren mittels Flüssigkeitschromatographie/Tandem-Massenspektrometrie (LC-MS/MS)

Qualité de l'eau - Détermination de substances per- et polyfluoroalkylées sélectionnées dans l'eau potable - Méthode par chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (LC-MS/MS)

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**Ta slovenski standard je istoveten z: EN 17892:2024**

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**ICS:**

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| 13.060.20 | Pitna voda                      | Drinking water                               |
| 13.060.50 | Preiskava vode na kemične snovi | Examination of water for chemical substances |

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Water quality - Determination of selected per- and polyfluoroalkyl substances in drinking water - Method using liquid chromatography/tandem-mass spectrometry (LC-MS/MS)

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This European Standard was approved by CEN on 19 May 2024.

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

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**EN 17892:2024 (E)****European foreword**

This document (EN 17892:2024) has been prepared by the Technical Committee CEN/TC 230 “Water analysis”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by December 2024, and conflicting national standards shall be withdrawn at the latest by December 2024.

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.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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## Introduction

Per- and polyfluoroalkyl substances (PFAS) are industrially manufactured chemicals, that contain at least one fully fluorinated methyl or methylene carbon atom (without any H/Cl/Br/I atom attached to it) [1]. This definition is also chosen in the document (see 3.1). According to the United States Environmental Protection Agency (U.S. EPA), PFAS is a chemical family consisting of more than 8 000 individual substances [2]. They are a group of widely used man-made chemicals. The perfluoroalkyl substances are persistent and can accumulate over time in humans and in the environment. Because of their special properties and stability, some of these compounds were widely used in industry, as components in firefighting foams or for consumer products and can now be found ubiquitous as background contamination in the environment [3].

PFAS - especially the shorter-chain - can enter the water cycle as a result of manufacture, application and disposal. PFAS are included in the EU Drinking Water Directive EU 2020/2184 [4] as parameter to be under surveillance with a maximum parametric limit value of 0,10 µg/l for the sum of 20 selected PFAS, i.e. the perfluorinated carbonic acids as well as the perfluorinated sulfonic acids with chain length of four to thirteen carbon atoms.

Longer-chain compounds such as PFOA, PFNA, PFHxS, and PFOS accumulate in the blood and the liver, and their half-lives in the human body amount to several years. In 2020 the European Food Safety Authority (EFSA) has derived a tolerably weekly intake (TWI) for the sum of the four substances PFOA, PFNA, PFHxS and PFOS of 4,4 ng/kg body weight based on epidemiological studies and the most sensitive effect on the human immune system [5].

Due to the low TWI the EFSA recommends for the four substances PFOA, PFNA, PFHxS, and PFOS, the analysis of at least these four EFSA-PFAS should be possible with a limit of detection far below the maximum parametric limit value of 0,10 µg/l.

**WARNING** — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

**IMPORTANT** — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

## EN 17892:2024 (E)

## 1 Scope

This document specifies a method for the determination of the dissolved fraction of selected perfluoroalkyl and polyfluoroalkyl substances (PFAS) in non-filtrated drinking water using liquid chromatography-tandem mass spectrometry (LC-MS/MS). The applicability of the method to other types of water like fresh waters (e.g. ground water, surface water) or treated wastewater can be validated separately for each individual case.

For each target compound both, eventually occurring branched isomers and the respective non-branched isomer, are quantified together. The selected set of substances determined by this method is representative for a wide variety of PFAS. This method has been validated for the analytes specified in Table 1. The list given in this table can be modified depending on the purpose and focus of the method. The lower application range of this method can vary depending on the sensitivity of the equipment used and the matrix of the samples. For many substances to which this document applies a limit of quantification (LOQ) of 1 ng/l can be achieved. Using high volume direct injection as described in part A or SPE as described in part B of the method allows lower LOQs. Analytical limitations can occur with short-chain PFAS or PFAS with more than ten carbon atoms in the carbon chain. Actual LOQs can depend on the blank values realized by individual laboratories as well.

**NOTE** This document enables the analysis of those 20 PFAS which are listed in point 3 of Part B of Annex III of the EU Drinking Water Directive, EU 2020/2184 [4], for the surveillance of the parametric limit value of 0,10 µg/l for the sum of PFAS.

Furthermore, alternatives and substitutes for these PFAS substances can be analysed using this document as well.

