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INTERNATIONAL STANDARD

NORME INTERNATIONALE

HORIZONTAL STANDARD

NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 10: Polycyclic aromatic hydrocarbons (PAHs) in polymers and electronics by gas chromatography-mass spectrometry (GC-MS)

Détermination de certaines substances dans les produits électrotechniques – Partie 10: Hydrocarbures aromatiques polycycliques (HAP) dans les polymères et les produits électroniques par chromatographie en phase gazeuse-spectrométrie de masse (GC MS)

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FOREWORD

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

FDIS	Report on voting
111/575/FDIS	111/580/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.

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 "http://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- withdrawn,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 this has resulted in the adoption of regulations affecting wastes, substances and energy use of electrotechnical products.

The use of certain substances (e.g. lead (Pb), cadmium (Cd) and polybrominated diphenyl ethers (PBDEs)) 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 62321-10 introduces a new subject covering polycyclic aromatic hydrocarbons (PAHs) 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 10: Polycyclic aromatic hydrocarbons (PAHs) in polymers and electronics by gas chromatography-mass spectrometry (GC-MS)

1 Scope

This part of IEC 62321 specifies one normative technique for the determination of polycyclic aromatic hydrocarbons (PAHs) in polymers of electrotechnical products. These PAHs can especially be found in the plastic and rubber parts of a wide range of consumer articles. They are present as impurities in some of the raw materials used in the production of such articles, in particular in extender oils and in carbon black. They are not added intentionally to the articles and do not perform any specific function as constituents of the plastic or rubber parts.

The gas chromatography-mass spectrometry (GC-MS) test method is suitable for the determination of polycyclic aromatic hydrocarbons (PAHs).

These test methods have been evaluated for use with plastics and rubbers. These test methods have been evaluated for use with ABS (acrylonitrile butadiene styrene) containing individual PAHs ranging from 37,2 mg/kg to 119 mg/kg and rubbers containing individual PAHs ranging from 1 mg/kg to 221,2 mg/kg.

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This horizontal standard is primarily intended for use by technical committees in the preparation of standards in accordance with the principles laid down in IEC Guide 108.

One of the responsibilities of a technical committee is, wherever applicable, to make use of horizontal standards in the preparation of its publications. The contents of this horizontal standard will not apply unless specifically referred to or included in the relevant publications.

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, *Determination of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*

ISO 3696, *Water for analytical laboratory use – Specification and test methods*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

No terms and definitions are listed in this document.

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

3.2 Abbreviated terms

ABS	acrylonitrile butadiene styrene
CCC	continuing calibration check standard
EI	electron ionization
GC-MS	gas chromatography-mass spectrometry
IS	internal standard
IUPAC	International Union of Pure and Applied Chemistry
LOD	limit of detection
LOQ	limit of quantification
MDL	method detection limit
PAH	polycyclic aromatic hydrocarbon
PBDE	polybrominated diphenyl ether
QC	quality control
RSD	relative standard deviation
SIM	selected ion monitoring
TICS	tentatively identified compounds
US EPA	United States Environmental Protection Agency

4 Principle

PAH compounds are quantitatively determined using ultrasonic extraction or Soxhlet extraction followed by gas chromatography-mass spectrometry (GC-MS) using single (or "selected") ion monitoring (SIM).

5 Reagents and materials

Use, as far as available, reagents of analytical quality, or better. Use only reagents with negligibly low concentrations of PAH and verify by blank determinations and, if necessary, apply additional cleaning steps (for calibrants, see 8.4):

a) Dichloromethane (GC grade or higher).

b) Helium (purity of greater than a volume fraction of 99,999 %).

c) Silica gel (purity of greater than a mass fraction of 99 %).

d) Toluene (GC grade or higher).

NOTE 1 The standards are acceptable when using a quadrupole-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 (see 8.4). Other stock solution concentrations can be utilized providing the standard solution concentrations given in 8.5.2 can be achieved.

e) Sodium sulphate (purity of greater than a mass fraction of 99 %).

f) Surrogate and internal standards:

- internal standard (to correct for injection errors, according to 8.4.2 a)), (e.g. naphthalene-d8, pyrene-d10, anthracene-d10, phenanthrene-d10, benzo(a)pyrene-d12, perylene-d12 or triphenylbenzene);

NOTE 2 At least three internal standards are preferably used to be mixed with toluene as extraction agent.

