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# ISO 23702-1

# IULTCS/IUC 39-1

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## Leather — Organic fluorine —

## Part 1: Determination of the non-volatile compound content by extraction method using liquid chromatography/ tandem mass spectrometry detector (LC-MS/MS)

*Cuir — Fluor organique —*

*Partie 1: Détermination de la teneur en composés non volatils par une méthode d'extraction utilisant la chromatographie en phase liquide couplée à un détecteur par spectrométrie de masse en tandem (LC-MS/MS)*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by the Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS) in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

IULTCS, originally formed in 1897, is a world-wide organization of professional leather societies to further the advancement of leather science and technology. IULTCS has three Commissions, which are responsible for establishing international methods for the sampling and testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

A list of all parts in the ISO 23702 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.



# Introduction

The group of per- and poly-fluorinated compounds (PFC) consists of more than 800 substances. The most well-known are perfluorooctanioc sulfonic acid (PFOS) and perfluorooctanooic acid (PFOA).

Perfluorooctanoic sulfonic acid (PFOS) is classified as persistent, bio-accumulative and toxic (PBT). PFOS and its salts are restricted and regulated in many countries regarding its marketing and use (see References [3] and [4]).

Perfluorooctanoic acid (PFOA) and its salts are suspected of having a similar risk profile to PFOS.

A number of long chain per- and poly-fluorinated compounds have been included in the EU Candidate List of Substances of Very High Concern (SVHC), which is available at https://echa.europa.eu/candidate-list-table.

The regulatory thresholds for restricted per- and poly-fluorinated compounds limit the use to a level below which they cannot be meaningfully used. The thresholds need to take into consideration the possible presence of unavoidable impurities and unintentional trace contaminants.

The long chain, fully fluorinated anions are non-volatile. They are heat stable and resistant to breaking down in the environment. The per- and poly-fluorinated compounds have been widely used in many industries, including in oil-, soil- and water-repellent finishes for textiles, leather products, paper, furniture and carpets.

# Leather — Organic fluorine —

# Part 1:
# Determination of the non-volatile compound content by extraction method using liquid chromatography/tandem mass spectrometry detector (LC-MS/MS)



## 1 Scope

This document specifies a test method for detection and quantification of extractable neutral, ionic, long, medium and short chain perfluorinated and poly-fluorinated substances in leather and coated leather.

This document, taking into account the three-dimensional distribution of the fibres within leather, makes the evaluation of the perfluorinated and poly-fluorinated substances with respect to the mass.

Classes of regulated compounds listed in Annex A, Table A.1, include acids, telomers, sulfonates and sulphonamide alcohols. Classes of other non-regulated compounds that can be determined by this document are defined in Annex B, Table B.1.

## 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 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4044:2017, *Leather — Chemical tests — Preparation of chemical test samples*

EN 15987, *Leather — Terminology — Key definitions for the leather trade.*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15987 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

**3.1**
**analyte**
substance or chemical constituent that is subjected to measurement

[SOURCE: CEN/TS 15968:2010, 3.1]

**3.2**
**constituent**
pure chemical material and substance of which a material is composed

**3.3**
**extract**
concentrated preparation of the analytes isolated from the treated material

**3.4**
**internal standard**
compound different from the analyte, present in the sample with known content or added to the sample, simultaneously detected with the analyte, with physical and chemical properties as similar as possible to the analyte

## 4 Principle

The classes of regulated compounds listed in Annex A, Table A.1, are extracted with methanol and the extract is analysed by high-performance liquid chromatograph with a **tandem** mass spectrometric detector (LC-MS/MS).

NOTE The classes of non-regulated compounds listed in Annex B, Table B.1, are processed according to the same principle as the regulated compounds.

## 5 Reagents

The chemicals used below shall be of the defined purity.

