भारतीय मानक

Indian Standard

IS 16197 (Part 12) : 2024
IEC 62321-12 : 2023

विद्युत तकनीकी उत्पादों में कुछ पदार्थों का निर्धारण

भाग 12 एक साथ निर्धारण — गैस क्रोमैटोग्राफी-मास स्पेक्ट्रोमेट्री द्वारा पॉलिमर में पॉलीब्रोमिनेटेड बाइफिनाइल, पॉलीब्रोमिनेटेड डाइफिनाइल ईथर और थैलेट्स

Determination of Certain Substances in Electrotechnical Products

Part 12 Simultaneous Determination — Polybrominated Biphenyls Polybrominated Diphenyl Ethers and Phthalates in Polymers by Gas Chromatography-Mass Spectrometry

ICS 01.110; 13.020.01; 29.100.01

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NATIONAL FOREWORD

This Indian Standard (Part 12) which is identical to IEC 62321-12 : 2023 'Determination of certain substances in electrotechnical products — Part 12: Simultaneous determination — Polybrominated biphenyls, polybrominated diphenyl ethers and phthalates in polymers by gas chromatography-mass spectrometry' issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Standardization of Environmental Aspects for Electrical and Electronics Products Sectional Committee and approval of the Electrotechnical Division Council.

This standard is published in ten parts. Other parts in this series are:

Part 1	Introduction and overview
Part 2	Disassembly, disjointment and mechanical sample preparation
Part 3	Screening
Part 4	Mercury in polymers, metals and electronics by cv-Aas, cv-Afs, icp-Oes and icp–Ms
Part 5	Cadmium, lead and chromium in polymers and electronics and cadmium and lead in metals by aas, afs, icp - Oes and icp – Ms
Part 6	Polybrominated biphenyls and polybrominated diphenyl ethers in polymers by gas chromatography-mass spectrometry (GC-MS)
Part 7	Hexavalent chromium
Part 8	Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py-TD-GC-MS)

The text of IEC standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminologies and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and

b) Comma (,) has been used as a decimal marker, while in Indian Standards the currentpractice is to use a point (.) as the decimal marker.

In this standard, reference appears to International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
IEC 62321-1 : 2013 Determination of certain substances in electrotechnical products — Part 1: Introduction and overview	IS 16197 (Part 1) : 2014/IEC 6232-1 : 2013 Determination of certain substances in electrotechnical products: Part 1 Introduction and overview	Identical
IEC 62321-2 Determination of certain substances in electrotechnical products — Part 2: Disassembly, disjointment and mechanical sample preparation	IS 16197 (Part 2) : 2014/ IEC 62321-2 : 2023 Determination of certain substances in electrotechnical products: Part 2 Disassembly, disjointment and mechanical sample preparation	Identical

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CONTENTS

INTRODUCTION

The widespread use of electrotechnical products has drawn increased attention to their impact on the environment. In many countries around the world it has been a contributing factor in adapting regulations that affect wastes, substances and energy use of electrotechnical products.

The use of certain substances (e.g. lead (Pb), cadmium (Cd), polybrominated diphenyl ethers (PBDEs) and specific phthalates) in electrotechnical products is a source of concern in current and proposed regional legislation.

The purpose of the IEC 62321 series is therefore to provide test methods that will allow the electrotechnical industry to determine the levels of certain substances of concern in electrotechnical products on a consistent global basis.

This first edition of IEC 62321-12 introduces a new part in the IEC 62321 series.

WARNING – Persons using this document should be familiar with normal laboratory practices. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

Indian Standard

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS

PART 12 SIMULTANEOUS DETERMINATION — POLYBROMINATED BIPHENYLS POLYBROMINATED DIPHENYL ETHERS AND PHTHALATES IN POLYMERS BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY

1 Scope

This part of IEC 62321 specifies a reference test method for the simultaneous determination of polybrominated biphenyls, polybrominated diphenyl ethers, and four phthalates: di-isobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzylbutyl phthalate (BBP), di-(2-ethylhexyl) phthalate (DEHP) in polymers of electrotechnical products.

The extraction technique described in this document is the ultrasonic-assisted extraction used for simultaneous extraction for sample preparation.

Gas chromatography-mass spectrometry (GC-MS) is considered as the reference technique for the measurement of the simultaneous determination of analytes in the range of 25 mg/kg to 2 000 mg/kg.

The test method using ultrasonic-assisted extraction followed by GC-MS detection has been evaluated by the tests of polypropylene (PP), polyvinylchloride (PVC), acrylonitrile butadiene styrene (ABS), acrylate rubber (ACM), polystyrene (PS), polyurethane (PU) and polyethylene (PE) materials.

This document has the status of a horizontal standard in accordance with IEC Guide 108.

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.

IEC 62321-1:2013, *Determination of certain substances in electrotechnical products – Part 1: Introduction and overview*

IEC 62321-2, *Determination of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1.1
simultaneous determination
same analysis and detection procedure to determine different classes of analytes that includes (but is not limited to): pretreatment, extraction, cleaning-up and detection

3.1.2
ultrasonic-assisted extraction
extraction technique using ultrasonic waves, which makes it possible to accelerate the speed of extraction of substances in the sample matrix (the extractant does not dissolve the sample matrix), in order to improve the extraction efficiency, for example in an ultrasonic bath

3.1.3
calibrant
calibration standard
substance in solid or liquid form with known and stable concentration(s) of the analyte(s) of interest used to establish instrument response (calibration curve) with respect to analyte(s) concentration(s)

[SOURCE: IEC 62321-8:2017, 3.1.3]

3.1.4
technical mixture
commercial product manufactured for industrial use whose purity is not as clearly defined as an individual high purity calibration standard

[SOURCE: IEC 62321-6:2015, 3.1.2, modified – "(e.g. flame retardants)" has been deleted.]

3.2 Abbreviated terms

ABS	acrylonitrile butadiene styrene
ACM	acrylate rubber
BBP	benzyl butyl phthalate
BDE	brominated diphenyl ether
BSA	bis(trimethylsilyl)acetamide
BSTFA	N,O-bis(trimethylsilyl)trifluoroacetamide
CCC	continuing calibration check standard
DBOFB	(4, 4'-dibromooctafluorobiphenyl) (n)
DBP	di-n-butyl phthalate
deca-BB	decabromobiphenyl
deca-BDE	decabromodiphenyl ether
DEHP	di-(2-ethylhexyl) phthalate
DIBP	di-isobutyl phthalate
DMDCS	dimethyldichlorosilane
EI	electron ionization
EPA	U.S. Environmental Protection Agency
GC-MS	gas chromatography-mass spectrometry
IS	internal standard
IUPAC	International Union of Pure and Applied Chemistry
LOD	limit of detection
LOQ	limit of quantification
MDL	method detection limit

PBB	polybrominated biphenyl
PBDE	polybrominated diphenyl ether
PE	polyethylene
PP	polypropylene
PS	polystyrene
PTFE	polytetrafluoroethylene
PTV	programmed temperature vaporizing
PU	polyurethane
PVC	polyvinylchloride
QC	quality control
RSD	relative standard deviation
SIM	selected ion monitoring
TICS	tentatively identified compounds

4 Principle

Different classes of analytes, i.e. PBBs, PBDEs, BBP, DBP, DEHP, and DIBP, in polymers, are simultaneously extracted by ultrasonic-assisted extraction and determined qualitatively and quantitatively by gas chromatography-mass spectrometry (GC-MS) using full scan mode and (or) single (or "selected") ion monitoring (SIM) mode.

5 Reagents and materials

All reagents chemicals shall be tested for contamination and blank values prior to application as follows:

a) n-hexane (GC grade or higher);

b) acetone (GC grade or higher);

c) acetone/n-hexane (1:1, v/v);

d) toluene (GC grade or higher);

e) helium (purity greater than a volume fraction of 99,999 %);

f) technical BDE-209 with BDE-209 ~ 96,9 % and BDE-206 ~ 1,5 % solution;

g) calibrants: refer to 8.4;

h) surrogate and internal standards:

- surrogate standard used to monitor analyte recovery according to 8.2.1 a), 8.5.2 and 8.5.3, e.g. DBOFB (4, 4'-dibromooctafluorobiphenyl) (n), dibutyl phthalate-3,4,5,6-d_4 or di-(2-ethylhexyl) phthalate-3,4,5,6-d_4;
- internal standard used to correct for injection errors, according to 8.2.1 b), 8.2.3 and 8.5.4, e.g. anthracene-d_{10} or CB209 (2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl).

Deuterium substituted target analytes are recommended as surrogate and internal standards. $_{13}$C-labelled nonaBDE and $_{13}$C-labelled decaBDE are recommended for the high-mass PBDEs. Other standards can be used as surrogate and internal standard, if they have been validated to give acceptable blank, recoveries and precision of analysis.

6 Equipment, apparatus and tools

The following items shall be used for the analysis:

a) analytical balance capable of measuring accurately to 0,000 1 g;

b) 1 ml, 5 ml, 10 ml, 25 ml, 100 ml volumetric flasks;

c) ultrasonic bath (450 W, 40 kHz, volume ~10 l, or equivalent);

NOTE 1 Much lower ultrasonic power and frequency, and a much larger bath volume can influence the extraction efficiency. Validation of extraction efficiency can be referred to in Annex B.[1]

d) glass centrifuge tube with a screw cap with polytetrafluoroethylene gasket (for extraction, ~10 ml);

e) centrifuge (capacity not less than 5 000 r/min);

f) deactivated injector liner (for GC-MS);

g) aluminium foil;

NOTE 2 Brown or amber vessels as indicated in the text of the procedure can also be used.

h) microlitre syringe or automatic pipettes;

i) Pasteur pipette;

j) 1,5 ml sample vials with 100 µl glass insert and a screw cap with polytetrafluoroethylene (PTFE) gasket or, depending on the analytical system, a comparable sample receptacle. Brown or amber vessels shall be used as indicated in the text of the procedure;

k) mini-shaker (also known as vortexer or vortex mixer);

l) a gas chromatograph with a capillary column coupled to a mass spectrometric detector (electron ionization, EI). The mass spectrometric detector shall be able to perform selective ion monitoring and have an upper mass range of at least 1 000 m/z. The high-range mass is required to unambiguously identify deca-BDE and nona-BDE. The use of an autosampler is strongly recommended to ensure repeatability;

m) a column length of approximately 15 m that has sufficient separation efficiency for PBB, PBDE and phthalate compounds (see 8.3 a)) for example of suitable column);

n) 0,45 µm PTFE filter membrane;

o) pre-cleaned filter paper, pre extracted using acetone/n-hexane (see Clause 5 c)) as extractant according to 8.2.2 d) for three cycles and dried in the air with a temperature below 45 °C.

7 Sampling

Sampling shall be as described in IEC 62321-2, unless indicated otherwise (e.g. "... using a nipper."). Cryogenic grinding with liquid nitrogen cooling is recommended and the samples shall be ground to pass through a 500 µm sieve before extraction. Otherwise, the sample shall be cut in pieces < 1 × 1 mm.

8 Procedure

8.1 General instructions for the analysis

The following general instructions shall be followed:

In order to reduce blank values, ensure the cleanliness of all glass equipment (excluding volumetric flasks) and deactivate glass wool by subjecting it to 450 °C for at least 30 min.

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[1] Jingu (China), Bandelin (Germany), SONOSWISS (Switzerland), Branson (USA), Shumei (China), SHARP (Japan) are examples of suitable ultrasonic bath or equipment available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

To avoid decomposition or debromination, or both, of PBDEs by UV light during extraction and analysis, glass equipment made from brown or amber glass shall be used after extraction for storage of the extract.

NOTE If no brown or amber glass is available, aluminium foil can be used for protection from light.

8.2 Sample preparation

8.2.1 Stock solution

The following stock solutions shall be prepared:

a) surrogate standard (to monitor analyte recovery): 1 000 µg/ml in an organic solvent (e.g. DBOFB, dibutyl phthalate-3,4,5,6-d_4 or di-(2-ethylhexyl)phthalate-3,4,5,6-d_4 in n-hexane);

b) internal standard (to correct for injection error): 1 000 µg/ml in an organic solvent (e.g. decachlorobiphenyl, anthracene-d_{10} in n-hexane);

c) polybrominated biphenyl (PBB) solution: 100 µg/ml in an organic solvent (e.g. toluene);

d) polybrominated diphenyl ether (PBDE) solution: 100 µg/ml in an organic solvent (e.g. toluene);

e) phthalate (DIBP, DBP, BBP and DEHP) solution: 1 000 µg/ml in an organic solvent (e.g. n-hexane).

f) matrix spiking solution for PBBs, PBDEs and phthalate; containing a total of five calibration congener standards in an organic solvent (e.g. n-hexane) as indicated in Table 1. The addition of 1 ml of a matrix spiking solution containing each of the five analytes in a concentration of 10 µg/ml is suitable for delivery of the required 10 µg (see 11.2 b)) in the matrix spike sample.

