BS ISO 16000-31:2014

BSI Standards Publication

Indoor air

Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid ester

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National foreword

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Indoor air —

Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid ester

Air intérieur —

Partie 31: Mesurage des ignifugeants basés sur des composés organophosphorés — Ester d'acide phosphorique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

ISO 16000 consists of the following parts, under the general title *Indoor air*:

— *Part 1: General aspects of sampling strategy*

— *Part 2: Sampling strategy for formaldehyde*

— *Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method*

— *Part 4: Determination of formaldehyde — Diffusive sampling method*

— *Part 5: Sampling strategy for volatile organic compounds (VOCs)*

— *Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS or MS-FID*

— *Part 7: Sampling strategy for determination of airborne asbestos fibre concentrations*

— *Part 8: Determination of local mean ages of air in buildings for characterizing ventilation conditions*

— *Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*

— *Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*

— *Part 11: Determination of the emission of volatile organic compounds from building products and furnishing — Sampling, storage of samples and preparation of test specimens*

— *Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)*

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— *Part 13: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) — Collection on sorbent-backed filters*

— *Part 14: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) — Extraction, clean-up and analysis by high-resolution gas chromatography and mass spectrometry*

— *Part 15: Sampling strategy for nitrogen dioxide (NO_2)*

— *Part 16: Detection and enumeration of moulds — Sampling by filtration*

— *Part 17: Detection and enumeration of moulds — Culture-based method*

— *Part 18: Detection and enumeration of moulds — Sampling by impaction*

— *Part 19: Sampling strategy for moulds*

— *Part 21: Detection and enumeration of moulds — Sampling from materials*

— *Part 23: Performance test for evaluating the reduction of formaldehyde concentrations by sorptive building materials*

— *Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials*

— *Part 25: Determination of the emission of semi-volatile organic compounds by building products — Micro-chamber method*

— *Part 26: Sampling strategy for carbon dioxide (CO_2)*

— *Part 28*: *Determination of odour emissions from building products using test chambers*

— *Part 29: Test methods for VOC detectors*

— *Part 30: Sensory testing of indoor air*

— *Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid ester*

— *Part 32: Investigation of buildings for pollutants and other injurious factors — Inspections*

The following parts are under preparation:

— *Part 33: Determination of phthalates with gas chromatography/mass spectrometry (GC/MS)*

— *Part 34: Strategies for the measurement of airborne particles (PM 2,5 fraction)*

— *Part 35: Measurement of polybrominated diphenylether, hexabromocyclododecane and hexabromobenzene*

— *Part 36: Test method for the reduction rate of airborne bacteria by air purifiers using a test chamber*

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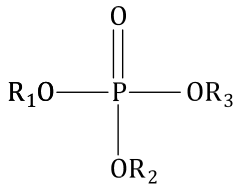
Introduction

In general terms, phosphoric acid esters are formed by the reaction of alcohols or phenols with phosphoryl chloride ($POCl_3$). A distinction is made between mono-, di-, and triesters (see Figure 1). The triesters with aryl and alkyl groups, as well as with mixed halogenated and halogen-free ester groups serve as plasticizers and flame retardants in plastics and varnishes. Parallel therewith, phosphoric acid esters are incorporated in hardeners and accelerants, in mordant and adhesion boosting agents, and serve as cleaning, corrosion inhibition, and adhesion-facilitating substances by the treatment of metal surfaces. They are used as auxiliary means in paper and textiles, as wetting and defoaming agents, emulsifiers, and stabilizers, as additives to cleaning agents and detergents, as not readily flammable hydraulic liquids, and as oil and fuel additives (see Table 1). Not considered in this regard are the groups of the thio- and dithiophosphoric acid esters (pesticides), as well as the phosphoric acid esters with cyanide and halogen groups, with a special impact as acetylcholinesterase inhibitors (nerve gases).[3]



Key

R aryl, alkyl, halogenated, and halogen-free ester groups

Figure 1 — Structural formula of the phosphoric acid esters

Such multiplex applicability of the organophosporus compounds (OPCs) leads to the effect that large indoor areas with OPC-containing building materials, varnishes, paints, floor care products, or fire protection coatings can be encountered. Due to their varying physical and physical-chemical properties, OPC can also be detected in the indoor transfer media (dust and air). As a rule, OPCs boiling at high temperatures such as tris(2-butoxyethyl) phosphate (TBEP, see Annex A) can be found in relevant concentrations in indoor air only if the emission source reaches elevated temperatures. In the case of such sources (e.g. screens or roller blinds for sun protection), the importance of higher temperatures must be taken into consideration when emission chamber tests are carried out.

There is an increasing public interest in the possible health effects of OPCs indoors. For indoor air, a Guideline Value II of 0,05 mg/m^3 was published for TCEP and, in simplified terms, for the sum of TCEP, TCPP, TBP, TBEP, TEHP, and TPP as well. The Guideline Value I derived there amounts to 0,005 mg/m^3.[4] A review article on the occurrence and evaluation of OPC in an indoor environment is available.[5]

This part of ISO 16000 is based on VDI 4301 Part 5.

