SLOVENSKI STANDARD
oSIST prEN IEC 62321-12:2022

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Določevanje posameznih snovi v elektrotehničnih izdelkih - 12. del: Sočasno določevanje polibromiranih bifenilov, polibromiranih difenil etrov in ftalatov v polimerih s plinsko kromatografijo-masno spektrometrijo

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

Ta slovenski standard je istoveten z: prEN IEC 62321-12:2022

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COMMITTEE DRAFT FOR VOTE (CDV)

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IEC TC 111 : ENVIRONMENTAL STANDARDIZATION FOR ELECTRICAL AND ELECTRONIC PRODUCTS AND SYSTEMS	
SECRETARIAT: Italy	SECRETARY: Mr Marco Iadevaia
OF INTEREST TO THE FOLLOWING COMMITTEES:	PROPOSED HORIZONTAL STANDARD: ☒ Other TC/SCs are requested to indicate their interest, if any, in this CDV to the secretary.
FUNCTIONS CONCERNED: ☐ EMC ☒ ENVIRONMENT ☐ QUALITY ASSURANCE ☐ SAFETY	
☒ SUBMITTED FOR CENELEC PARALLEL VOTING	☐ NOT SUBMITTED FOR CENELEC PARALLEL VOTING
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TITLE:

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

PROPOSED STABILITY DATE: 2027

NOTE FROM TC/SC OFFICERS:

This CDV was submitted based on the agreement of the 33rd WG3 meeting in September 2021.

INTERNATIONAL ELECTROTECHNICAL COMMISSION

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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:

FDIS	Report on voting
XX/XX/FDIS	XX/XX/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://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.

The National Committees are requested to note that for this document the stability date is 20XX.

THIS TEXT IS INCLUDED FOR THE INFORMATION OF THE NATIONAL COMMITTEES AND WILL BE DELETED AT THE PUBLICATION STAGE.

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 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 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 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 one normative extraction and measurement technique for 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.

Ultrasonic-assisted extraction is described for simultaneous extraction for sample preparation in this document.

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

This test method has been evaluated by the test of PP, PVC, ABS, ACM, PS, PU and PE materials.

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, *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 terminological 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

Use the same analysis and detection procedure to determine different classes of analytes. The same analysis and detection procedure includes (but not limited to): pretreatment, extraction, cleaning-up and detection.

[Defined by referring to ISO 21470: 2020]

3.1.2 Ultrasonic-assisted extraction

An extraction technique using ultrasonic waves to accelerate the speed of extracting substances in the sample matrix (the extractant does not dissolve the sample matrix) so as to improve the extraction efficiency, e.g., in an ultrasonic bath.

[Defined by referring to Method EPA 3550C: 2007]

3.1.3 Calibrant

Calibration standard

Substance in solid, liquid or gas 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]

3.2 Abbreviated terms

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
PTFE	Polytetrafluoroethylene
PTV	Programmed temperature vaporising
QC	Quality control
RSD	Relative standard deviation
SIM	Selected ion monitoring
TICS	Tentatively identified compounds

4. Principle

Different classes of analytes, ie., PBBs, PBDEs, BBP, DBP, DEHP, and DIBP, in polymers are simultaneously ultrasonic assisted extracted 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 may 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.);