Table 1 – Matrix spiking solution

Number of PBDEs congeners		Number of PBBs congeners		Number of phthalate congeners
Mono to penta	1	Mono to penta	1	1
Hexa to deca	1	Hexa to deca	1	

All brominated species from mono- to deca-brominated biphenyl (PBB) and mono- to deca-brominated diphenyl ether (PBDE) shall be included in the PBB and PBDE stock solutions (see 8.4). Other stock solution concentrations can be utilized providing the standard solution concentrations given in 8.5.2 can be achieved. All the standard solutions should be stored at a temperature lower than −10 °C before use.

8.2.2 Extraction

The following steps shall be followed for sample extraction:

a) Transfer 100 mg ± 10 mg of the sample into the centrifuge tube (see Clause 6 d)). Record the sample mass to the nearest 0,1 mg. The sample is allowed to be wrapped up by a pre-cleaned filter paper (see Clause 6 o)) to help in isolating the supernatant, so as to avoid centrifugation (see e) below) after extraction. In this way, the centrifuge tube can be replaced with other glass containers in which the sample can be soaked (see 8.2.2 b)).

b) Add 4 ml acetone/n-hexane (see Clause 5 c)) into the tube and shake for a moment so that the sample is soaked.

NOTE 1 Different extractants can give different extraction efficiencies (see Annex A).

c) Add 25 µl of the surrogate standard (1 000 µg/ml) (see 8.2.1 a)).

d) Extract for 15 min in an ultrasonic bath (see Clause 6 c). The temperature of the ultrasonic bath should not be higher than 40 °C. The temperature of the bath can usually be kept below 40 °C during extraction. The temperature control can be taken by adding an ice-pack or by

changing the water in the bath. The water level in the ultrasonic bath should be higher than the extractant level in the tube during extraction.

WARNING – A temperature of the bath that is too high can be dangerous due to the volatilization of the organic solvent in the sealed tube.

e) Centrifuge the tube at 5 000 r/min for 5 min. Transfer the supernatant into a 25 ml volumetric flask.

f) Repeat b), d) and e) twice. All the supernatants are collected into the same 25 ml volumetric flask.

NOTE 2 An insufficient number of extraction cycles will give lower recoveries of the analytes. See Annex B for details.

g) The volumetric flask is filled with extraction solvent to the mark.

8.2.3 Addition of the internal standard (IS)

Prepare a 1 ml aliquot of each sample extract to be analysed and place it in an appropriate sample vial. Add 20 µl of internal standard solution (see 8.5.3) to the vial and cap the vial. Shake the vial by hand to mix thoroughly.

Inject 1 µl of the sample solution into the GC-MS and analyse it according to the parameters described in 8.3.

8.3 Instrumental parameters

Different conditions can be necessary to optimize a specific GC-MS system to achieve effective separation of all calibration congeners and meet the quality control (QC) and limits of detection (LOD) requirements. The following parameters have been found suitable and are provided as an example (see the chromatograms and mass spectrograms in Annex C and Annex D, respectively):

a) GC column: non-polar (phenyl-arylene-polymer equivalent to 5 % phenyl-methylpolysiloxane); length 15 m; internal diameter 0,25 mm; film thickness 0,1 µm. A high temperature column (maximum = 400 °C) shall be used for the stated GC conditions in the method.

b) PTV (programmed temperature vaporizing), cool on-column, split/splitless injector or comparable injection systems can be used.

   The use of an on-column injector can also be suggested as another way of introducing the sample. This is particularly beneficial for the sensitivity of heavier congeners like octa-BDE and nona-BDE. Be aware of sensitivity to matrix effects.

c) Injector liner: 4 mm single bottom taper glass liner with glass wool at bottom (deactivated).

NOTE 1 Additional deactivation of a purchased deactivated injector liner can be performed. This is especially useful if the "PR-206" quality control requirements in 11.3 cannot be achieved. An example of a chemical deactivation procedure is as follows: a commercially available, factory-deactivated liner (split/splitless single-taper with glass wool at the bottom) is taken and immersed in 5 % dimethyldichlorosilane (DMDCS) in dichloromethane or toluene for 15 min. It is picked up with forceps and drained and immersed three times in the DMDCS to make sure the glass wool has been thoroughly covered and flushed. It is drained once more and the residue solution is blotted onto a clean wiper. The liner is immersed in methanol for 10 min to 15 min, and again drained and immersed three times. It is rinsed inside and out with methanol from a squeeze bottle, followed by dichloromethane from a squeeze bottle. The liner is transferred to a vacuum oven purged with nitrogen and dried at 110 °C for at least 15 min. Once dry it is ready for use.

d) Carrier: helium (see Clause 5 e)), 1,0 ml/min, constant flow.

e) Oven: 100 °C for 2 min, 20 °C/min ramp to 320 °C for 3 min.

f) Transfer line: 300 °C, direct.

g) Ion source temperature: 230 °C.

h) Ionization method: electron ionization (EI), 70 eV.

i) Dwell time: 50 ms in SIM mode.

NOTE 2 To achieve the required data quality for a PBB, PBDE or phthalate GC peak, three to four scans of the quantification ions selected can be acquired per second. This will give the appropriate dwell time for each ion (m/z) to be monitored. The scan rate will result in a dwell time in the range of 50 ms per ion. It is noted that by default some software sets the dwell time as a function of the scan rate. The analysis of PBBs and PBDEs is carried out in selected ion monitoring (SIM) mode with the mass traces given in Table 2 to Table 4. These have been found suitable and are provided as examples.

Table 2 – Reference for the quantification of PBBs

Type of PBBs	Identification ions	Quantification ions
Mono	152 232 233	232
Di	152 310 312	312
Tri	390 230 149	390
Tetra	470 310 308	310
Penta	548 227 388	388
Hexa	628 468 308	468
Hepta	705 546 544	705
Octa	785 546 707	785
Nona	864 786 705	705
Deca	943 783 781	783

Table 3 –Reference for the quantification of PBDEs

Type of PBDEs	Identification ions	Quantification ions
Mono	250 248 141	248
Di	328 221 168	328
Tri	406 248 139	406
Tetra	488 486 326	486
Penta	564 406 404	564
Hexa	644 484 242	644
Hepta	722 562 455	562
Octa	799 642 564	642
Nona	880 721 719	721
Deca	959 799 797	799

Table 4 – Reference for the quantification of each phthalate

Type of phthalate	Identification ions	Quantification ions
DIBP	149 57 104	149
DBP	149 223 205	149
BBP	149 91 296	149
DEHP	149 167 57	149

A full scan run using a total ion current ("full scan") MS method for each sample is also recommended for checking for the existence of target compounds not present in the calibration (tentatively identified compounds or "TICS") or not seen in the SIM window. If present, identify the peak and determine the class of compound (e.g. octabromobiphenyl, pentabromodiphenyl ether) by evaluation of the total ion spectra.

8.4 Calibrants

Reference solutions are used as calibrants. All brominated species from mono-BB to deca-BB, mono-BDE to deca-BDE and phthalates shall be included in the calibration. The following Table 5 is an example list of commercially available reference solutions that have been found suitable for this analysis.

Table 5 – Example of commercially available reference solutions

Group	Compound	CAS No.
PBBs	2-Bromobiphenyl	2052-07-5
	2,5-Dibromobiphenyl	57422-77-2
	2,4,6-Tribromobiphenyl	59080-33-0
	2,2’,5,5’-Tetrabromobiphenyl	59080-37-4
	2,2’,4,5’,6-Pentabromobiphenyl	59080-39-6
	2,2’,4,4’,6,6’-Hexabromobiphenyl	59261-08-4
	2,2’,3,4,4’,5,5’-Heptabromobiphenyl	67733-52-2
	Octabromobiphenyl, technology (hepta + octa + nona)	27858-07-7
	2,2’,3,3’,4,4’,5,5’,6-Nonabromobiphenyl	69278-62-2
	Decabromobiphenyl	13654-09-6
PBDEs	4-Bromodiphenyl ether	101-55-3
	4,4’-Dibromobiphenyl ether	2050-47-7
	3,3’,4-Tribromobiphenyl ether	147217-80-9
	3,3’,4,4’-Tetrabromobiphenyl ether	93703-48-1
	2,2’,4,4’,6-Pentabromobiphenyl ether	189084-64-8
	2,2’,4,4’,5,6’-Hexabromodiphenyl ether	207122-15-4
	2,2’,3,4,4’,5,6-Heptabromodiphenyl ether	189084-67-1
	2,2’,3,4,4’,5,5’,6’-Octabromodiphenyl ether	337513-72-1
	2,2’,3,3’,4,4’,5,5’,6-Nonabromodiphenyl ether	63387-28-0
	Decabromodiphenyl ether	1163-19-5
Phthalates	Butyl benzyl phthalate	85-68-7
	Di-n-butyl phthalate	84-74-2
	Di-(2-ethylhexyl) phthalate	117-81-7
	Di-isobutyl phthalate	84-69-5

8.5 Calibration

8.5.1 General

Wherever possible, the solvent used for the sample and standard solutions shall be the same to avoid any potential solvent effects.

A calibration curve shall be developed for quantitative analysis. At least five calibration solutions shall be prepared in equidistant concentration steps. Quantification is made on the basis of the measurement of the peak areas. The linear regression fit of each calibration curve is required to have a relative standard deviation (RSD) of less than or equal to 15 % of the linear calibration function.

NOTE Linear regression calibration is most desirable. In the event that the linear regression fit requirement (a relative standard deviation (RSD) of less than or equal to 15 %) cannot be achieved, the use of a polynomial calibration is suitable if another statistical treatment (e.g. coefficient of correlation or curve fit of 0,995 or better) can demonstrate acceptability.

8.5.2 Mixing stock solutions for PBB (10 µg/ml for each congener), PBDE (10 µg/ml for each congener), phthalate (10 µg/ml for each analyte) and surrogate standard (10 µg/ml)

Transfer 1,0 ml of each PBB (see 8.2.1 c)) and each PBDE (see 8.2.1 d)) stock solution (100 µg/ml), 100 µl of each phthalate (see 8.2.1 e)) stock solution (1 000 µg/ml) and 100 µl of the surrogate stock solution (see 8.2.1 a)) (1 000 µg/ml) in a 10 ml volumetric flask and fill up with extraction solvent (see Clause 5 c)) up to the mark. All the standard solutions should be stored at a temperature lower than −10 °C before use.

8.5.3 Internal standard solution (100 µg/ml of CB209, anthracene-d_{10})

Transfer 1,0 ml of internal standard (see 8.2.1 b)) solution (1 000 µg/ml) into a 10 ml volumetric flask and fill with solvent (see Clause 5 a)) up to the mark. All the standard solutions should be stored at a temperature lower than −10 °C before use.

8.5.4 Standard solutions

The following calibration solutions are produced from the stock solution of the PBB (10 µg/ml for each congener), PBDE (10 µg/ml for each congener), phthalate (10 µg/ml for each analyte) and surrogate standard (10 µg/ml) (see 8.5.2). The volumes indicated in Table 6 are placed in a 1 ml volumetric flask with a pipette and filled with extraction solvent (see Clause 5 c)) up to the mark. 20 µl of 100 µg/ml internal standard solution (see 8.5.3) is then added.

For deca-BDE, it is possible that the calibration range suggested in Table 6 will have to be modified. When establishing a calibration curve for deca-BDE, the lower range should be set according to the instrument's sensitivity. A higher concentration can be used for the upper range to account for the generally high (a mass fraction of 10 % to 12 %) levels of deca-BDE normally found in samples.

Table 6 – Examples of calibration ranges of PBBs, PBDEs and phthalates

No.	Volume µl		Concentration (per analyte) ng/ml		
	PBB + PBDE + phthalate + surrogate(ul) (10µg/ml) see 8.5.2	CB209, anthracene-d_{10} (100 µg/ml) see 8.5.3	Concentration (PBB), Concentration (PBDE)	Concentration (phthalate)	Concentration (surrogate)
1	10	20	100	100	100
2	25	20	250	250	250
3	50	20	500	500	500
4	100	20	1 000	1 000	1 000
5	200	20	2 000	2 000	2 000
6	400	20	4 000	4 000	4 000
7	800	20	8 000	8 000	8 000

The internal standard is used for the correction of the injection error. Therefore the evaluation of the response factor or ratio is carried out by A/A_{IS}.

To produce the calibration straight lines, the response A/A_{IS} is plotted against the concentration ratio C/C_{IS}.

