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Textiles — Determination of certain preservatives —

Part 2: Determination of triclosan residues method using LC-MS/MS

Textiles — Détermination de certains conservateurs —

Partie 2: Détermination des résidus de triclosan par une méthode utilisant LC-MS/MS

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

A list of all parts in the ISO 22992 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Textiles — Determination of certain preservatives —

Part 2: Determination of triclosan residues method using LC-MS/MS

WARNING — This document calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this document that the execution of its provisions is entrusted to appropriately qualified and experienced people.

1 Scope

This document specifies a method for determination of triclosan residues in textiles by high performance liquid chromatography — tandem mass spectrometry (HPLC-MS/MS).

This method is applicable to all kinds of textile products.

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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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:

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

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

4 Principle

The triclosan is extracted from textile specimen by ultrasonic generator with methanol. After being concentrated and diluted to volume, the residue is determined by HPLC-MS/MS, quantified by external standard method.

Statistical data of interlaboratory trial is given in Annex B.

5 Reagents

5.1 Water, grade 1 water specified in ISO 3696.

5.2 Methanol, HPLC grade.

5.3 Triclosan, CAS No. 3380-34-5, with a purity of more than 98 %.

6 Apparatus and materials

6.1 High performance liquid chromatography — tandem mass spectrometry (HPLC-MS/MS), equipped with electrospray ionization source.

6.2 Rotary vacuum evaporator.

6.3 Ultrasonic bath, with controllable heating capable of maintaining a temperature of about 40 °C.

6.4 Nitrogen blowing device.

6.5 Balance, with a resolution of 0,01 g used for preparing test specimen.

6.6 Balance, with a resolution of 0,1 mg used for preparing standard solution.

6.7 Glass vial, with a capacity of 40 ml with tight closure.

6.8 Round bottom flask, with a capacity of 100 ml.

6.9 Organic phase filtration membrane, with a pore size of 0,22 µm.

7 Test procedure

7.1 Preparation of standard solution

7.1.1 Standard stock solution

Prepare 1 000 mg/l stock standard solution of the triclosan (5.3) in methanol (5.2).

7.1.2 Working solution

Prepare a working solution of the triclosan in methanol and dilute it to a series of suitable concentrations depending on test needs. Select at least five appropriate dilutions of the calibration sets to create calibration curve.

NOTE Standard stock solution is kept in the refrigerator at 0 °C to 4 °C for up to 6 months. Working solution is kept in the refrigerator at 0 °C to 4 °C for up to 3 months.

7.2 Preparation of test specimen

Prepare a representative test specimen of the sample. Cut it into small pieces with a maximum dimension less than 5 mm and mix them homogeneously. Weigh (1,00 ± 0,01) g of the pieces with a balance (6.5).

7.3 Ultrasonic wave extraction

Put the pieces into a vial with tight closure (6.7) and add 25 ml of methanol (5.2). Place the vial in an ultrasonic generator (6.3) at about 40 °C for (30 ± 2) min. Filter and transfer the extract into 100 ml flask (6.8). Add 25 ml of methanol to the residue and place the vial in the ultrasonic generator to extract the residue at about 40 °C for (30 ± 2) min. Filter and merge the extract into the flask (6.8).

Concentrate the extract with the rotary vacuum evaporator (6.2) at 40 °C to almost 1 ml. Blow slowly to nearly dry with nitrogen (6.4). Add 2 ml of methanol to dissolve the residue and then filter by filtration membrane (6.9). The filtrate is ready for determining the triclosan.

7.4 Determination of triclosan

Determine the triclosan in solution (7.3) by HPLC-MS/MS (6.1).

The test parameters by HPLC-MS/MS are given in Annex A as an example.

When the triclosan level is very low, it is necessary to increase the mass of the test pieces or concentrate the specimen liquid.

When the triclosan level is beyond the linear detector response range of the equipment, it is necessary to dilute the specimen liquid properly.

8 Blank test

Run a blank test to control contamination.

9 Calculation

The content of the triclosan is expressed by the mass ratio of the triclosan to test specimen, in mg/kg. Calculate the result by using Formula (1), and the result is rounded at two significant figures:

$$X = \frac{(C - C_0) \times V \times F}{m} \tag{1}$$

where

X is the content of the triclosan in the textile specimen, in mg/kg;

C is the concentration of the triclosan in the specimen solution, in mg/l;

C_0 is the concentration of the triclosan in the blank solution, in mg/l;

V is the final volume of the specimen solution, in ml;

F is dilution factor;

m is the mass of the test specimen, in g.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 22992-2:2020;

b) all details necessary for identification of the sample tested;

c) the content of the triclosan, in mg/kg;

d) any deviation from the procedure specified.

Annex A
(informative)

Test parameters by HPLC-MS/MS

A.1 General

As the instrumental equipment of the laboratories can vary, no generally applicable parameters can be provided for chromatographic analyses. The following parameters have been found successfully.

