SLOVENSKI STANDARD
SIST EN 17134:2019

01-september-2019

Tekstilije in tekstilni izdelki - Določevanje nekaterih konzervansov, metoda z uporabo tekočinske kromatografije

Textiles and textile products - Determination of certain preservatives, method using liquid chromatography

Textilien und textile Erzeugnisse - Bestimmung bestimmter Konservierungsmittel, Verfahren mittels Flüssigkeitschromatographie

Textiles et produits textiles - Détermination de certains agents de conservation, méthode par chromatographie en phase liquide

Ta slovenski standard je istoveten z: **EN 17134:2019**

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59.080.01	Tekstilije na splošno	Textiles in general
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English Version

Textiles and textile products - Determination of certain preservatives, method using liquid chromatography

Textiles et produits textiles - Détermination de certains agents de conservation, méthode par chromatographie en phase liquide

Textilien und textile Erzeugnisse - Bestimmung bestimmter Konservierungsmittel, Verfahren mittels Flüssigkeitschromatographie

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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 17134:2019) 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of an identical text or by endorsement, at the latest by January 2020, and conflicting national standards shall be withdrawn at the latest by January 2020.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 EN ISO 13365 prepared by the Technical Committee CEN/TC 309, “Footwear”, in collaboration with ISO 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.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The European Biocidal Product Regulation [2] concerns the placing on the market and use of biocidal products, which are used to protect humans, animals, materials or articles against harmful organisms, like pests or bacteria, by the action of the active substances contained in the biocidal product. The Biocidal Products Regulation (BPR) also sets rules for the use of articles treated with, or intentionally incorporating, one or more biocidal products.

Article 17 "Making available on the market and use of biocidal products" states that biocidal products are not to be made available on the market or used unless authorized in accordance with this regulation.

For placing on the market of Treated Articles, Article 58 defines that a Treated Article is not to be placed on the market unless all active substances contained in the biocidal products that it was treated with or incorporates are approved active substances and included in a Union list of approved active substances for the relevant product type and use or listed in Annex I ("treated article" means any substance, mixture or article which has been treated with, or intentionally incorporates, one or more biocidal products).

This test method was established to verify the compliance of textiles and textile products with the Biocidal Product Regulation for particular product-type 9 preservatives. (Product-type 9: Fibre, leather, rubber and polymerised materials preservatives: Products used for the preservation of fibrous or polymerised materials, such as leather, rubber or paper or textile products by the control of microbiological deterioration.

This product-type includes biocidal products which antagonise the settlement of microorganisms on the surface of materials and therefore hamper or prevent the development of odour and/or offer other kinds of benefits.)

WARNING — The use of this document involves hazardous chemicals. It does not purport to address all of the safety or environmental problems associated with their 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 test method for the determination of the content of the preservative agents (biocidal products) 2-phenylphenol (OPP) and triclosan in textile materials and articles composed of textile products, by liquid chromatography.

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

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

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 of the component of the textile product is extracted with acetonitrile using ultrasonic waves. The filtered extract is analysed by liquid chromatography (LC) with Diode Array Detector (DAD) and/or Mass Selective detector.

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 2-Phenylphenol (OPP), CAS no.: 90-43-7, highest available defined purity standard or certified stock solution.

5.2 Triclosan, CAS no.: 3380-34-5, highest available defined purity standard or certified stock solution.

5.3 Stock solutions

5.3.1 OPP stock solution (500 mg/l in acetonitrile)

Weigh 5 mg of OPP (5.1) with an accuracy of 0,1 mg into a 10 ml volumetric flask and fill to the mark with acetonitrile (5.4).

5.3.2 Triclosan stock solution, (500 mg/l in acetonitrile)

Weigh 5 mg of Triclosan (5.2) with an accuracy of 0,1 mg into a 10 ml volumetric flask and fill to the mark with acetonitrile (5.4).

5.4 Acetonitrile, HPLC grade.

5.5 Water, HPLC grade.

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 Liquid chromatograph with reverse phase C8 or C18 column, with corresponding pre-column, Diode Array Detector (DAD) and/or Mass Selective Detector (MS).

6.3 Ultrasonic bath, with adjustable temperature suitable for operation at about 40 °C.

6.4 Membrane filter, polyamide, pore size 0,45 µm.

6.5 Glass vial, with screw cap that can be tightly sealed (e.g. volume 40 ml).

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

7 Preparation of test specimens

7.1 Sampling

If possible, sample in accordance with EN ISO 5089.

7.2 Specimen preparation

Cut the sample in small pieces of about 0,3 cm to 0,5 cm edge length.

8 Procedure

8.1 Extraction

Weigh (1,00 ± 0,01) g of small pieces of the test specimen to the nearest 0,001 g in a glass vial (6.5). Pipette 20 ml of acetonitrile (5.4) and add it to the test specimen and seal the vial. The test specimen is extracted in an ultrasonic bath (6.3) for 1 h ± 5 min at (40 ± 5) °C.

Subsequently, the extract is filtered through a membrane filter (6.4) into a suitable vial (6.5).

8.2 Determination with LC

8.2.1 Calibration

Calibration is carried out by means of an external standard. Prepare adequate dilutions (in acetonitrile) of preservative stock solutions (5.3.1, 5.3.2). Calibration shall be done using at least three concentration levels.

8.2.2 Determination with LC

Transfer an aliquot of the extraction solution (8.1) into an LC vial (6.6) and determine the content of each preservative by LC (6.2). See Annex A for example of chromatographic conditions.

9 Expression of results

Calculate the mass fraction, w_i, of each preservative detected, in milligrams per kilogram (mg/kg) of material, using the following equation:

$$w_i = \frac{\rho \times V \times F \times 1000}{m \times 1000}$$

where

w_i is the mass fraction, expressed in milligrams per kilogram (mg/kg), of a certain preservative in material;

ρ is the mass concentration of preservative obtained from the calibration, in micrograms per millilitre (µg/ml);

V is the extract volume, in millilitres (ml);

F is the dilution factor;

m is the quantity of sample weighed, in grams (g).

The mass fraction of each preservative is given in milligrams per kilogram (mg/kg), rounded to the nearest 0,1 mg.

10 Test report

The test report shall include the following information:

a) a reference to this European Standard, i.e. EN 17134:2019

b) date of the test;

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

d) the type of liquid chromatography detection;

e) the analytical result for each mass fraction of preservative, in milligrams per kilogram (mg/kg);

f) any deviation from the analytical procedure.