SLOVENSKI STANDARD
oSIST prEN 17134-1:2023

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Tekstilije in tekstilni izdelki - Določevanje biocidnega dodatka - 1. del: 2-fenilfenol in triklosan, metoda z uporabo tekočinske kromatografije

Textiles and textile products - Determination of biocide additives - Part 1: 2-Phenylphenol and triclosan, method using liquid chromatography

Textilien und textile Erzeugnisse - Bestimmung von Biozid-Zusatzstoffen - Teil 1: 2-Phenylphenol und Triclosan, Verfahren mittels Flüssigkeitschromatographie

Ta slovenski standard je istoveten z: **prEN 17134-1**

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59.080.01	Tekstilije na splošno	Textiles in general
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Textiles and textile products - Determination of biocide additives - Part 1: 2-Phenylphenol and triclosan, method using liquid chromatography

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

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European foreword

This document (prEN 17134-1:2023) 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 will supersede EN 17134:2019.

The main changes compared to the previous edition are listed below:

— The previous edition has been split into 3 parts;

— The title has been changed;

— Clause 4 has been rephrased;

— Clause 5 has been rephrased;

— 5.3.1, 5.3.2 and 5.5 have been technically changed and rephrased;

— 6.2 and 6.4 has been technically changed and rephrased;

— 8.1 has been technically changed and rephrased;

— 9.1: The title has been added;

— Formula (1): The factors 1000 of the numerator and denominator have been deleted;

— 9.2 has been added;

— Clause 10 has been amended and rephrased;

— Bibliography; Reference [2] has been replaced.

This document is Part 1 the EN 17134 series of standards under the title *Textiles and textile products - Determination of biocide additives*:

— *Part 1: Part 1: 2-Phenylphenol and triclosan, method using liquid chromatography* (this document)

— *Part 2: Chlorophenol-based preservatives, method using gas chromatography*

— *Part 3: Permethrin, method using liquid chromatography*

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 glass and plastic ware - 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 terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/

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

4 Principle

The sample is cut into small pieces and extracted with acetonitrile using an ultrasonic bath. The filtered extract is analysed by liquid chromatography (LC) with Diode Array Detector (DAD) and/or Mass Selective detector (MS).

5 Reagents

Unless otherwise specified, all reagents shall be of a recognized analytical grade.

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

Prepare an OPP stock solution of an appropriate concentration.

EXAMPLE For an OPP stock solution with a concentration of 500 mg/l, weigh 10 mg of OPP (5.1) with an accuracy of 0,1 mg into a 20 ml volumetric flask and fill to the mark with acetonitrile (5.4).

5.3.2 Triclosan stock solution

Prepare a Triclosan stock solution of an appropriate concentration.

EXAMPLE For a Triclosan stock solution with a concentration of 500 mg/l, weigh 10 mg of Triclosan (5.2) with an accuracy of 0,1 mg into a 20 ml volumetric flask and fill to the mark with acetonitrile (5.4).

5.4 Acetonitrile, HPLC grade.

5.5 Water, grade 3 according to ISO 3696.

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 (LC) coupled with 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, e.g. polyamide or regenerated cellulose, 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 approximately 1 g of small pieces of the test specimen to the nearest 0,01 g in a glass vial (6.5). Pipette 20 ml of acetonitrile (5.4), 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.

If not enough test specimen is available, its mass and solvent volume can be reduced proportionally.

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

9.1 Calculation

Calculate the mass fraction, w_i, of each preservative detected, in milligrams per kilogram (mg/kg) of material, using the following Formula (1):

$$w_i = \frac{\rho \times V \times F}{m} \quad (1)$$

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, if used;

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 a maximum of 2 significant figures with a maximum of 2 decimal points.

9.2 Precision of the method

This method is able to determine the concentrations of OPP and triclosan with a limit of quantification (LOQ) of 10 mg/kg or lower if a LC-DAD is used. If a LC-MS is used this method is able to determine the concentrations of OPP with a LOQ of 0,02 mg/kg or lower and the concentrations of triclosan with a LOQ of 0,2 mg/kg or lower.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. prEN 17134-1:2023;

b) identification of the submitted sample;

c) date of the test;

d) the type of liquid chromatography detection;

e) mass fraction of each identified and quantified preservative (9.1), in milligrams per kilogram (mg/kg);

f) any deviation from the analytical procedure;

g) any unusual features observed.

Annex A
(informative)

Example of chromatographic conditions

High performance liquid chromatography with diode array detection (DAD) and mass selective detection (MS)

Binary system

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

Injection volume: 10 µl

Constant flow rate: 0,8 ml/min

Table A.1 — Solvent gradient

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

Table A.2 — DAD wavelength, quantifier ions and retention times

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