SLOVENSKI STANDARD
SIST EN 17131:2019

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Tekstilije in tekstilni izdelki - Določevanje dimetilformamida (DMF), metoda z uporabo plinske kromatografije

Textiles and textile products - Determination of dimethylformamide (DMF), method using gas chromatography

Textilien und textile Erzeugnisse - Bestimmung von Dimethylformamid (DMF), Verfahren mittels Gaschromatographie

Textiles et produits textiles - Détermination du diméthylformamide (DMF), méthode par chromatographie en phase gazeuse

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Textiles and textile products - Determination of dimethylformamide (DMF), method using gas chromatography

Textiles et produits textiles - Détermination du diméthylformamide (DMF), méthode par chromatographie en phase gazeuse

Textilien und textile Erzeugnisse - Kritische Stoffe, die in Bestandteilen von Materialien textiler Erzeugnisse vorhanden sein können - Bestimmung von Dimethlyformamid (DMF), gaschromatographisches Verfahren

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 17131:2019) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by January 2020, and conflicting national standards shall be withdrawn at the latest by January 2020.

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.

This document is adapted from CEN ISO/TS 16189, it was prepared by Technical Committee CEN/TC 309 “*Footwear*”, in collaboration with Technical Committee ISO/TC 216 “*Footwear*”, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement). The adaptation is based on the extension of the scope to textile products.

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Introduction

In Europe according to Regulation (EU) 2018/1513 amending Annex XVII to Regulation (EC) No 1907/2006 (REACH) by Entry 72, (a) clothing or related accessories; (b) textiles other than clothing which, under normal or reasonably foreseeable conditions of use, come into contact with human skin to an extent similar to clothing; (c) footwear are not to be placed on the market after 1 November 2020, if they contain more than 3 000 mg/kg of dimethylformamide (DMF).

This restriction does not apply to: (a) clothing, related accessories or footwear, or parts of clothing, related accessories or footwear, made exclusively of natural leather, fur or hide; (b) non-textile fasteners and non-textile decorative attachments; (c) second-hand clothing, related accessories, textiles other than clothing or footwear; (d) wall-to-wall carpets and textile floor coverings for indoor use, rugs and runners. It also does not apply to clothing, related accessories, textiles other than clothing, or footwear within the scope of Regulation (EU) 2016/425 or Regulation (EU) 2017/745. The restriction on textiles other than clothing does not apply to disposable textiles. "Disposable textiles" means textiles that are designed to be used only once or for a limited time and are not intended for subsequent use for the same or a similar purpose.

Although the Regulation excludes non-textile fastenings and decorative items, this method is also suitable for such materials.

WARNING — The use of this document involves hazardous materials. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the document and fulfil statutory and regulatory requirements for this purpose.

1 Scope

This document specifies a method to determine the amounts of extractable dimethylformamide (DMF) in components of textile products containing polyurethane or acrylic.

NOTE Further information can be found in CEN/TR 16741:2015, Tables 1 and 3 that define which materials are applicable to 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.

EN ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use (ISO 4787)*

EN ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing (ISO 5089)*

3 Terms and definitions

No terms and definitions are listed in this document.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle of method

The sample is cut into small pieces and extracted with methanol, in a sealed vial at 70 °C, in an ultrasonic bath. An aliquot of the extract is analysed using a gas chromatograph with mass selective detector (GC-MS).

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

5.4 Stock solutions

5.4.1 Internal standard — Stock solution (1 000 mg/l)

Weigh 10 mg of DMF-d7 (5.2), with an accuracy of 0,1 mg, into a 10 ml volumetric flask, fill to the mark with methanol (5.3) and mix. Transfer the content into an amber 10 ml vial with cap (6.6) and keep in a refrigerator at about 6 °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).

5.4.3 Target compound — Stock solution (1 000 mg/l)

Weigh 10 mg of DMF (5.1), with an accuracy of 0,1 mg, into a 10 ml volumetric flask, fill up to the mark with methanol (5.3) and mix. Transfer the content into an amber 10 ml vial with cap (6.6) and keep in a refrigerator at about 6 °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).

6 Equipment

The usual laboratory equipment and laboratory glassware, according to EN ISO 4787, 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 a screw cap that can be tightly sealed (e.g. volume of 20 ml),

6.3 Ultrasonic bath with adjustable temperature suitable for operation at about 70 °C,

6.4 PTFE-membrane filter, pore width 0,45 µm,

6.5 GC vial, capped (e.g. volume of 2 ml),

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

6.7 Micropipettes,

6.8 Gas chromatograph with mass selective detector (GC-MS),

7 Sampling

7.1 Samples

DMF is volatile and care should be taken to minimize loss. It is recommended that samples are individually wrapped in aluminium foil before shipping.

7.2 Preparation of test specimens

If possible, sampling is carried out according to EN ISO 5089. Samples of materials are cut into pieces of about 0,3 cm to 0,5 cm edge length.

NOTE Up to three test specimens (of equal mass) of the same material type can be tested together taking into consideration the limits of detection and quantification.

8 Procedure

8.1 Extraction

Weigh (1,0 ± 0,1) g of the sample in a glass vial (6.2), record the mass to the nearest 1 mg, add 1 ml of the solution of internal standard (5.4.2) and 9 ml of methanol (5.3), and seal the vial. Extract the sample at (70 ± 5) °C for 1 h ± 5 min in an ultrasonic bath.

WARNING – Do not open the vial before cooling as the contents may be under pressure.

After cooling to 27 °C, or below, filter this solution through a PTFE membrane filter (6.4).

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

8.2 Determination with GC-MS

8.2.1 Preparation of the calibration solutions

Prepare at least 3 calibration solutions of DMF, including internal standard, from the working solutions (5.4.2 and 5.4.4), in methanol (5.3), at suitable concentrations for the analysis.

8.2.2 Determination with GC-MS

Determine the DMF extracted in 8.1 by GC-MS (6.8).

An example of chromatographic conditions is given in Annex A.

9 Expression of results

Plot a calibration graph of the response against the known standard concentration (corrected for the response for the internal standard). From the calibration graph, interpolate the concentration of DMF in µg/ml (ρ_S).

The DMF level is calculated as mass portion w in mg/kg of the tested material according to the following equation:

$$w = \frac{\rho_s \times V}{m_E}$$

where

ρ_S is the interpolated concentration of DMF, in µg/ml;

V is the volume of the extract in ml;

m_E is the mass of the tested material, in g.

10 Test report

The test report shall include at least the following:

a) reference to this test method, i.e. EN 17131:2019;

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) result of the extracted dimethylformamide, in mg/kg;

f) any deviation from the given procedure.