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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)

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Détermination de certaines substances dans les produits électrotechniques – Partie 8: Analyse des phtalates 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)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

COMMISSION ELECTROTECHNIQUE INTERNATIONALE

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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)

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It has the status of a horizontal standard in accordance with IEC Guide 108.

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

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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.

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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-isononyl 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.

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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.

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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 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*

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
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 %

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

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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
BBP	Benzyl butyl phthalate
BSA	N,O-bis(trimethylsilyl)acetamide
BSTFA	N,O-bis(trimethylsilyl)trifluoroacetamide
CRM	Certified reference material
DBP	Di-n-butyl phthalate
DEHP	Di-(2-ethylhexyl) phthalate
DIBP	Di-isobutyl phthalate
DIDP	Di-iso-decyl phthalate
DINP	Di-isononyl phthalate
DIP	Direct injection probe
DNOP	Di-n-octyl phthalate
EGA	Evolved gas analysis
EI	Electron ionization
GC-MS	Gas chromatography – mass spectrometry

IAMS	Ion attachment mass spectrometry
IS	Internal standard
LC-MS	Liquid chromatography – mass spectrometry
LOD	Limit of detection
LOQ	Limit of quantification
MDL	Method detection limit
PVC	Polyvinyl chloride
Py	Pyrolyzer
QC	Quality control
SIM	Selected ion monitoring
TD	Thermal desorption
THF	Tetrahydrofuran

4 Principle

In the GC-MS method, DIBP, DBP, BBP, DEHP, DNOP, DINP and DIDP are quantitatively determined using ultrasonic dissolution and precipitation of the sample matrix or Soxhlet extraction from polymers with separation by gas chromatography separation and mass spectrometry detection.

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The Py/TD-GC-MS uses gas chromatography-mass spectrometry coupled with a pyrolyzer/thermal desorption accessory to screen for the presence of DIBP, DBP, BBP, DEHP, DNOP, DINP and DIDP in polymeric materials. The polymer sample is directly introduced into the pyrolyzer/thermal desorption accessory to thermally extract phthalates from the polymer using a specified heating programme. Thermally desorbed phthalates are then transferred to the gas chromatograph, separated by a capillary column and detected by a mass spectrometer. The respective phthalates are identified based on the retention times , m/z (quantitative and confirmation ions) and ion ratios. A SIM mode is used to improve the limits of detection. A one-point calibration is applied for screening and semi-quanititative analysis of phthalates in the sample.

NOTE A full scan run using a total ion current ("full scan") MS method for each sample can be used in order to check for the existence of negative matrix interference from other additives in the polymer. Negative matrix interference causes ion suppression which provides lower concentration results. Scan/SIM measurement (simultaneous measurements) is also applicable.

5 Reagents and materials

5.1 GC-MS method

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

a) ACN (HPLC grade);

b) THF (GC grade or higher);

c) n-Hexane (GC grade or higher);

d) helium (purity of greater than a volume fraction of 99,999 %);

e) calibrants: refer to 8.4;

f) surrogate and internal standards:

  – surrogate standard used to monitor analytes recovery according to 8.2.1.1, 8.2.1.3, and 8.5.1.1, for example dibutyl phthalate-3,4,5,6-d_4 or di-(2-ethylhexyl)phthalate-3,4,5,6-d_4 standard;

– internal standard used to correct for injection errors, according to 8.2.1.1 and 8.5.1.2, for example anthracene-d_{10} or benzyl benzoate.

The standards are acceptable when using a quadruple-type mass spectrometer. A high-resolution mass spectrometer will require the use of other suitable standard substances having a mass and elution time similar to that of the analyte. Deuterium substituted DBP and DEHP are recommended for specified phthalates.

NOTE Commercially available surrogates and internal standards are listed in Annex I.

5.2 Py/TD-GC-MS method

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

a) helium (purity of greater than a volume fraction of 99,999 %);
b) reference polymer materials:
   one contains approximately 100 mg/kg and the other 1 000 mg/kg of phthalates;
c) blank polymer material (no phthalates shall be included).

