
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

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31.020	Elektronske komponente na splošno	Electronic components in general
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111/645/CDV

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: <input checked="" type="checkbox"/> Other TC/SCs are requested to indicate their interest, if any, in this CDV to the secretary.
FUNCTIONS CONCERNED: <input type="checkbox"/> EMC <input checked="" type="checkbox"/> ENVIRONMENT <input type="checkbox"/> QUALITY ASSURANCE <input type="checkbox"/> SAFETY	
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<p>Attention IEC-CENELEC parallel voting</p> <p>The attention of IEC National Committees, members of CENELEC, is drawn to the fact that this Committee Draft for Vote (CDV) is submitted for parallel voting.</p> <p>The CENELEC members are invited to vote through the CENELEC online voting system.</p>	

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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.

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

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**Part 12: Simultaneous determination– polybrominated biphenyls,
polybrominated diphenyl ethers and phthalates in polymers by gas
chromatography-mass spectrometry**

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FOREWORD

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125 co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and
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154 International Standard IEC 62321-12 has been prepared by IEC technical committee 111:
155 Environmental standardization for electrical and electronic products and systems.

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

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

157

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

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

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

- 164 • reconfirmed,
165 • withdrawn,
166 • replaced by a revised edition, or
167 • amended.

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

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

172

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INTRODUCTION

174 The widespread use of electrotechnical products has drawn increased attention to their impact
175 on the environment. In many countries all over the world this has resulted in the adaptation of
176 regulations affecting wastes, substances and energy use of electrotechnical products.

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

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

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

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

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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

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* 2022

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]

229 **3.1.2 Ultrasonic-assisted extraction**

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

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

234 **3.1.3 Calibrant**

235 Calibration standard

236 Substance in solid, liquid or gas form with known and stable concentration(s) of the analyte(s)
237 of interest used to establish instrument response (calibration curve) with respect to analyte(s)
238 concentration(s).

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

240 **3.1.4 Technical mixture**

241 Commercial product manufactured for industrial use whose purity is not as clearly defined as
242 an individual high purity calibration standard.

243 [SOURCE: IEC 62321-6: 2015, 3.1.2]

244 **3.2 Abbreviated terms**

245 BBP Benzyl butyl phthalate

246 BDE Brominated diphenyl ether

247 BSA Bis(trimethylsilyl)acetamide

248 BSTFA N,O-Bis(trimethylsilyl)trifluoroacetamide

249 CCC Continuing calibration check standard

250 DBOFB (4, 4'-dibromooctafluorobiphenyl) (n)

251 DBP Di-n-butyl phthalate

252 Deca-BB Decabromobiphenyl

253 Deca-BDE Decabromodiphenyl ether

254 DEHP Di-(2-ethylhexyl) phthalate

255 DIBP Di-isobutyl phthalate

256 DMDCS Dimethyldichlorosilane

257 EI Electron ionization

258 EPA U.S. Environmental Protection Agency

259 GC-MS Gas chromatography-mass spectrometry

260 IS Internal standard

261 IUPAC International Union of Pure and Applied Chemistry

262 LOD Limit of detection

263 LOQ Limit of quantification

264 MDL Method detection limit

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265	PBB	Polybrominated biphenyl
266	PBDE	Polybrominated diphenyl ether
267	PTFE	Polytetrafluoroethylene
268	PTV	Programmed temperature vaporising
269	QC	Quality control
270	RSD	Relative standard deviation
271	SIM	Selected ion monitoring
272	TICS	Tentatively identified compounds

273 4. Principle

274 Different classes of analytes, ie., PBBs, PBDEs, BBP, DBP, DEHP, and DIBP, in polymers are
 275 simultaneously ultrasonic assisted extracted and determined qualitatively and quantitatively by
 276 gas chromatography-mass spectrometry (GC-MS) using full scan mode and (or) single (or
 277 “selected”) ion monitoring (SIM) mode.

278 5. Reagents and materials

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

- 281 a) n-hexane (GC grade or higher);
- 282 b) acetone (GC grade or higher);
- 283 c) acetone/n-hexane (1:1, v/v);
- 284 d) toluene (GC grade or higher);
- 285 e) helium (purity greater than a volume fraction of 99,999 %);
- 286 f) technical BDE-209 with BDE-209 ~ 96,9 % and BDE-206 ~ 1,5 % solution;
- 287 g) calibrants: refer to 8.4;
- 288 h) surrogate and internal standards

289 – surrogate standard used to monitor analyte recovery according to 8.2.1 a), 8.5.2 and 8.5.3,
 290 e.g. DBOFB (4, 4'-dibromooctafluorobiphenyl) (n), dibutyl phthalate-3,4,5,6-d₄ or di-(2-
 291 ethylhexyl) phthalate-3,4,5,6-d₄.

292 – internal standard used to correct for injection errors, according to 8.2.1 b), 8.2.3 and 8.5.4,
 293 e.g. anthracene-d₁₀ or CB209 (2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl).

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

298 6. Equipment, apparatus and tools

299 The following items shall be used for the analysis:

- 300 a) analytical balance capable of measuring accurately to 0,000 1 g;
- 301 b) 1 ml, 5 ml, 10 ml, 25 ml, 100 ml volumetric flasks;
- 302 c) ultrasonic bath (450 W, 40 kHz, Volume ~10 l, or equivalent.);