**5.1 Distilled or deionized water**, at least Grade 3 quality as specified in ISO 3696.

**5.2 Methanol,** CAS 67-56-1, HPLC grade.

**5.3 Ammonium acetate,** CAS 631-61-8, analytical grade.

**5.4 Stock solutions of reference compounds**, purity > 95 % for the pure substance.

Solutions of the reference compounds listed in Annex A and Annex B are available commercially. They should be diluted to the required concentrations. If reference compounds are obtained pure, for example weigh 100 mg of each standard separately into a 100 ml volumetric flask and make up to the mark with methanol (5.2). Dilute this solution with methanol at a ratio of 1:1 000 to prepare a 1 000 µg/l stock solution.

**5.5 Target compound solutions.**

Prepare a 500 µg/l solution of each target compound by diluting the 1 000 µg/l reference compound stock solutions (5.3) with methanol.

For the preparation of the target compound solution, certified solutions are commercially available. The purity level and the solvent shall be checked in order to be in accordance with the present standard.

**5.6 Internal standard.**

**A suitable internal standard shall be used.** The impurity level of the internal standard should be determined prior to the use of every new lot.

Examples of suitable mass-labelled internal standards are:

— Perfluoro[1,2,3,4-$^{13}C_4$ ]-octanoic-acid, [mass-labelled PFOA];

— $^{13}C_x$-PFOS (e.g. [$F(CF_2)_8SO_3^-$ $H^+$]-, 1,2,3,4-$^{13}C_4$);

— $^{18}O_x$-PFOS (e.g. [$F(CF_2)_8SO_3^-$ $H^+$]-, $^{18}O_2$).

When other types of labelled internal standards become available, they may be used.

Prepare a 1 000 µg/l solution of the internal standard by diluting the commercial solution with methanol.

### 5.7 Preparation of calibration solutions.

Materials and liquids shall be stored at 4 °C and in clean containers.

Prepare suitable calibration solutions using methanol (5.2), target compound solutions (5.4) and the internal standard solution (5.6). At least five calibration solutions shall be prepared with a concentration range to match the limits given. For example, prepare according to the volumes given in Table 1.

**Table 1 — Example of calibration solutions**

| **Concentration (µg/l)** | **2,5** | **5** | **10** | **20** | **30** |
|---|---|---|---|---|---|
| **Volume methanol (µl)** | 975 | 970 | 960 | 940 | 920 |
| **Volume target compound (µl)** | 5 | 10 | 20 | 40 | 60 |
| **Volume internal standard (µl)** | 20 | 20 | 20 | 20 | 20 |

### 5.8 Eluent for the LC-MS/MS.

10 mM ammonium acetate solution is prepared by dissolving 0,771 g of ammonium acetate (5.3) in 1 000 ml deionized water (5.1).

## 6 Apparatus

Equipment or any accessible part of it that may come into contact with the sample or the extract should be free from interfering compounds, see Annex D.

Use equipment free from all types of fluoropolymer plastics, including polytetrafluoroethene (PTFE). For example, use equipment made of polypropylene (PP) or polyethylene (PE).

Clean all labware and accessible parts of the extraction apparatus by rinsing with methanol (5.2).

Sample containers shall be rinsed thoroughly with water (5.1) and methanol (5.2) prior to use.

Sample containers shall be checked for possible background contamination before use.

**6.1** Suitable device with a **sharp blade** to cut leather sample.

**6.2** **Volumetric flasks**, PP or PE, with inert stopper may be used.

**6.3** **Extraction vials**, suitable PP or PE vials, volume at least 20 ml and able to be used in a centrifuge.

**6.4** **Laboratory centrifuge,** suitable for the extraction vials (6.3).

**6.5** **Ultrasonic bath**, equipped with adjustable bath temperature control, up to at least 60 °C.

**6.6** **Analytical balance,** weighing up to 0,001 g.

**6.7** **High-performance liquid chromatograph**, (LC-MS/MS), temperature-controlled and with all required accessories including gases, LC columns, injector and a tandem mass spectrometry detector.

**6.8** **Membrane filter equipment** and **polyamide or polypropylene membrane filter,** 0,22 µm pore size.

# 7 Testing procedure

## 7.1 Sampling and preparation of the sample

The chosen leather sample should be representative of the lot it is taken from. Sample in accordance with ISO 2418. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from finished products like shoes or garments), details about sampling shall be given in the report. When sampling leather products that have separate distinct parts, the product shall be taken apart and each part shall be analysed separately.

