
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

Air intérieur —

*Partie 13: Dosage des polychlorobiphényles (PCB) de type dioxine et des polychlorodibenzo-*p*-dioxines (PCDD)/polychlorodibenzofuranes (PCDF) totaux (en phase gazeuse et en phase particulaire) — Collecte sur des filtres adsorbants*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 16000-13 was prepared by Technical Committee 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 — 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/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)*
- *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 (NO₂)*
- *Part 16: Detection and enumeration of moulds — Sampling by filtration*
- *Part 17: Detection and enumeration of moulds — Culture-based method*
- *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 compounds and carbonyl compounds without formaldehyde concentrations by sorptive building materials*

The following parts are under preparation:

- *Part 18: Detection and enumeration of moulds — Sampling by impaction*
- *Part 19: Sampling strategy for moulds*
- *Part 25: Determination of the emission of semi-volatile organic compounds for building products — Micro-chamber method*
- *Part 26: Measurement strategy for carbon dioxide (CO₂)*
- *Part 28: Sensory evaluation of emissions from building materials and products*

The following parts are planned:

- *Part 20: Detection and enumeration of moulds — Sampling from house dust*
- *Part 21: Detection and enumeration of moulds — Sampling from materials*
- *Part 22: Detection and enumeration of moulds — Molecular methods*
- *Part 27: Standard method for the quantitative analysis of asbestos fibres in settled dust*

Furthermore,

- ISO 12219-1^[2] (under preparation), *Indoor air — Road vehicles — Part 1: Whole vehicle test chamber — Specification and method for the determination of volatile organic compounds in car interiors*,
- ISO 16017-1^[3], *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography — Part 1: Pumped sampling, and*
- ISO 16017-2^[4], *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography — Part 2: Diffusive sampling*

focus on volatile organic compound (VOC) measurements.

Introduction

The different parts of ISO 16000 describe general requirements relating to the measurement of indoor air pollutants and the important conditions to be observed before or during the sampling of individual pollutants or groups of pollutants as well as the measurement procedures themselves (see Foreword).

This part of ISO 16000 is applicable to the collection from indoor air of dioxin-like polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) and to the preparation of sampling media required for sampling. The same sampling methods described here are also applicable to polycyclic aromatic hydrocarbons (PAHs) made up of two or more fused aromatic rings. Procedures for preparation and extraction of sampling media for PAHs and for the analytical determination of PAHs are given in ISO 12884, and therefore are not included here.

The sampling strategy to analyse PCBs, PCDDs/PCDFs and PAHs in indoor air is described in ISO 16000-12.

Several PCBs and PCDDs/PCDFs are considered to be potential human carcinogens. There are 209 individual PCBs (congeners), 75 PCDDs and 135 PCDFs. The most toxic PCBs are those that are coplanar and structurally similar to PCDDs. The most toxic PCDD is 2,3,7,8-tetrachlorodibenzodibenzo-*p*-dioxin (2,3,7,8-TCDD).

PCBs are emitted into the indoor air primarily from concrete sealers, certain paints, or electrical capacitors, all of which have been banned in recent years. The principal sources of PCDDs/PCDFs in indoor air are impurities in wood preservatives containing pentachlorophenol (PCP) and emissions from fires involving chlorinated products. Tracked-in soil and emissions from nearby landfills and abandoned industrial sites may also contribute PCBs and PCDDs/PCDFs to the indoor environment.

Total PCB concentrations (six marker PCBs: PCB-28, -52, -101, -138, -153, and -180 multiplied by 5) in urban outdoor air typically range from 10 to several hundred picograms per cubic metre (pg/m³). PCDDs/PCDFs are usually found in urban outdoor air at extremely low concentrations; e.g. femtograms per cubic metre (fg/m³) to picograms per cubic metre (pg/m³). PCBs and PCDDs/PCDFs may be distributed between the gas and particle-associated phases in ambient or indoor air, depending on the temperature, humidity, degree of chlorination, their concentration, and their capacity to associate with suspended particulate matter. These compounds, especially those having vapour pressures above 10⁻⁸ kPa, will tend to vaporize from particle filters during sampling. Consequently, a back-up sorbent trap is included for efficient sampling. Separate analyses of the filter and sorbent trap will not reflect the original atmospheric phase distributions at normal ambient temperatures because of volatilization of compounds from the filter and should not be attempted.

Shipping of PCDD/PCDF standards has to comply with the national legal regulations. They have to be transported in special containers, which are commercially available. Handling should only be done by trained operators.

This part of ISO 16000 describes sampling of polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs). Extraction, clean-up and analysis are described in ISO 16000-14.

