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Determination of certain substances in electrotechnical products – Part 3-3: Screening – Polybrominated biphenyls, polybrominated diphenyl ethers and phthalates in polymers by gas chromatography-mass spectrometry using a pyrolyser/thermal desorption accessory (Py/TD-GC-MS)

Détermination de certaines substances dans les produits électrotechniques – Partie 3-3: Détection – Diphényles polybromés, diphényléthers polybromés et phtalates dans les polymères par chromatographie en phase gazeuse-spectrométrie de masse par pyrolyse/thermodésorption (Py/TD-GC-MS)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

FDIS	Report on voting
111/626/FDIS	111/632/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- withdrawn,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 all over the world, this has resulted in the adaptation of regulations affecting wastes, substances and energy use of electrotechnical products.

The use of polybrominated biphenyls (PBBs), polybrominated diphenyl ethers (PBDEs) and certain phthalates in electrotechnical products is of concern in many regions of the world.

The purpose of this document is therefore to provide a test method that will allow the electrotechnical industry to determine the levels of polybrominated biphenyls (PBBs), polybrominated diphenyl ethers (PBDEs), di-isobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzylbutyl phthalate (BBP), di-(2-ethylhexyl) phthalate (DEHP), di-n-octyl phthalate (DNOP), di-isononyl phthalate (DINP) and di-isodecyl phthalate (DIDP) in electrotechnical products on a consistent global basis.

WARNING – Persons using this document should be familiar with normal laboratory practice. 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 3-3: Screening – Polybrominated biphenyls, polybrominated diphenyl ethers and phthalates in polymers by gas chromatography-mass spectrometry using a pyrolyser/thermal desorption accessory (Py/TD-GC-MS)

1 Scope

This part of IEC 62321 specifies the screening analysis of polybrominated biphenyls (PBBs), polybrominated diphenyl ethers (PBDEs), di-isobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzylbutyl phthalate (BBP), di-(2-ethylhexyl) phthalate (DEHP), di-n-octyl phthalate (DNOP), di-isononyl phthalate (DINP), and di-isodecyl phthalate (DIDP) in polymers of electrotechnical products using the analytical technique of gas chromatography-mass spectrometry using a pyrolyser/thermal desorption accessory (Py/TD-GC-MS).

This test method has been evaluated through the analysis of PP (polypropylene), PS (polystyrene), and PVC (polyvinyl chloride) materials containing deca-BDE between 100 mg/kg and 1 000 mg/kg and individual phthalates between 100 mg/kg to 4 000 mg/kg as depicted in Annex J. Use of the methods described in this document for other polymer types, PBBs (mono-deca), PBDEs (mono-deca) and phthalates or concentration ranges other than those specified above has not been specifically evaluated.

This document has the status of a horizontal standard in accordance with IEC Guide 108 [1][1].

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

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

[1] Numbers in square brackets refer to the bibliography.

3.1.1
reference material
material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

[SOURCE: IEC 62321-1:2013, 3.1.7 [2]]

3.1.2
screening
analytical procedure to determine the presence or absence of substances in the representative part or section of a product, relative to the value or values chosen as the criterion for presence, absence or further testing

Note 1 to entry: If the screening method produces values that are not conclusive, then additional analysis or other follow-up actions may be necessary to make a final presence/absence decision.

[SOURCE: IEC 62321-1:2013, 3.1.10]

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 with respect to analyte(s) concentration(s) or mass

3.1.4
response factor
RF
ratio between the mass of the compound being analysed and the peak area of that compound in Equation (1)

$$\mathrm{RF} = A \,/\, m \tag{1}$$

where

RF is the response factor;

A is the peak area of a compound;

m is the mass of a compound

3.1.5
relative response factor
RRF
ratio between the RFs of two compounds – compound A and compound B – in Equation (2)

$$\mathrm{RRF_{A/B}} = \mathrm{RF_A} \,/\, \mathrm{RF_B} \tag{2}$$

where

$\mathrm{RRF_{A/B}}$ is the relative response factor of compound A to compound B;