In the case of coated leather, separate, if possible, the coating from the leather substrate. The leather substrate shall be analysed according to the procedure in this document. If separation of the coating from the leather cannot be carried out, the entire article shall be analysed according to this procedure.

NOTE The coating can be analysed according to CEN/TS 15986[2].

Take a leather sample by mass using ≥ 1 g of leather. The results shall be reported in units of mg/kg.

Cut (6.1) the leather sample into small pieces according to the method specified in ISO 4044:2017, 6.3.

Accurately weigh (6.6) 1,0 g ± 0,1 g of the leather pieces into an appropriate extraction vial (6.3). Record the mass of the leather test sample, *m*.

## 7.2 Extraction procedure

### 7.2.1 Extraction

Add 10 ml methanol (5.2) and 100 µl of the internal standard solution (5.6) to the extraction vial containing the leather pieces. Extract the test specimen in an ultrasonic bath (6.5) at a temperature of 60 °C for 1 hour.

If necessary, centrifuge (6.4) the extraction vial at approximately 2 000 rpm for 5 min. Transfer and filter (6.8) a liquid sample to a vial for LC-MS/MS chromatographic analysis (6.7).

### 7.2.2 Interferences

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, see Annex D.

## 7.3 Analytical procedure

After extraction with methanol the extract is analysed by LC-MS/MS (6.7).

Various types of high-performance liquid chromatographic equipment with mass spectrometric detector (6.7) can be used. Guidelines for suitable chromatographic conditions are given in Annex C. In C.2 and C.3 two LC-MS/MS chromatographic techniques that have been found to be suitable for this analysis are suggested.

# 8 Calculation and expression of results

## 8.1 Calibration curve

For each of the target PFCs, set up individually the linear regression function, by using the following ratio (Ae/Ais) and (Ce/Cis) with the help of the formula:

$$\frac{A_e}{A_{is}} = a \cdot \left( \frac{C_e}{C_{is}} \right) + b$$

where:

$A_e$ is the peak area for the corresponding target PFC compound;

$A_{is}$ is the peak area for the internal standard chosen;

$C_e$ is the concentration of the target PFC in the calibration standard in micrograms per litre;

$C_{is}$ is concentration of internal standard in the calibration standard in micrograms per litre;

$b$ is the ordinate intercept of the calibration curve of the specific PFC;

$a$ is the slope of the calibration curve of the specific PFC.

## 8.2 Calculation of the result

The result is to be given in mg/kg rounded to the nearest 0,1 mg/kg.

The content of each PFC is calculated as mass portion, $w$, in milligrams per kilogram (mg/kg) of the leather sample according to the following formula:

$$w = \frac{V}{1\,000 \cdot m} \cdot \frac{\left( \frac{A_s}{A_{is\_sample}} - b \right) C_{is\_sample}}{a}$$

where

$A_s$ is the peak area of the corresponding PFC in the extraction solution;

$A_{is\_sample}$ is the peak area of the corresponding internal standard in the extraction solution;

$C_{is\_sample}$ is the concentration of the corresponding internal standard in the extraction solution in micrograms per litre;

$b$ is the ordinate intercept of the calibration curve of the specific PFC determinate in 8.1;

$a$ is the slope of the calibration curve of the specific PFC determinate in 8.1;

$V$ is the volume used according to 7.2.1 in millilitres (ml);

$m$ is the mass of the leather test sample according to 7.1 in grams (g).

# 9 Precision

With this procedure a quantification limit for PFOS of 0,2 mg/kg is achieved, considering the 50 % uncertainty level in an interlaboratory trial, see Annex E.

## 10 Test report

The report shall contain at least the following information:

a) identity of the sample and, if necessary, details of sampling (7.1);

b) the method used, by reference to this document;

c) the sample amount (in grams);

d) identification and quantification of individual components;

e) expression of results in mg/kg;

f) any deviation from this procedure and all circumstances that may have influenced the result;

g) the limit of quantification (LOQ).