The following reagent chemicals, when used for preparing the polymer sample, shall be similarly tested as the above.

d) n-hexane for preparing the phthalate standard solution (GC grade or higher);
e) THF, or a solvent suitable for preparing the polymer sample (GC grade or higher).

NOTE Commercially available reference materials are listed in Annex I.

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6 Apparatus

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6.1 GC-MS method

The following items shall be used for the analysis:

a) analytical balance capable of measuring accurately to 0,000 1 g;
b) cryogenic grinding/milling with liquid N_2 cooling;
c) 1 ml, 5 ml, 10 ml, 100 ml volumetric flasks;
d) Soxhlet extractors:
   – 30 ml Soxhlet extractors;
   – 250 ml round-bottomed flask;
   – ground-in stopper NS 29/32;
   – Dimroth condenser NS 29/32;
   – boiling stones (e.g. glass pearls or Raschig rings);
e) extraction thimble (cellulose 30 ml, ID 22 mm, height 80 mm);
f) glass wool (for extraction thimble);
g) ultrasonic bath;
h) deactivated injector liner (for GC-MS);
i) heating jackets;
j) funnels;
k) aluminium foil;
l) cork rings;
m) 0,45 μm PTFE filter;
n) microlitre syringe or automatic pipettes;

o) rotary evaporator with vacuum capability;

p) Pasteur pipettes;

q) 1,5 ml sample vials and a screw cap with polytetrafluoroethylene (PTFE) gasket or, depending on the analytical system, a comparable sample receptacle;

r) mini-shaker (also known as vortexer or vortex mixer);

s) a gas chromatograph – mass spectrometer, split/splitless inlet and a programmable temperature controlled oven. The mass spectrometer shall be able to perform selected ion monitoring (SIM) and a total ion current ("full scan"). The ionization box shall be treated for chemical stability, and controlled at 230 °C. A 70 eV energy shall be applied in electron ionization (EI) mode.

The use of an autosampler is strongly recommended to ensure repeatability.

t) a capillary column.

NOTE A liquid phase 100 % dimethyl polysiloxane or 5 % diphenyl, 95 % dimethyl polysiloxane has been found suitable. The preferred column dimension length is 30 m, internal diameter is 0,25 mm, and the film thickness is 0,25 μm (see Annex J).

6.2 Py/TD-GC-MS method

The following items shall be used for the analysis:

a) analytical balance capable of measuring accurately to 0,000 01 g (0,01 mg)

b) cryogenic grinding/milling with liquid N_2 cooling;

c) nipper (a hand tool for cutting samples);

d) micro spatula;

e) tweezers;

f) cutter;

g) file;

h) micro puncher;

i) deactivated glass wool;

j) microlitre syringe or automated pipettes;

k) a gas chromatograph – mass spectrometer equipped with pyrolyzer/thermal desorption accessory, a split/splitless inlet and a programmable temperature controlled oven. The mass spectrometer shall be able to perform selected ion monitoring (SIM) and a total ion current ("full scan"). The ionization box shall be treated for chemical stability, and controlled at 230 °C. A 70 eV energy shall be applied in electron ionization (EI) mode.

The use of an auto-sampler is strongly recommended to ensure repeatability.

NOTE 1 For example, the ion box is heated in a furnace at 400 °C for 1 h for the treatment of chemical stability.

l) the pyrolyzer/thermal desorption accessory shall be capable of a temperature rise of 1 °C to 100 °C per minute across a temperature range from 40 °C to 500 °C. The pyrolyzer/thermal desorption sample cup shall be treated for chemical stability, be capable of accommodating both liquid and solid samples and maintaining the interface between the pyrolyzer/ thermal desorption unit and the gas chromatograph inlet up to 400 °C.

m) capillary column.

NOTE 2 A liquid phase 100 % dimethyl polysiloxane or 5 % diphenyl, 95 % dimethyl polysiloxane has been found suitable. The preferred column dimension length is 15 m or 30 m, the internal diameter is 0,25 mm, and the film thickness is 0,25 μm to 0,05 μm (see Annex J).

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