SLOVENSKI STANDARD
oSIST prEN ISO 16189:2021

01-marec-2021

Obutev - Kritične snovi, ki so lahko v obutvi in delih obutve - Preskusna metoda za kvantitativno ugotavljanje dimetilformamida v obutvenih materialih (ISO/DIS 16189:2021)

Footwear - Critical substances potentially present in footwear and footwear components - Test method to quantitatively determine dimethylformamide in footwear materials (ISO/DIS 16189:2021)

Schuhe - Möglicherweise in Schuhen und Schuhbestandteilen vorhandene kritische Substanzen - Prüfverfahren zur quantitativen Bestimmung von Dimethylformamid in Schuhwerkstoffen (ISO/DIS 16189:2021)

Chaussures - Substances critiques potentiellement présentes dans les chaussures et les composants de chaussures - Méthode d'essai pour déterminer quantitativement le diméthylformamide dans les matériaux de chaussures(ISO/DIS 16189:2021)

Ta slovenski standard je istoveten z: **prEN ISO 16189**

ICS:

61.060	Obuvala	Footwear

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Footwear — Critical substances potentially present in footwear and footwear components — Test method to quantitatively determine dimethylformamide in footwear materials

Chaussures — Substances critiques potentiellement présentes dans la chaussure et les composants de chaussure — Méthodes d'essai pour déterminer quantitativement le diméthylformamide dans les matériaux de chaussure

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 216, *Footwear*.

This second edition cancels and replaces the first edition (ISO/TS 16189:2013), which has been technically revised.

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.

Footwear — Critical substances potentially present in footwear and footwear components — Test method to quantitatively determine dimethylformamide in footwear materials

1 Scope

This document specifies a method to determine the amounts of dimethylformamide (DMF) in footwear and footwear components containing polyurethane (PU) coated material.

NOTE 1 In footwear industry, when PU is injected (reaction moulded), this process does not need the use of DMF. For PU coated material, the use of DMF is possible.

NOTE 2 Several abbreviations can be used for dimethylformamide DMF, DMFa, DMFo. This document recommends to use DMF.

CEN ISO/TR 16178, table 1 defines which materials are concerned by this determination.

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 4787:2010, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

ISO/TR 16178:2020, *Footwear — Critical substances potentially present in footwear and footwear components*

ISO/DIS 21061:2020, *Footwear — Chemicals tests — General principles on the preparation of samples*

3 Terms and definitions

For the purposes of this document, 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 https://www.iso.org/obp

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

3.1
CAS
Chemical Abstract Service

4 Method principle

The sample is cut in small pieces and extracted with methanol in a sealed vial at 70 °C in an ultrasonic bath for 1 h. An aliquot is then analysed using a gas chromatograph with mass selective detector.

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 Dimethylformamide (DMF), CAS number 68-12-2, highest available defined purity standard

5.2 Dimethylformamide – d7 (DMF-d7), CAS number 4472-41-7, highest available defined purity standard

5.3 Methanol, CAS number 67-56-1, analytical grade

5.4 Stock solutions

5.4.1 Internal standard – stock solution (1 000 mg/l)

10 mg of DMF-d7 (5.2) is weighted with an accuracy of 0,1 mg in a 10 ml volumetric flask and filled up to the mark with methanol. The content is further transferred in an amber 10 ml vial with PTFE stopcock. The stock solution shall be stored in a refrigerator at about 4 °C.

5.4.2 Internal standard - working solution (200 mg/l)

The solution is prepared by 1:5 dilution of the stock solution (5.4.1) with methanol (5.3). This solution shall be stored in a refrigerator at about 4 °C.

5.4.3 Target compound – stock solution (1 000 mg/l)

10 mg of DMF (5.1) is weighed with an accuracy of 0,1 mg in in a 10 l volumetric flask and filled up to the mark with methanol. The content is further transferred in an amber 10 ml vial with PTFE stopcock. This solution shall be stored in a refrigerator at about 4 °C.

5.4.4 Target compound – working solution (200 mg/l)

The solution is prepared by 1:5 dilution of the stock solution (5.4.3) with methanol (5.3). This solution shall be stored in a refrigerator at about 4 °C.