A linear regression is carried out using Equation (1):

$$\frac{A}{A_{IS}} = a \times \frac{C}{C_{IS}} + b \quad (1)$$

where

A is the peak area of PBB, PBDE, phthalates or the surrogate in the calibration solution;

A_{IS} is the peak area of the internal standard;

C is the concentration of PBB, PBDE, phthalates or the surrogate per congener (ng/ml);

C_{IS} is the concentration of the internal standard (ng/ml);

NOTE 1 It is common practice to set the internal standard concentration to 1 ng/ml for the internal standard methods when the amount and concentration of the internal standard added to the sample and calibrants prior to injection are the same.

a is the slope of the calibration curve;

b is the intercept on the y-axis of the calibration curve.

NOTE 2 A polynomial (e.g. second-order) regression can be utilized in the event that the relative standard deviation curve requirements cannot be achieved using linear regression. All quality control requirements are still in effect when using polynomial regression.

9 Calculation of analyte concentration

9.1 General

Only detected PBB and PBDE compounds shall be included in a total summation.

When there are no PBDEs or no PBBs detected in the sample, the total PBDE or PBB shall be reported as a function of the congener(s) with the highest method detection limits. For example, if the method detection limit is 20 mg/kg for deca-BB and 10 mg/kg for all other PBBs, and no PBBs are found in the sample, the total PBB shall be reported as < 20 mg/kg.

Analytes detected below the LOQ and above the LOD shall be summed using the limit of quantification for the analyte detected. For example, if deca-BB is found above the limit of detection but below the limit of quantification, and if the limit of quantification is 60 mg/kg for deca-BB and no other PBBs are found above the limit of detection in the sample, the total PBB shall be reported as < 60 mg/kg.

Isomers of each PBB and PBDE can give different retention times during GC-MS analysis. Qualification should include all the isomers, not only those in the standard solutions which possibly contain only one isomer of each PBB and PBDE (see Table 5).

Phthalate concentration shall be calculated individually.

9.2 Calculation

Quantify the samples using the calibration curve. The instrument software usually performs the quantification. Normally, the calibration level of the internal standard for all five calibration levels are set to 1 in the instrument method, but it can also be performed manually using the equation of the fit from the calibration.

For a linear fit, the equation takes the form of Equation (2):

$$y = ax + b \quad (2)$$

where

y is the response factor or ratio (A/A_{IS}) for the congener in the sample;

a is the slope of the line that best fits the calibration obtained in Equation (1);

x is the instrumental result (C/C_{IS} where C_{IS} is commonly = 1) in ng/ml (the concentration of the congener in the extract);

b is the intercept on the y-axis of the calibration curve.

For a quadratic fit the equation takes the form of Equation (3):

$$y = ax^2 + bx + c \quad (3)$$

where

y is the response factor or ratio (A/A_{IS}) for the congener in the sample;

a and b are constants that correspond to the curve that best fits the calibration;

x is the instrumental result in ng/ml (the concentration of the congener in the extract);

c is the y intercept or the concentration when the response factor equals 0.

Equation (2), which is in the form of a linear equation, can be rewritten in the form of Equation (4):

$$C = \left(\frac{A}{A_{IS}} - b \right) \times \left(\frac{C_{IS}}{a} \right) \quad (4)$$

where

A is the peak area of each analyte or surrogate;

A_{IS} is the peak area of the internal standard;

C is the (intermediate) concentration of each analyte or surrogate in ng/ml;

C_{IS} is the concentration of the internal standard in ng/ml.

NOTE 1 It is common practice to set the internal standard concentration to 1 ng/ml for the internal standard methods when the amount and concentration of internal standard added to the sample and calibrants prior to injection are the same.

a is the slope of the calibration curve;

b is the intercept on the y-axis of the calibration curve.

A polynomial (e.g. second-order) regression can be utilized in the event that the relative standard deviation curve requirements cannot be achieved using linear regression. All quality control requirements are still in effect when using polynomial regression.

If the concentration of each congener in a sample does not fall within the range of its respective calibrants, prepare a serial sample dilution that will bring the concentration of the congener to

the midpoint of the calibration. Analyse the dilution and use the dilution factor to quantify the concentration of those congeners that were not within the calibration range in the original analysis. The dilution factor (D) can be calculated by dividing the final volume of the dilution by the volume of the aliquot, which is calculated according to Equation (5):

$$D = \frac{V_{\mathrm{f}}}{V_{\mathrm{a}}} \tag{5}$$

where

D is the dilution factor;

V_{f} is the final volume in ml;

V_{a} is the volume of the aliquot in ml.

Equation (4) does not give the final concentration as the volume of the organic solvent, the mass of the sample and the volume of the extract and any dilution factor shall be taken into account. A conversion factor (F) to convert the units from ng to µg is also necessary. The final concentration of each analyte or surrogate in the sample can be calculated by using Equation (6):

$$C_{\mathrm{final}} = \left(\frac{A}{A_{\mathrm{IS}}} - b \right) \times \frac{C_{\mathrm{IS}}}{a} \times \frac{V}{M} \times F \tag{6}$$

where

C_{final} is the concentration of PBB, PBDE, phthalate or surrogate per congener in the sample in µg/g;

V is the final extraction volume (25 ml);

a is the slope of the calibration curve;

b is the intercept on the y-axis of the calibration curve;

M is the mass of the sample in grams;

F is a conversion factor for ng to µg (1×10^{-3}).

The calculation example (Equation (6)) is for linear regression calibration only. A separate calculation is required if polynomial regression calibration is utilized.

The total results are the sum of the concentration of each PBB (total PBBs) and the sum of the concentrations of each PBDE (total PBDEs).

The total PBDEs or the total PBBs can be calculated by summing the measured concentrations of all of the signals identified as a PBDE or PBB. The PBBs and the PBDEs that are included in the total results shall include all the signals with appropriate mass, retention time and ion ratios for a PBB or a PBDE. The PBBs and PBDEs included in the totals shall not be limited only to those used in the calibration solutions since most entities are interested in the concentration of the total PBBs and total PBDEs, not specific isomers.

The calibration solutions can be used to establish an average response factor for each degree of bromination within the PBDEs and PBBs. The average response factors can then be used in the calculation of the measured concentration of detected congeners in the samples that are not included in the calibration (e.g. tentatively identified compounds or "TICS", see also 8.3). Automatic integration of signals meeting the criteria for a PBB or a PBDE is a common function of software used in GC-MS trace analysis.

The PBDEs and phthalates isolated from the sample extraction (see 8.2.3) are quantified by adding the internal standard (CB209 and anthracene-d_{10}) (see 8.2.1 b)) to an extract aliquot, injecting the solution into the GC-MS, measuring the area of the analyte peak(s) and the area of the CB209 peak and calculating the concentration of the analyte according to Equations (4) and (6). Data on the surrogate standard (DBOFB) (see 8.2.1 a)) are used for quality control purposes (see 11.2 d)) and are not used in the calculation of the analyte concentration(s) in the sample.

10 Precision

When the values of two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, lie within the range of the mean values cited in Table 7 below, the absolute deviation between the two test results obtained will not exceed the repeatability limit r deduced by statistical analysis of the international interlaboratory study 12 (IIS12) results in more than 5 % of cases.

When the values of two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, lie within the range of the values cited in Table 7 below, the absolute deviation between the two results will not be greater than the reproducibility limit R by statistical analysis of the international interlaboratory study 12 (IIS12) results in more than 5 % of cases.

Table 7 – IIS12 repeatability and reproducibility

Analytes	v mg/kg	r mg/kg	R mg/kg
PBBs	85	18	34
PBDEs	125	27	63
DEHP	753	137	386
PBDEs	1 094	204	550
DIBP	909	108	362
DBP	934	109	410
BBP	880	161	377
DEHP	1 022	198	398
PBBs	2 326	248	749
PBDEs	1 773	227	663
DIBP	740	91	394
DBP	745	99	439
BBP	901	119	423
DEHP	860	138	362

Key

v = expected value in mg/kg

r = repeatability

R = reproducibility

NOTE See Annex E for supporting data.

11 Quality assurance and control

11.1 Resolution

At least annually (or any time instrumental parameters are changed), a 5 µg/ml solution of technical deca-BDE (with BDE-209 ~ 96,9 % and BDE-206 ~ 1,5 %) with internal standard shall be analysed to confirm that the GC-MS system and parameters are suitable for the accurate determination of nona-BDE in the presence of BDE-209 and to demonstrate that congener degradation is not occurring. After the concentration (in µg/ml) of BDE-206 and BDE-209 measured in the injection solution is measured, the 206/(206 + 209) per cent ratio ("PR – 206") is calculated as shown below.

$$PR = \frac{C_A}{C_A + C_B} \times 100 \quad (7)$$

where

PR is the per cent ratio, "PR-206";

C_A is the measured concentration of BDE-206 in µg/ml;

C_B is the measured concentration of BDE-209 in µg/ml.

Table 8 gives an example calculation.

Table 8 – Example calculation

BDE congener	Theoretical injection concentration µg/ml	Measured concentration µg/ml	PR-206 %
BDE-209	4,845	5,200	(0,107/5,307) × 100 = 2,01
BDE-206	0,076	0,107	
Total		5,307	

A calculated PR-206 in the injection < 4,0 is acceptable and samples can be tested. A calculated PR-206 > 4,0 is unacceptable and samples shall not be tested until this condition is corrected. Effective corrections include replacement of the injection liner, reduction of the injection temperature, reduction of oven temperature or times, etc. New limits of detection (LOD) studies are required if the instrumental parameters are changed.

11.2 Performance

The following steps are taken for the quality control:

a) At least one reagent blank shall be extracted with each sequence of samples. The reagent blank comprises only solvent taken through the entire extraction procedure according to 8.2.2. The concentration of any analyte compounds found in the method blank shall be less than the method detection limits (see 11.3) for each compound.

b) At least one sample per sequence or one every ten samples, depending on the sample load, shall be spiked with 10 µg of each analyte in the matrix spiking solution (see 8.2.1 f)). The following formula shall be used for calculation:

$$R = \frac{C_m - C}{C_s} \times 100 \quad (8)$$

where

R is the recovery of each analyte in %;

C_m is the concentration of each analyte in the matrix spike in ng/ml;

C is the concentration of each analyte in the original sample in ng/ml;

C_s is the concentration of analyte spike solution in ng/ml.

Table 9 shows the recovery results of IIS12. The per cent recovery for each analyte shall be between 80 % and 120 %. The per cent recovery for each matrix spike shall be recorded and tracked in a spreadsheet to determine possible matrix effects in the analysis.

Table 9 – IIS12 mean, recovery and relative standard deviation

Analytes	*m* mg/kg	*v* mg/kg	*m/v* %	*RSD* %
PBBs	82	85	97	14,7
PBDEs	131	125	104	16,6
DEHP	759	753	101	17,9
PBDEs	1 083	1 094	99	17,7
DIBP	837	909	92	15,2
DBP	861	934	92	16,7
BBP	870	880	99	15,2
DEHP	956	1 022	94	14,7
PBBs	2 367	2 326	102	18,0
PBDEs	1 749	1 773	99	17,1
DIBP	774	740	105	17,9
DBP	803	745	108	19,2
BBP	944	901	105	15,7
DEHP	880	860	102	14,5

Key

m = general mean of the test property in mg/kg

v = expected value in mg/kg

m/v = recovery in %

RSD = relative standard deviation of the IIS12 results

NOTE See Annex E for supporting data.

c) After every tenth sample run and at the end of each sample set, analyse a continuing calibration check standard (CCC). A CCC is an unextracted mid-range calibrant that is analysed as a sample. The per cent recovery for each analyte shall be between 80 % and 120 %. If the per cent recovery for any analyte in the CCC standard falls outside of this range, the CCC standard should be reinjected within 12 h. If the recovery is still out of range after re-injection of the CCC standard, the analysis is stopped and maintenance shall be performed on the system to return it to optimal operating conditions. All samples injected before the last successful CCC standard can be reported. But samples between the last successful and failing CCC standard and all samples after the failing CCC standard shall be re-analysed with a new calibration.

d) The surrogate recovery shall be monitored for each sample. Per cent (%) surrogate recovery shall be calculated by the following formula:

$$SR = \frac{ms}{25\,\mu g} \times 100 \quad (9)$$

where

SR is the surrogate recovery, as a percentage (%);

ms is the total mass (µg) of surrogate measured in the final sample solution.

