SLOVENSKI STANDARD
SIST EN 17130:2019

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Tekstilije in tekstilni izdelki - Določevanje dimetilfumarata (DMFu), metoda z uporabo plinske kromatografije

Textiles and textile products - Determination of dimethylfumarate (DMFu), method using gas chromatography

Textilien und textile Erzeugnisse - Bestimmung von Dimethylfumarat (DMFu), Verfahren mittels Gaschromatographie

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

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Textiles and textile products - Determination of dimethylfumarate (DMFu), method using gas chromatography

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 17130:2019) 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of an identical text or by endorsement, at the latest by January 2020, and conflicting national standards shall be withdrawn at the latest by January 2020.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 16186 prepared by Technical Committee CEN/TC 309, “Footwear”, in collaboration with ISO Technical Committee 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 (EC) No 1907/2006 (REACH), Annex XVII, Entry 61, Dimethylfumarate is not to be used in articles, or any parts thereof, in concentrations greater than 0,1 mg/kg. Articles, or any parts thereof, containing DMFu in concentrations greater than 0,1 mg/kg are not to be placed on the market.

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 gives a test method for determining the amounts of dimethyl fumarate (DMFu) in textile materials and textile articles. It also includes desiccant sachets that can be present.

The test method is not applicable to metal parts. The materials to which it is applicable are given in CEN/TR 16741:2015, Tables 1 and 3.

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.

CEN/TR 16741:2015, *Textiles and textile products — Guidance on health and environmental issues related to chemical content of textile products intended for clothing, interior textiles and upholstery*

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

The sample is cut into small pieces and extracted with acetone in a sealed vial at a defined temperature in an ultrasonic bath. Two different procedures are proposed to be used, depending on the material being tested:

a) the first procedure, without purification (concentration of the extracted solution is optional), can be used for samples giving a simple chromatograph, for example, textiles;

b) the second procedure, with purification and concentration of the extract, can be used for samples with a complex matrix effect, such as leather.

An aliquot of the extract is analysed using a gas chromatograph with mass selective detector.

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 Dimethyl fumarate, CAS number 624-49-7, highest available defined purity standard,

5.2 Dimethyl maleate, CAS number 624-48-6, highest available defined purity standard,

5.3 Dimethyl fumarate-d2, CAS number 23057-98-9, highest available defined purity standard,

5.4 Acetone, CAS number 67-64-1,

5.5 Stock solutions and working solutions,

5.5.1 Internal standard – Stock solution (1 g/l)

Weigh (10 ± 0,1) mg of Dimethyl fumarate-d2 (5.3) into a 10 ml volumetric flask and fill to the mark with acetone (5.4). Transfer the content into an amber 10 ml vial with cap and keep in a refrigerator at about 6 °C.

5.5.2 Internal standard – Working solution (1 mg/l)

Prepare this solution by means of 1:1 000 dilution of the stock solution of internal standard (5.5.1) with acetone (5.4).

5.5.3 Target compounds – Stock solution (1 g/l)

Weigh (50 ± 0,1) mg of dimethyl fumarate (5.1) and (50 ± 0,1) mg of dimethyl maleate (5.2) in a 50 ml volumetric flask, and fill to the mark with acetone (5.4).

5.5.4 Target compounds – Working solution (1 mg/l)

Prepare this solution by means of 1:1 000 dilution of the stock solution (5.5.3) with acetone (5.4).

6 Apparatus

The usual laboratory apparatus 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 screw cap that can be tightly sealed (e.g. volume of 40 ml),

6.3 Ultrasonic bath, with adjustable temperature, suitable for operation at about 60°C,

6.4 PTFE membrane filter, with a pore size of 0,45 µm,

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

6.6 GC vials, with cap (e.g. volume of 2 ml),

6.7 Activated magnesium silicate cartridge (2 g/6 ml),

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

6.9 Nitrogen evaporator, with conical tubes and with adjustable temperature, suitable for operation up to 40 °C.

7 Sample preparation

7.1 Sampling

If possible, sampling is carried out according to EN ISO 5089.

7.2 Preparation of test specimens

The test specimen shall consist of a single material type (made of textile, leather, polymer or other organic material), which is tested separately.

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.

Cut the homogenous textile (or leather or polymer or organic material) samples into pieces of about 0,3 cm to 0,5 cm edge length. Desiccant samples may be used without any processing.

8 Procedures

8.1 Standard procedure - 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 (5.5.2) and 9 ml of acetone, and seal the vial. Extract the sample at (60 ± 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 below at least 27 °C, decant the solution and, if necessary, reduce to 1,0 ml under a gentle stream of nitrogen (6.9) at maximum 40 °C.

Filter this solution through a PTFE membrane filter (6.4).

Transfer an aliquot of the extract to a GC-MS vial (6.6) and seal with a cap.

8.2 Procedure for complex matrix

8.2.1 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 (5.5.2) and 9 ml of acetone, and seal the vial. Extract the sample at (60 ± 5) °C for 1 h ± 5 min in an ultrasonic bath.