**Table 1 — Analytes for which a determination was validated in accordance with this method**

| Analyte                              | IUPAC name <sup>a)</sup>  | Formula   | Abbreviation | CAS-RN <sup>b)</sup> |
|--------------------------------------|---|---|--------------|----------------------|
| Perfluoro- <i>n</i> -butanoic acid   | 2,2,3,3,4,4,4-Heptafluorobutanoic acid  | C <sub>4</sub> HF <sub>7</sub> O <sub>2</sub>   | PFBA         | 375-22-4             |
| Perfluoro- <i>n</i> -pentanoic acid  | 2,2,3,3,4,4,5,5,5-Nonafluoropentanoic acid  | C <sub>5</sub> HF <sub>9</sub> O <sub>2</sub>   | PFPeA        | 2706-90-3            |
| Perfluoro- <i>n</i> -hexanoic acid   | 2,2,3,3,4,4,5,5,6,6,6-Undecafluorohexanoic acid                                   | C <sub>6</sub> HF <sub>11</sub> O <sub>2</sub>  | PFHxA        | 307-24-4             |
| Perfluoro- <i>n</i> -heptanoic acid  | 2,2,3,3,4,4,5,5,6,6,7,7,7-Tridecafluoroheptanoic acid                             | C <sub>7</sub> HF <sub>13</sub> O <sub>2</sub>  | PFHpA        | 375-85-9             |
| Perfluoro- <i>n</i> -octanoic acid   | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-Pentadecafluorooctanoic acid                        | C <sub>8</sub> HF <sub>15</sub> O <sub>2</sub>  | PFOA         | 335-67-1             |
| Perfluoro- <i>n</i> -nonanoic acid   | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Heptadecafluorononanoic acid                    | C <sub>9</sub> HF <sub>17</sub> O <sub>2</sub>  | PFNA         | 375-95-1             |
| Perfluoro- <i>n</i> -decanoic acid   | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Nonadecafluorodecanoic acid              | C <sub>10</sub> HF <sub>19</sub> O <sub>2</sub> | PFDA         | 335-76-2             |
| Perfluoro- <i>n</i> -undecanoic acid | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heneicosafluoroundecanoic acid     | C <sub>11</sub> HF <sub>21</sub> O <sub>2</sub> | PFUnDA       | 2058-94-8            |
| Perfluoro- <i>n</i> -dodecanoic acid | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,12-Tricosafluorododecanoic acid | C <sub>12</sub> HF <sub>23</sub> O <sub>2</sub> | PFDoDA       | 307-55-1             |



| Analyte                                     | IUPAC name <sup>a)</sup>   | Formula   | Abbreviation | CAS-RN <sup>b)</sup> |
|---|--|---|--------------|----------------------|
| Perfluoro- <i>n</i> -tridecanoic acid       | 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Pentacosfluorotridecanoic acid              | C <sub>13</sub> HF <sub>25</sub> O <sub>2</sub>                 | PFTrDA       | 72629-94-8           |
| Perfluoro- <i>n</i> -butanesulfonic acid    | 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonic acid   | C <sub>4</sub> HF <sub>9</sub> O <sub>3</sub> S                 | PFBS         | 375-73-5             |
| Perfluoro- <i>n</i> -pentanesulfonic acid   | 1,1,2,2,3,3,4,4,5,5,5-Undecafluoropentane-1-sulfonic acid  | C <sub>5</sub> HF <sub>11</sub> O <sub>3</sub> S                | PFPeS        | 2706-91-4            |
| Perfluoro- <i>n</i> -hexanesulfonic acid    | 1,1,2,2,3,3,4,4,5,5,6,6,6-Tridecafluorohexane-1-sulfonic acid  | C <sub>6</sub> HF <sub>13</sub> O <sub>3</sub> S                | PFHxS        | 355-46-4             |
| Perfluoro- <i>n</i> -heptanesulfonic acid   | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,7-Pentadecafluoroheptane-1-sulfonic acid                                   | C <sub>7</sub> HF <sub>15</sub> O <sub>3</sub> S                | PFHpS        | 375-92-8             |
| Perfluoro- <i>n</i> -octanesulfonic acid    | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-Heptadecafluorooctane-1-sulfonic acid                                | C <sub>8</sub> HF <sub>17</sub> O <sub>3</sub> S                | PFOS         | 1763-23-1            |
| Perfluoro- <i>n</i> -nonanesulfonic acid    | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Nonaadecafluorononane-1-sulfonic acid                            | C <sub>9</sub> HF <sub>19</sub> O <sub>3</sub> S                | PFNS         | 68259-12-1           |
| Perfluoro- <i>n</i> -decanesulfonic acid    | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heneicosfluorodecane-1-sulfonic acid                      | C <sub>10</sub> HF <sub>21</sub> O <sub>3</sub> S               | PFDS         | 335-77-3             |
| Perfluoro- <i>n</i> -undecanesulfonic acid  | 1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Tricosfluoroundecane-1-sulfonic acid                  | C <sub>11</sub> HF <sub>23</sub> O <sub>3</sub> S               | PFUnDS       | 749786-16-1          |
| Perfluoro- <i>n</i> -dodecanesulfonic acid  | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,12-Pentacosfluorododecane-1-sulfonic acid        | C <sub>12</sub> HF <sub>25</sub> O <sub>3</sub> S               | PFDoDS       | 79780-39-5           |
| Perfluoro- <i>n</i> -tridecanesulfonic acid | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Heptacosfluorotridecane-1-sulfonic acid | C <sub>13</sub> HF <sub>27</sub> O <sub>3</sub> S               | PFTrDS       | 791563-89-8          |
| 4:2 Fluorotelomer sulfonic acid             | 3,3,4,4,5,5,6,6,6-Nonafluorohexane-1-sulfonic acid   | C <sub>6</sub> H <sub>5</sub> F <sub>9</sub> O <sub>3</sub> S   | 4:2 FTSA     | 757124-72-4          |
| 6:2 Fluorotelomer sulfonic acid             | 3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctane-1-sulfonic acid  | C <sub>8</sub> H <sub>5</sub> F <sub>13</sub> O <sub>3</sub> S  | 6:2 FTSA     | 27619-97-2           |
| 8:2 Fluorotelomer sulfonic acid             | 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecane-1-sulfonic acid                             | C <sub>10</sub> H <sub>5</sub> F <sub>17</sub> O <sub>3</sub> S | 8:2 FTSA     | 39108-34-4           |
| Perfluorooctanesulfonamide                  | 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-Heptadecafluoro-1-octanesulfonamide                                  | C <sub>8</sub> H <sub>2</sub> F <sub>17</sub> NO <sub>2</sub> S | FOSA         | 754-91-6             |

