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INTERNATIONAL STANDARD

NORME INTERNATIONALE

Determination of certain substances In electrotechnical products – Part 3-4: Screening – Phthalates in polymers of electrotechnical products by high performance liquid chromatography with ultraviolet detector (HPLC-UV), thin layer chromatography (TLC) and thermal desorption mass spectrometry (TD-MS)

Détermination de certaines substances dans les produits électrotechniques – Partie 3-4: Détection – Phtalates dans les polymères des produits électrotechniques par chromatographie en phase liquide à haute performance avec détecteur d'ultraviolets (HPLC-UV), par chromatographie sur couche mince (CCM) et par spectrométrie de masse par désorption thermique (TD-MS)



IEC 62321-3-4:2023-05(en-fr)



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Détermination de certaines substances dans les produits électrotechniques – Partie 3-4: Détection – Phtalates dans les polymères des produits électrotechniques par chromatographie en phase liquide à haute performance avec détecteur d'ultraviolets (HPLC-UV), par chromatographie sur couche mince (CCM) et par spectrométrie de masse par désorption thermique (TD-MS)

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CONTENTS

FOREWORD 5
INTRODUCTION 7
1 Scope 8
2 Normative references 9
3 Terms, definitions and abbreviated terms 9
3.1 Terms and definitions 9
3.2 Abbreviated terms 10
4 Principle 10
5 HPLC-UV and TLC method 11
5.1 Reagents and materials 11
5.1.1 Reagents and materials of HPLC-UV method 11
5.1.2 Reagents and materials of TLC method 11
5.2 Equipment, apparatus and tools 12
5.2.1 Equipment, apparatus and tools for HPLC-UV method 12
5.2.2 Equipment, apparatus and tools for TLC method 12
5.3 Sampling 12
5.4 Procedure 13
5.4.1 Procedure of HPLC-UV method 13
5.4.2 Procedure of TLC method 15
5.5 Calculation of phthalates concentration 17
5.6 Precision 17
5.6.1 Precision of HPLC-UV method 17
5.6.2 Precision of TLC method 18
5.7 Quality assurance and control 19
5.7.1 Quality assurance and control of HPLC-UV method 19
5.7.2 Quality assurance and control of TLC method 21
5.8 Test report 22
6 TD-MS method 22
6.1 Reagents and materials 22
6.2 Equipment, apparatus and tools 22
6.2.1 Equipment 22
6.2.2 Apparatus and tools 22
6.3 Sampling 22
6.4 Procedure 23
6.4.1 Procedure of APCI-MS method 23
6.4.2 Procedure of IA-MS method 25
6.5 Calculation of phthalates concentration 27
6.6 Precision 27
6.7 Quality assurance and control 28
6.7.1 Sensitivity 28
6.7.2 Stability test 28
6.7.3 Blank test 29
6.7.4 Limit of detection (LOD) or method detection limit (MDL) and limit of quantification (LOQ) 29
6.8 Test report 29
Annex A (informative) FT-IR method 30

A.1 Principle 30
A.2 Reagents and materials 32
A.3 Apparatus 32
A.4 Sampling 33
A.5 Procedure 33
A.5.1 Sample preparation 33
A.5.2 Instrumental parameters 33
A.5.3 Calibration 33
A.6 Calculation of phthalates concentration 34
A.7 Precision 34
A.8 Quality assurance and control 35
A.9 Test report 35
Annex B (informative) Details of analysis by TLC method 36
B.1 Separation by TLC 36
B.2 Detection by image analysis 36
B.3 Re-measurement 38
Annex C (informative) Examples of spectrums and chromatograms at suggested conditions 41
C.1 FT-IR spectrum 41
C.2 HPLC-UV chromatogram 41
C.3 TLC chromatogram 42
C.4 APCI-MS mass spectrum 42
C.5 IA-MS mass spectrum 43
Annex D (informative) Commercially available reference materials and solutions considered suitable for the suggested methods 44
Annex E (informative) Flowchart of test methods 45
Annex F (informative) Commonly used phthalates 46
Annex G (informative) Results of international inter-laboratory study 3-4 (IIS 3-4) 47
Bibliography 51