Acceptable surrogate recovery shall be between 70 % and 130 %. If the surrogate recovery for any sample is outside of these limits, the sample shall be re-analysed. If, after re-analysis, the surrogate recovery is not within these limits, the sample shall be re-extracted and re-analysed.

e) From the results of the five calibrants (see Table 5), calculate the average response (peak area) for the internal standard. The internal standard (IS) response for each sample shall be monitored throughout the analysis and compared to the average. If, at any point in the analysis, the IS response fluctuates below 50 % or above 150 % of the average, the sample is deemed out of control and shall be re-analysed. If the IS response is still out of range, check the results of the duplicate extract. If both are out of range and biased in the same direction, report data as suspect due to matrix effects.

f) A solvent blank run between each injection is recommended in order to be certain that there is no analyte carry-over from sample to sample. This is particularly important when samples containing high levels of analyte or potentially interfering brominated flame retardants or phthalates are analysed. When no check has been carried out to examine that the instrument is free of contamination, it can lead to falsely interpretation of the results. The solvent can contain a small amount of silylating agent (e.g. BSA, BSTFA) to maintain the inertness of the injector liner.

g) The retention time of analytes having an identification mass corresponding to BDE-209 and BDE-206 shall be within ±20 s of the BDE-209 and BDE-206 standards used in the calibration solutions and the corresponding retention time deviation between BDE-209 and BDE-206 shall be less than 130 % of the deviation between BDE-209 and BDE-206 standards used in the calibration solutions in order to be confirmed as being BDE-209 or BDE-206. Peaks eluting outside this range cannot be identified as BDE-209 or BDE-206. (Samples containing deca-BDE will have BDE-206 as the dominant nona-BDE.) For the other analytes, the retention time of analytes having an identification mass corresponding to each phthalate shall be within ±1 % of standards used in the calibration solutions. The use of retention times as a confirmation criterion is a widely accepted practice.

11.3 Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ)

A limit of detection (LOD) or method detection limit (MDL) study shall be completed before conducting testing and each time there is a significant change in the method or instrument type. The LOD or MDL is most appropriately determined experimentally by performing replicate, independent measurements on low-level or fortified sample matrices (e.g. plastic) carried out through the entire test procedure, including extraction. A minimum of six replicates and analyte concentrations of three to five times the estimated LOD or MDL shall be performed for this analysis. The complete LOD or MDL for an entire test procedure is determined by multiplying the standard deviation of the replicates by an appropriate factor. IUPAC recommends a factor of 3 for a minimum of six replicates, whilst EPA utilizes a one-sided confidence interval with the multiplier equal to Student's t value chosen for the number of replicates and the level of confidence (e.g. t = 3,36 for six replicates for 99 % confidence).

a) Mill approximately 2 g of suitable polymer from a pure source known not to contain the analytes or other compounds that can interfere with the analysis.

b) Weigh out 100 mg of the milled polymer and place it in a new extraction tube (centrifuge tube). Repeat this step six more times.

c) Spike the tube with 5 µg of each calibration analyte approximating the concentration of the lowest concentration calibrant.

d) Place the centrifuge tube in the ultrasonic bath apparatus.

e) Use the procedure (extraction according to 8.2.2) to extract each of the samples. Analyse accordingly.

f) The per cent recovery of each analyte shall be between 80 % and 120 %. If the recovery is above or below these limits, the analysis shall be repeated. If the recovery is outside of these limits a second time, the entire extraction and analysis procedure shall be repeated.

g) Each analyte shall have a calculated LOD or MDL of less than or equal to 100 mg/kg. If the calculated LOD or MDL for any of the analytes is above these limits, the procedure, extraction and analysis shall be repeated for that/those analyte(s).

h) The limits of quantification (LOQ) for each analyte shall be, at a minimum, three times the respective LOD or MDL. Unlike the LOD or MDL, which relates to detection only, the limit of quantification (LOQ) is a concentration that can be accurately quantified for a given compound.

NOTE If the required LOD or MDL cannot be met, a concentration step can be added to the extraction procedure. Since the concentration step will also increase the resin concentration (matrix) in the extract, a clean-up step can also be used for each sample.

12 Test report

For the purposes of this document, IEC 62321-1:2013, 4.8 (Test report) applies in addition to the following:

- identification of technical mixtures (if any) used for calibration.

Annex A
(informative)

Example of extraction efficiency in different extractants

The following Figure A.1 shows the extraction efficiency using detection response of the analytes in toluene, terahydrofuran, acetic ether and acetone/n-hexane (1:1, v/v), respectively. The four extractants get similar responses (extraction efficiencies), while acetone/n-hexane (1:1, v/v) gets the highest response and lowest standard deviation for deca-brominated diphenyl ether.

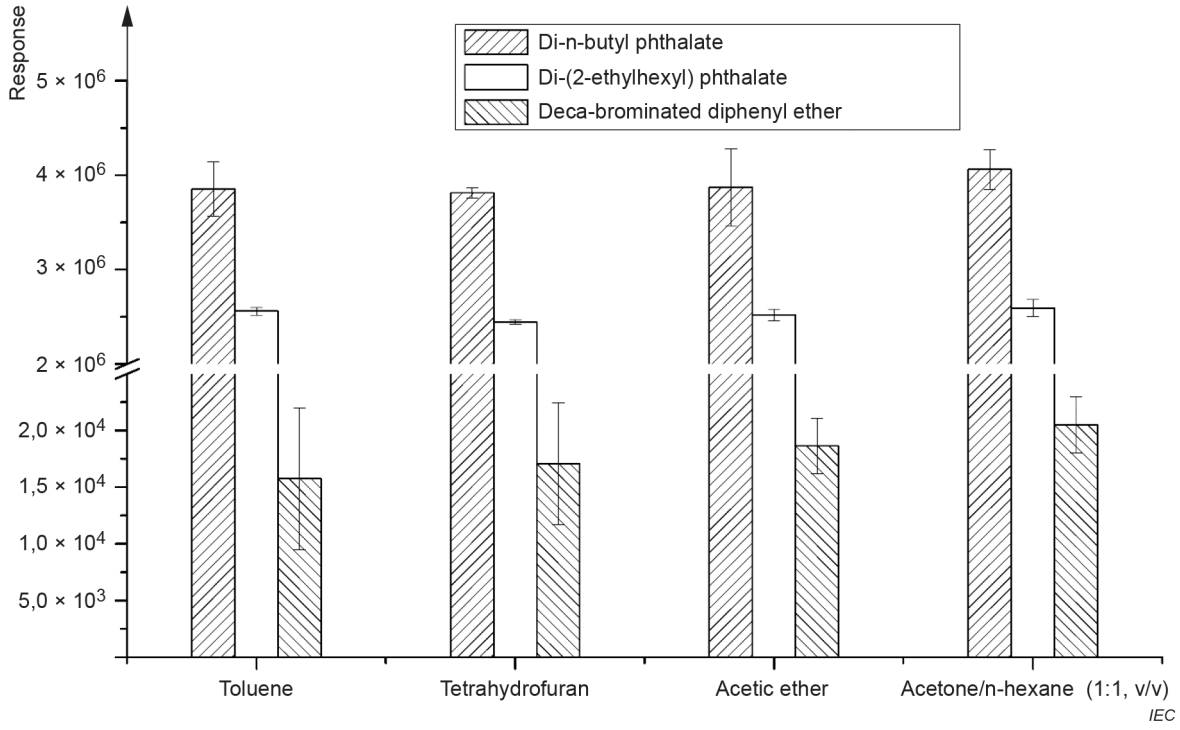

Figure A.1 – Extraction efficiency using detection response of the analytes in different extractants

Annex B
(informative)

Example of extraction efficiency in different cycles

The following Table B.1 shows extraction efficiency of the analytes in different extraction cycles using ultrasonic-assisted extraction. The extracted amount in three cycles can be > 99,5 %.

Table B.1 – Extraction efficiency of the analytes in different cycles

Extraction cycle	Extracted amount %, n = 3		
	Di-n-butyl phthalate	Di-(2-ethylhexyl) phthalate	Deca-brominated diphenyl ether
1st cycle	85,3	86,3	77,6
2nd cycle	12,6	12,1	22,4
3rd cycle	1,8	1,5	0
4th cycle	0,3	0,2	0
5th cycle	0	0	0

Annex C
(informative)

Examples of chromatograms

The following chromatograms (see Figure C.1 and Figure C.2) were obtained by GC-MS analysis using the parameters described in 8.3.

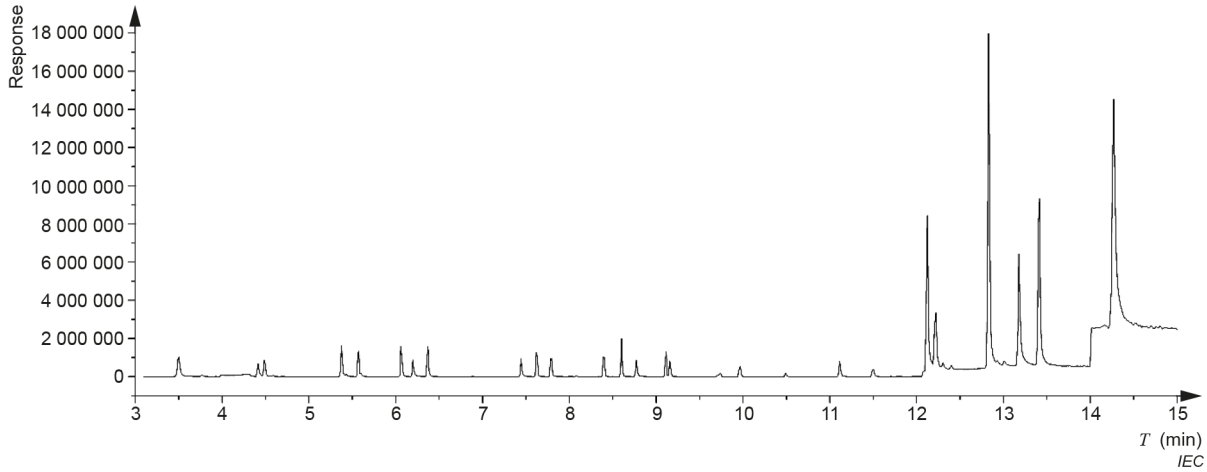

Figure C.1 – Total ion current chromatogram of each analyte (1,5 µg/ml, 1 µl, splitless)

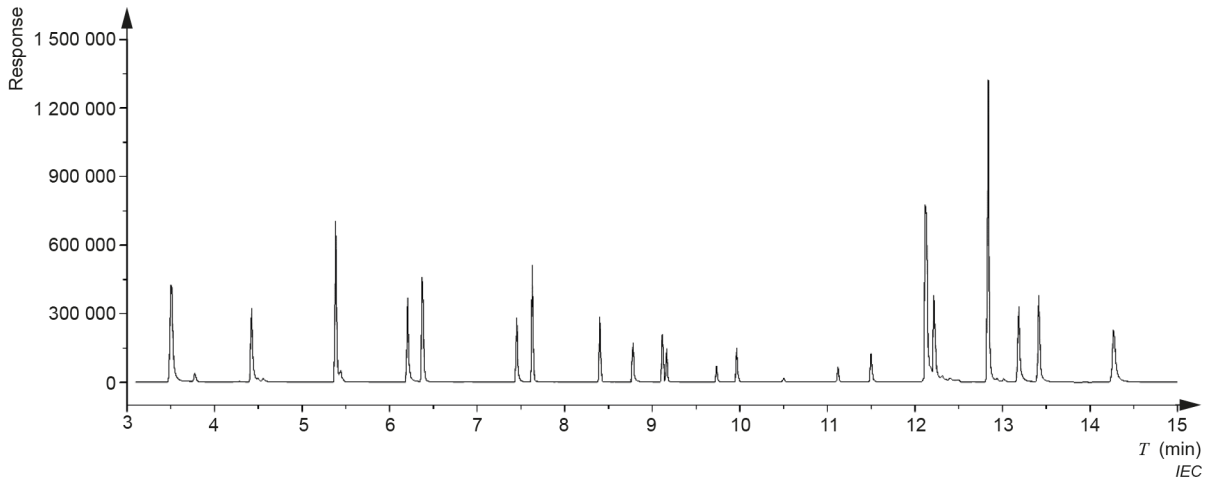

Figure C.2 – SIM ion chromatogram of PBBs, PBDEs and phthalate (1,5 µg/ml, 1 µl, splitless)

Annex D
(informative)

Examples of mass spectrograms

The following mass spectrograms (see Figure D.1 to Figure D.24) of polybrominated biphenyl (PBB), polybrominated diphenyl ether (PBDE) and phthalates were obtained by GC-MS analysis using the parameters described in 8.3.

