

© Copyright SEK. Reproduction in any form without permission is prohibited.

Bestämning av vissa ämnen i elektriska och elektroniska produkter – Del 8: Ftalater i polymerer genom gaskromatografi-masspektrometri (GC-MS), gaskromatografi-masspektrometri med utrustning för pyrolysis/termisk desorption (Py-TD-GC-MS)

*Determination of certain substances in electrotechnical products –
Part 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas
chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py-TD-GC-MS)*

Som svensk standard gäller europastandarden EN 62321-8:2017. Den svenska standarden innehåller den officiella engelska språkversionen av EN 62321-8:2017.

Nationellt förord

Europastandarden EN 62321-8:2017

består av:

- **europastandardens ikraftsättningsdokument**, utarbetat inom CENELEC
- **IEC 62321-8, First edition, 2017 - Determination of certain substances in electrotechnical products - Part 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py-TD-GC-MS)**

utarbetad inom International Electrotechnical Commission, IEC.

Standarder underlättar utvecklingen och höjer elsäkerheten

Det finns många fördelar med att ha gemensamma tekniska regler för bl a mätning, säkerhet och provning och för utförande, skötsel och dokumentation av elprodukter och elanläggningar.

Genom att utforma sådana standarder blir säkerhetsfordringar tydliga och utvecklingskostnaderna rimliga samtidigt som marknadens acceptans för produkten eller tjänsten ökar.

Många standarder inom elområdet beskriver tekniska lösningar och metoder som åstadkommer den elsäkerhet som föreskrivs av svenska myndigheter och av EU.

SEK är Sveriges röst i standardiseringsarbetet inom elområdet

SEK Svensk Elstandard svarar för standardiseringen inom elområdet i Sverige och samordnar svensk medverkan i internationell och europeisk standardisering. SEK är en ideell organisation med frivilligt deltagande från svenska myndigheter, företag och organisationer som vill medverka till och påverka utformningen av tekniska regler inom elektrotekniken.

SEK samordnar svenska intressenters medverkan i SEKs tekniska kommittéer och stödjer svenska experters medverkan i internationella och europeiska projekt.

Stora delar av arbetet sker internationellt

Utformningen av standarder sker i allt väsentligt i internationellt och europeiskt samarbete. SEK är svensk nationalkommitté av International Electrotechnical Commission (IEC) och Comité Européen de Normalisation Electrotechnique (CENELEC).

Standardiseringsarbetet inom SEK är organiserat i referensgrupper bestående av ett antal tekniska kommittéer som speglar hur arbetet inom IEC och CENELEC är organiserat.

Arbetet i de tekniska kommittéerna är öppet för alla svenska organisationer, företag, institutioner, myndigheter och statliga verk. Den årliga avgiften för deltagandet och intäkter från försäljning finansierar SEKs standardiseringsverksamhet och medlemsavgift till IEC och CENELEC.

Var med och påverka!

Den som deltar i SEKs tekniska kommittéarbete har möjlighet att påverka framtida standarder och får tidig tillgång till information och dokumentation om utvecklingen inom sitt teknikområde. Arbetet och kontakterna med kollegor, kunder och konkurrenter kan gynnsamt påverka enskilda företags affärsutveckling och bidrar till deltagarnas egen kompetensutveckling.

Du som vill dra nytta av dessa möjligheter är välkommen att kontakta SEKs kansli för mer information.

SEK Svensk Elstandard

Box 1284
164 29 Kista
Tel 08-444 14 00
www.elstandard.se

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 62321-8

June 2017

ICS 31.020; 71.040.50

English Version

**Determination of certain substances in electrotechnical products
- Part 8: Phthalates in polymers by gas chromatography-mass
spectrometry (GC-MS), gas chromatography-mass spectrometry
using a pyrolyzer/thermal desorption accessory (Py/TD-GC-MS)
(IEC 62321-8:2017)**

Détermination de certaines substances dans les produits
électrotechniques - Partie 8: Analyse des phthalates dans les
polymères par chromatographie en phase gazeuse-
spectrométrie de masse (GC-MS), chromatographie en
phase gazeuse-spectrométrie de masse par
pyrolyse/thermodésorption (Py/TD-GC-MS)
(IEC 62321-8:2017)

Verfahren zur Bestimmung von bestimmten Substanzen in
Produkten der Elektrotechnik - Teil 8: Phthalate in
Polymeren mit Pyrolyse-Gaschromatographie-
Massenspektrometrie (Py-GC-MS), Ionen-Anlagerungs-
Massenspektrometrie (IAMS), Gaschromatographie-
Massenspektrometrie (GC-MS) und
Flüssigchromatographie-Massenspektrometrie (LC-MS)
(IEC 62321-8:2017)

This European Standard was approved by CENELEC on 2017-05-02. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CENELEC member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CENELEC member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CENELEC members are the national electrotechnical committees of Austria, Belgium, Bulgaria, Croatia, Cyprus, the Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, the Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.