A.2 HPLC test parameters

a) Chromatographic column, C18 reversed-phase column, 150 mm (column length) × 2,1 mm (inside diameter) × 3,5 µm (particle size), or equivalent column.

b) Mobile phase, methanol +H_2O (90:10, volume fraction).

c) Flow rate, 0,25 ml/min.

d) Column temperature, room temperature.

e) Injection volume, 20,0 µl.

A.3 Tandem mass spectrometry test parameters

a) Ionization mode, Electrospray ionization (ESI).

b) Scanning mode, negative ion scanning.

c) Detection mode, multiple reaction monitoring (MRM).

d) Electrospray voltage, −4 500 V.

e) Curtain gas (CUR), 30 Psi.

f) Atomized gas (GS1), 40 Psi.

g) Auxiliary gas (GS2), 30 Psi.

h) Collision gas (CAD), 6,0 Psi.

i) Ion source temperature, 550 °C.

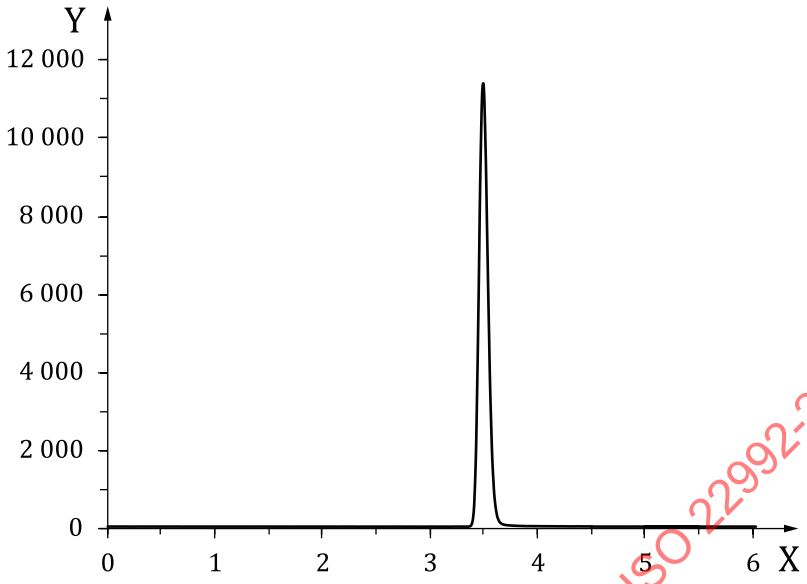
j) Typical transitions and detection limit shown in Table A.1.

Table A.1 — Typical transitions and detection limit

Object name	Precursor ion (m/z)	Product ion (m/z)	Declustering potential (V)	Collision energy (eV)	Detection limit (mg/kg)
Triclosan	286,7	35,0[a]	−35	−35,3	0,5
		142,1	−35	−41,5	

[a] Quantitative transition

HPLC-MS/MS chromatograms are shown in Figures A.1 and A.2.

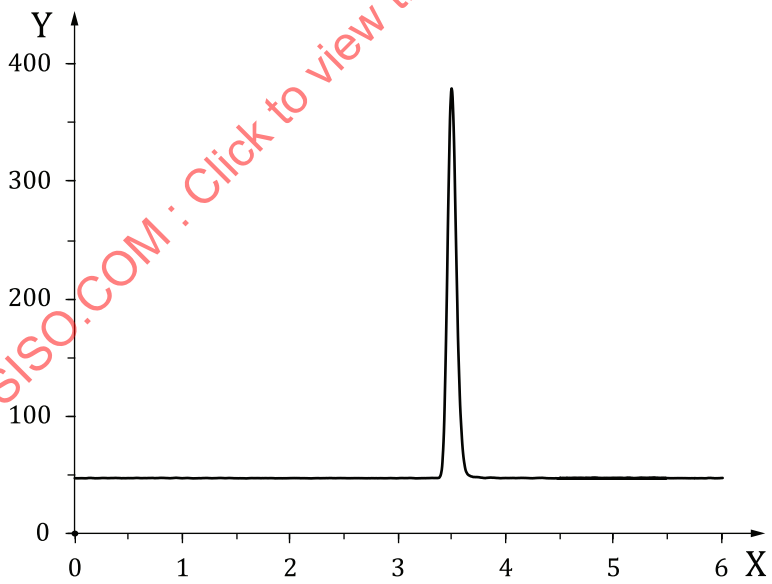


Key

Y abundance

X time, in min

Figure A.1 — HPLC-MS/MS chromatogram of triclosan (transitions 286,7/35,0)



Key

Y abundance

X time, in min

Figure A.2 — HPLC-MS/MS chromatogram of triclosan (transitions 286,7/142,1)