# Annex A
(informative)

# Classes of highly fluorinated regulated compounds determinable by this document

Long chain ionic per- and poly-fluorinated compounds from C7 to C14 have been used in the leather industry for oil, soil and water repellent finishes. Classes of regulated compounds in Table A.1 include acids, telomers, sulfonates and sulphonamide alcohols.

**Table A.1 — Classes of highly fluorinated regulated compounds determinable by this document**

| Compound name | Acronym[a] | CAS Number |
|---|---|---|
| Perfluorooctane-sulfonic-acid | PFOS | 1763–23–1 |
| Perfluorohexane sulfonique | PFHxS | 355–46–4 |
| Perfluorooctanoic-acid | PFOA | 335–67–1 |
| Perfluorononanoic-acid | PFNA | 375–95–1 |
| Perfluoroundecanoic-acid | PFUnDA (or PFUdA) | 2058–94–8 |
| Perfluorododecanoic-acid | PFDoDA (or PFDoA) | 307–55–1 |
| Perfluorotridecanoic-acid | PFTrDA | 72629–94–8 |
| Perfluorotetradecanoic-acid | PFTeDA | 376–06–7 |

[a] The anion is the analyte.

# Annex B
(informative)

# Other relevant compounds of concern

Classes of other compounds of concern. This list is not exhaustive and this method can be used to determine the amount of other PFC in leather after an analytical validation.

**Table B.1 — Examples of classes of highly fluorinated compounds of concern determinable by this document**

| Compound name | Acronym | CAS Number |
|---|---|---|
| Perfluorobutanesulfonic acid | PFBS | 375–73–5 |
| Perfluorobutanoic acid | PFBA | 375–22–4 |
| Perfluoropentanoic acid | PFPeA | 2706–90–3 |
| Perfluorohexanoic acid | PFHxA | 307–24–4 |
| Perfluoroheptanoic acid | PFHpA | 375–85–9 |
| Perfluorodecanoic acid | PFDA | 335–76–2 |
| Perfluorooctanesulfonamide | FOSA (or PFOSA) | 754–91–6 |

# Annex C
(informative)

# LC-MS/MS chromatographic conditions

## C.1 General

Various types of high-performance liquid chromatographic equipment with mass spectrometric detector (LC-MS/MS) can be used. In C.2 and C.3 there are two examples of LC-MS/MS chromatographic equipment, column and operating conditions with parameters (Tables C.1 to C.6) and chromatograms (Figures C.1 and C.2) that have been found suitable for this analysis.

**Table C.1 — Reagents for LC-MS/MS**

| Chemical | Purity |
|---|---|
| Deionized water | ≥ grade 3, ISO 3696 |
| Acetic acid, CAS 64-19-7 | $w(CH_3COOH) \geq 99{,}9$ %. |
| Ammonium acetate, CAS 631-61-8 | $w(CH_3COONH_4) \geq 97$ %. |
| Formic acid, CAS 64-18-6 | $w(HCOOH) \geq 99$ %. |
| Methanol, CAS 67-56-1 | HPLC grade |
| Reference solutions | $^{13}C_x$-PFOS (e.g. $[F(CF_2)_8SO_3^-\ H^+]$-, 1,2,3,4-$^{13}C_4$); or $^{18}O_x$-PFOS (e.g. $[F(CF_2)_8SO_3^-\ H^+]$-, $^{18}O_2$). |

For the LC-MS/MS eluent, a 10 mM ammonium acetate solution is prepared by dissolving 0,771 g of ammonium acetate in 1 000 ml deionized water.

## C.2 Example 1 of LC-MS/MS operating conditions

### C.2.1 LC-MS/MS chromatographic conditions

Column: Kinetex® C18, 150 mm × 2,1 mm, 2,6 µm, 100 Å

Mobile phase: Eluent A: 10 mM ammonium acetate solution

Eluent B: methanol

**Table C.2 — Gradient**

| Time<br>min | Eluent A<br>% | Eluent B<br>% |
|---|---|---|
| 0 | 99 | 1 |
| 10 | 1 | 99 |
| 13 | 1 | 99 |
| 13,5 | 99 | 1 |
| 25 | 99 | 1 |

Flow: 0,15 ml/min

Injection volume: 20 µl

Temperature column oven: 40 °C

Run injection: Every batch of 20 samples or less requires four calibration points and a blank.