Figure 1 – Polymer samples in glass vials with acetonitrile (tightened with sealing tape) 15
Figure A.1 – Phthalate analysis in polymers (check) 31
Figure A.2 – Phthalate analysis in polymers with pre-treatment 31
Figure B.1 – Usage of TLC plate (20 cm × 10 cm) 36
Figure B.2 – Set-up of camera-equipment for TLC (inside of darkroom) 37
Figure B.3 – TLC chromatogram 38
Figure B.4 – Separation by re-measurement conditions (in case of pattern a)) 39
Figure B.5 – Peak shift affected by large amount of DEHA 39
Figure B.6 – TLC re-measurement by standard addition method (in case of pattern b)) 40
Figure C.1 – Spectrum of FT-IR 41
Figure C.2 – Chromatogram of HPLC-UV 41
Figure C.3 – Developed TLC plate exposed to UV light of 254 nm 42
Figure C.4 – Image processed TLC chromatogram of Figure C.3 42
Figure C.5 – Mass spectrums of APCI-MS 43
Figure C.6 – Mass spectrums of IA-MS 43
Figure E.1 – Flowchart for screening step and verification test step 45

Table 1 – Standard mixture solution concentrations 13
Table 2 – Measurement conditions of HPLC-UV 14
Table 3 – Standard mixture solution concentrations 15
Table 4 – Measurement conditions of TLC 16
Table 5 – IIS 3-4 Repeatability and reproducibility of HPLC-UV 18
Table 6 – IIS 3-4 Repeatability and reproducibility of TLC 19
Table 7 – Measurement conditions of APCI-MS 24
Table 8 – Measurement conditions of IA-MS 26
Table 9 – IIS 3-4 Repeatability and reproducibility of TD-MS 28
Table A.1 – IIS 3-4 Repeatability and reproducibility of FT-IR 34
Table B.1 – Conditions of photography 37
Table B.2 – Range of Rf values of target phthalates 38
Table D.1 – Example list of commercially available reference materials 44
Table F.1 – Example list of commonly used phthalates in products 46
Table G.1 – Formulation of samples 47
Table G.2 – Statistical data for HPLC-UV 48
Table G.3 – Statistical data for TLC 49
Table G.4 – Statistical data for TD-MS 50
Table G.5 – Statistical data for FT-IR 50

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 3-4: Screening – Phthalates in polymers of electrotechnical products by high performance liquid chromatography with ultraviolet detector (HPLC-UV), thin layer chromatography (TLC) and thermal desorption mass spectrometry (TD-MS)

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

Draft	Report on voting
111/695/FDIS	111/701/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/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 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 62321-3-4 introduces a new part in the IEC 62321 series.

Appropriate test methods are required in order to facilitate the monitoring of the contents of certain substances in affected materials. Faced with the enormous task of testing a diversity of electronic and electric equipment, the industry adopted the concept of 'screening' in order to reduce the amount of testing. As defined in IEC 62321-1:2013, 3.1.10, *"...screening is an 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 further testing"*. Executed as a predecessor to any other test analysis of the product, the main objective of screening is to quickly, expediently, inexpensively and preferably in a non-destructive manner, determine whether the screened product:

– contains a certain substance at a concentration significantly higher than its value accepted as criterion, and therefore can be rejected as being above the threshold;

– contains a certain substance at a concentration significantly lower than its value accepted as criterion, and therefore can be considered below the threshold;

– contains a certain substance at a concentration so close to the value accepted as criterion that when all possible errors of measurement and safety factors and measurement uncertainty are considered, no conclusive decision can be made about the absence or presence of substance and, therefore, a follow-up action can be required, such as another, more specific or more precise and accurate analysis.

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 3-4: Screening – Phthalates in polymers of electrotechnical products by high performance liquid chromatography with ultraviolet detector (HPLC-UV), thin layer chromatography (TLC) and thermal desorption mass spectrometry (TD-MS)

1 Scope

This part of IEC 62321 specifies procedures for the screening of di-isobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzyl butyl phthalate (BBP), di-(2-ethylhexyl) phthalate (DEHP) in polymers of electrotechnical products by using high performance liquid chromatography with ultraviolet detector (HPLC-UV), thin layer chromatography (TLC) and thermal desorption mass spectrometry (TD-MS).