European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

European foreword

The text of document 111/416/CDV, future edition 1 of IEC 62321-8, prepared by IEC/TC 111 "Environmental standardization for electrical and electronic products and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62321-8:2017.

The following dates are fixed:

- latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2018-02-02
- latest date by which the national standards conflicting with the document have to be withdrawn (dow) 2020-05-02

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC shall not be held responsible for identifying any or all such patent rights.

Endorsement notice

The text of the International Standard IEC 62321-8:2017 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following notes have to be added for the standards indicated:

ISO 3696	NOTE	Harmonized as EN ISO 3696.
ISO/IEC 17025	NOTE	Harmonized as EN ISO/IEC 17025.
IEC 62321-2:2013	NOTE	Harmonized as EN 62321-2:2014.
IEC 62321-6:2015	NOTE	Harmonized as EN 62321-6:2016

Annex ZA

(normative)

**Normative references to international publications
with their corresponding European publications**

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE 1 When an International Publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

NOTE 2 Up-to-date information on the latest versions of the European Standards listed in this annex is available here: www.cenelec.eu.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
IEC 62321-1	2013	Determination of certain substances in electrotechnical products -- Part 1: Introduction and overview	EN 62321-1	2013

CONTENTS

FOREWORD.....	6
INTRODUCTION.....	8
1 Scope	9
2 Normative references	9
3 Terms, definitions and abbreviated terms	10
3.1 Terms and definitions.....	10
3.2 Abbreviated terms.....	10
4 Principle	11
5 Reagents and materials	11
5.1 GC-MS method	11
5.2 Py/TD-GC-MS method	12
6 Apparatus.....	12
6.1 GC-MS method	12
6.2 Py/TD-GC-MS method	13
7 Sampling	14
7.1 General.....	14
7.2 GC-MS method	14
7.3 Py/TD-GC-MS method	14
8 Procedure.....	14
8.1 General instructions for the analysis	14
8.1.1 Overview	14
8.1.2 GC-MS method.....	14
8.1.3 Py/TD-GC-MS method	14
8.2 Sample preparation.....	15
8.2.1 GC-MS method.....	15
8.2.2 Py/TD-GC-MS method	16
8.3 Instrumental parameters	16
8.3.1 GC-MS method.....	16
8.3.2 Py/TD-GC-MS method	18
8.4 Calibrants	18
8.5 Calibration	19
8.5.1 GC-MS method.....	19
8.5.2 Py/TD-GC-MS method	20
9 Calculation of phthalate concentration	21
9.1 GC-MS method	21
9.2 Py/TD-GC-MS method	22
10 Precision	23
10.1 GC-MS method	23
10.1.1 Threshold judgement	23
10.1.2 Repeatability and reproducibility	24
10.2 Py/TD-GC-MS method	25
10.2.1 Screening judgement.....	25
10.2.2 Repeatability and reproducibility	26
11 Quality assurance and control	27
11.1 General.....	27

11.2	GC-MS method	27
11.2.1	Performance.....	27
11.2.2	Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ)	28
11.3	Py/TD-GC-MS method	29
11.3.1	Sensitivity.....	29
11.3.2	Blank test	29
11.3.3	Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ)	29
12	Test report.....	30
Annex A (informative) Determination of phthalates in polymers by ion attachment mass spectrometry (IAMS)		31
A.1	Principle	31
A.2	Reagents and materials	31
A.3	Apparatus	31
A.4	Sampling.....	32
A.5	Procedure	32
A.5.1	General instructions for the analysis	32
A.5.2	Sample preparation	32
A.5.3	Instrumental parameters	33
A.5.4	Calibrants	34
A.5.5	Calibration	34
A.6	Calculation of phthalates concentration.....	35
A.7	Quality assurance and control	35
A.7.1	General	35
A.7.2	Sensitivity.....	35
A.7.3	Recovery	35
A.7.4	Blank test	36
A.7.5	Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ)	37
A.8	Test report.....	37
Annex B (informative) Determination of phthalates in polymers by liquid chromatography-mass spectrometry(LC-MS)		38
B.1	Principle	38
B.2	Reagents and materials	38
B.3	Apparatus	38
B.4	Sampling.....	39
B.5	Procedure	39
B.5.1	General instructions for the analysis	39
B.5.2	Sample preparation	39
B.5.3	Instrumental parameters	40
B.5.4	Calibrants	42
B.5.5	Calibration	42
B.6	Calculation of phthalates concentration.....	43
B.7	Quality assurance and control	44
B.7.1	General	44
B.7.2	Performance.....	44
B.7.3	Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ)	44
B.8	Test report.....	45