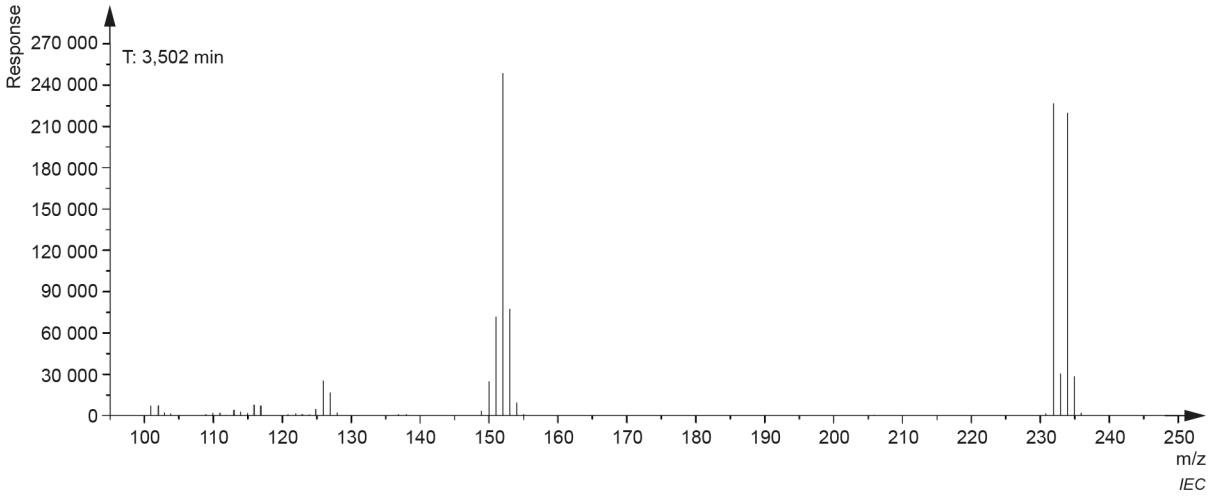

Figure D.1 – 2-Bromobiphenyl (Mono-BB)

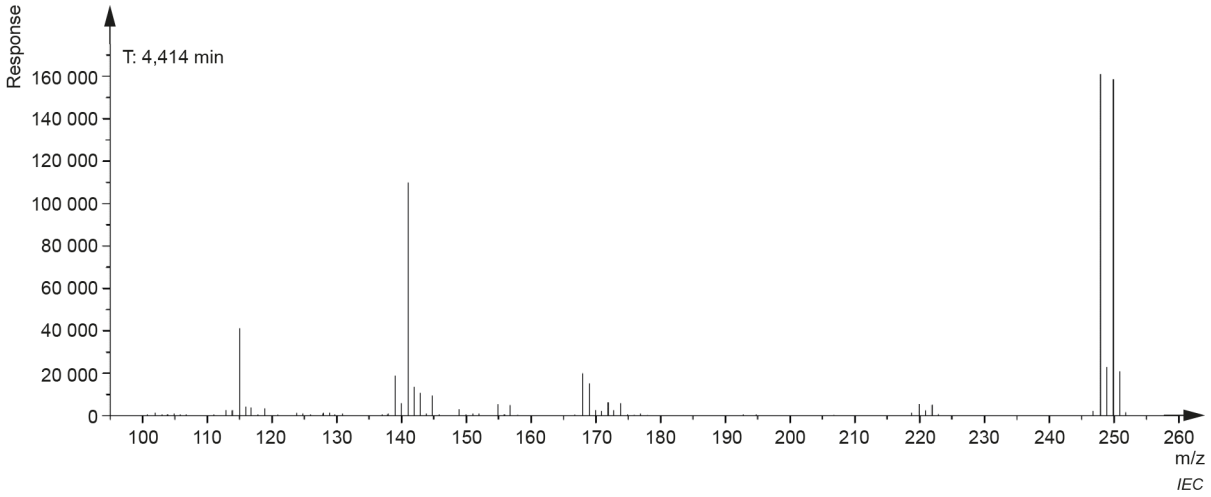

Figure D.2 – 4-Bromodiphenyl ether (Mono-BDE)

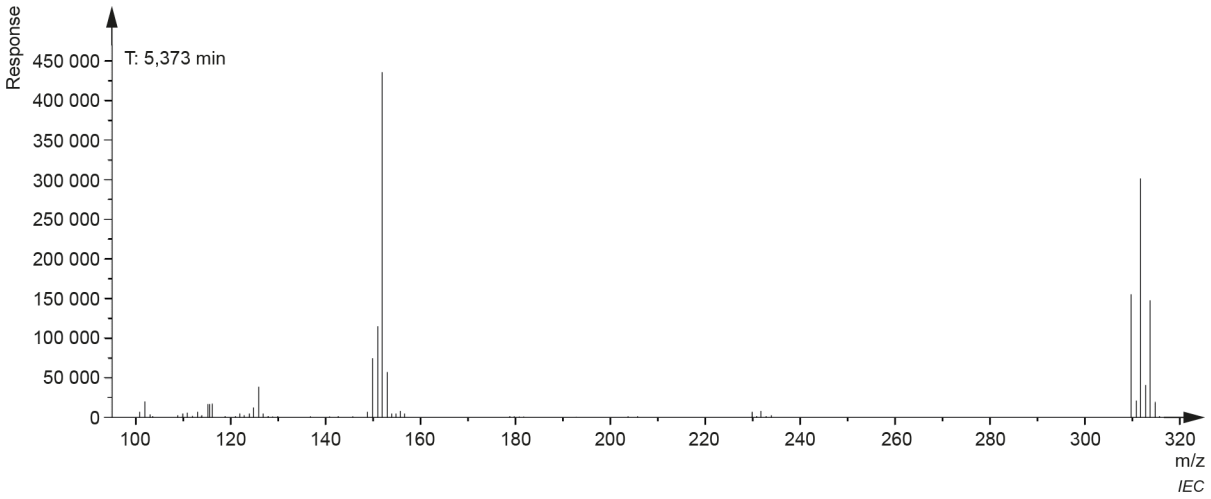

Figure D.3 – 2,5-Dibromobiphenyl (Di-BB)

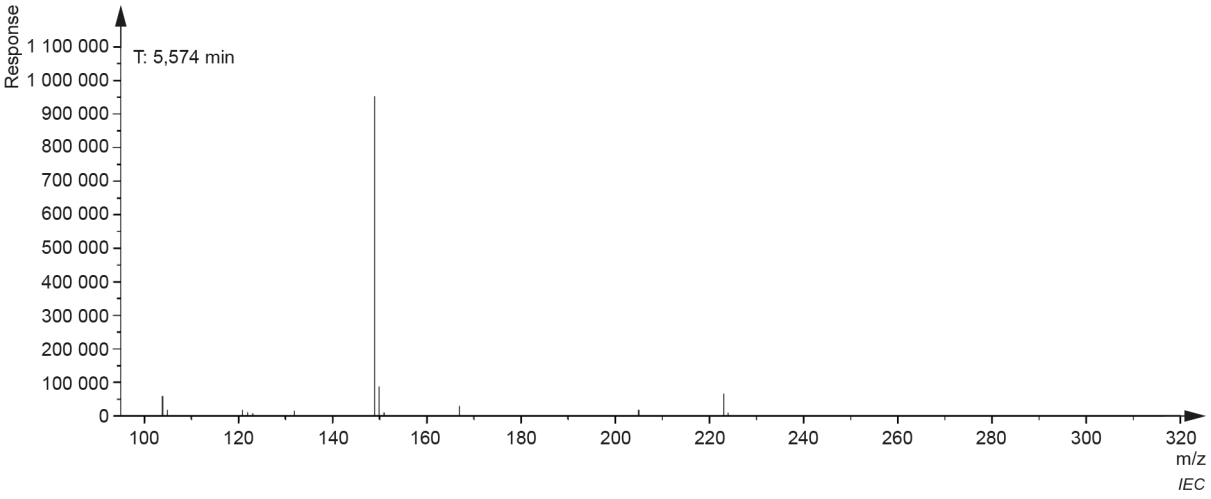

Figure D.4 – Di-isobutyl phthalate (DIBP)

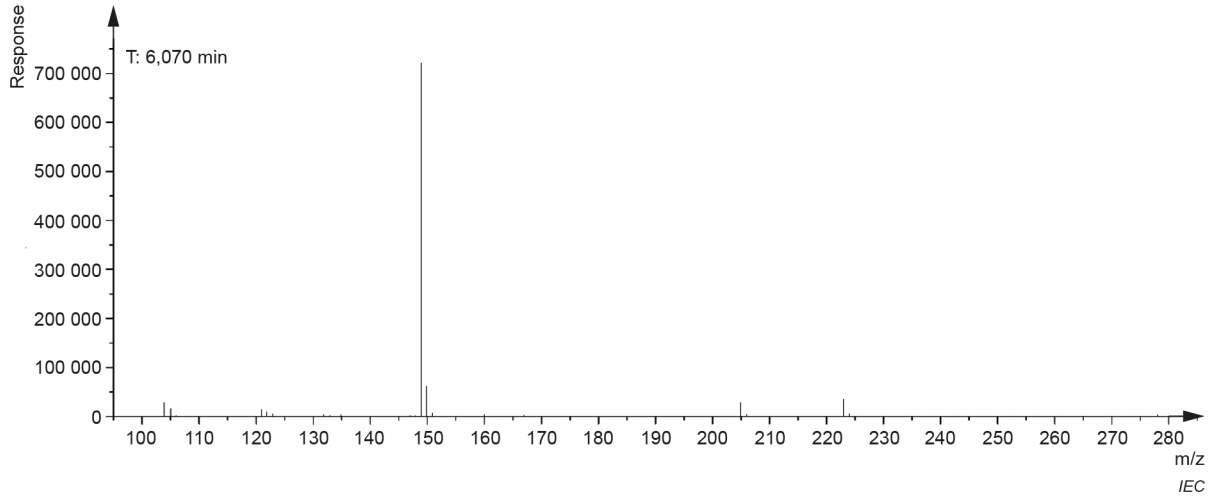

Figure D.5 – Di-n-butyl phthalate (DBP)

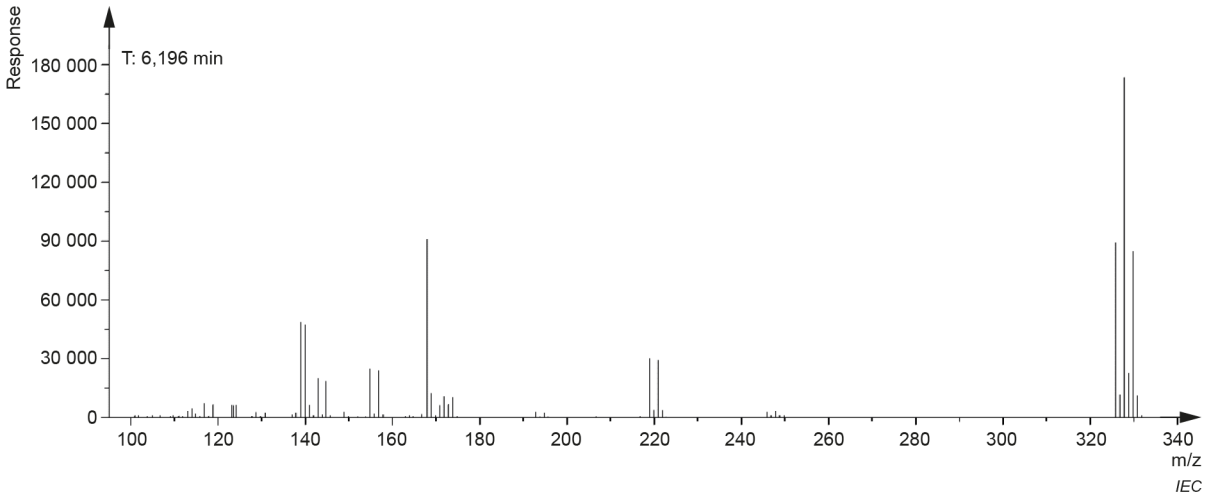

Figure D.6 – 4,4'-Dibromobiphenyl ether (Di-BDE)

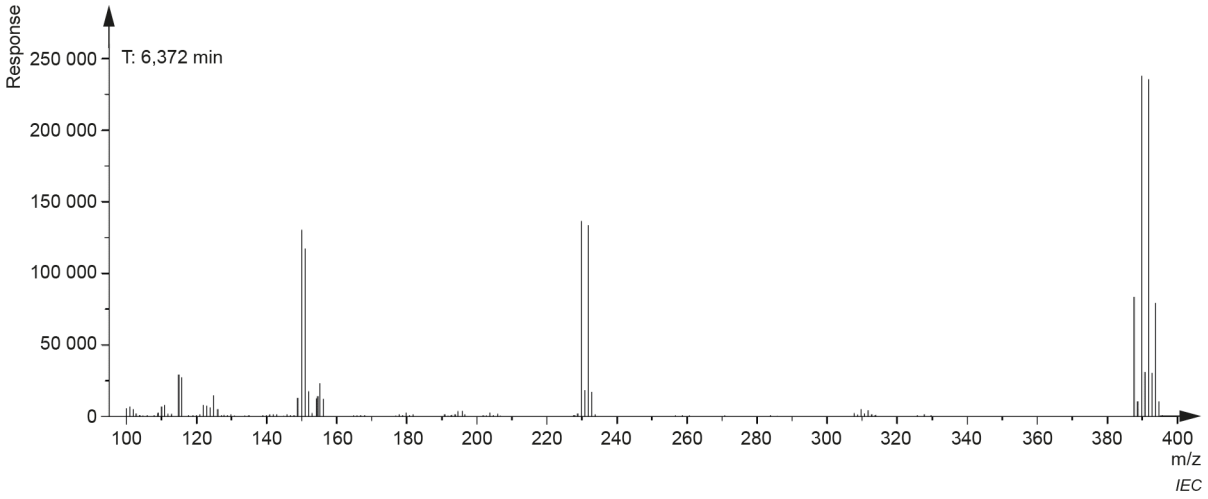

Figure D.7 – 2,4,6-Tribromobiphenyl (Tri-BB)

