
Določevanje posameznih snovi v elektrotehničnih izdelkih - 11. del: Določevanje tris (2-kloroetil) fosfata (TCEP) v polimernih materialih s plinsko kromatografijo-masno spektrometrijo (GC-MS) in tekočinsko kromatografijo-masno spektrometrijo (LC-MS)

Determination of certain substances in electrotechnical products - Part 11: Tris (2-chloroethyl) phosphate (TCEP) in plastics by gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS)

Določevanje posameznih snovi v elektrotehničnih izdelkih - 11. del: Določevanje tris (2-kloroetil) fosfata (TCEP) v plastiki in polimernih materialih s plinsko kromatografijo-masno spektrometrijo (GC-MS) in tekočinsko kromatografijo-masno spektrometrijo (LC-MS)

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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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TITLE: Determination of certain substances in electrotechnical products - Part 11: Tris (2-chloroethyl) phosphate (TCEP) in plastics by gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS)
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PROPOSED STABILITY DATE: 2025

NOTE FROM TC/SC OFFICERS:

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**DETERMINATION OF CERTAIN SUBSTANCES
IN ELECTROTECHNICAL PRODUCTS –**

**Part 11: Tris(2-chloroethyl) phosphate (TCEP) in plastics by gas
chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass
spectrometry (LC-MS)**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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**DETERMINATION OF CERTAIN SUBSTANCES
IN ELECTROTECHNICAL PRODUCTS –**

**Part 11: Tris(2-chloroethyl) phosphate (TCEP) in plastics by gas
chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass
spectrometry (LC-MS)**

FOREWORD

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International Standard IEC 62321-11 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems.

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.

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

- 140 – reconfirmed,
- 141 – withdrawn,
- 142 – replaced by a revised edition, or
- 143 – amended.

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

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

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148 INTRODUCTION

149 The widespread use of electrotechnical products has drawn increased attention to their impact on the
150 environment. In many countries this has resulted in the adoption of regulations affecting wastes,
151 substances and energy use of electrotechnical products.

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

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

157 This first edition of IEC 62321-11 introduces a new subject covering Tris(2-chloroethyl) phosphate
158 (TCEP) in the IEC 62321 series.

159 TCEP is a halogenated phosphorus-based flame retardant that is disclosable as a Substance of Very
160 High Concern (SVHC) as it is classified as toxic to reproduction category 2 (R60) and was included in
161 the candidate list for authorisation on 13 January 2010, following ECHA's decision ED/68/2009 and in
162 regulation (EC) No 1907/2006 ANNEX XVI.

163 TCEP is used as a flame retardant in plastics such as polyester and polyurethane foam, and as a
164 plasticizer in Polyvinyl chloride. Additionally, TCEP is used as an alternative for brominated flame
165 retardants that have been restricted. No applicable testing standard exists for TCEP analysis in plastics.

166 As a result, analysis criteria have been established by IEC/TC 111/JWG 14 joint-development with
167 ISO/TC 61/SC 5 as ISO/IEC double logo International Standard; to provide a test method that will allow
168 industry to determine the levels of TCEP in plastics.

169 **WARNING — Persons using this International Standard should be familiar with normal**
170 **laboratory practice. This International Standard does not purport to address all of the safety**
171 **problems, if any, associated with its use. It is the responsibility of the user to establish**
172 **appropriate safety and health practices and to ensure compliance with any national regulatory**
173 **conditions.**

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 11: Tris(2-chloroethyl) phosphate (TCEP) in plastics by gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS)

1. Scope

This part of IEC 62321 specifies two different techniques for the determination of TCEP tris(2-chloroethyl) phosphate (TCEP) in plastics, the GC-MS or LC-MS method; both of which are suitable for quantitative analysis.

These two techniques have been evaluated for use with polyurethane, Polyvinyl chloride and polyethylene materials containing TCEP between 200 mg/kg to 2 000 mg/kg. Use of the methods described in International Standard for other polymers and concentration ranges has not been specifically evaluated.

These test methods do not apply to plastics materials having a processing temperature higher than 230 °C.

NOTE TCEP starts thermal decomposition at approximately 230 °C. Polymer types which have a processing temperature into shapes of plastics (e.g. pellets, moulded parts, or sheets etc.) not exceeding the decomposition temperature can contain TCEP.

Py-TD-GC-MS is another technique, suitable for the screening of TCEP in plastics (See Annex A).

2. Normative references (standards.iteh.ai)

The following referenced documents are indispensable for the application 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 62321-2:2013, *Determination of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*

ISO 472:2013, *Plastics – Vocabulary*

ISO 1043-1:2011, *Plastics – Symbols and abbreviated terms – Part 1: Basic polymers and their special characteristics*

ISO 1382:2012, *Rubber-Vocabulary*

3. Terms, definitions and abbreviations

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. 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

217 further testing.

