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DRAFT

INTERNATIONAL
STANDARD

**ISO/FDIS
17881-2**

ISO/TC **38**

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on: **2015-11-10**

Textiles — Determination of certain flame retardants —

Part 2:
Phosphorus flame retardants

Textiles — Détermination de certains retardateurs de flamme —

Partie 2: Retardateurs de flamme phosphorés

Please see the administrative notes on page iii

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ISO/CEN PARALLEL PROCESSING

This final draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-lead** mode of collaboration as defined in the Vienna Agreement. The final draft was established on the basis of comments received during a parallel enquiry on the draft.

This final draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel two-month approval vote in ISO and formal vote in CEN.

Positive votes shall not be accompanied by comments.

Negative votes shall be accompanied by the relevant technical reasons.

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 38, *Textiles*.

ISO 17881 consists of the following parts, under the general title *Textiles — Determination of certain flame retardants*:

— *Part 1: Brominated flame retardants*

— *Part 2: Phosphorus flame retardants*

The following parts are under preparation:

— *Part 3: Chlorinated paraffin flame retardants*

Textiles — Determination of certain flame retardants —

Part 2: Phosphorus flame retardants

WARNING — This International Standard calls for the use of substances and/or procedures that may 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 International Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

1 Scope

This part of ISO 17881 specifies a test method for determining some phosphorous flame retardants in textiles by high performance liquid chromatography – tandem mass spectrometry (HPLC-MS/MS).

The method is applicable to all kinds of textile products.

NOTE For tris (1-aziridinyl) phosphineoxide (TEPA), only unbonded TEPA is extractable.

2 Principle

The flame retardants are extracted from textile specimen by ultrasonic generator with acetone. The flame retardants in the specimen are identified and quantified by HPLC-MS/MS.

3 Reagents

Unless otherwise specified, use only reagents of recognized analytical grade.

3.1 Tris (2,3-dibromopropyl) phosphate (TRIS), CAS no. 126-72-7.

3.2 Tris (1-aziridinyl) phosphineoxide (TEPA), CAS no. 545-55-1.

3.3 Tris (2-chloroethyl) phosphate (TCEP), CAS no. 115-96-8.

3.4 Acetone.

3.5 Acetonitrile (ACN).

3.6 Ammonium acetate solution, 10 mmol/l.

4 Apparatus

4.1 High performance liquid chromatography – tandem mass spectrometry (HPLC-MS/MS).

4.2 Ultrasonic generator, with a frequency from 35 kHz to 45 kHz.

4.3 Evaporator device, with water bath at 40 °C.

4.4 Glass vial, 40 ml with tight closure.

4.5 Flask, 100 ml.

4.6 Filtration membrane, 0,45 µm.

4.7 Balance, an accuracy of 0,1 mg.

5 Procedure

5.1 Preparation of standard solutions

5.1.1 Stock standard solution

Prepare 1 000 µg/ml stock standard solutions of the individual flame retardant (3.1 to 3.3) in acetonitrile (3.5).

5.1.2 Working solution

Prepare an admixture working solution of three flame retardants in acetonitrile 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 and perform HPLC-MS/MS analysis.

5.2 Preparation of test specimen

Prepare a representative test specimen of the sample. Cut it into small pieces and weigh (1,00 ± 0,01) g of the pieces with a balance (4.7).

5.3 Ultrasonic wave extraction

Put the pieces into a vial with tight closure (4.4) and add 20 ml of acetone. Place the vial in an ultrasonic generator (4.2) at 40 °C for 40 min. Filter and transfer the extract into 100 ml flask (4.5). Add 20 ml of acetone to the residue and place the vial in the ultrasonic generator to extract the residue at 40 °C for 20 min. Filter and merge the extract into the flask (4.5).

Evaporate the extract to near dryness by evaporator device (4.3). Add 2 ml of acetonitrile to dissolve the residue and then filter by filtration membrane (4.6). The filtrate is ready for determination of flame retardants.

5.4 Flame retardants determination

Determinate the flame retardants in solution (5.3) by HPLC-MS/MS (4.1). The test parameters by HPLC-MS/MS are given in Annex A as an example. Run a blank to control contamination.

When the flame retardants level is very low, it is necessary to increase the mass of the pieces in order to reach at least three times the detection limit.

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

6 Calculation

Quantify the concentration of each flame retardant by using the calibration curve. The content of each flame retardant is expressed by the mass ratio of flame retardant to test specimen, in µg/g. Calculate the result by using Formula (1).

$$X_i = \frac{(C_i - C_0) \times V}{m} \quad (1)$$

where

X_i is the content of the flame retardant, *i*, in the textile specimen, in µg/g;

C_i is the concentration of the flame retardant, *i*, in the specimen solution, in µg/ml;

C_0 is the concentration of the flame retardant, *i*, in the blank solution, in µg/ml;

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

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

7 Test report

The test report shall include the following information:

a) a reference to this part of ISO 17881, i.e. ISO 17881-2:2015;

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

c) the content of each flame retardant;

d) any deviation from the procedure specified.

Annex A
(informative)

Test parameters by HPLC-MS/MS

A.1 Instrument parameters

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

a) Eluent 1: 10 mmol/l ammonium acetate solution;

b) Eluent 2: Acetonitrile;

c) Chromatographic column: Pursuit XRs C18, 100 mm × 2,0 mm, 5 µm;

d) Column temperature: 30 °C;

e) Flow rate: 0,2 ml/min;

f) Injection volume: 5,0 µl;

g) Gradient:

Time (min)	Eluent 2 (%)
0	10
3	70
10	80
12	95
17	95
17,1	10
25	10

h) Detection mode: Quadrupole tandem mass spectrometers;
Multiple Reaction Monitoring (MRM);

i) Ionizing mode: ESI electro spray ionizing method and positive-ion detection;

j) Impressed voltage: 5 500 V;

k) Temperature of spray: 400 °C;

l) Spray gas: Nitrogen.

A.2 Typical transitions and detection limit

Typical transitions and detection limit for flame retardants are shown in Table A.1.

Table A.1 — Typical transitions and detection limit

No.	Flame retardant	Precursor ion (m/z)	Product ion (m/z)	Detection limit (µg/g)
1	Tris (2,3-dibromopropyl) phosphate (TRIS)	698,6	99,1[a]	1
			299,2	
2	Tris (1-aziridinyl) phosphineoxide (TEPA)	174,0	131,0[a]	1
			90,0	
3	Tris (2-chloroethyl) phosphate (TCEP)	284,9	99,0	1
			63,0[a]	

[a] Quantitative transition.