### C.2.2 LC-MS/MS mass spectrometry conditions

Polarity: Negative API-ES (ESI)

Mode: MRM

Fragmentor: 72 V

Dry gas temperature: 350 °C

Nebulizer pressure: 40 psi

Gas flow: 12 l/min

Capillary voltage: 3 000 V

### C.2.3 Fragment for quantification

| Analytes | MRM Transitions |
|---|---|
| PFHpA | 363,1 → 319,0 |
| | 363,1 → 169,1 |
| PFOA | 412,96 → 369,0 |
| | 412,96 → 169,0[a] |
| PFNA | 463,0 → 419,0 |
| | 463,0 → 219,1 |
| PFDA | 513,0 → 468,9 |
| | 513,0 → 219,0 |
| PFUnDA | 562,9 → 518,9 |
| | 562,9 → 269,0 |
| PFDoDA | 612,9 → 568,9 |
| | 612,9 → 319,0 [a] |
| PFTrDA | 662,9 → 618,9 |
| | 662,9 → 269,0 [a] |
| PFTeDA | 712,9 → 668,9 |
| | 712,9 → 269 [a] |
| PFOS | 499,0 → 80,0 |
| | 499,0 → 99,1 |
| FOSA | 498,1 → 78,0 |
| | 498,1→ 47,9 |

[a] Only for confirmation of identification.

## C.3 Example 2 of LC-MS/MS operating conditions

### C.3.1 LC-MS/MS chromatographic conditions for PFOS and PFOA

#### C.3.1.1 General

Column: Gemini® C18, 100 × 2,1 mm, 2,6 µm, 100 Å

Mobile phase: Eluent A – ammonium acetate 10 mM solution

Eluent B – methanol

**Table C.3 — Gradient**

| Time<br>min | Eluent A<br>% | Eluent B<br>% |
|---|---|---|
| 0,01 | 65 | 35 |
| 15,00 | 15 | 85 |
| 20,00 | 10 | 90 |
| 25,01 | 65 | 35 |
| 30,00 | 65 | 35 |