- surrogate standard (to monitor analyte recovery according to 8.4.2 b), (e.g. chrysene-d12 or p-terphenyl-d14).

g) Petroleum ether (purity of greater than a mass fraction of 99 %).

h) Water (Grade 1 specified in ISO 3696 used for preparation of labware and others).

6 Apparatus

The following items shall be used for the analysis:

a) 0,45 µm PTFE filter membrane.

b) 1 ml, 5 ml, 10 ml, 100 ml volumetric flasks.

c) Aluminium foil.

d) Analytical balance capable of measuring accurately to 0,000 1 g.

e) 40 ml brown or amber vessel.

f) Cryogenic grinding with liquid N_2 cooling.

g) Dry oven.

h) Furnace.

i) Extraction thimble (cellulose 30 ml, ID 22 mm, height 80 mm).

j) Funnel.

k) Glass column (size: 220 mm × 15 mm).

l) Glass wool (for extraction thimble).

m) Heating jackets.

n) Microlitre syringe or automatic pipettes.

o) Mini-shaker (also known as vortexer or vortex mixer).

p) Pasteur pipette.

q) Rotary evaporator.

r) Soxhlet extractors:

- 30 ml Soxhlet extractors,
- 250 ml round-bottomed flasks,
- ground-in stopper NS 29/32,
- Dimroth condenser NS 29/32,
- boiling stones (e.g. glass pearls or Raschig rings).

s) Ultrasonic extractors:

Ultrasonic bath with a minimum power of 200 W and a bath area of 706 cm^2, corresponding to 0,28 W/cm^2, without a basket and with an internal or external thermostat.

t) Vial for GC-MS:

2 ml sample vials with 100 µl glass insert and a screw cap with polytetrafluoroethylene (PTFE) gasket or depending on the analytical system, a comparable sample receptacle. Brown or amber vessels shall be used as indicated in the text of the procedure.

u) GC-MS:

A gas chromatograph with a capillary column coupled to a mass spectrometric detector (electron ionization, EI) is used for the analysis. The mass spectrometric detector shall be able to perform selective ion monitoring and have an upper mass range of at least 550 m/z. The use of an autosampler is strongly recommended to ensure repeatability. Ferrules used shall not contain more than 40 % graphite (a suitable ferrule is made of 60 % polyimide and 40 % graphite) to decrease the risk that PAHs are absorbed.

v) GC column for PAH analysis:

A column length of 20 m or longer has sufficient separation efficiency for PAH compounds. An example of suitable column and its separation results is given in Annex A, see Table A.2, Table A.3 and Figure A.1.

For the capillary column, 5 % phenyl, 95 % methyl polysiloxane (e.g. such as HT8, DB-EUPAH and ZB-PAH) is recommended. The preferred dimensions are 20 m in length, 0,25 mm or 0,18 mm in internal diameter, and 0,25 µm or 0,14 µm in film thickness.

NOTE Based on the AfPS-GS-2014-01-PAK method, a nonpolar DB-5MS column is not suited for a separation of the different benzofluoranthenes listed in Table 1.

7 Sampling

As described in IEC 62321-2, unless indicated otherwise (e.g. "using a knife"), cryogenic grinding with liquid nitrogen cooling is recommended and the samples shall be ground to pass through a 500 µm sieve before extraction.

If samples are not tested immediately, they shall be stored in tightly sealed glass vessels and in a cool and dark place.

It shall be confirmed that glassware is thoroughly cleaned and that all new materials that may come into contact with the sample are checked by blank analysis that they give no interference.

NOTE Interferences which can affect the results can occur due to contaminations from glassware, solvents and other materials that can come into contact with the sample. Such interferences will form an artifact or will increase the detector baseline. Interferences can also come from components in samples that co-elute with the specific PAHs of interest.

8 Procedure

8.1 General instructions for the analysis

The following general instructions shall be followed:

The validation of the instrumentation shall include testing of potential cross contaminations between sequential samples. Additional blanks or an inverted sequence of testing will help to identify cross contaminations.

See Annex C for guidance regarding labware cleaning procedures for PAH testing.

To avoid decomposition of PAHs by UV light during extraction and analysis, glass equipment made from brown or amber glass shall be used.

NOTE If no brown or amber glass is available, aluminium foil can be used for protection from light.