$\mathrm{RF_A}$ is the response factor of compound A;

$\mathrm{RF_B}$ is the response factor of compound B

3.1.6
substitute compound
compound used to calculate RRFs of each analyte

Note 1 to entry: More than one compound can be selected as a substitute compound. The RRF of the analyte is the ratio of the RF of the analyte to this compound. In Equation (3), compound B corresponds to this. The role is the same as internal standards to correct the response factor. However, this is not included in test samples and is analysed before analysis of test samples. From the RF of the substitute compound and the RRF of the analyte, the RF of each analyte is calculated.

$$RF_A = RRF_{A/B} \times RF_B \quad (3)$$

where

RF_A is the response factor of compound A;

RF_B is the response factor of compound B; substitute compound

3.2 Abbreviated terms

BB-003	4-bromobiphenyl
BB-015	4,4'-dibromobiphenyl
BB-029	2,4,5-tribromobiphenyl
BB-049	2,2',4,5'-tetrabromobiphenyl
BB-103	2,2',4,5',6-pentabromobiphenyl
BB-153	2,2',4,4',5,5'-hexabromobiphenyl
BB-189	2,3,3',4,4',5,5'-heptabromobiphenyl
BB-194	2,2',3,3',4,4',5,5'-octabromobiphenyl
BB-206	2,2',3,3',4,4',5,5',6- nonabromobiphenyl
BB-209	decabromo biphenyl
BBP	benzyl butyl phthalate
BDE-003	4-bromodiphenyl ether
BDE-015	4,4'-dibromodiphenyl ether
BDE-028	2,4,4'-tribromodihenyl ether
BDE-047	2,2',4,4'-tetrabromodiphenyl ether
BDE-099	2,2',4,4',5-pentabromodiphenyl ether
BDE-153	2,2',4,4',5,5'-hexabromodiphenyl ether
BDE-183	2,2',3,4,4',5',6-heptabromodiphenyl ether
BDE-203	2,2',3,4,4',5,5',6-octabromodiphenyl ether
BDE-206	2,2',3,3',4,4',5,5',6-nonabromodiphenyl ether
BDE-209	decabromodiphenyl ether
CRM	certified reference material
DBP	di-n-butyl phthalate
DEHP	di-(2-ethylhexyl) phthalate
DIBP	di-isobutyl phthalate
DIDP	di-isodecyl phthalate
DINP	di-isononyl phthalate
DNOP	di-n-octyl phthalate
EGA	evolved gas analysis
EI	electron ionization

GC	gas chromatography
LOQ	limit of quantification
MDL	method detection limit
MS	mass spectrometry
PBB	polybrominated biphenyl
PBDE	polybrominated diphenyl ether
PBMS	performance-based measurement system
PE	polyethylene
PP	polypropylene
PS	polystyrene
PVC	polyvinyl chloride
Py/TD-GC-MS	gas chromatography-mass spectrometry using a pyrolyser/thermal desorption accessory
QC	quality control
RF	response factor
RRF	relative response factor
SIM	selected ion monitoring
THF	tetrahydrofuran

4 Principle

4.1 Overview

The concept of 'screening' has been developed to reduce the amount of testing. Executed as a predecessor to any other test analysis, the main objective of screening is to quickly determine whether the screened part or section of a product:

- contains a certain substance at a concentration significantly higher than its value or values chosen as criterion, and therefore may be deemed unacceptable;
- contains a certain substance at a concentration significantly lower than its value or values chosen as criterion, and therefore may be deemed acceptable;
- contains a certain substance at a concentration so close to the value or values chosen as criterion that when all possible errors of measurement and safety factors are considered, no conclusive decision can be made about the acceptable absence or presence of a certain substance and, therefore, a follow-up action may be required, including further analysis using verification testing procedures.