Flow: 0,2 ml/min

Injection volume: 1,0 µl

Nebulizing gas flow: 1,5 l/min

DL temperature: 250 °C

Heat block temperature: 200 °C

Drying gas flow: 15 l/min

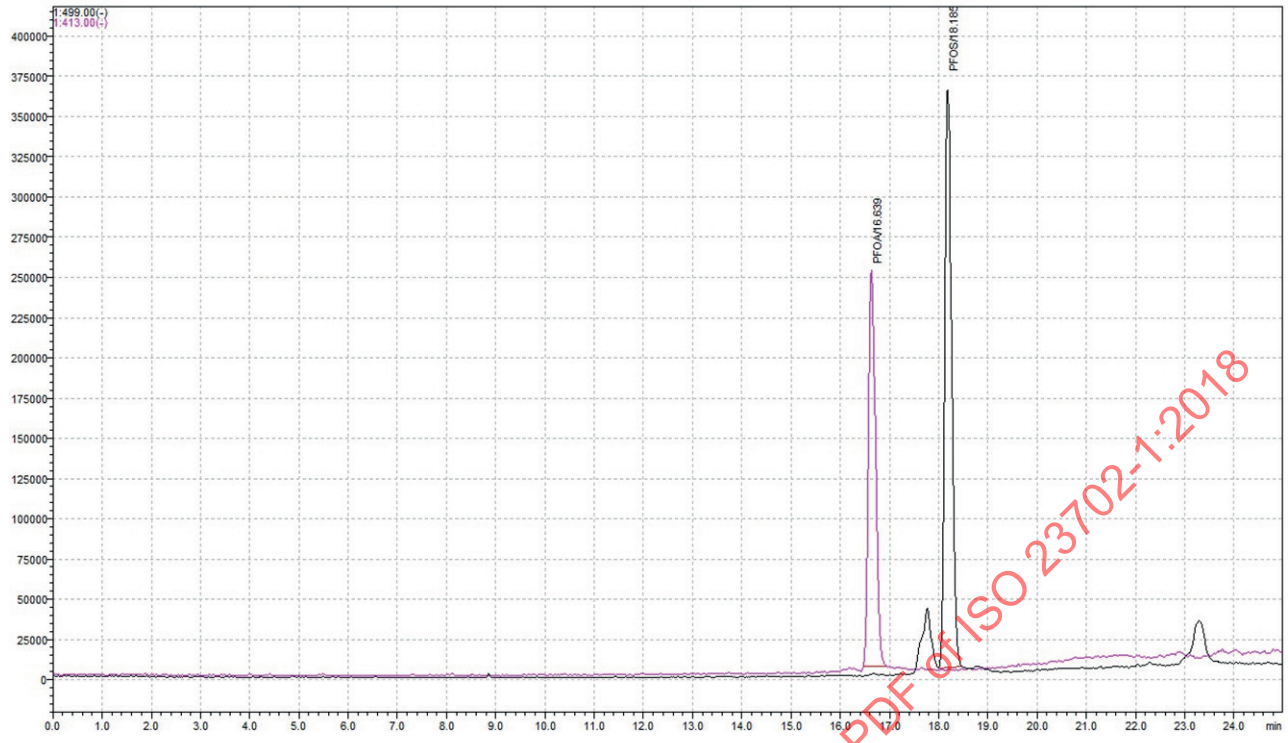



**Figure C.1 — Chromatogram of PFOS and PFOA**

PFOS retention time: 18,172 min

PFOA retention time: 16,668 min

### C.3.1.2 Identification of PFOS and PFOA

**Table C.4 — Conditions MRM – PFOS**

| Precursor m/z | Product m/z | Dwell time msec | Q1 Pre-bias V | CE | Q1 Pre-bias V |
|---|---|---|---|---|---|
| 498,90 | 80,00 | 7,0 | 14,0 | 55,0 | 29,0 |
| 498,90 | 99,00 | 7,0 | 14,0 | 44,0 | 16,0 |

**Table C.5 — Conditions MRM – PFOA**

| Precursor m/z | Product m/z | Dwell time msec | Q1 Pre-bias V | CE | Q1 Pre-bias V |
|---|---|---|---|---|---|
| 412,90 | 369,05 | 7,0 | 15,0 | 11,0 | 24,0 |
| 413,10 | 169,00 | 7,0 | 20,0 | 19,0 | 30,0 |

