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DRAFT

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STANDARD

**ISO/FDIS
17881-1**

ISO/TC **38**

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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

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).

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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 part is under preparation:

— *Part 3: Chlorinated paraffin 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 17881-1:2015;

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 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