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

1 Scope

This part of ISO 16000 specifies sampling and preparation of sampling media for dioxin-like polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in indoor air (see Table C.1). The method incorporates a sampling procedure that collects PCBs and PCDFs/PCDDs from air on a fine-particle filter backed up by a sorbent trap. The method cannot be used to separately determine gaseous PCBs and PCDFs/PCDDs and those that are particle-associated because some of the compounds will volatilize from the filter and be collected by the sorbent trap. This part of ISO 16000 does not address analytical methods for PCBs, PCDDs and PCDFs, which are included in ISO 16000-14. (ISO 16000-13 and ISO 16000-14 are both parts of the overall measurement procedure for the determination of PCBs and PCDDs/PCDFs.)

Combined with the analytical procedures specified in ISO 16000-14, which incorporate specific analyses by high-resolution gas chromatography combined with high-resolution mass spectrometry (HRGC/HRMS), this method is capable of detecting 0,2 pg/m³ or lower concentrations of most PCBs and PCDFs/PCDDs. However, it may not be possible to detect all analytes at 0,2 pg/m³ or lower, especially at lower sampling rates. To achieve adequate detection limits, no less than 50 m³ of air should be sampled. For normal measurement tasks, sampling volumes up to 360 m³ are employed (equivalent to a sampling period of 24 h at 16 m³/h or 7 d at 2 m³/h). The method has been validated for sampling periods up to seven days. With the use of larger sorbent traps, sampling volumes up to 1 000 m³ have been validated.

Sampling precision under normal conditions can be expected to be ± 25 % or better and uncertainty ± 50 % or better.

If the appropriate restrictions are applied, this method can be used for small rooms, for example children's rooms as well as for offices.

2 Normative references

The following referenced documents are indispensable for the application 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.

ISO 12884, *Ambient air — Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons — Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analyses*

ISO 16000-12, *Indoor air — Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)*

ISO 16000-14, *Indoor air — 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*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

sampling efficiency

$\% E_s$

ability of the sampler to trap and retain targeted compounds

NOTE The $\% E_s$ is the percentage of the analyte of interest collected and retained by the sampling medium when it is introduced into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use.

3.2

dynamic retention efficiency

E_r

ability of the sampling medium to retain a given PCB, PCDD or PCDF that has been added to the sorbent trap in a spiking solution when air is drawn through the sampler under normal conditions for a period of time equal to or greater than that required for the intended use

3.3

dioxin-like PCB

non- and mono-*ortho* PCB having an affinity to the aryl hydrocarbon (Ah) receptor, showing similar toxicological effects as the 2,3,7,8-substituted PCDDs/PCDFs according to WHO

NOTE 1 See Reference [5].

NOTE 2 See also Tables C.1 and C.2.

3.4

marker PCB

one of six PCBs

NOTE The six marker PCBs are PCB-28, PCB-52, PCB-101, PCB-138, PCB-153, and PCB-180.

3.5

spiking

addition of $^{13}\text{C}_{12}$ -labelled PCB standards

3.6

operational performance characteristic

measurement that deals with the influence of the physical and chemical environment and maintenance problems

EXAMPLE Main line voltage, temperature, supply of certain substances, set-up time, period of unattended operation (see Reference [1]).

3.7

field blank

unexposed but spiked sample of the sampling medium [e.g. filter, polyurethane foam (PUF) trap, or complete sampling cartridge], that is carried to the field and through the complete analytical procedure, including the extraction, clean-up, and identification steps

NOTE The measurement value is needed to ensure that no significant contamination has occurred during all steps of the measurement process and to check that the operator can achieve a quantification level adapted to the task.

3.8**analytical blank**

unexposed but spiked sample of a reagent or sampling medium that is carried through the complete analytical procedure including the extraction, clean-up and identification steps

3.9**sampling standard**

marker agent that is added to a sampling medium before sampling to determine the overall efficiency of the method

EXAMPLE $^{13}\text{C}_{12}$ -labelled PCB and PCDD/PCDF.

3.10**extraction standard**

marker agent, added to a sampling medium before extraction and used for calculation of results

EXAMPLE $^{13}\text{C}_{12}$ -labelled PCB and PCDD/PCDF.

3.11**congener**

substance which belongs to the chemical group of PCB or PCDD or PCDF

NOTE Includes the 209 individual PCBs, 75 individual PCDDs, and 135 individual PCDFs.

3.12**isomer**

PCB or PCDD or PCDF with identical elemental composition but different structure

EXAMPLE 1-chlorobiphenyl and 2-chlorobiphenyl are isomers.

4 Abbreviated terms

(See also Tables C.1 and C.2.)

PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo- <i>p</i> -dioxin
PCDF	polychlorinated dibenzofuran
TeCB	tetrachlorobiphenyl
PeCB	pentachlorobiphenyl
HxCB	hexachlorobiphenyl
HpCB	heptachlorobiphenyl
TCDD	tetrachlorodibenzo- <i>p</i> -dioxin
PeCDD	pentachlorodibenzo- <i>p</i> -dioxin
HxCDD	hexachlorodibenzo- <i>p</i> -dioxin
HpCDD	heptachlorodibenzo- <i>p</i> -dioxin
OCDD	octachlorodibenzo- <i>p</i> -dioxin

TCDF	tetrachlorodibenzofuran
PeCDF	pentachlorodibenzofuran
HxCDF	hexachlorodibenzofuran
HpCDF	heptachlorodibenzofuran
OCDF	octachlorodibenzofuran
PTFE	polytetrafluoroethylene

5 Principle

PCBs, PCDDs, and PCDFs are collected directly from indoor atmospheres by pulling a sample of air through a fine particle filter backed up with a sorbent trap consisting of polyurethane foam (PUF). Low-volatility compounds associated with airborne particulate matter are collected primarily on the particle filter. Gaseous compounds that pass through the filter and compounds that vaporize from the filter are collected by the polyurethane foam trap.

The method is applicable to rooms in residences, office buildings, and public or commercial facilities including those with large interior spaces (e.g. factories, warehouses, enclosed stadiums). The sampling flow rate and volume of air collected will depend on the size of the room or interior space being sampled. It may range from 1,5 m³/h to 16 m³/h according to the measurement task.

The sampling media (filter plus PUF) are combined and extracted together. Extraction procedures are normally based on Soxhlet extraction with toluene or an equivalent solvent. ¹³C₁₂-labelled PCB/PCDD/PCDF standards are added before sampling and extraction to monitor sampling efficiency and recovery. Spiking with ¹³C₁₂-labelled PCBs/PCDDs/PCDFs before sampling is necessary to determine the overall recoveries of the PCB/PCDD/PCDF congeners. Losses during extraction and clean-up are detected and compensated by using these isotopically-labelled surrogates as internal extraction standards for quantification, together with recovery standards that are added just before the HRGC/HRMS analysis.

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6 Apparatus and materials

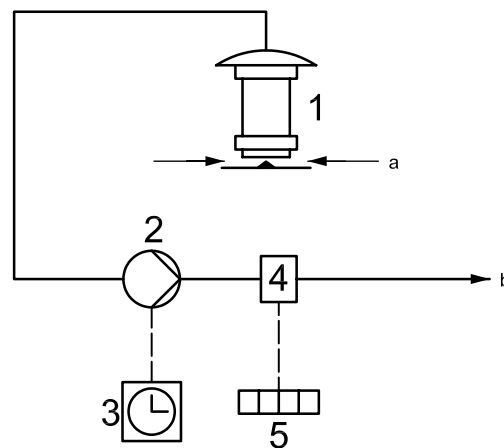
6.1 Sampling apparatus

A schematic of a typical sampling system is shown in Figure 1 and that of a typical sampling module is shown in Figure 2.

The sampling module shown in Figure 2 is the basis of the outdoor sampler for polycyclic aromatic hydrocarbons (PAHs) described in ISO 12884. It is based on a published design (Reference [6]) and is also used for determination of PCDDs and PCDFs in ambient air (Reference [7]). A module of this design having the same dimensions as those described in ISO 12884 may be used for sampling rates from 1,5 m³/h to 16 m³/h and total air volumes up to 360 m³ depending on the capacity of the air sampling system used. Modules of this basic design having various dimensions and configurations may be used if validated.

NOTE The particle capture velocity of a sampler will depend on the face velocity during sampling. For example, it can be estimated by Stokes Law that pulling air through a 100 mm filter at 15 m³/h will collect particles smaller than about 40 µm. At a sampling rate of 6 m³/h, particles less than about 20 µm would be captured. Reducing the sampling rate further to 1,5 m³/h would collect particles smaller than 10 µm to 12 µm. Experience shows for indoor air measurements that there is little or no significant differences in what is measured with the same sampling module at different flow rates down to 1,5 m³/h. Likewise, if a size-selective inlet (e.g. PM_{2,5} or PM₁₀) is used, there should be little effect on the total PCB/PCDD/PCDF content of the sample unless there is a nearby emission source of particle-associated compounds.

There are a number of sampling systems capable of providing air flows from 1,5 m³/h to 16 m³/h, some of which may have air flow controls that can be set over a wide range to provide the desired sampling rate. Limitations on permissible air sampling rates and volumes are discussed in Clause 8. Some examples of sampling systems are presented in Annex A.



Key

- 1 sampling module (see Figure 2)
- 2 air sampling pump
- 3 timer
- 4 flow controller (with measuring electronics)
- 5 elapsed time counter

- a Air intake.
- b Exhaust.

Figure 1 — Schematic drawing of a typical sampling system

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