8.2 Sample preparation

8.2.1 Ultrasonic extraction

The following steps shall be followed for sample extraction:

The samples shall be pre-cut less than 5 mm × 5 mm and/or milled by cryogenic grinding with liquid N_2 cooling or cut sample materials to 2 mm to 3 mm. Quantitatively transfer 500 mg ± 10 mg of the sample into the vessel (Clause 6 e)).

a) Weigh 500 mg ± 10 mg of the sample into a 40 ml amber vessel (Clause 6 e)). Record the mass to the nearest 0,1 mg.

b) Add 20 µl of the surrogate standard (Clause 5 f)) (100 µg/ml) into the 40 ml amber vessel.

c) Transfer 20 ml of toluene (Clause 5 d)) and 20 µl of internal standard (8.4.4 c)) (100 µg/ml) to the 40 ml amber vessels (Clause 6 e)).

d) Place it in an ultrasonic extractor (Clause 6 s)) and sonicate it for about 1 h at 60 °C and then allow to cool at room temperature after the extraction of the sample.

e) Allow the polymer to settle or filter the mixture through a 0,45 µm PTFE membrane.

8.2.2 Soxhlet extraction

For the Soxhlet extraction step the following procedure is applied:

a) Quantitatively transfer 500 mg ± 10 mg of the sample into a cellulose extraction thimble for Soxhlet extraction. Record the mass to the nearest 0,1 mg.

b) Allow the sample to be transferred through a funnel into the extraction thimble. To ensure a quantitative transfer, the funnel should be rinsed with approximately 10 ml of toluene.

c) 10 µl of the surrogate standard (8.4.5 d)) (50 µg/ml) is added.

d) Cover the thimble with glass wool to prevent the sample from floating.

e) Approximately 120 ml of toluene is used for extraction under reflux. Allow the sample to be extracted for at least 6 h with 6 to 8 cycles per hour. Shorter extraction times may result in lower recoveries of the analyses.

f) After six hours of reflux, the extract is concentrated to about 2 ml using a vacuum rotary evaporator. 10 µl of the internal standard (8.4.4 d)) (50 µg/ml) is then added and the extract is diluted with toluene to 5 ml.

g) The diluted sample is transferred into a 2 ml GC sample/auto sample vial with a PTFE coated seal.

8.2.3 Sample clean-up

If the interference is caused by relatively polar compounds of the same boiling range as the analytes, then multiple column or cartridge clean-ups may be required.

a) The silica gel (Clause 5 c)) is deactivated beforehand by adding 10 % water (the corresponding volume of water is added to the silica gel in a glass flask, and the mixture is homogenized on the rotary evaporator for 1 h at standard pressure and room temperature. The silica gel can then be stored in the sealed glass flask at room temperature).

b) The packed column is conditioned with 10 ml of petroleum ether (Clause 5 g)).

c) The aliquot of toluene extract is then evaporated to a volume of approximately 1 ml on the rotary evaporator and poured into the column.

d) The pointed flask is rinsed out with approximately 20 ml of eluent, which is then also transferred to the clean-up column.

e) Elution is performed with 50 ml of petroleum ether.

f) The collected petroleum ether eluent is amended with 1 ml of toluene and evaporated to a volume of approximately 1 ml under a nitrogen stream (e.g. on the TurboVap).

g) This is then made up to a defined volume with toluene, and the extract is analysed by GC-MS.

8.3 Instrumental parameters

Different conditions might be necessary to optimize a specific GC-MS system to achieve effective separation of all calibration congeners and meet the QC and limits of detection (LOD) requirements. The following parameters have been found suitable and are provided as an example:

a) GC column: a column length of approximately 20 m or longer has sufficient separation efficiency for PAH compounds (see Clause A.2 for an example of suitable column and its separation results). For the capillary column, 5 % phenyl, 95 % methyl polysiloxane (e.g. such as HT8, DB-EUPAH and ZB-PAH) is recommended. The preferred dimensions are length 20 m, internal diameter 0,25 mm or 0,18 mm, and film thickness 0,25 µm or 0,14 µm.

b) Carrier: helium (see Clause 5 b)), 1,0 ml/min, constant flow.

c) Oven: 50 °C (initial temperature), 300 °C (final temperature), 10 °C/min ramp to 300 °C.

d) Injection temperature: 280 °C.

e) Injection volume: 1 µl.

