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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

Détermination de certaines substances dans les produits électrotechniques – Partie 12: Détermination simultanée – Biphényles polybromés, diphényléthers polybromés et phtalates dans les polymères par chromatographie en phase gazeuse-spectrométrie de masse

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The text of this International Standard is based on the following documents:

Draft	Report on voting
111/689/FDIS	111/696/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

A list of all parts in the IEC 62321 series, published under the general title *Determination of certain substances in electrotechnical products*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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.

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.

[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.