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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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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

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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- withdrawn,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.