Annex C (informative) Examples of chromatograms at suggested conditions	46
C.1 GC-MS method	46
C.2 Py/TD-GC-MS method	47
C.3 LC-MS method.....	47
C.4 IAMS method	48
Annex D (informative) Verification of the EGA thermal desorption zone	51
Annex E (informative) Example of IAMS and Py/TD-GC-MS instruments.....	52
Annex F (informative) Example of false positive detection of phthalates.....	54
Annex G (informative) Examples of sample preparation for quantitative analysis of phthalates by GC-MS.....	55
G.1 General.....	55
G.2 Soxhlet extraction of phthalates using proper organic solvents	55
Annex H (informative) Extraction of phthalates by dissolution in THF using sonication and precipitation of polymer matrix	58
Annex I (informative) Commercially available reference materials considered suitable for GC-MS and Py/TD-GC-MS.....	60
I.1 GC-MS.....	60
I.2 Py/TD-GC-MS.....	60
Annex J (informative) Commercially available capillary columns considered suitable for GC-MS and Py-GC-MS	62
Annex K (informative) Labware cleaning procedure for phthalate testing.....	63
K.1 With the use of a furnace (non-volumetric glassware only)	63
K.2 Without the use of a furnace (glassware and plastic-ware).....	63
K.3 Estimation of cleanliness of the inner areas of volumetric glassware	64
Annex L (informative) Results of international inter-laboratory study 5	65
Annex M (informative) Sample analysis sequence.....	70
M.1 GC-MS.....	70
Annex N (informative) Flow chart	71
Bibliography.....	72

Figure C.1 – Total ion current chromatogram of each phthalate (10 µg/ml, 1 µl, splitless)	46
Figure C.2 – Extracted ion chromatogram of DINP (10 µg/ml, 1 µl, splitless).....	46
Figure C.3 – Extracted ion chromatogram of DIDP (10 µg/ml, 1 µl, splitless).....	47
Figure C.4 – Total ion current chromatogram of 100 µg/ml of phthalate mixture by Py/TD-GC-MS	47
Figure C.5 – Total ion current chromatogram of 5 µg/ml of phthalate mixture by LC-MS.....	48
Figure C.6 – Mass spectrum of each phthalate by IAMS	49
Figure C.7 – Total ion current chromatogram of each absolute amount (0,08 µg) of phthalate mixture by IAMS	50
Figure C.8 – Total ion current chromatogram of approximately 0,3 mg of PVC which contains 300 mg/kg of each phthalate mixture by IAMS (Absolute amount: 0,09 µg)	50
Figure D.1 – Example of EGA thermogram of a PVC sample containing phthalates.....	51
Figure E.1 – Example of IAMS instrument.....	52
Figure E.2 – Example of Py/TD-GC-MS instrument	53
Figure F.1 – Typical laboratory wares made of plastic materials that may cause phthalate contamination	54