A full scan run using a total ion current ("full scan") MS method for each sample is also recommended for checking for the existence of peaks/congeners not present in the calibration (tentatively identified compounds or "TICS") or not seen in the SIM window. If present, identify the peak and determine the class of compound (e.g. benzo[e]pyrene, benzo[a]pyrene) by evaluation of the total ion spectra.

Table 1 lists GC-MS parameters related to reference masses for the quantification of PAHs. Additional detailed GC-MS instrument parameters are described in Table A.1.

Table 1 – List of reference masses for the quantification of PAHs

Type of PAHs	Ions (m/z) monitored in the extract		
	Target ions (m/z)	Qualifier ions (m/z)	
Internal standard			
Naphthalene-d8	136	108	137
Anthracene-d10	188	178	187
Benzo[a]pyrene-d12	264	260	265
Substances			
Naphthalene	128	102	129
Acenaphthylene	152	76	151
Acenaphthene	154	76	153
Fluorene	166	83	165
Phenanthrene	178	76	179
Anthracene	178	89	176
Fluoranthene	202	101	200
Pyrene	202	101	200
Benzo[a]anthracene	228	114	226
Chrysene	228	114	226
Benzo[b]fluoranthene	252	126	253
Benzo[j]fluoranthene	252	126	253
Benzo[k]fluoranthene	252	126	253
Benzo[e]pyrene	252	126	253
Benzo[a]pyrene	252	126	253
Indeno[1,2,3cd]pyrene	276	138	274
Dibenzo[a,h]anthracene	278	139	276
Benzo[ghi]perylene	276	138	274

8.4 Calibrants

8.4.1 General

All PAH species from naphthalene- to benzo(g,h,i)perylene shall be included in the calibration. The availability of calibration standards for a particular PAH (e.g. benzo(a)pyrene) may vary from region to region. The following Table 2 is an example list of typically available calibration chemicals which are suitable for this analysis.

8.4.2 Stock solution

The following stock solutions shall be prepared:

a) Internal standard (to correct for injection error): 50 µg/ml, 100 µg/ml in toluene (e.g. naphthalene-d8, anthracene-d10 and benzo[a]pyrene-d12).

b) Surrogate standard (to monitor analyte recovery): 50 µg/ml, 100 µg/ml in toluene (e.g. chrysene-d12).

c) A PAH solution can be utilized providing the standard solution concentrations given in 8.5.2 can be achieved.

8.4.3 Preparation of calibration standard

Table 2 – Example list of commercially available calibration chemicals considered suitable for this analysis

Abbreviation	Compound name	CAS number	Formula	Molecular mass (g/mol)
ACE	Acenaphthene	83-32-9	$C_{12}H_{10}$	154,20
ACY	Acenaphthylene	208-96-8	$C_{12}H_8$	152,20
ANT	Anthracene	120-12-7	$C_{14}H_{10}$	178,24
BaA	Benzo[a]anthracene	56-55-3	$C_{18}H_{12}$	228,30
BaP	Benzo[a]pyrene	50-32-8	$C_{20}H_{12}$	252,32
BeP	Benzo[e]pyrene	192-97-2	$C_{20}H_{12}$	252,32
BbF	Benzo[b]fluoranthene	205-99-2	$C_{20}H_{12}$	252,32
BjF	Benzo[j]fluoranthene	205-82-3	$C_{20}H_{12}$	252,32
BkF	Benzo[k]fluoranthene	207-08-9	$C_{20}H_{12}$	252,32
BghiP	Benzo[ghi]perylene	191-24-2	$C_{22}H_{12}$	276,34
CHR	Chrysene	218-01-9	$C_{18}H_{12}$	228,30
DBahA	Dibenzo[a,h]anthracene	53-70-3	$C_{22}H_{14}$	278,35
FLU	Fluoranthene	206-44-0	$C_{16}H_{10}$	202,26
FLN	Fluorene	86-73-7	$C_{13}H_{10}$	166,23
IcdP	Indeno[1,2,3cd]pyrene	193-39-5	$C_{22}H_{12}$	276,34
NP	Naphthalene	91-20-3	$C_{10}H_8$	128,18
PHE	Phenanthrene	85-01-8	$C_{14}H_{10}$	178,24
PYR	Pyrene	129-00-0	$C_{16}H_{10}$	202,26