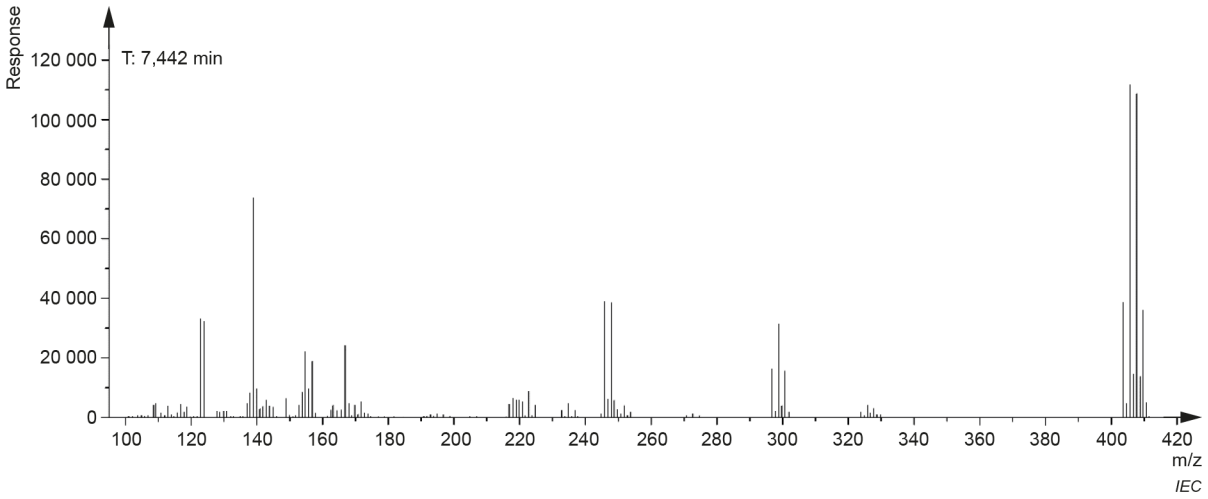

Figure D.8 – 3,3',4-Tribromobiphenyl ether (Tri-BDE)

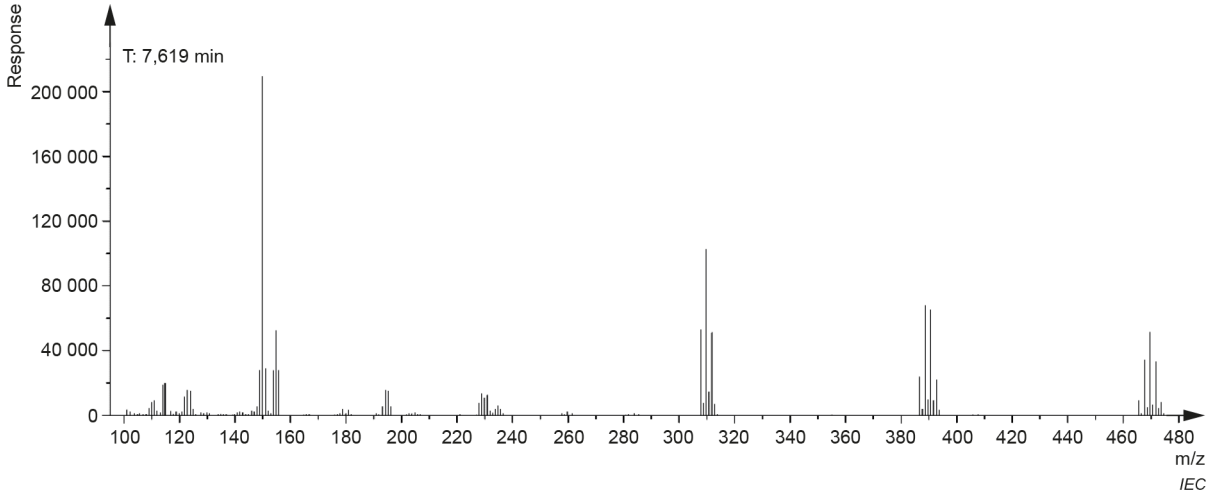

Figure D.9 – 2,2’,5,5’-Tetrabromobiphenyl (Tetra-BB)

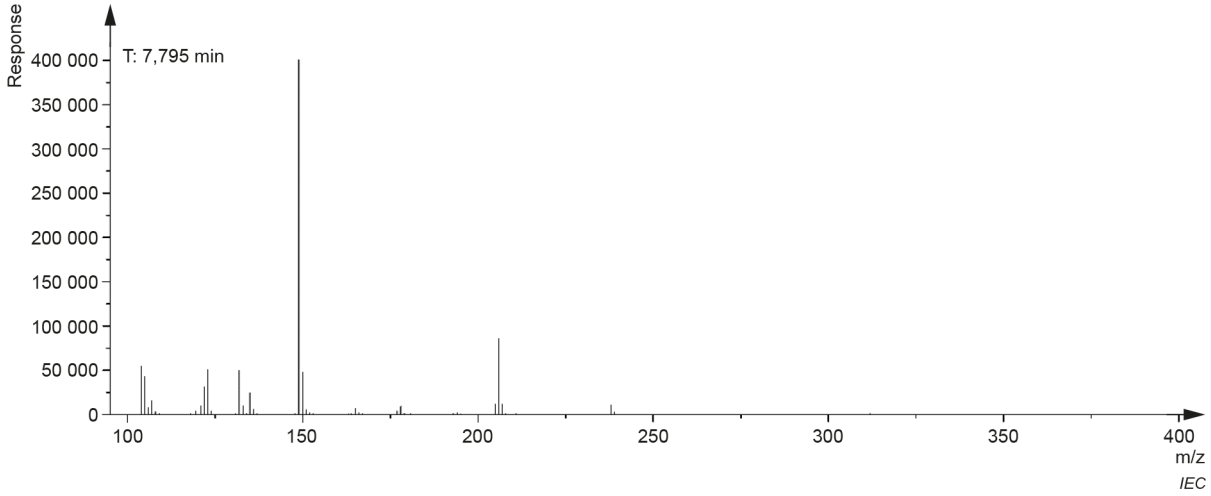

Figure D.10 – Butyl benzyl phthalate (BBP)

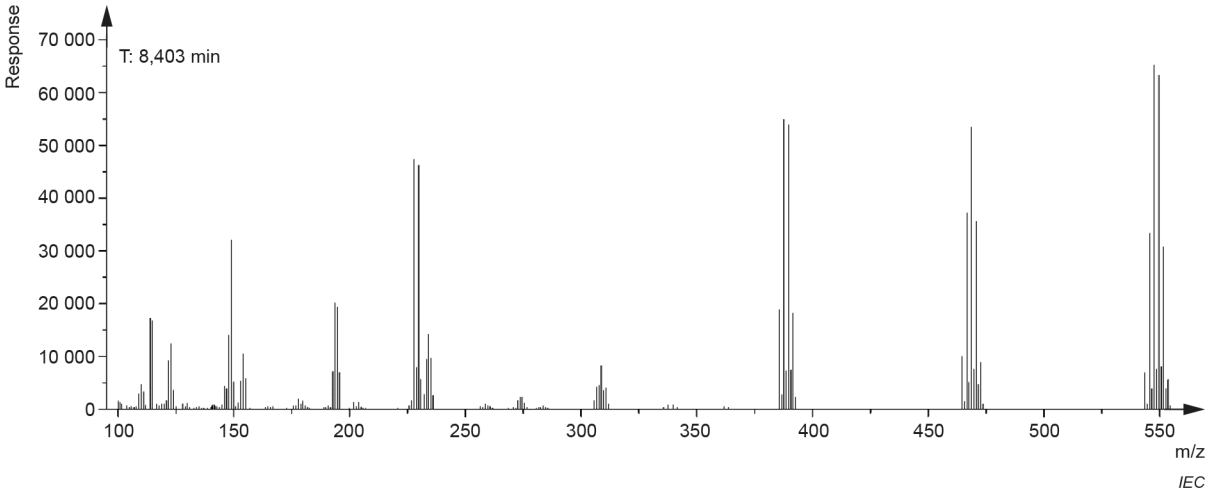

Figure D.11 – 2,2’,4,5’,6-Pentabromobiphenyl (Penta-BB)

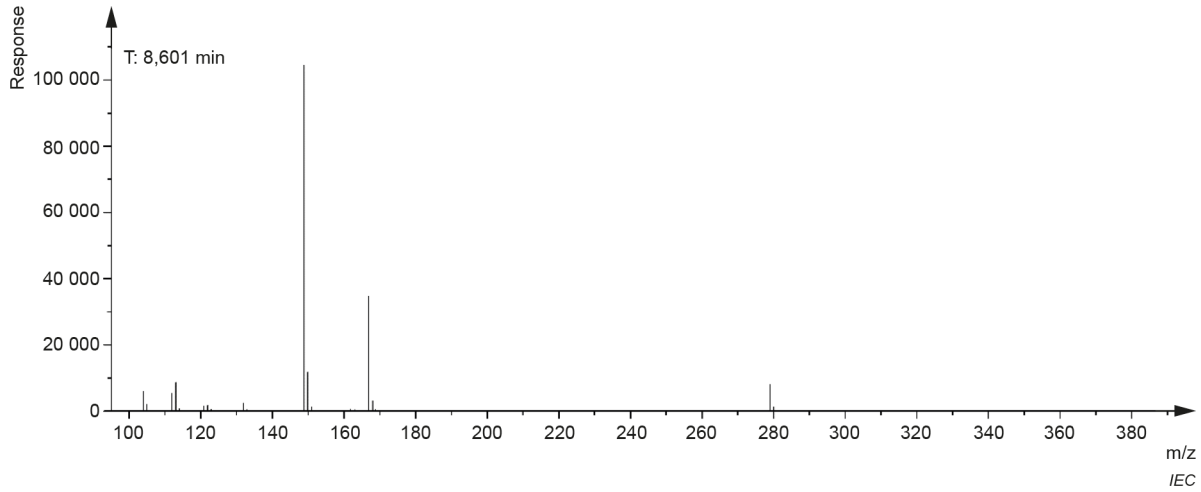

Figure D.12 – Di-(2-ethylhexyl) phthalate (DEHP)

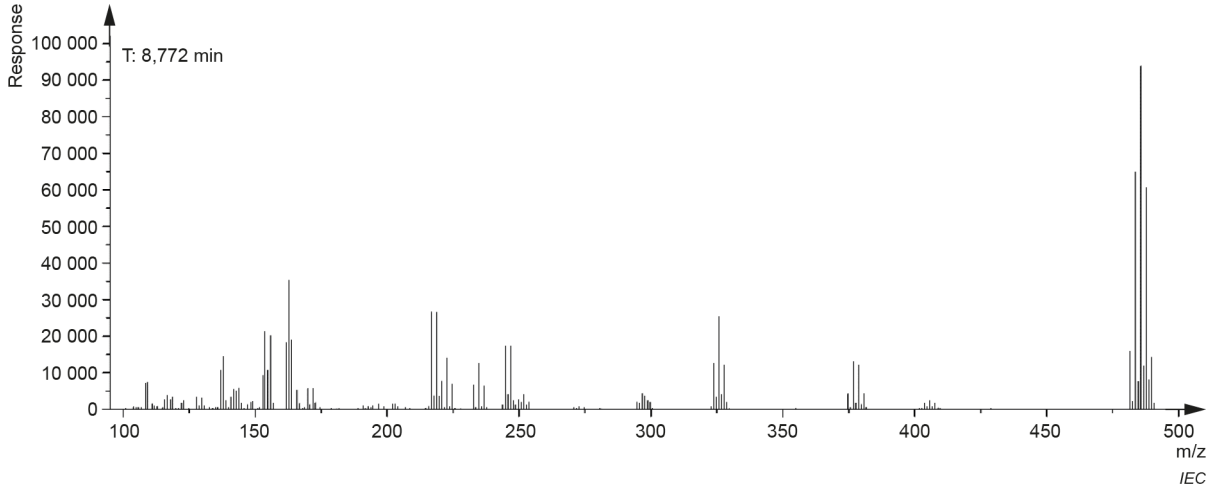

Figure D.13 – 3,3’,4,4’-Tetrabromobiphenyl ether (Tetra-BDE)

