INTERNATIONAL STANDARD

ISO 17881-2

First edition
2016-02-01

Textiles — Determination of certain flame retardants —

Part 2: Phosphorus flame retardants

Textiles — Détermination de certains retardateurs de flamme — Partie 2: Retardateurs de flamme phosphorés

Reference number
ISO 17881-2:2016(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*

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 17881-2:2016;

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.

Annex B
(informative)

Round Robin test

The Round Robin test was carried out by five textile laboratories in China.

A total of nine samples were prepared, three from cotton woven fabrics (no. 1 to 3), three from polyester woven fabrics (no. 4 to 6) and three from 50 cotton/50 polyester woven fabrics (no. 7 to 9) each containing three flame retardants, i.e. TRIS, TCEP, and TEPA. The three samples from each fabric were at three concentration levels of 10 mg/kg, 200 mg/kg, and 500 mg/kg. Three test specimens from each sample were tested in each laboratory.

The results of the Round Robin test are shown in Table B.1.

Table B.1 — Results of the Round Robin test

Sample no.	Substance	Expected value (mg/kg)	Mean (mg/kg)	Recovery (%)	Repeatability		Reproducibility	
					S_r (mg/kg)	CV_r (%)	S_R (mg/kg)	CV_R (%)
1	TRIS	10	8,59	85,90	0,35	4,08	0,32	3,71
	TCEP	10	8,98	89,78	0,34	3,82	0,97	10,85
	TEPA	10	7,53	75,27	0,27	3,58	1,54	20,44
2	TRIS	200	189,56	94,78	13,02	6,87	20,98	11,07
	TCEP	200	194,92	97,46	9,52	4,88	18,44	9,46
	TEPA	200	206,04	103,02	15,73	7,64	14,84	7,20
3	TRIS	500	443,73	88,75	10,86	2,45	31,28	7,05
	TCEP	500	454,77	90,95	6,82	1,73	32,30	7,10
	TEPA	500	528,33	105,67	37,03	7,01	42,00	7,95
4	TRIS	10	8,42	84,24	0,49	5,79	1,00	11,87
	TCEP	10	7,95	79,48	0,71	8,89	1,76	22,13
	TEPA	10	7,64	76,40	0,27	3,54	1,83	23,95
5	TRIS	200	176,51	88,26	7,45	4,22	15,53	8,80
	TCEP	200	188,70	94,35	8,18	4,33	11,37	6,02
	TEPA	200	204,53	102,27	13,13	6,42	12,71	6,22
6	TRIS	500	437,35	87,47	15,69	3,59	27,04	6,18
	TCEP	500	456,61	91,32	12,68	2,78	20,77	4,55
	TEPA	500	475,22	95,04	24,75	5,21	17,54	3,69
7	TRIS	10	8,04	80,36	0,28	3,51	1,72	21,43
	TCEP	10	8,52	85,24	0,40	4,74	0,92	10,76
	TEPA	10	7,36	73,60	0,37	5,05	2,86	38,88
8	TRIS	200	183,53	91,76	5,71	3,11	9,54	5,20
	TCEP	200	200,93	100,46	7,05	3,51	19,75	9,83
	TEPA	200	185,82	92,91	7,45	4,01	19,20	10,33

NOTE The results of TEPA are from three laboratories.

Table B.1 *(continued)*

Sample no.	Substance	Expected value (mg/kg)	Mean (mg/kg)	Recovery (%)	Repeatability		Reproducibility	
					S_r (mg/kg)	CV_r (%)	S_R (mg/kg)	CV_R (%)
9	TRIS	500	475,96	95,19	13,70	2,88	34,45	7,24
	TCEP	500	476,92	95,38	15,60	3,27	52,00	10,90
	TEPA	500	507,44	101,49	36,68	7,23	21,05	4,15
NOTE The results of TEPA are from three laboratories.								

ICS 59.080.01

Price based on 7 pages