This test method is designed specifically to screen for PBBs, PBDEs, DIBP, DBP, BBP, DEHP, DNOP, DINP, and DIDP in polymers in electrotechnical products by using the analytical technique of Py/TD-GC-MS. Annex A provides a flow chart as an example of how this method can be used for screening.

4.2 Principle of test

Py/TD-GC-MS uses gas chromatography-mass spectrometry coupled with a pyrolyser/thermal desorption accessory (see Annex B, Figure B.1) to screen the presence of PBBs, PBDEs, DIBP, DBP, BBP, DEHP, DNOP, DINP and DIDP in polymers in electrotechnical products. Since Py/TD-GC-MS does not require any prolonged solvent extraction process, a fast screening of PBBs, PBDEs and phthalates is available. The polymer sample is directly introduced into the pyrolyser, which thermally extracts PBBs, PBDEs and phthalates from a polymer under a specific heat zone. Thermally desorbed PBBs, PBDEs and phthalates are then transferred to the gas chromatograph. PBBs, PBDEs and phthalates are separated by a gas chromatographic capillary column and detected by a mass spectrometer. The respective PBBs, PBDEs and phthalates are identified based on the retention times, m/z (quantification and confirmation ions), and ion ratio as a result of standard specimen analysis. The selected ion monitoring (SIM) mode is used as the measuring mode of MS to improve detection limits. Calculation of the PBBs, PBDEs and phthalates concentration in the original sample is achieved by using response factors (RFs) and relative response factors (RRFs) normalized by RF of the substitute compound. RRFs can be continuously used by verifying the accuracy. Moreover, when the accuracy satisfies the recovery rate between 70 % and 130 % by the test in 11.2.5, the RRFs which are determined by a different Py/TD-GC-MS system can be applied.

NOTE 1 Additionally, scan measurement of MS is suitable to check negative matrix interference from other additives in the polymer. Negative matrix interference causes ion suppression which provides lower concentration results. Scan/SIM measurement (simultaneous measurements) is also applicable.

NOTE 2 Annex C provides the potential alternative test methods for the screening.

NOTE 3 Since IEC 62321 (all parts) employs PBMS, test methods that provide equivalent performance criteria required in this document do not prevent its use.

5 Reagents and materials

All reagent chemicals shall be tested for contamination and blank values prior to application as follows.

When measuring PBBs and/or PBDEs, low degradable materials (such as PP and PS) shall be used as a standard sample for the determination of response factors, refer to Annex D:

NOTE 1 Deca-BB and deca-BDE are known to become highly degradable in some types of polymers.

a) blank polymer material from a pure source which does not contain the specific analytes and other compounds that may interfere with the analysis by peak overlapping or ion suppression: refer to Annex D;

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

NOTE 2 The nitrogen gas can be used if it is confirmed that the required performance is satisfied.

c) calibrants: refer to Annex D;

d) polymer reference materials: One contains approximately 100 mg/kg of analytes (PBBs, PBDEs and/or phthalates) and the other 1 000 mg/kg;

The following reagent chemicals, when used for preparing the polymer sample, shall be similarly tested as the above:

e) toluene for preparing the PBBs, PBDEs and phthalates standard solution, GC grade or higher;

f) THF for preparing the polymer solution, GC grade or higher.

6 Apparatus

The following items shall be used for the analysis:

a) analytical balance capable of measuring accurately to 0,000 01 g (0,01 mg);
b) deactivated glass wool;
c) deactivated sample cup; if a sample cup is re-used, analyte carry-over shall be confirmed by analysing without a sample;

NOTE 1 Before a sample cup is re-used, it is baked out to prevent cross contamination. However, if a re-used sample cup causes certain PBDE and PBB decomposition, the sample cup is not re-used.

d) gas chromatograph – mass spectrometer equipped with a pyrolyser/thermal desorption accessory, a split/splitless inlet and a programmable temperature controlled oven. The mass spectrometer shall be able to perform selected ion monitoring (SIM) and a total ion monitoring ("full scan");