High performance liquid chromatography with ultraviolet detector (HPLC-UV), thin layer chromatography (TLC) and thermal desorption mass spectrometry (TD-MS) techniques are described in the normative part of this document. Fourier transform infrared spectroscopy (FT-IR) is described in the informative annexes of this document.

The HPLC-UV and TLC techniques are suitable for screening and semi-quantitative analysis of DIBP, DBP, BBP and DEHP in polymers that are used as parts in electrotechnical products above 300 mg/kg.

Theu TD-MS technique is suitable for screening and semi-quantitative analysis of DIBP, DBP, BBP and DEHP in polymers that are used as parts in electrotechnical products above 300 mg/kg.

The FT-IR technique is suitable for preliminary screening of total phthalates (DIBP, DBP, BBP, DEHP and so forth) in polymers that are used as parts in electrotechnical products above 50 000 mg/kg.

These test methods have been evaluated by testing polyethylene (PE), polyvinyl chloride (PVC) materials containing individual phthalates between 500 mg/kg to 3 000 mg/kg as depicted in this document. The use of the methods described in this document for other polymer types, phthalate compounds or concentration ranges other than those specified above has not been specifically evaluated.

A flow chart is given as an example of how each method included in this document can be used for screening. The test methods in this document differ from those given in IEC 62321-8 [1][1] in that not all phthalates in this scope are separated from each other. Detectable combinations are DIBP + DBP + BBP and DEHP for the HPLC-UV technique, DIBP + DBP, BBP and DEHP for the TLC technique and TD-MS technique, total phthalates for the FT-IR technique. FT-IR is a suitable analytical technique for preliminary screening in the first step of phthalates screening. These test methods are characterized by a shorter measuring time compared with IEC 62321-8 because all phthalates in this scope are not separated from each other.

NOTE See Annex F for commonly used phthalates in products.

This document has the status of a horizontal publication in accordance with IEC Guide 108 [2].

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1 Numbers in square brackets refer to the Bibliography.

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 62321-2:2021, *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 https://www.electropedia.org/
- ISO Online browsing platform: available at https://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 further testing

Note 1 to entry: If the screening method produces values that are not conclusive, then additional analysis or other follow-up actions may be necessary to make a final presence/absence decision.

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

3.1.2
semi-quantitative
level of accuracy in a measurement amount where the relative uncertainty of the result is typically 30 % or better at a defined level of confidence of 68 %

[SOURCE: IEC 62321-6:2015, 3.1.1 [3]]

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)

3.2 Abbreviated terms

ACN	acetonitrile
APCI	atmospheric pressure chemical ionization
APCI-MS	atmospheric pressure chemical ionization mass spectrometry
BBP	benzyl butyl phthalate
CRM	certified reference material
DBP	di-n-butyl phthalate
DEHP	di-(2-ethylhexyl) phthalate
DIBP	di-isobutyl phthalate
DIP	direct injection probe
DNOP	di-n-octyl phthalate
FT-IR	Fourier Transform infrared spectroscopy
HPLC-UV	high performance liquid chromatography with ultraviolet detector
IA-MS	ion attachment mass spectrometry
TLC	thin layer chromatography
IS	internal standard
LOD	limit of detection
LOQ	limit of quantification
MDL	method detection limit
MS	mass spectrometry
PVC	polyvinyl chloride
QC	quality control
SIM	selected ion monitoring
TD-MS	thermal desorption mass spectrometry
THF	tetrahydrofuran

4 Principle

In the HPLC-UV method, DIBP, DBP, BBP and DEHP are determined using ultrasonic extraction followed by high-pressure, liquid chromatography separation and ultraviolet detection. Owing to the peak overlapping of DIBP, DBP and BBP, occurrence of the peak indicates only qualitative information of possible presence of DIBP, DBP and BBP or a combination of one or two of either of these phthalates.

The TLC method, as well as the HPLC method, is one of the liquid chromatography methods and can be performed with simple instruments. In the TLC method, DIBP, DBP, BBP and DEHP in the polymer are separated by TLC after ultrasonic extraction and detected by image analysis after photography under UV light. DIBP and DBP are detected as sum peaks because it is difficult to separate them by TLC.