Figure F.2 – Example of a chromatogram of a blank solvent (THF) in a plastic bottle showing DEHP contamination	54
Figure G.1 – Recovery ratios of Di-(2-ethylhexyl) phthalate using Soxhlet extraction with different organic solvents.....	57
Figure G.2 – Comparison of recovery ratios of phthalates using different extracting conditions	57
Figure I.1 – Sample preparation of reference materials	61
Figure N.1 – Flow chart for screening step and quantitative step	71
Table 1 – Measurement condition of GC-MS	17
Table 2 – Reference masses for the quantification of each phthalate	17
Table 3 – Measurement condition of Py/TD-GC-MS	18
Table 4 – Calibration standard solution of phthalates	19
Table 5 – IIS5 Threshold judgement.....	23
Table 6 – IIS5 Repeatability and reproducibility	24
Table 7 – IIS5 screening and threshold judgement.....	25
Table 8 – IIS5 Repeatability and reproducibility	26
Table A.1 – Measurement condition of IAMS.....	34
Table A.2 – Certified value of constituent phthalates in KRISS CRM 113-03-006	36
Table B.1 – Measurement condition of LC-MS	42
Table B.2 – Standard stock solution concentrations	43
Table G.1 – Recovery ratios of phthalates according to different Soxhlet extraction times (extracting solvent: n-hexane)	56
Table H.1 – Comparison of the efficiency of the sample preparation method of dissolution in THF using sonication and precipitation of polymeric matrix with that of Soxhlet extraction for soluble sample	58
Table H.2 – Comparison of the efficiency of the sample preparation method of dissolution in THF using sonication and precipitation of polymeric matrix with that of Soxhlet extraction for insoluble samples	59
Table I.1 – Example list of commercially available reference solutions considered suitable for GC-MS	60
Table I.2 – Example list of commercially available reference materials considered suitable for Py/TD-GC-MS.....	61
Table J.1 – Example list of commercially available capillary columns considered suitable for GC-MS and Py-GC-MS analysis	62
Table L.1 – Statistical data for Py/TD-GC-MS	65
Table L.2 – Statistical data for GC-MS	67
Table L.3 – Statistical data for IAMS	68
Table L.4 – Statistical Data For LC-MS	69
Table M.1 – Sample analysis sequence for GC-MS analysis	70
Table M.2 – Sample analysis sequence for Py/TD-GC-MS analysis	70

INTERNATIONAL ELECTROTECHNICAL COMMISSION

**DETERMINATION OF CERTAIN SUBSTANCES
IN ELECTROTECHNICAL PRODUCTS –****Part 8: Phthalates in polymers by gas chromatography-mass spectrometry
(GC-MS), gas chromatography-mass spectrometry using
a pyrolyzer/thermal desorption accessory (Py/TD-GC-MS)****FOREWORD**

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 62321-8 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:

CDV	Report on voting
111/416/CDV	111/430/RVC

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.

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 publication will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication 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 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 62321-8 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 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py/TD-GC-MS)

1 Scope

This part of IEC 62321 specifies two normative and two informative techniques for the determination of di-isobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzylbutyl phthalate (BBP), di-(2-ethylhexyl) phthalate (DEHP), di-n-octyl phthalate (DNOP), di-isonyl phthalate (DINP) and di-iso-decyl phthalate (DIDP) in polymers of electrotechnical products.

Gas chromatography-mass spectrometry (GC-MS) and gas chromatography-mass spectrometry (Py/TD-GC-MS) techniques are described in the normative part of this document.

The GC-MS method is considered the referee technique for the quantitative determination of DIBP, DBP, BBP, DEHP, DNOP, DINP and DIDP in the range of 50 mg/kg to 2 000 mg/kg.

The GC-MS coupled with a pyrolyzer/thermal desorption (TD) accessory is suitable for screening and semi-quantitative analysis of DIBP, DBP, BBP, DEHP, DNOP, DINP, and DIDP in polymers that are used as parts of the electrotechnical products in the range of 100 mg/kg to 2 000 mg/kg.

The IAMS technique is suitable for screening and semi-quantitative analysis of DIBP, DBP, BBP, DEHP, DNOP, DINP, and DIDP. Determination of DBP and DIBP, DEHP and DNOP by IAMS has not been established due to peak and mass spectral resolution limitations.

The LC-MS technique is limited to the determination of BBP, DEHP, DNOP, DINP, and DIDP. Determination of DBP and DIBP by LC-MS has not been established due to peak and mass spectral resolution limitations.

A flow chart depicting how the normative Py/TD-GC-MS and GC-MS methods and informative methods using ion attachment mass spectrometry (IAMS) coupled with direct injection probe (DIP) and liquid chromatography-mass spectrometry (LC-MS) can be used are provided in annexes of this document.

These four test methods have been evaluated by the test of PE (polyethylene) and PVC (polyvinyl chloride) materials containing individual phthalates between ~450 mg/kg to 30 000 mg/kg as depicted in the normative and informative parts of this document. The use of the four methods described in this document for other polymer types, phthalate compounds or concentration ranges other than those specified above has not been specifically evaluated.

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*