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| Analyte  | IUPAC name <sup>a)</sup>   | Formula  | Abbreviation    | CAS-RN <sup>b)</sup> |
|--|--|--|-----------------|----------------------|
| <i>N</i> -ethyl perfluorooctanesulfonamidoacetic acid  | 2-[Ethyl(1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-hepta-decafluorooctylsulfonyl)amino]acetic acid     | C <sub>12</sub> H <sub>8</sub> F <sub>17</sub> NO <sub>4</sub> S | EtFOSAA         | 2991-50-6            |
| Hexafluoropropylene oxide dimer acid   | 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoic acid                         | C <sub>6</sub> HF <sub>11</sub> O <sub>3</sub>                   | HFPO-DA         | 13252-13-6           |
| 4,8-Dioxa-3 <i>H</i> -perfluorononanoic acid   | 2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]propanoic acid           | C <sub>7</sub> H <sub>2</sub> F <sub>12</sub> O <sub>4</sub>     | DONA            | 919005-14-4          |
| Perfluoro-3-methoxypropanoic acid  | 2,2,3,3-Tetrafluoro-3-(trifluoromethoxy)propanoic acid   | C <sub>4</sub> HF <sub>7</sub> O <sub>3</sub>                    | PFMPA (PF40PeA) | 377-73-1             |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid   | 2-(6-Chloro-1,1,2,2,3,3,4,4,5,5,6,6-dodecafluorohexoxy)-1,1,2,2-tetrafluoroethanesulfonic acid | C <sub>8</sub> HClF <sub>16</sub> O <sub>4</sub> S               | 9Cl-PF3ONS      | 73606-19-6           |
| <sup>a)</sup> IUPAC: International Union of Pure and Applied Chemistry<br><sup>b)</sup> CAS-RN: Chemical Abstract Services Registry Number |  |  |                 |                      |

## 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 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-5, *Water quality — Sampling — Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

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

### 3.1

#### **per- and polyfluoroalkyl substance PFAS**

fluorinated substance that contains at least one fully fluorinated methyl or methylene carbon atom (without any H/Cl/Br/I atom attached to it), i.e. with a few noted exceptions, any chemical with at least a perfluorinated methyl group ( $-\text{CF}_3$ ) or a perfluorinated methylene group ( $-\text{CF}_2-$ )

Note 1 to entry: The term is used in the broader sense for substances that contain chlorine and oxygen in a polyfluoroalkyl structure as well [1].

## 4 Principle

Part A of this document comprises a method using direct injection liquid chromatography coupled with tandem mass-spectrometry detection (LC-MS/MS): An aliquot of the unfiltered water sample is diluted with methanol and amended with a solution of isotopically labelled internal standards. The diluted water sample is injected directly into the analysis system. The identification and quantitative determination of the substances listed in Table 1 is performed using LC-MS/MS.