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Indoor air —

Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid ester

1 Scope

This part of ISO 16000 specifies a test method for the sampling and analysis of the phosphoric acid esters in indoor air and in test chamber air through gas chromatography/mass spectrometry. Both principle methods of the low-resolution mass spectrometry and the high-resolution mass spectrometry are described.

The methods described in this part of ISO 16000 are not suitable for the determination of the phosphoric acid esters in materials.

NOTE ISO 16000-6 can be applied for the determination of the more volatile compounds covered by this standard.

2 Normative references

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.

ISO 16000-1, *Indoor air — Part 1: General aspects of sampling strategy*

ISO 16000-9, *Indoor air — Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*

ISO 16000-13, *Indoor air — Part 13: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) — Collection on sorbent-backed filters*

3 Occurrence

The following compounds belonging to the halogen-free organophosphates are frequently detected in indoor air:

— triphenyl phosphate (TPP);

— tris(2-butoxyethyl) phosphate (TBEP);

— tri-n-butyl phosphate (TBP);

— tris(2-ethylhexyl) phosphate (TEHP);

— cresyl diphenyl phosphate (CDP);

— triethyl phosphate (TEP);

— tricresyl phosphate (TCP).

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The following halogenated phosphoric acid esters are detected predominantly in house dust and indoor air:

— tris(2-chloroethyl) phosphate (TCEP);

— tris(chloropropyl) phosphate (TCPP);

— tris(1,3-dichloro-2-propyl) phosphate (TDCPP).

Bridged diphosphates based on resorcinol- and bisphenol-A are also increasingly implemented as flame retardants, e. g.

— tetraphenyl resorcinol diphosphate (RDP) or

— tetraphenyl bisphenol-A diphosphate (BDP).

So far, there is little information about the release of these compounds from primary sources.

Table 1 provides an overview of the organophosphates frequently detected in indoor air. The nomenclature and the chemical and physical data on the organophosphates are listed in Annex A.

Table 1 — Organophosphates and their intended use (see References[5] and[6])

OPC Abbreviation	Organophosphates	Intended use as flame retardant and plasticizer
CDP	Cresyl diphenyl phosphate	Flame retardant coatings
RDP	Tetraphenyl resorcinol diphosphate	Casing materials, special cables
TBEP	Tris(2-butoxyethyl) phosphate	Floor care products, floor wax, additives in elastomers, solvents for resins, antifoaming agents, additives for plastic, synthetic rubber, and varnishes
TBP	Tri-n-butyl phosphate	Defoaming agents for concrete additives, wall paper, dispersion paints, varnishes, rubber, plastic, and vinyl resins, antifoaming agents
TCEP	Tris(2-chloroethyl) phosphate	Paints, coatings, soundproofing ceilings, fibre glass wall paper, expanding foam, casing materials
TCPP	Tris(1-chloro-2-propyl)-phosphate[a]	Sound-absorbing polyurethane foam, expansion foam, soft foam (seats, mattresses), rear lining of textile, casing materials
TDBPP	Tris(2,3-dibromo-propyl) phosphate	Foamed rubber, varnishes, paper, paints, cellulose-, triacetate-, and polyester fabrics, carpets, resins, furniture, car interior details
TDCPP	Tris(1,3-dichloro-2-propyl) phosphate	PU foam, casing materials, rear lining of textile
TEHP	Tris(2-ethylhexyl) phosphate	Stabilizers, fat additives, additives for synthetic rubber
TCP	Tricresyl phosphate	Lubricants, car interiors, additives to floor covering and wall cladding
TPP	Triphenyl phosphate	Expanding foam, paints and varnishes, brighteners, casing materials, photographic films, phenol resins, additives for lubricants, car interior details

[a] Technical TCPP does not exist as a pure isomer. Hence it usually leads to up to three GC signals.

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The published concentrations of indoor air measurements are compiled in Table 2. Citing of occasion-relevant measurement values cannot be excluded.

Table 2 — Published results on OPC in indoor air

Substance	Concentration in indoor air µg/m³		
	Range	Median	Reference
TCEP	< 0,005 to 6	0,10 to 0,38	[8][9][10]
TCPP	0,019 to 0,058	–	[10]
TBP	0,01 to 0,064	–	[10]
TBEP	0,001 to 0,03	–	[9] and[10]
TEHP	< 0,001 to 0,01	–	[10]
TPP	< 0,01	–	[9] and[10]
TCP	< 0,01	–	[9]

4 Indoor air

4.1 Measurement planning

The measurement planning for the determination of the indoor air for phosphoric acid esters is performed according to the requirements of ISO 16000-1.

4.2 Apparatus and materials for sampling and sample preparation

4.2.1 Sampling apparatus

The sampling system is described in detail in ISO 16000-13.

4.2.1.1 Sampling head, with a sleeve for holding the PU foams (Figures 2a and 2b) (see also ISO 16000-13).

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