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Textiles — Determination of certain flame retardants —

Part 1: Brominated flame retardants

Textiles — Détermination de certains retardateurs de flamme —

Partie 1: Retardateurs de flamme bromés

Reference number
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Contents

Page

Foreword

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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 1:
Brominated 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 brominated flame retardants in textiles by gas chromatography – mass spectrometry (GC-MS).

The method is applicable to all kinds of textile products.

2 Principle

The flame retardants are extracted from textile specimen by ultrasonic generator with toluene. The flame retardants in the specimen are identified by GC-MS and quantified by using internal standard method.

3 Reagents

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

3.1 Monobromobiphenyl (MonoBB), CAS no. 2052-07-5.

3.2 Dibromobiphenyl (DiBB), CAS no. 57422-77-2.

3.3 Tribromobiphenyl (TriBB), CAS no. 59080-34-1.

3.4 Tetrabromobiphenyl (TetraBB), CAS no. 60044-24-8.

3.5 Pentabromo-1,1'-biphenyl (PentaBB), CAS no. 59080-39-6.

3.6 Hexabromobiphenyl (HexaBB), CAS no. 60044-26-0.

3.7 Heptabromo-1,1'-biphenyl (HeptaBB), CAS no. 88700-06-5.

3.8 Octabromobiphenyl (OctaBB), CAS no. 67889-00-3.

3.9 Nonabromobiphenyl (NonaBB), CAS no. 69278-62-2.

3.10 Decabromobiphenyl (DecaBB), CAS no. 13654-09-6.

3.11 Tetrabromodiphenylether (TetraBDE), CAS no. 5436-43-1.

3.12 Pentabromodiphenylether (PentaBDE), CAS no.32534-81-9.

3.13 Hexabromodiphenylether (HexaBDE), CAS no. 207122-15-4.

3.14 Heptabromodiphenylether (HeptaBDE), CAS no. 207122-16-5.

3.15 Octabromodiphenylether (OctaBDE), CAS no. 337513-72-1.

3.16 Decabromodiphenylether (DecaBDE), CAS no. 1163-19-5.

3.17 Hexabromocyclododecane (HBCDD), CAS no. 25637-99-4.

3.18 Decachlorobiphenyl, CAS no.2051-24-3, internal standard (IS).

3.19 Toluene.

NOTE Since brominated flame retardants have many isomers, this method might not cover all of them. Determination of the isomers of flame retardants in Clause 3 can refer to this method according to the principle.

4 Apparatus

4.1 Gas chromatography – mass spectrometry (GC-MS).

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

4.3 Evaporator device, with water bath at 50 °C.

4.4 Brown 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 of stock standard solutions with individual flame retardant (3.1 to 3.17) and internal standard (3.18) in toluene (3.19).

Some commercial reference material solutions may be available in a different solvent.

5.1.2 Internal standard solution

Prepare 10 µg/ml standard solution of decachlorobiphenyl in toluene.

5.1.3 Working solution

Prepare an admixture working solution of 17 flame retardants in internal standard solution (5.1.2) and dilute it to a series of suitable concentrations depending on test needs. Select at least five dilutions of the calibration sets to create calibration curve and perform GC-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 in a vial with tight closure (4.4) and add 20 ml of toluene. Place the vial in an ultrasonic generator (4.2) and extract the pieces for 30 min at room temperature. Filter and transfer the extract into 100 ml flask (4.5). Add 10 ml of toluene to the residue in the vial and place the vial in the ultrasonic generator to extract the residue for 15 min at room temperature. 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 internal standard solution (5.1.2) 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

Determine the flame retardants in the solution (5.3) by GC-MS (4.1). The test parameters by GC-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 17881-1:2016;

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 GC-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) Capillary column: VF-5ht, length 15 m, inside diameter 0,25 mm, film thickness 0,1 µm or equivalent;

b) Temperature programme: 100 °C for 2 min, 100 °C to 310 °C (20 °C/min), 310 °C for 5 min;

c) Injector temperature: 280 °C;

d) Transfer line temperature: 300 °C;

e) Carrier gas: Helium with a purity of no less than 99,999 % delivered at 1,5 ml/min;

f) Ionization mode: EI;

g) Ionization energy: 70 eV;

h) Detection mode: Selected ion monitor detection;

i) Injector system: Splitless, split at 1 min;

j) Injector volume: 1 µl.

A.2 Typical ions and detection limit

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

Table A.1 — Typical ions and detection limit

No.	Flame retardant	Typical ions/amu		Detection limit (µg/g)
		Target ion	Target ion	
1	Monobromobiphenyl (MonoBB)	152	234, 232, 152	5
2	Dibromobiphenyl (DiBB)	152	312, 310, 152	5
3	Tribromobiphenyl (TriBB)	230	392, 390, 230	5
4	Tetrabromobiphenyl (TetraBB)	310	470, 310, 308	5
5	Pentabromo-1,1'-biphenyl (PentaBB)	388	550, 390, 388	5
6	Hexabromobiphenyl (HexaBB)	468	628, 468, 466	5
7	Heptabromo-1,1'-biphenyl (HeptaBB)	546	705, 546, 544	10
8	Octabromobiphenyl (OctaBB)	544	785, 546, 544	10
9	Nonabromobiphenyl (NonaBB)	705	864, 705, 703	10