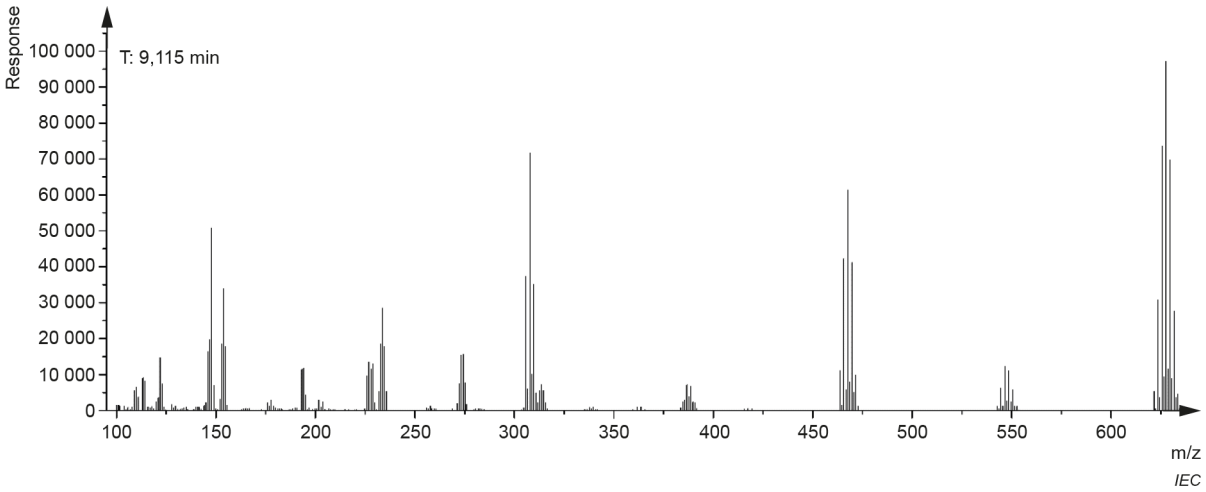

Figure D.14 – 2,2’,4,4’,6,6’-Hexabromobiphenyl (Hexa-BB)

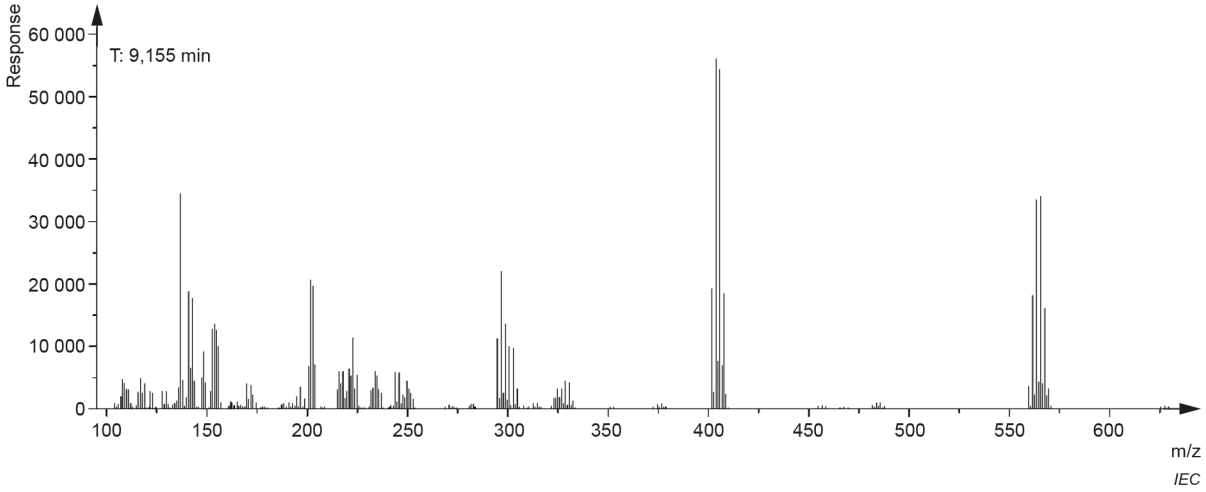

Figure D.15 – 2,2’,4,4’,6-Pentabromobiphenyl ether (Penta-BDE)

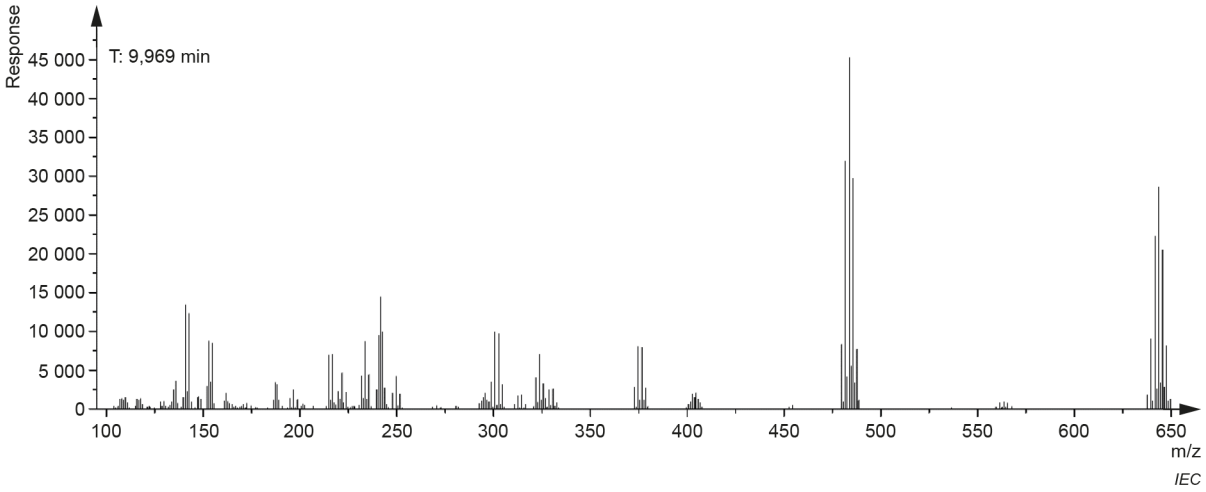

Figure D.16 – 2,2’,4,4’,5,6’-Hexabromodiphenyl ether (Hexa-BDE)

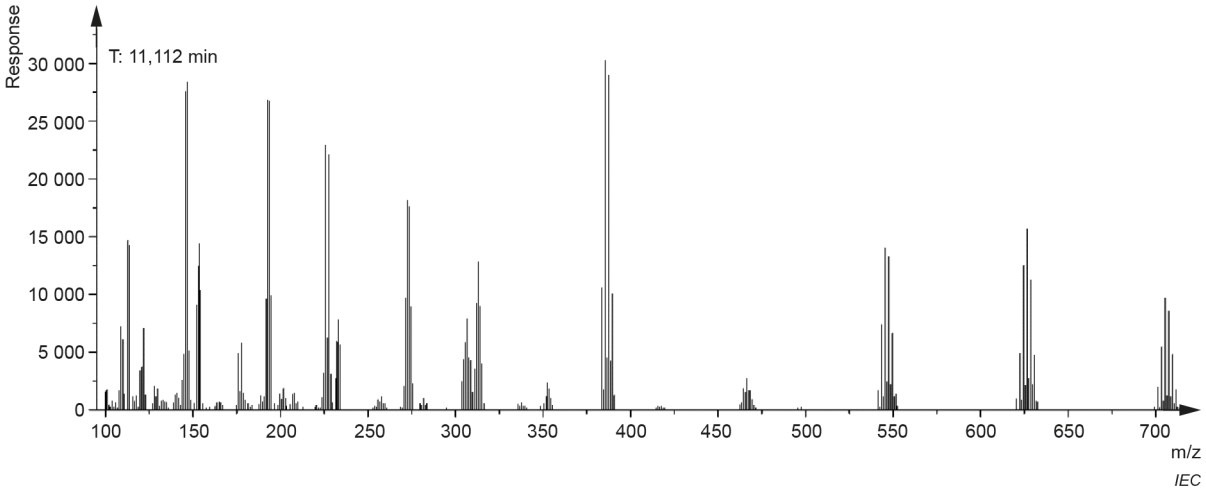

Figure D.17 – 2,2’,3,4,4’,5,5’-Heptabromobiphenyl (Hepta-BB)

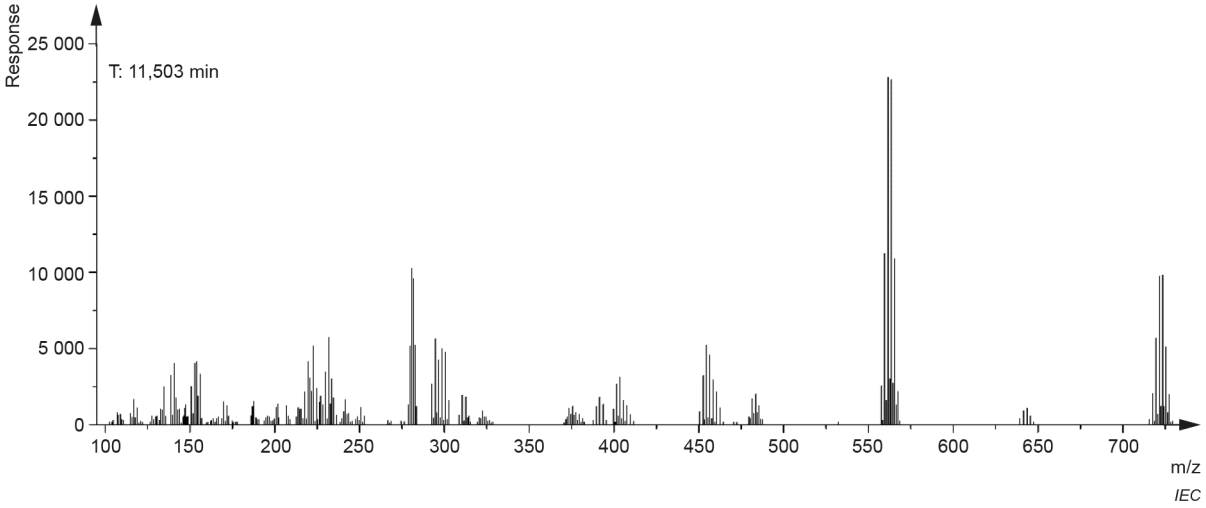

Figure D.18 – 2,2’,3,4,4’,5,6-Heptabromodiphenyl ether (Hepta-BDE)

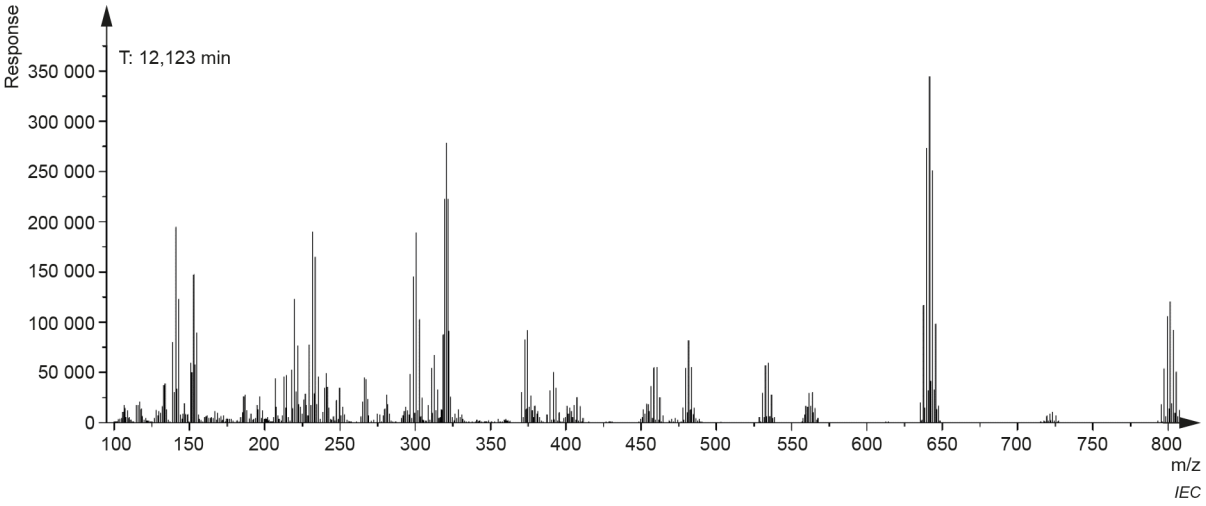

Figure D.19 – 2,2’,3,4,4’,5,5’,6’-Octabromodiphenyl ether (Octa-BDE)