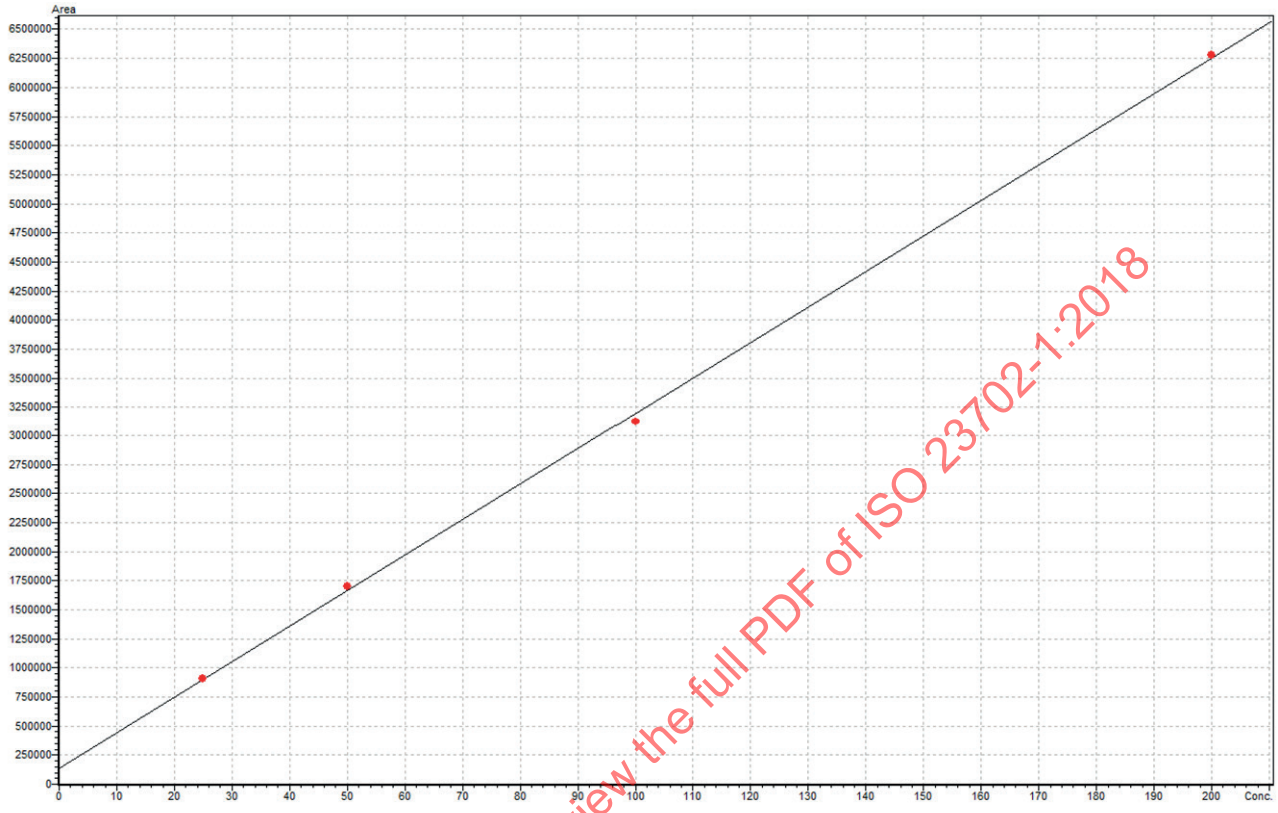
### C.3.1.3 Quantification of PFOS and PFOA

#### C.3.1.3.1 Quantification PFOS (SIM -)

| Name | m/z | Retention time min | Conc STD1 µg/l | Conc STD2 µg/l | Conc STD3 µg/l | Conc STD4 µg/l | Acquisition |
|---|---|---|---|---|---|---|---|
| PFOS | 499,00 | 18,173 | 25 | 50 | 100 | 200 | 1:Q3 SIM(-) |

