



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 23702-1:2017**  
**01-september-2017**

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**Usnje - Organski fluor - 1. del: Določevanje nehlapnih spojin z metodo ekstrakcije z uporabo tekoče kromatografije (ISO/DIS 23702-1:2017)**

Leather - Organic fluorine - Part 1: Determination of non-volatile compounds by extraction method using liquid chromatography (ISO/DIS 23702-1:2017)

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**ICS:**

59.140.30      Usnje in krzno      Leather and furs

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**ISO/DIS 23702-1**  
**IULTCS/IUC 39-1**

IULTCS

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**Leather - Organic fluorine —**

**Part 1:  
Determination of non-volatile compounds by extraction  
method using liquid chromatography**

*Titre manque*

ICS: 59.140.30

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**ISO/DIS 23702-1:2017(E)**  
**IULTCS/IUC 39-1:2017(E)****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.

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](http://www.iso.org/directives)).

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ISO yyyy 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 Standardisation (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the agreement on technical co-operation 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.

[Annexes A](#) is a normative annex.

[Annexes B, C](#) and [D](#) are informative annexes.

## Introduction

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

Perfluorooctanoic sulfonic acid (PFOS) is classified as persistent, bio-accumulative and toxic (PBT). PFOS and its salts are restricted in the EU Regulation 1907/2006 (REACH) Annex XVII regarding its marketing and use. PFOS and its salts were added to [Annex B](#) of the Stockholm Convention on Persistent Organic Pollutants (POP) in May 2009. Consequently the use of PFOS and its salts are now restricted in many national regulations, for example the EU Regulation 757/2010.

Perfluorooctanoic acid (PFOA) and its salts are suspected to have 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 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.

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# Leather - Organic fluorine —

## Part 1:

# Determination of non-volatile compounds by extraction method using liquid chromatography

## 1 Scope

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

This standard, 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 [Table A.1](#) of the normative [Annex A](#) include acids, telomers, sulfonates and sulphonamide alcohols. Classes of other non-regulated compounds that can be determined by this standard are defined in [Table B.1](#) in [Annex B](#).

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

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

ISO 4044, *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 leather terms and definitions given in EN 15987 and the following terms and definitions apply.

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

- ISO Online browsing platform: available at <http://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]<sup>[1]</sup>

### 3.2

#### **blank matrix**

matrix which is largely identical with the analytical one, but does not contain the analyte or contains it in low and known content.

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[SOURCE: CEN/TS 15968:2010, 3.2] [1]

### 3.3

#### **constituents**

all pure chemical materials and substances of which a material is composed.

### 3.4

#### **extract**

concentrated preparation of the analytes isolated from the treated material.

### 3.5

#### **internal standard (IS)**

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 [Table A.1](#) in [Annex A](#) are extracted with methanol and the extract is analyzed by high performance liquid chromatograph with a mass spectrometric detector (LC-MS/MS).

NOTE The classes of non-regulated compounds listed in [Table B.1](#) in [Annex B](#) 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 Stock solutions of reference compounds**, purity > 95 % for the pure substance.

Solutions of the reference compounds are available commercially. They should be diluted to the required concentrations. If reference compounds are obtained pure, 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 1:1 000 to prepare a 1 000 µg/L stock solution.

**5.4 Target compound solutions**

Prepare a 500 µg/L solution of each target compound by diluting the 1 000 µg/L reference compound stock solutions (5.1) with methanol.

**5.5 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 four 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 — Calibration solutions

Concentration (µg/L)	2,5	5	10	20	30
Volume methanol (µL)	990	985	975	955	935
Volume Target compound (µL)	5	10	20	40	60
Volume Internal Standard (µL)	5	5	5	5	5

## 5.6 Internal Standard

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

Examples of internal standards (IS) are:

- 1) perfluorononanoic-acid, PFNA.
- 2) suitable mass-labeled internal standard are:
  - Perfluoro-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]-octanoic-acid, MPFOA [mass-labeled PFOA];
  - <sup>13</sup>C<sub>x</sub>-PFOS (e.g. [F(CF<sub>2</sub>)<sub>8</sub>SO<sub>3</sub><sup>-</sup> H<sup>+</sup>]<sup>-</sup>, 1,2,3,4-<sup>13</sup>C<sub>4</sub>);
  - <sup>18</sup>O<sub>x</sub>-PFOS (e.g. [F(CF<sub>2</sub>)<sub>8</sub>SO<sub>3</sub><sup>-</sup> H<sup>+</sup>]<sup>-</sup>, <sup>18</sup>O<sub>2</sub>).

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

Prepare a 500 µg/L solution of the internal standard by diluting the commercial solution with methanol.

For the LC-MS/MS eluent, a 10 mM ammonium acetate solution is prepared by dissolving 0,771 g of ammonium acetate (Table 3) in 1000 ml deionized water.

## 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 glass and all types of fluoropolymer plastics, including polytetrafluoroethene (PTFE). For example, use equipment made of polypropylene (PP) or polyethylene (PE).

Clean all lab-ware 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.

**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** **Concentration equipment** (e.g. hydrophilic lipophilic base, SPE, weak anion exchange resin or rotary evaporator).

**6.7** **Analytical balance**, weighing to 0,001 g.