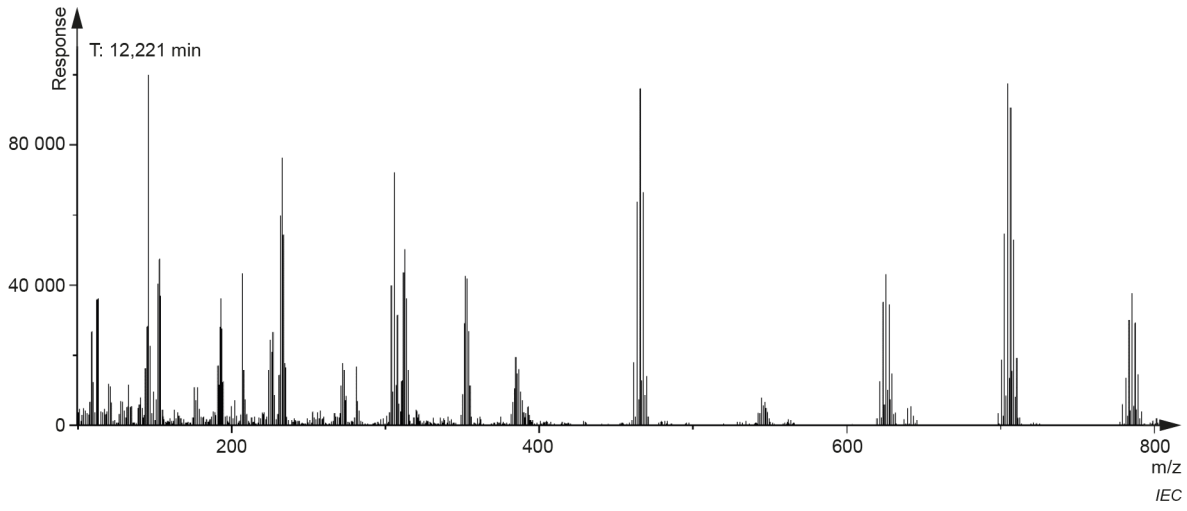

Figure D.20 – Octabromobiphenyl, technology (hepta + octa + nona) (Octa-BB)

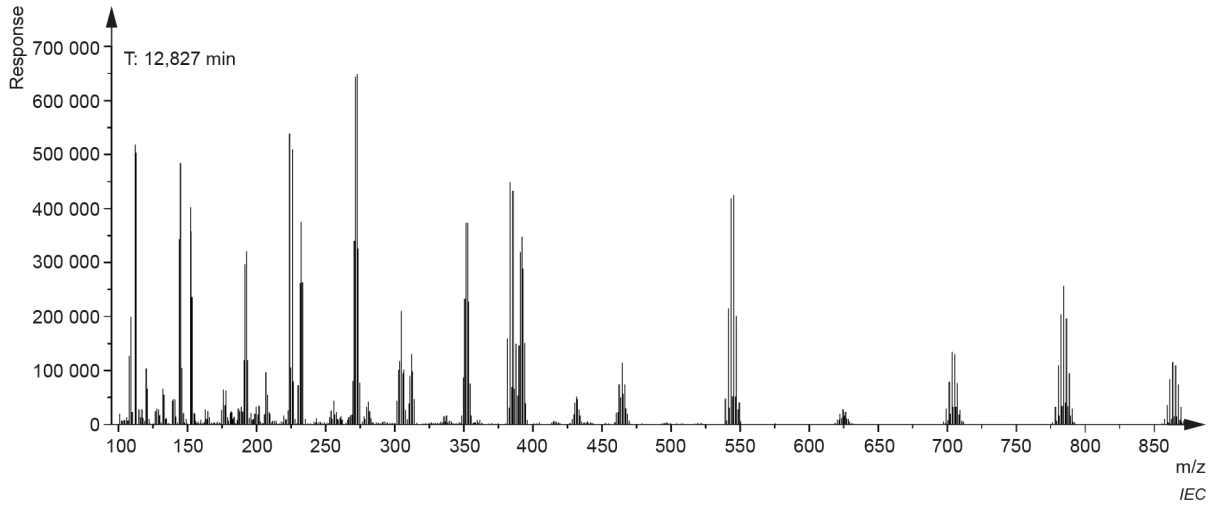

Figure D.21 – 2,2’,3,3’,4,4’,5,5’,6-Nonabromobiphenyl (Nona-BB)

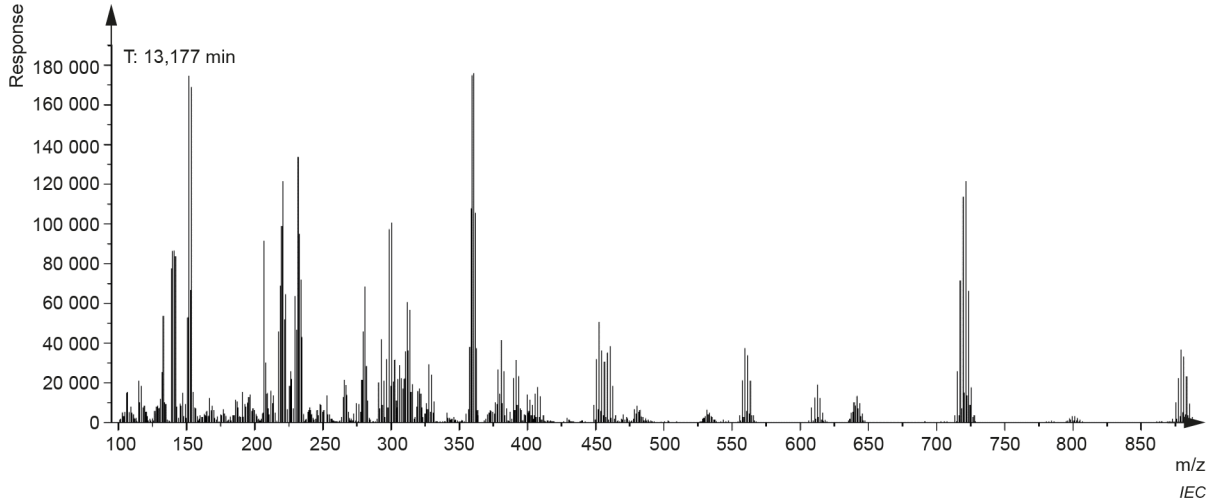

Figure D.22 – 2,2’,3,3’,4,4’,5,5’,6-Nonabromodiphenyl ether (Nona-BDE)

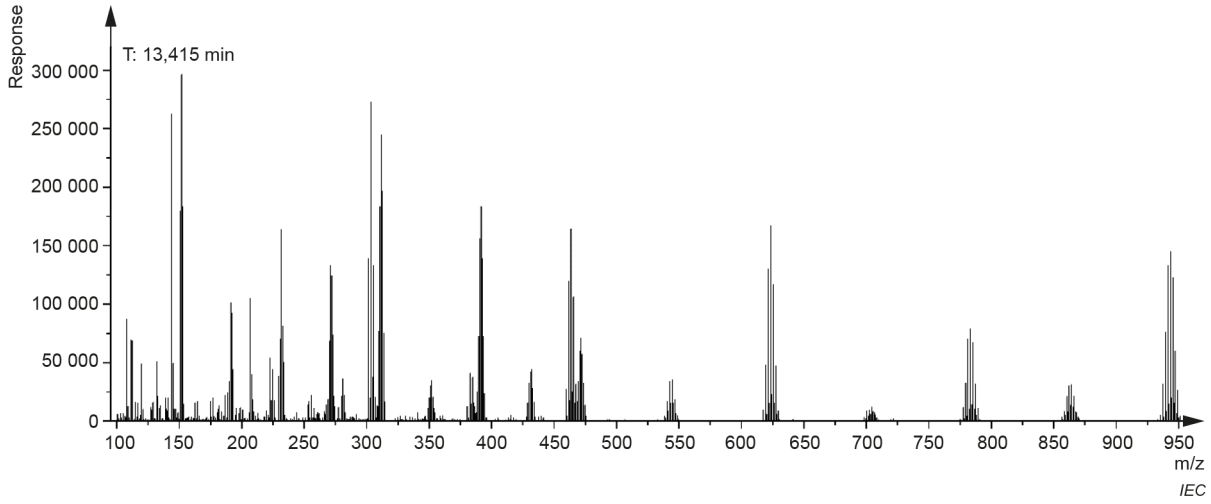

Figure D.23 – Decabromobiphenyl (Deca-BB)

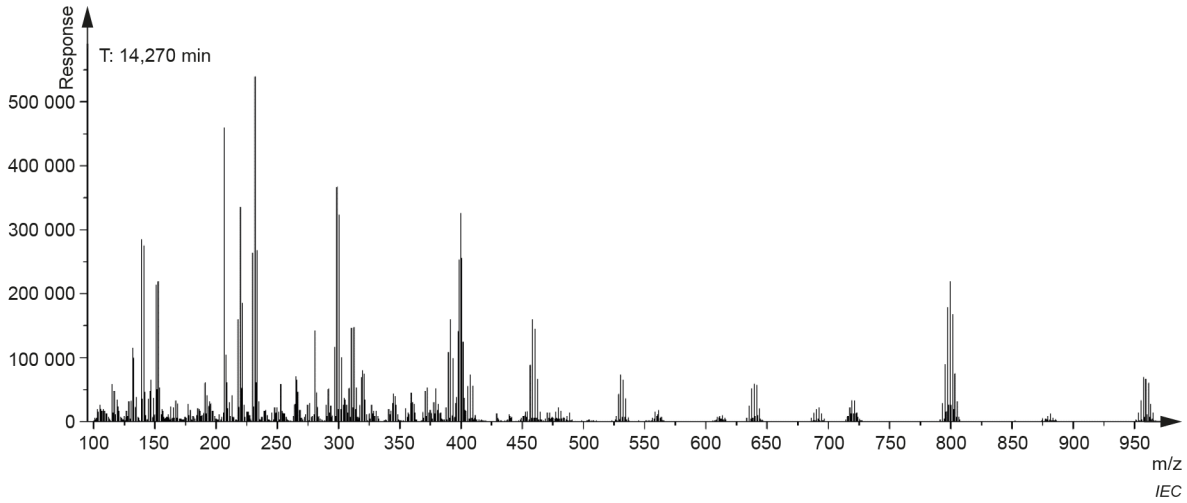

Figure D.24 – Decabromodiphenyl ether (Deca-BDE)

Annex E
(informative)

Statistics results of the international interlaboratory study 12 (IIS12)

Sample	Analytes	m (mg/kg)	v (mg/kg)	m/v (%)	RSD (%)	n	$s(r)$ (mg/kg)	r (mg/kg)	$s(R)$ (mg/kg)	R (mg/kg)	p	Outlier labs
IIS12-S01	PBBs	82	85	97	14,7	40	6	18	12	34	13	3
IIS12-S01	PBDEs	131	125	104	16,6	31	10	27	22	63	10	6
IIS12-S02	DEHP	759	753	101	17,9	71	49	137	138	386	22	0
IIS12-S03	PBDEs	1 083	1 094	99	17,7	44	73	204	197	550	14	4
IIS12-S04	DIBP	837	909	92	15,2	60	38	108	129	362	19	3
IIS12-S04	DBP	861	934	92	16,7	60	39	109	147	410	19	3
IIS12-S04	BBP	870	880	99	15,2	58	58	161	135	377	18	4
IIS12-S04	DEHP	956	1 022	94	14,7	61	71	198	142	398	19	3
IIS12-S05	PBBs	2 367	2 326	102	18,0	42	89	248	268	749	15	3
IIS12-S05	PBDEs	1 749	1 773	99	17,1	45	81	227	237	663	16	2
IIS12-S05	DIBP	774	740	105	17,9	65	33	91	141	394	20	2
IIS12-S05	DBP	803	745	108	19,2	65	35	99	157	439	20	2
IIS12-S05	BBP	944	901	105	15,7	62	42	119	151	423	19	3
IIS12-S05	DEHP	880	860	102	14,5	68	49	138	129	362	21	1

Key

m = general mean of the test property in mg/kg

v = expected value in mg/kg

m/v = recovery in %

RSD = relative standard deviation of the results taken into calculation

n = number of test results taken into calculation

$s(r)$ = repeatability standard deviation

r = repeatability

$s(R)$ = reproducibility standard deviation

R = reproducibility

p = number of labs taken into calculation

Bibliography

IEC 62321-6:2015, *Determination of certain substances in electrotechnical products – Part 6: Polybrominated biphenyls and polybrominated diphenyl ethers in polymers by gas chromatograhy-mass spectometry (GC-MS)*

IEC 62321-8:2017, *Determination of certain substances in electrotechnical products – Part 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py-TD-GC-MS)*

IEC GUIDE 108, *Guidelines for ensuring the coherence of IEC publications – Horizontal functions, horizontal publications and their application*

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(*Continued from second cover*)

Only the English language text has been retained while adopting it in this Indian Standard, and as such, the page numbers given here are not the same as in the IEC publication.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding of numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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