**Equation:** Y = (30 578,5)X + (136 271)

R = 0,999 8  $R^2$ = 0,999 5



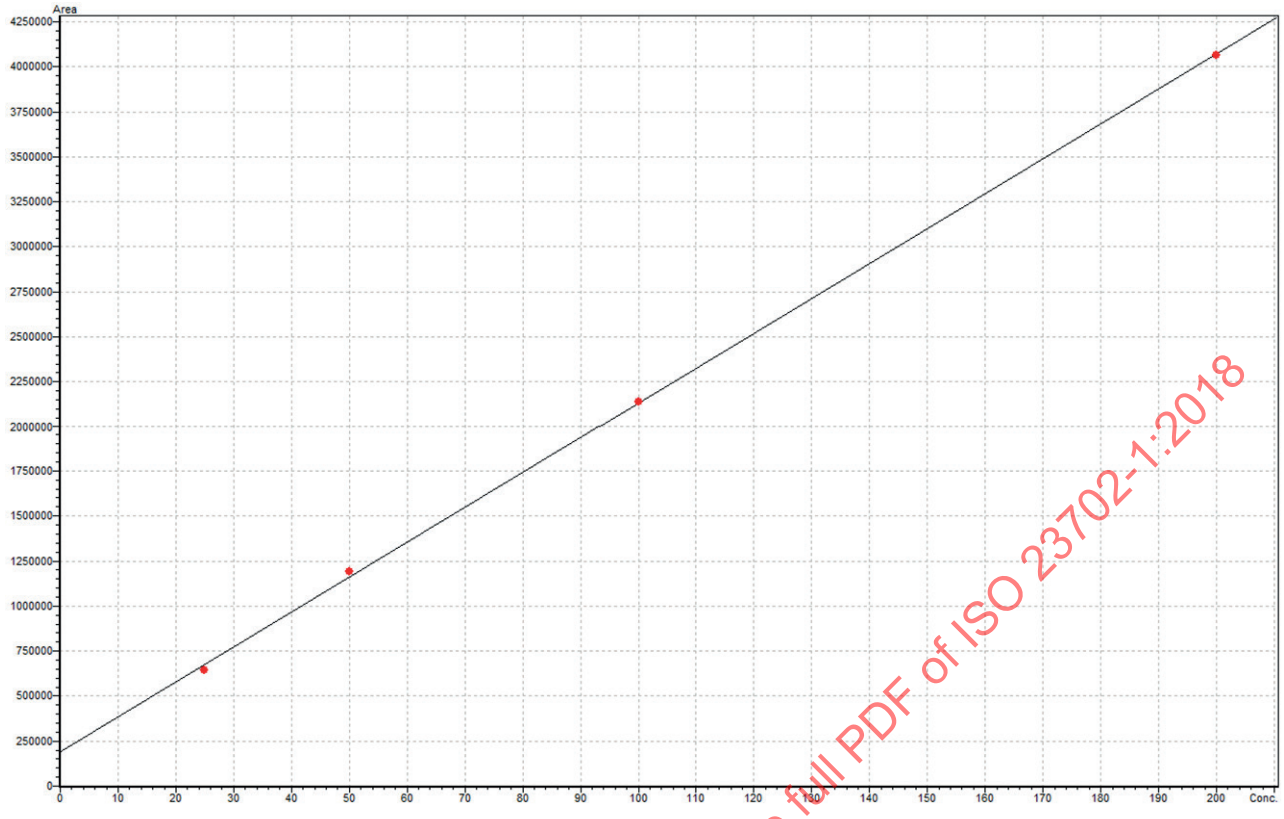

| Data# | Data File-name | Retention time min | Area | Conc. ug/l | Std. conc. ug/l | Accuracy % | S/N |
|---|---|---|---|---|---|---|---|
| 1 | STD1.lcd | 18,133 | 910 423 | 25,317 | 25 | 101,3 | 235,46 |
| 2 | STD3.lcd | 18,145 | 1 703 753 | 51,261 | 50 | 102,5 | 388,43 |
| 3 | STD2.lcd | 18,172 | 3 119 326 | 97,554 | 100 | 97,6 | 274,84 |
| 4 | STD4.lcd | 18,310 | 6 278 514 | 200,868 | 200 | 100,4 | 129,63 |

### C.3.1.3.2 Quantification PFOA (SIM -)

| Name | m/z | Retention time min | Conc STD5 µg/l | Conc STD6 µg/l | Conc STD7 µg/l | Conc STD8 µg/l | Acquisition |
|---|---|---|---|---|---|---|---|
| PFOA | 413,00 | 16,624 | 25 | 50 | 100 | 200 | 1:Q3 SIM(-) |

**Equation:** Y = (19 402,8)X + (190 650)

R = 0,999 8  $R^2$ = 0,999 7




| Data# | Data file-name | Retention time min | Area | Conc. ug/l | Std. conc. ug/l | Accuracy % | S/N |
|---|---|---|---|---|---|---|---|
| 1 | STD5.lcd | 16,575 | 645 776 | 23,457 | 25 | 93,8 | 29,35 |
| 2 | STD6.lcd | 16,587 | 1 190 955 | 51,555 | 50 | 103,1 | 151,88 |
| 3 | STD7.lcd | 16,606 | 2 138 086 | 100,369 | 100 | 100,4 | 391,9 |
| 4 | STD8.lcd | 16,735 | 4 063 836 | 199,620 | 200 | 99,8 | 417,81 |

### C.3.2 LC-MS/MS chromatographic conditions for other polyfluorinated compounds (PFC): PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFTrDA and PFTeDA

LC-MS/MS parameters as given in C.3.1.

**Table C.6 — Gradient**

| Time min | Eluent A % | Eluent B % |
|---|---|---|
| 0,01 | 70 | 30 |
| 10,00 | 10 | 90 |
| 15,00 | 10 | 90 |
| 16,00 | 70 | 30 |
| 20,00 | 70 | 30 |