SLOVENSKI STANDARD
oSIST prEN 17134:2018

01-marec-2018

Tekstilije in tekstilni izdelki - Kritične snovi, ki so potencialno prisotne v sestavinah materialov tekstilnih izdelkov - Določevanje nekaterih konzervansov, metoda z uporabo tekočinske kromatografije

Textiles and textile products - Critical substances potentially present in components of textile product materials - Determination of certain preservatives, method using liquid chromatography

Textilien und textile Erzeugnisse - Kritische Stoffe, die potentiell in Bestandteilen von Materialien textiler Erzeugnisse vorhanden sind - Bestimmung gewisser Konservierungsmittel, Verfahren unter Verwendung von Flüssigkeitschromatographie

Produits textiles - Substances critiques potentiellement présentes dans les composants des produits textiles - Méthodes d'essai pour déterminer quantitativement la teneur en agents de conservation (TCMTB, PCMC, OPP, OIT) par chromatographie liquide

Ta slovenski standard je istoveten z: **prEN 17134**

ICS:

59.080.01	Tekstilije na splošno	Textiles in general
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

oSIST prEN 17134:2018 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 17134

February 2018

ICS 59.080.01

English Version

Textiles and textile products - Critical substances potentially present in components of textile product materials - Determination of certain preservatives, method using liquid chromatography

Produits textiles - Substances critiques potentiellement présentes dans les composants des produits textiles - Méthodes d'essai pour déterminer quantitativement la teneur en agents de conservation (TCMTB, PCMC, OPP, OIT) par chromatographie liquide

Textilien und textile Erzeugnisse - Kritische Stoffe, die potentiell in Bestandteilen von Materialien textiler Erzeugnisse vorhanden sind - Bestimmung gewisser Konservierungsmittel, Verfahren unter Verwendung von Flüssigkeitschromatographie

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 248.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents

European foreword

This document (prEN 17134:2018) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

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.

1 Scope

This document specifies a test method for the determination of the content of the following preservative agents (bioactive agents):

— 2-phenylphenol (OPP);

— triclosan

in textile products by liquid chromatography.

NOTE The preservative agent 2-phenylphenol (OPP) can also be determined according to EN ISO 17070 and quantified by means of gas chromatography/mass spectroscopy (GC-MS).

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 2418, *Leather - Chemical, physical and mechanical and fastness test - Sampling location (ISO 2418)*

EN ISO 4044, *Leather - Chemical tests - Preparation of chemical test samples (ISO 4044)*

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

EN ISO 4684, *Leather - Chemical tests - Determination of volatile matter (ISO 4684)*

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 a suitable solvent using ultrasonic waves. The filtered extract is analysed by high-performance liquid chromatography (HPLC) with DAD-MS detection.

5 Reagents

5.1 OPP, minimum 99,5 %.

5.2 OPP stock solution, 500 mg/l in acetonitrile.

5.3 Triclosan, certified reference material

5.4 Triclosan stock solution,

5.5 **Acetonitrile,** HPLC grade.

5.6 **Water,** HPLC grade.

6 Apparatus and materials

Usual laboratory apparatus and laboratory glassware, according to EN ISO 4787, is required and, in particular, the following:

6.1 **Analytical balance,** weighing to an accuracy of 0,1 mg.

6.2 **HPLC system,** with DAD detection or other suitable detectors, e.g. MS.

6.3 **Separation column,** reversed phase C8 or C18 with corresponding pre-column.

6.4 Ultrasonic bath, **e.g. 40 kHz.**

6.5 Membrane filter, **polyamide, 0,45 µm.**

7 Procedure

7.1 Sampling and sample preparation

If possible, sample in accordance with EN ISO 5089. Cut the sample in small pieces.

In the case of a leather component, sample in accordance with EN ISO 2418 and grind in accordance with EN ISO 4044. If sampling in accordance with EN ISO 2418 is not possible (e.g. leathers from finished products, such as garments), details of the sampling shall be given, together with the test report.

7.2 Preparation of analytical solution

Weigh (1,00 ± 0,01) g of small pieces or (when relevant) ground leather to the nearest 0,001 g in a 100 ml conical flask. Pipette 20 ml of acetonitrile (5.5) and add it to the test specimen. The test specimen is extracted in an ultrasonic bath (6.4) for 1 h ± 5 min at room temperature.

NOTE During extraction, the temperature in the mixture increases to about 35 °C.

Subsequently, a part of the extract is filtered through a membrane filter (6.5) into a suitable vial. The filtrate is analysed by HPLC (6.2) and detected preservatives are quantified.

7.3 Chromatographic conditions

See Annex A for example of chromatographic conditions.

7.4 Calibration

Calibration is carried out by means of an external standard. Prepare adequate dilutions (in acetonitrile) of preservative stock solutions (5.2, 5.4). Calibration shall be done using five concentration levels. The calibration is effected by plotting a graph of the preservative peak area versus its concentration, in micrograms per millilitre (µg/ml).

As very different preservative concentrations can be expected, it is not possible to cover the whole range with a single calibration curve.

8 Calculation

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.

The mass fraction of preservative referred to dry matter, according to EN ISO 4684, can be calculated using the following equation:

$$w_{dm} = w \times D$$

where

w_{dm} is the mass fraction of preservative, expressed in milligrams per kilogram (mg/kg), of sample referred to dry matter;

w is the mass fraction of preservative, expressed in milligrams per kilogram (mg/kg), of the sample being tested;

D is the recalculation factor for dry matter

$$D = \frac{100}{100 - w_V}$$

w_V is the mass fraction of volatile matter, based on EN ISO 4684, expressed as a percentage.

9 Test report

The test report shall include the following information:

a) a reference to this European Standard, i.e. EN 17134:201X;

b) the type, origin and designation of the analysed product and the sampling method used;

c) the type of liquid chromatography detection;

d) the analytical result for each mass fraction of preservative, in milligrams per kilogram (mg/kg) rounded to one decimal place;

e) any deviation from the analytical procedure.

Annex A
(informative)

Example of chromatographic conditions

High perfomance liquid chromatography diode array detecton (DAD) and mass selective detection (MS)

Binary system

Column:SB-C18 1,8 µm, Diameter: 4,6 mm, length 100 mm

Injection volume: 10µl

Constant flow rate: 0,8 ml/min

Time min	0,0	5,0	5,3	8,0	8,5	10
Channel A Ammonium Acetate 0,77 g/L	40 %	40 %	20 %	20 %	40 %	40 %
Channel B Acetonitrile	60 %	60 %	80 %	80 %	60 %	60 %

Substance//Signal	DAD 230 nm	DAD 285 nm	MS-Esi negative amu	Retention time min
o-Phenylphenol	x	x	169	2,91
Triclosan	x	x	289/287	7,38

Bibliography

[1] EN ISO 13365:2011, *Leather - Chemical tests - Determination of the preservative (TCMTB, PCMC, OPP, OIT) content in leather by liquid chromatography (ISO 13365:2011)*

[2] EN ISO 17070:2015, *Leather - Chemical tests - Determination of tetrachlorophenol-, trichlorophenol-, dichlorophenol-, monochlorophenol-isomers and pentachlorophenol content (ISO 17070:2015)*