NOTE 2 An auto-sampler is used to ensure repeatability.

e) pyrolyser/thermal desorption accessory;
f) capillary column;

The following items should be used for sample preparation as necessary:

g) cryogenic grinding mill with liquid nitrogen cooling;
h) polymer sample preparation tools to cut or file polymer such as nipper, micro spatula, tweezers, cutter, file, and micro puncher;
i) micro syringe or automated pipettes;
j) glass equipment; made from brown or amber glass for long-term storage of PBDEs.

NOTE 3 To avoid decomposition and/or debromination of PBDEs by UV light during long-term sample storage, glass equipment made from brown or amber glass is used.

7 Sampling

The sample can either be cut into small pieces using a cutter or filed off.

8 Procedure

8.1 General instructions for the analysis

The validation of the instrumentation should include testing of potential cross contaminations between sequential samples. Additional blanks or an inverted sequence of testing will help to identify cross contaminations.

The following general instructions should be followed:

a) After analysis of test samples with high analyte concentration, blank samples should be analysed until the background level of PBBs, PBDEs and phthalates is decreased to a value equivalent to 100 mg/kg or even lower.

NOTE A blank polymer material or blank sample cup is used for blank-sample analysis.

b) In order to reduce blank values, ensure the cleanliness of all tools used in the sample preparation.

8.2 Sample preparation

8.2.1 General

The sample preparation requires clean labware (e.g. cutter, tweezers) to avoid cross contamination.

NOTE If the distribution of the analyte is not uniform and a sample mass of 0,5 mg is not guaranteed to represent the degree of concentration, the sample is taken from several locations and mixed well using a cryogenic mill or completely dissolved using an optimal solvent such as THF. Refer to IEC 62321-2 [3].

8.2.2 Polymer sample

a) Place approximately 0,5 mg of the cut or powdered sample into a pre-weighed sample cup using a micro spatula or tweezers.

b) Record the total weight of the cup with the sample in it to the nearest 0,01 mg and record the sample weight by subtracting the weight of the sample cup from the total weight.

c) Place an appropriate amount of deactivated glass wool into the sample cup to ensure that the sample powder will not spill out.

NOTE Refer to Annex E.

8.2.3 Stock solution or polymer reference material

The PS solution and standard mixture solution are available for the calibration and sensitivity check. When polymer reference materials are available, they shall be used for the calibration and sensitivity check. A thinly stretched polymer reference sheet or film would also be available (see Annex D).

NOTE 1 The following solution concentrations and solvent types are examples and can be changed.

a) PS solution: 50 mg/ml in THF solvent;

NOTE 2 The other type of base polymer is available if it is dissolved completely by a suitable solvent.

NOTE 3 When measuring PBBs and/or PBDEs, deca-BDE is known to become highly degradable in some types of polymers and a PS solution is used as a standard sample for the determination of response factors; refer Annex D.

b) PBB solution: 50 µg/ml mono to deca-BB in toluene;

c) PBDE solution: 50 µg/ml mono to deca-BDE in toluene;

d) phthalate solution: 100 µg/ml in organic solvent such as hexane or toluene; the phthalate solution should contain all phthalates necessary for analysis of DIBP, DBP, BBP, DEHP, DNOP, DINP and DIDP;

e) stock solution of substitute compound such as DEHP: 100 µg/ml in organics solvent such as hexane or toluene.

NOTE 4 When more than one compound is used as a substitute compound, each stock solution or mixture solution is prepared.

8.3 Instrumental parameters

Different conditions may be necessary to optimize a specific Py/TD-GC-MS system to achieve effective separation of each substance and meet the quality control (QC) and method detection limits (MDL) requirements. The following parameters have been found suitable and are provided as an example (see Table 1). The total ion current chromatogram and mass chromatogram is shown in Annex G (see Figure G.1).