6 Equipment

The usual laboratory apparatus and amber laboratory glassware, according to ISO 4787:2010, shall be used, in addition to the following.

6.1 Analytical balance with a precision of at least 0,1 mg

6.2 Glass vial with screw cap that can be tightly sealed (e.g. volume of 20 ml)

6.3 Ultrasonic bath with adjustable temperature suitable for operation at 70 ± 5 °C

6.4 PTFE-membrane filter pore width 0,45 µm

6.5 GC vials capped (e.g. volume 2 ml)

6.6 Amber glass vials with a screw cap that can be tightly sealed (e.g. volume of 10 ml)

6.7 Gas chromatograph with mass selective detector (GC-MS)

7 Preparation of sample

7.1 Sampling

Samples of PU coated materials are cut in pieces of about 3 mm to 5 mm edge length.

NOTE Up to three test specimens (of equal mass) of the same material classification (see ISO 21061 may be tested together taking into consideration the limits of detection and quantification.

7.2 Extraction

Weigh (1,0 ± 0,1) g of the sample with the analytical balance (6.1) in a glass vial (6.2), record the mass to the nearest 1 mg, add 1 ml of the internal standard working solution (5.2.2) and 9 ml of methanol (5.3), and seal the vial.

Extract the sample at (70 ± 5) °C for (60 ± 5) min in an ultrasonic bath.

After cooling to room temperature, the solution is filtered (if necessary) through a PTFE membrane filter (6.4).

Transfer an aliquot of the the extract to an GC-MS vial (6.5) and sealed with a PTFE-cap.

8 Determination with GC-MS

8.1 Calibration standard

At least 4 calibrations points shall be used to establish the calibration curve. Examples of calibration solutions are given in Table 1.

Table 1 — Examples of calibration solution

Standard	L1	L2	L3	L4	L5	L6
Volume of target compound working solution (5.2.4)	25 µl	50 µl	100 µl	250 µl	500 µl	1 000 µl
Concentration of target compound in the calibration solutions (mg/l)	0,5	1	2	5	10	20
Volume of the methanol working solution of internal standard (5.2.2)	1 000 µl	1 000 µl	1 000 µl	1 000 µl	1 000 µl	1 000 µl
Concentration of the internal standard (mg/l)	20	20	20	20	20	20
Filled to the mark with methanol						

8.2 Examples of instrumental method

Informative example is given in Annex A.

9 Expression of results

9.1 Calibration curve

Set up the linear regression function by using the following ratio (A_e/A_{is}) and (C_e/C_{is}) with the help of the formula:

$$\frac{A_e}{A_{is}} = a \times \frac{C_e}{C_{is}} + b$$

where

A_e is the area of the peak of Dimethylformamide;

A_{is} is the area of the peak of Dimethylformamide-D7;

C_e is concentration of Dimethylformamide in the calibration standard in microgram per liter;

C_{is} is concentration of Dimethylformamide-D7 in the calibration standard in microgram per liter;

a is the slope of the linear function;

b is the ordinate intercept of the calibration curve. The units depend of the evaluation.

The content of DMF is calculated according to the following equation as a mass fraction w in mg/kg:

$$W = (V/m) \times \frac{\left(\frac{A_{ech}}{A_{is}} - b \right)}{a} \times C_{is}$$

where

W is the content of the Dimethylformamide in the specimen (mg/kg);

V is the volume of the solvent use for the extraction (ml) (in most case this value will be equal to 10 ml);

m is the mass of the tested specimen (g);

A_{ech} is the area of the peak of Dimethylformamide in the sample;

A_{is} is the area of the peak of Dimethylformamide-D7 in the sample;

C_{is} is concentration of Dimethylformamide-D7 in the sample in microgram per liter;

a is the slope of the linear function;

b is the ordinate intercept of the calibration curve. The units depend of the evaluation.

10 Performance of the method

This method is able to detect DMF with a limit of quantification of 5 mg/kg or lower in PU coated materials.

11 Test report

The test report shall include at least the following:

a) reference to this test method, ISO 16189;

b) date of the test;

c) all details necessary for complete identification of the sample tested;

d) condition of sample receipt and storage before the test;

e) the amount determined of the extracted dimethylformamide in mg/kg;

f) any deviation from the given procedure.