TD-MS techniques use a thermal desorption system directly connected to mass spectrometry with ionization systems such as an atmospheric pressure chemical ionization or ion attachment to screen for the presence of DIBP, DBP, BBP and DEHP in polymers. This method allows for the direct analysis of a polymer sample without pre-treatment process. For example:

– The APCI-MS method has an ion source that attaches H^+ to target molecules by a corona discharge under atmospheric pressure and is coupled with a furnace stabilized at 330 °C and a sample heater. The sample heater is programmed to heat up to 230 °C to thermally desorb sample molecules. The thermally desorbed sample molecules (M) form adducts ($M + H^+$) with H^+ in the reaction and are analysed by a mass spectrometer via select ion monitoring.
– The IA-MS method includes a Li^+ attachment reaction chamber with a Li^+ emitter and is coupled with a direct injection probe (DIP). The DIP is programmed to heat up to 350 °C to thermally desorb sample molecules. The thermally desorbed sample molecules (M) form adducts ($M + Li^+$) with Li^+ in the reaction chamber and those adducts are analysed by a mass spectrometer via select ion monitoring.

Therefore, target molecules with the same molecular weight such as DBP and DIBP, DEHP and Di-n-octyl phthalate (DNOP) are detected as sum peaks by TD-MS techniques.

The principle of phthalate detection by FT-IR can be referenced in Annex A.

These test methods are based on the concept of performance. Apparatus, sampling and calibration are specified in this document in relatively general terms. It is the responsibility of the user to document all procedures developed in the laboratory that uses the test methods described in this document. The user shall establish a written procedure for all cases denoted in the test methods described in this document by the term "work instructions". A flowchart is provided in Annex E as an example of how these methods can be used for screening.

5 HPLC-UV and TLC method

5.1 Reagents and materials

5.1.1 Reagents and materials of HPLC-UV method

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

a) methanol (HPLC grade, purity greater than a volume fraction of 99,7 %);
b) THF (HPLC grade, purity greater than a volume fraction of 99,7 %);
c) ethanol (HPLC grade, purity greater than a volume fraction of 99,7 %);
d) ultrapure water (HPLC grade);
e) standard mixture solution or reference polymer materials as calibrants: one contains approximately 1 000 mg/kg of phthalates.

NOTE Commercially available reference materials are listed in Annex D.

5.1.2 Reagents and materials of TLC method

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

a) acetonitrile;
b) methanol;
c) standard mixture solution or reference polymer materials as calibrants: one contains approximately 1 000 mg/kg of phthalates;

NOTE Commercially available reference materials are listed in Annex D.

5.2 Equipment, apparatus and tools

5.2.1 Equipment, apparatus and tools for HPLC-UV method

The following equipment shall be used for the analysis:

a) high-performance liquid chromatography (HPLC) system equipped with a UV or PDA/UV detector, auto sampler, pump and column oven;
b) analytical balance capable of measuring accurately to 0,000 1 g (0,1 mg);
c) ultrasonic bath (capable of heating above 50 °C);

The following equipment should be used for sample preparation as necessary:

d) cryogenic grinding or milling with liquid N_2 cooling.

The following items shall be used for the analysis:

e) column;
f) glass vials for HPLC-UV;
g) glass vials for extraction (40 ml volume is recommended);
h) volumetric flask;
i) adjustable pipettes;
j) paper filters, medium-fast filtration, general laboratory use.

NOTE The size of the required glass vial for HPLC depends on the instrument.

5.2.2 Equipment, apparatus and tools for TLC method

The following equipment shall be used for the analysis:

a) ultrasonic bath (capable of heating above 60 °C);
b) analytical balance (capable of measuring accurately to 0,000 1 g (0,1 mg));
c) TLC plate (stationary phase C18, size 20 cm × 20 cm, cut in half to 20 cm × 10 cm);
d) TLC developing chamber;
e) UV lamp (λ = 254 nm, 2 units required);
f) digital camera (with UV lens filter for ultraviolet adsorption as optional);
g) clamp (for fixing UV lamps and a camera);
h) desktop darkroom.

The following items shall be used for the analysis:

a) glass vials for extraction (4 ml volume is recommended);
d) capillary (capacity 1 µl);
c) volumetric flask;
d) adjustable pipettes or micro syringes;
e) scissors or cutter knife.

5.3 Sampling

Unless otherwise specified in this document, the sampling procedure described in IEC 62321-2 shall be referred to.