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

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

221 3.1.2. Plastic, noun

222 Material which contains as an essential ingredient a high polymer and which, at some stage in its processing into
223 finished products, can be shaped by flow

224 Note 1 to entry Elastomeric materials, which are also shaped by flow, are not considered to be plastics.

225 Note 2 to entry In some countries, particularly the United Kingdom, the term "plastics" is used as the singular form as well as the plural form.

226 [SOURCE: ISO 472:2013, 2.702]

227 3.1.3. Polymer

228 substance composed of molecules characterized by the multiple repetition of one or more species of atoms or
229 groups of atoms (constitutional units) linked to each other in amounts sufficient to provide a set of properties that
230 do not vary markedly with the addition or removal of one or a few of the constitutional units. [SOURCE: ISO
231 1382:2012, 2.341]

232 3.2. Abbreviations

233 ACN Acetonitrile

234 API-ES Atmospheric pressure ionization – electrostatic

235 BSA *N,O*-Bis(trimethylsilyl)acetamide

236 BSTFA *N,O*-Bis(trimethylsilyl)trifluoroacetamide

237 CCC Continuing calibration check standard

238 CRM Certified reference material

239 D Dilution factor

240 DEHP Di-(2-ethylhexyl) phthalate

241 DIP Direct injection probe [oSIST prEN IEC 62321-11:2022](https://standards.iteh.ai/catalog/standards/sist/845cdf03-h50-4147-80c5-119d054f3feb/osist-pren-iec-62321-11-2022)

242 DMDCS Dimethyldichlorosilane [https://standards.iteh.ai/catalog/standards/sist/845cdf03-](https://standards.iteh.ai/catalog/standards/sist/845cdf03-h50-4147-80c5-119d054f3feb/osist-pren-iec-62321-11-2022)

243 EI Electron ionization [h50-4147-80c5-119d054f3feb/osist-pren-iec-62321-11-](https://standards.iteh.ai/catalog/standards/sist/845cdf03-h50-4147-80c5-119d054f3feb/osist-pren-iec-62321-11-2022)

244 GC-MS Gas chromatography – mass spectrometry

245 HPLC High-performance liquid chromatography

246 ID Internal diameter

247 IS Internal standard

248 LC-MS Liquid chromatography – mass spectrometry

249 LOD Limit of detection

250 LOQ Limit of quantification

251 MDL Method detection limit

252 MS Mass spectrometry

253 NS Neck size

254 PBB Polybrominated biphenyl

255 PBDE Polybrominated diphenyl ether

256 PS Polystyrene

257 PTFE Polytetrafluoroethylene

258 PUR Polyurethane

259	PVC	Polyvinyl chloride
260	Py/TD-GC-MS	Gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption
261		accessory
262	QC	Quality control
263	RF	Response factor
264	RRF	Relative response factor
265	RSD	Relative standard deviation
266	SIM	Single (or “selected”) ion monitoring
267	TCEP	Tris(2-chloroethyl) phosphate, (CAS Number: 115-96-8)
268	TD	Thermal desorption
269	THF	Tetrahydrofuran
270	TICS	Tentatively identified compounds

271

272 4. Principle

273 The samples are dissolved by THF using ultra-sonication, or extracted using Soxhlet. The matrix polymer of
 274 dissolved sample is separated by precipitation with non-solvent, methanol.

275 TCEP in the sample solution is determined quantitatively and qualitatively using GC-MS or LC-MS. It is possible
 276 to analyze high concentrations by diluting the sample solution.

277

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278 5. Reagents and materials

279 All chemicals are tested for contamination and blank values prior to application.

280 a) TCEP (tris(2-chloroethyl) phosphate); CAS No. 115-96-8 (purity of greater than a mass fraction of
 281 98 %) <https://standards.iteh.ai/catalog/standards/sist/845cdf03-6150-4147-80c5-119d054b1eb/osist-pr-en-iec-62321-11-2022>

282 b) THF (GC grade or higher, higher than 99,9 %)

283 c) n-Hexane (GC grade or higher, higher than 98,5 %)

284 d) Methanol (GC grade or higher, higher than 99,9 %)

285 e) Mixed solvent (THF mixed with methanol, the ratio of THF/methanol is 1/4)

286 f) Helium (purity of greater than 99,999 %)

287 g) Calibrants; reference materials of TCEP (purity of greater than 98 %)

288 h) Surrogate and internal standards:

289 - Surrogate standard used to monitor analytes recovery according to 8.2.1.1 and 8.2.2.1,
 290 for example TCEP-d₁₂;

291 - Internal standard used to correct injection errors, according to 8.2.1.1 and 8.2.2.1,
 292 for example anthracene d-₁₀

293 The standards are acceptable when using a quadruple-type mass spectrometer. A high-resolution mass
 294 spectrometer will require the use of other suitable standard substances having a mass and elution time
 295 similar to that of the analyte.

296