

STN	Kvalita vody Stanovenie vybraných per- a polyfluóralkylových látok v pitnej vode Metóda kvapalinovej chromatografie/tandemovej hmotnostnej spektrometrie (LC-MS/MS)	STN EN 17892 75 7568
------------	--	--

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

Táto norma obsahuje anglickú verziu európskej normy.
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 09/24

Obsahuje: EN 17892:2024

EUROPEAN STANDARD

EN 17892

NORME EUROPÉENNE

EUROPÄISCHE NORM

June 2024

ICS 13.060.50

English Version

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

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

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

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists 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.



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

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

EN 17892:2024 (E)

Contents	Page
European foreword	4
Introduction	5
1 Scope	6
2 Normative references	8
3 Terms and definitions	8
4 Principle	9
5 Interferences	9
5.1 Sampling	9
5.2 Background contamination	9
5.3 Interferences encountered during liquid chromatography and mass spectrometry	10
6 Reagents	10
7 Apparatus	12
8 Sampling	14
9 Procedure	14
9.1 Part A: Method using direct injection	14
9.1.1 General	14
9.1.2 Sampling	14
9.1.3 Sample preparation	14
9.2 Part B: Method using SPE	15
9.2.1 General	15
9.2.2 Sampling	15
9.2.3 Sample preparation	15
9.2.4 Extraction	16
9.3 LC MS/MS operating conditions	17
9.4 Blank determination	20
9.4.1 General	20
9.4.2 Method using direct injection	20
9.4.3 Method using SPE	20
9.5 Identification	20
9.6 Calibration	21
9.6.1 General requirements	21
9.6.2 Calibration using an external standard	22
9.6.3 Calibration using an internal standard	23
9.6.4 Calibration check	24
10 Calculation	24
10.1 Use of a calibration curve to determine concentration	24
10.2 Calculation of concentration using calibration with external standards	24
10.3 Calculation of concentration using calibration with internal standards	25
10.4 Treatment of results outside the calibration range	25
10.5 Quantification of branched isomers	25
11 Determination of analyte recovery	26
11.1 Recovery	26

11.2	Recovery of internal standards	27
12	Expression of results	27
13	Test report	27
Annex A (informative)	Performance data.....	28
Annex B (informative)	Instrumental conditions and chromatograms.....	35
Bibliography		41

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.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: 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 the United Kingdom.

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 ^{a)}	Formula	Abbreviation	CAS-RN ^{b)}
Perfluoro- <i>n</i> -butanoic acid	2,2,3,3,4,4,4-Heptafluorobutanoic acid	C ₄ HF ₇ O ₂	PFBA	375-22-4
Perfluoro- <i>n</i> -pentanoic acid	2,2,3,3,4,4,5,5,5-Nonafluoropentanoic acid	C ₅ HF ₉ O ₂	PFPeA	2706-90-3
Perfluoro- <i>n</i> -hexanoic acid	2,2,3,3,4,4,5,5,6,6,6-Undecafluorohexanoic acid	C ₆ HF ₁₁ O ₂	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 ₇ HF ₁₃ O ₂	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 ₈ HF ₁₅ O ₂	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 ₉ HF ₁₇ O ₂	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 ₁₀ HF ₁₉ O ₂	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 ₁₁ HF ₂₁ O ₂	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 ₁₂ HF ₂₃ O ₂	PFDoDA	307-55-1

Analyte	IUPAC name ^{a)}	Formula	Abbreviation	CAS-RN ^{b)}
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-Pentacos-fluorotridecanoic acid	C ₁₃ HF ₂₅ O ₂	PFT _r DA	72629-94-8
Perfluoro- <i>n</i> -butanesulfonic acid	1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonic acid	C ₄ HF ₉ O ₃ 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 ₅ HF ₁₁ O ₃ 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 ₆ HF ₁₃ O ₃ S	PFH _x S	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 ₇ HF ₁₅ O ₃ S	PFH _p S	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 ₈ HF ₁₇ O ₃ 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 ₉ HF ₁₉ O ₃ 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 ₁₀ HF ₂₁ O ₃ 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 ₁₁ HF ₂₃ O ₃ 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 ₁₂ HF ₂₅ O ₃ 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 ₁₃ HF ₂₇ O ₃ S	PFT _r DS	791563-89-8
4:2 Fluorotelomer sulfonic acid	3,3,4,4,5,5,6,6,6-Nonafluorohexane-1-sulfonic acid	C ₆ H ₅ F ₉ O ₃ 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 ₈ H ₅ F ₁₃ O ₃ 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 ₁₀ H ₅ F ₁₇ O ₃ 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 ₈ H ₂ F ₁₇ NO ₂ S	FOSA	754-91-6

EN 17892:2024 (E)

Analyte	IUPAC name ^{a)}	Formula	Abbreviation	CAS-RN ^{b)}
<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 ₁₂ H ₈ F ₁₇ NO ₄ 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 ₆ HF ₁₁ O ₃	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 ₇ H ₂ F ₁₂ O ₄	DONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	2,2,3,3-Tetrafluoro-3-(trifluoromethoxy)propanoic acid	C ₄ HF ₇ O ₃	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 ₈ HClF ₁₆ O ₄ S	9Cl-PF3ONS	73606-19-6
^{a)} IUPAC: International Union of Pure and Applied Chemistry ^{b)} 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*

koniec náhľadu – text ďalej pokračuje v platenej verzii STN