Part B of this document comprises a method using a solid-phase extraction protocol similar to ISO 21675 before LC-MS/MS measurement: The analytes listed in Table 1 are extracted from the water sample by solid-phase extraction using a weak anion exchange sorbent. The identification and quantitative determination of the substances in the extract is performed using LC-MS/MS.

Online solid-phase extraction protocols can be applied if they use weak anion exchange sorbents or hydrophilic-lipophilic balanced (HLB) SPE material and deal with the fact that long chain PFAS are adsorbed on the surface of vials and tubing if the methanol content is too low. The complete method shall be validated.

The method can be used to determine additional PFAS. Accuracy shall be tested and verified for each case as well as storage conditions of both samples and reference solutions. Criteria to be fulfilled depend on the specification for the analysis. ISO 21253-1 and ISO 21253-2 can be used as guidelines.

## 5 Interferences

### 5.1 Sampling

Long chain PFAS (e.g. perfluoroalkyl carboxylic acids with  $x \geq 7$  and perfluoroalkane sulfonic acids with  $x \geq 6$ ;  $x$  being the number of perfluorinated C-atoms in the chain) may distribute to the water/vessel and water/air interfaces. These interferences depend on the geometry and material of the sample vessels and can be reduced by minimizing the sample surface, e.g. by using narrow vessels with a small surface area.

### 5.2 Background contamination

Efforts should be taken to minimize background levels in materials and reagents. Handling of unavoidable blank values is described in 9.4.

Low molecular residuals in fluoropolymer-containing materials such as polyvinylidene fluoride or polyvinylidene difluoride (PVDF), polytetrafluoroethylene (PTFE), etc., which are often used in LC-equipment, may cause blank values, e.g. in the case of PFOA. In order to minimize these contributions to blank values, objects made of glass, steel, polyether ether ketone (PEEK), polystyrene (PS), polypropylene (PP), polyethylene (PE), poly(ethylene terephthalate) (PET) or silicone shall preferably be used for sampling, extraction and analysis. Sampling vessels (7.1) and sample vials (7.11) shall be checked for possible blank values before use.

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Typical contamination sources in the LC-system include vacuum degassers, frits and hoses as well as pump head seals. In the case of such interferences, degassing of the eluents, for example, can be carried out using helium instead of vacuum degassers. Fluoropolymer frits and hoses shall be replaced by frits and hoses made of stainless steel or PEEK. If available, pump head seals made of PE shall be used.

Background contamination, originating from the instrument and/or mobile phases can be controlled by using a delay column (7.13.3).

**5.3 Interferences encountered during liquid chromatography and mass spectrometry**

Peak tailing, peak fronting and/or broadened peaks are indicative of interferences in chromatography.

Particles in the measurement solution, e.g. fine fractions of the SPE material in case of analytical protocol B of this document or solid phase matter in the sample in case of analytical protocol A of this document, can block the inlet sieves or frits, respectively, of the LC column and result in interferences due to the increase in pressure; in such cases the measurement solutions shall be centrifuged (7.10) prior to the analysis.

Sample matrix components can affect the ionization of the target substances. This may result in suppression or enhancement of the ionization. Such matrix interferences can be detected and corrected by using authentic isotopically labelled internal standards or by the standard addition method.

Substances with similar retention times that can produce ions with similar mass to charge ratios ( $m/z$ ) to those produced by the target analytes may interfere with the determination. These interferences may lead to incompletely resolved signals and/or additional signals in the mass chromatograms of target substances. Depending on their levels in the sample, such substances may affect the accuracy and precision of the results. If the peak of interest can be separately integrated from interferences, it can be used.

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**6 Reagents**

As far as available, analytical grade or residue-analytical grade reagents shall be used. The content of impurities contributing to blank values or causing interfering signals shall be negligible. This shall be checked in regular intervals (9.4).

Reagents, solvents, and water used as eluents shall be suitable for LC and mass spectrometry.

**6.1 Water, H<sub>2</sub>O.**

Purified laboratory water can be used. The quality of water shall be checked using the blank determination procedure given in 9.4.

**6.2 Methanol, CH<sub>3</sub>OH.****6.3 Acetonitrile, CH<sub>3</sub>CN.****6.4 Acetic acid,  $w(\text{CH}_3\text{COOH})$ , 999 g/kg.****6.5 Formic acid,  $w(\text{HCOOH})$ , minimum 980 g/kg (SPE method only)****6.6 Acetone, C<sub>3</sub>H<sub>6</sub>O (SPE method only).****6.7 Ammonia solution,  $w(\text{NH}_3)$ , minimum 250 g/kg (SPE method only).****6.8 Ammonium acetate, CH<sub>3</sub>COONH<sub>4</sub>.****6.9 Sodium hydroxide solution, NaOH, 1 mol/l or other suitable concentration.**