# INTERNATIONAL STANDARD

# ISO 25101

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# Water quality — Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) — Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry

*Qualité de l'eau — Détermination du sulfonate de perfluorooctane (PFOS) et de l'octanoate perfluoré (PFOA) — Méthode par extraction en phase solide et chromatographie liquide/spectrométrie de masse pour des échantillons non filtrés*



Reference number
ISO 25101:2009(E)

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 25101 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

# Water quality — Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) — Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry

**WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 and to ensure compliance with any national regulatory conditions.**

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



## 1 Scope

This International Standard specifies a method for the determination of the linear isomers of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) in unfiltered samples of drinking water, ground water and surface water (fresh water and sea water) using high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS). Other isomers may be reported separately as non-linear isomers and qualified as such. The analytes specified in Table 1 can be determined by this method. The method is applicable to a concentration range of 2,0 ng/l to 10 000 ng/l for PFOS and 10 ng/l to 10 000 ng/l for PFOA. Depending on the matrix, the method may also be applicable to higher concentrations ranging from 100 ng/l to 200 000 ng/l after suitable dilution of the sample or reduction in sample size.

The user should be aware that particular problems could require the specification of additional conditions.

## 2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

## 3 Principle

The analytes listed in Table 1 are extracted from the water sample by solid-phase extraction followed by solvent elution and then determined by liquid chromatography with tandem mass-spectrometric detection.

NOTE This method is also applicable, with some limitations, to determination using high-performance liquid chromatography with single mass-spectrometric (HPLC-MS) detection (see Annex D).

**Table 1 — Analytes determinable by this method**

| Analyte | Formula [a] | Abbreviation | CAS [b] No. |
|---|---|---|---|
| Perfluoro-*n*-octanesulfonic acid (1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-*n*-octanesulfonic acid) | $CF_3(CF_2)_7SO_3H$ | PFOS | 1763-23-1 |
| Perfluoro-*n*-octanoic acid (pentadecafluoro-*n*-octanoic acid) | $CF_3(CF_2)_6COOH$ | PFOA [c] | 335-67-1 |

[a] The anion is the analyte.

[b] CAS = Chemical Abstract System.

[c] PFOA includes the acid and its salts.

## 4 Interferences

### 4.1 Interferences with sampling and extraction

Sampling containers shall consist of materials that do not change the composition of the sample during sample storage. All types of fluoropolymer plastics, including polytetrafluoroethene (PTFE) and fluoroelastomer materials, shall be avoided during sampling, sample storage and extraction. Glassware shall be avoided for sampling due to potential analyte loss due to adsorption. Sample containers shall be rinsed thoroughly with water (5.1) and methanol (5.5) prior to use. Sample containers shall be checked for possible background contamination before use.

Commercially available adsorbent materials are often of varying quality. Considerable batch-to-batch differences in quality and selectivity of these materials are possible. The recovery of a single substance may vary with the concentration. Therefore, check analyte recovery periodically at different concentrations and whenever new batches/lots of reagents or labware are used.

### 4.2 Interferences with HPLC-MS/MS

Substances with similar retention times and producing ions similar to those produced by the analytes of interest may interfere with the determination.

These interferences may lead to incompletely resolved signals or additional signals in the chromatographic pattern of target analytes, or both. Depending on their levels in the sample, such substances may affect the accuracy and precision of the results.

Matrix interferences may be caused by contaminants that are co-extracted from the samples. The extent of matrix interferences varies considerably, depending on the nature of the samples. In drinking water and ground water, matrix interferences are usually negligible, whereas wastewater and sea water matrices can be affected by matrix interferences that lead to ionization suppression or enhancement.

Interferences from instruments are significant for normal HPLC systems because many parts are made of PTFE and other fluoropolymers. It is necessary to check for possible blank contamination from individual parts, such as tubing, solvent inlet filters, valve seals and the degassing equipment, and replace these with materials such as stainless steel and polyetheretherketone (PEEK), where possible. The HPLC-vial caps should preferably be free of fluoropolymer material. The procedural blank including the instrumental blank should preferably be at least 10-fold less than the expected concentrations in real samples.