Table A.1 *(continued)*

No.	Flame retardant	Typical ions/amu		Detection limit (μg/g)
		Target ion	Target ion	
10	Decabromobiphenyl (DecaBB)	783	944, 783, 781	10
11	Tetrabromodiphenylether (TetraBDE)	326	488, 486, 326	5
12	Pentabromodiphenylether (PentaBDE)	404	564, 406, 404	5
13	Hexabromodiphenylether (HexaBDE)	484	643, 484, 482	5
14	Heptabromodiphenylether(HeptaBDE)	562	722, 562, 456	5
15	Octabromodiphenylether (OctaBDE)	642	801, 642, 639	5
16	Decabromodiphenylether (DecaBDE)	799	959, 799, 797	10
17	Hexabromocyclododecane (HBCDD)	157	319, 239, 157	10
18	Decachlorobiphenyl (IS)	498	498, 428, 214	—

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 six flame retardants, i.e. DiBB, PentaBB, DecaBB, PentaBDE, DecaBDE, and HBCDD. The three samples from each fabric were at three concentration levels of 50mg/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	DiBB	50	49,62	99,24	2,08	4,20	5,72	11,52
	PentaBB	50	45,00	90,00	1,97	4,37	6,16	13,69
	DecaBB	50	44,76	89,52	1,63	3,64	6,401	14,30
	PentaBDE	50	42,52	85,03	1,46	3,42	7,57	17,82
	DecaBDE	50	46,23	92,46	1,82	3,94	7,97	17,24
	HBCDD	50	46,56	93,12	2,78	5,96	1,79	3,85
2	DiBB	200	199,37	99,68	6,71	3,36	14,04	7,04
	PentaBB	200	198,84	99,42	6,77	3,41	12,49	6,28
	DecaBB	200	191,39	95,70	6,28	3,28	10,35	5,41
	PentaBDE	200	189,96	94,98	8,26	4,35	15,69	8,26
	DecaBDE	200	183,85	91,93	9,51	5,17	20,68	11,25
	HBCDD	200	192,05	96,03	6,16	3,21	7,70	4,01
3	DiBB	500	491,48	98,30	13,99	2,85	46,44	9,45
	PentaBB	500	482,46	96,49	21,19	4,39	12,12	2,51
	DecaBB	500	478,75	95,75	20,72	4,33	27,11	5,66
	PentaBDE	500	479,23	95,85	9,47	1,98	19,74	4,12
	DecaBDE	500	480,40	96,08	18,65	3,88	23,59	4,91
	HBCDD	500	475,28	95,06	35,11	7,39	28,18	5,93
4	DiBB	50	46,14	92,28	2,38	5,17	3,50	7,59
	PentaBB	50	44,90	89,79	1,30	2,90	6,30	14,03
	DecaBB	50	47,07	94,14	2,14	4,55	7,42	15,77
	PentaBDE	50	41,70	83,40	1,43	3,43	7,82	18,76
	DecaBDE	50	46,35	92,70	2,00	4,31	8,85	19,11
	HBCDD	50	43,84	87,68	2,23	5,08	6,59	15,04

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 (%)
5	DiBB	200	192,47	96,24	7,96	4,14	10,64	5,53
	PentaBB	200	191,08	95,54	10,80	5,65	11,75	6,15
	DecaBB	200	185,75	92,87	6,57	3,54	16,27	8,76
	PentaBDE	200	189,28	94,64	7,65	4,04	15,06	7,96
	DecaBDE	200	187,99	94,00	9,46	5,03	15,05	8,00
	HBCDD	200	180,58	90,29	6,55	3,63	12,76	7,07
6	DiBB	500	480,93	96,19	17,75	3,69	30,69	6,38
	PentaBB	500	483,82	96,76	13,65	2,82	13,84	2,86
	DecaBB	500	479,54	95,91	12,94	2,70	25,24	5,26
	PentaBDE	500	473,41	94,68	7,21	1,52	28,69	6,06
	DecaBDE	500	468,39	93,68	16,59	3,54	18,11	3,87
	HBCDD	500	485,21	97,04	21,12	4,35	17,67	3,64
7	DiBB	50	47,14	94,29	1,52	3,23	4,31	9,15
	PentaBB	50	44,67	89,34	1,79	4,01	6,99	15,65
	DecaBB	50	45,03	90,06	2,73	6,05	3,06	6,79
	PentaBDE	50	40,33	80,66	2,06	5,11	8,93	22,15
	DecaBDE	50	45,43	90,86	2,70	5,94	5,61	12,34
	HBCDD	50	42,52	85,04	2,21	5,19	4,54	10,69
8	DiBB	200	189,40	94,70	7,37	3,89	10,50	5,55
	PentaBB	200	202,38	101,19	5,89	2,91	12,36	6,11
	DecaBB	200	189,63	94,82	8,12	4,28	5,80	3,06
	PentaBDE	200	189,05	94,53	3,99	2,11	15,31	8,10
	DecaBDE	200	191,94	95,97	7,07	3,68	7,78	4,05
	HBCDD	200	179,18	89,59	7,65	4,27	15,56	8,69
9	DiBB	500	475,97	95,19	31,27	6,57	41,19	8,65
	PentaBB	500	494,46	98,89	11,23	2,27	23,03	4,66
	DecaBB	500	503,78	100,76	16,22	3,22	24,95	4,95
	PentaBDE	500	477,93	95,59	12,26	2,57	8,95	1,87
	DecaBDE	500	479,87	95,97	19,73	4,11	35,29	7,35
	HBCDD	500	489,33	97,87	20,70	4,23	13,04	2,67

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