
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.

6 Apparatus and materials

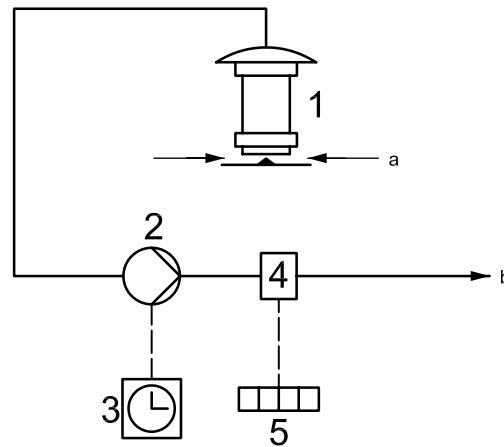
6.1 Sampling apparatus

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.

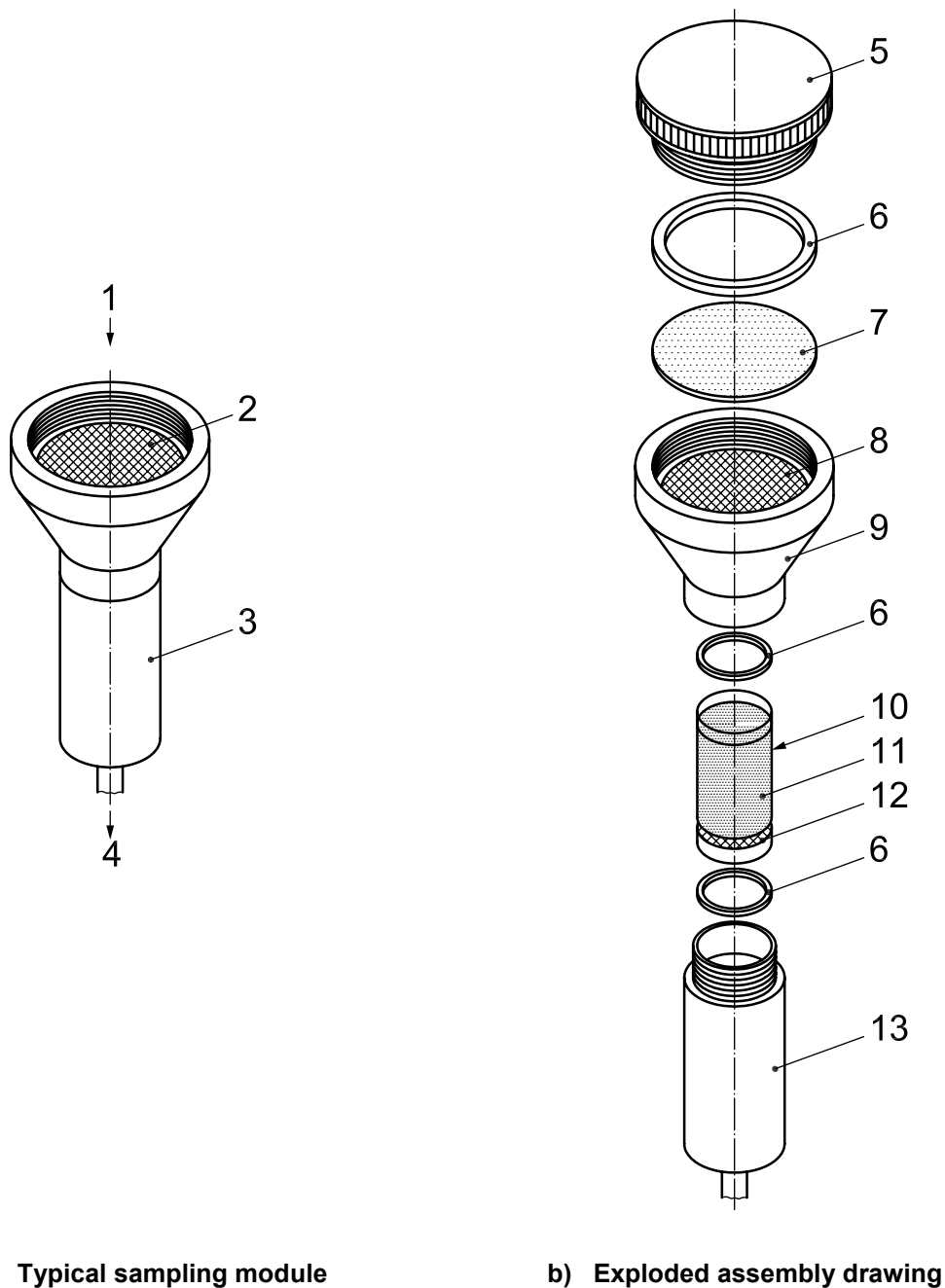
There are a number of sampling systems capable of providing air flows from 1,5 m³/h to 16 m³/h, some of which may have air flow controls that can be set over a wide range to provide the desired sampling rate. Limitations on permissible air sampling rates and volumes are discussed in Clause 8. Some examples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



a) Typical sampling module

b) Exploded assembly drawing

Key

- | | | | |
|---|--|----|------------------------------------|
| 1 | air flow inlet | 8 | filter support screen |
| 2 | filter support screen (identical to 8) | 9 | filter holder |
| 3 | assembled sampling module | 10 | glass sorbent cartridge (PUF trap) |
| 4 | air flow exhaust | 11 | PUF trap |
| 5 | filter retainer | 12 | support screen cartridge holder |
| 6 | sealing gaskets | 13 | cartridge holder |
| 7 | quartz- or glass-fibre filter | | |

Figure 2 — Typical sampling module that integrates particle filter and PUF trap

6.2 Sampling materials

6.2.1 Polyurethane foam, open-cell, polyether type, density 22 mg/cm³, cut into cylinders 76 mm long × 62 mm diameter, or other appropriate size depending on the specific sampling module used. They are available from several commercial sources. The PUF cylinders should be slightly larger in diameter than the internal diameter of the sorbent cartridge so that the sampled air does not flow around it instead of through it. Cleaning is performed according to Clause 7.

6.2.2 Filter, micro-quartz- or glass-fibre, binderless, acid-washed, with a filtration efficiency of 99,99 % mass fraction or better for particles below 0,5 µm, or other appropriate size filter depending on the specific sampling module used. This efficiency shall be certified by the filter supplier.

6.2.3 ¹³C-labelled sampling standards, see Tables 1 and 2 for composition.

6.2.4 Aluminium foil, for packing the filters and PUF traps before and after sampling.

6.2.5 Forceps and latex or neoprene gloves, for handling the filter and PUF traps.

6.2.6 Microlitre syringes, for applying recovery standards on the filter media.

6.2.7 PTFE tape, for sealing the suction tube in the screw-in port of the protective housing.

6.2.8 Glass jar or other suitable containers, sealable, for storing and transporting PUF traps and particle filters.

6.2.9 Desiccator or vacuum drying cabinet.

6.2.10 Diaphragm pump or other suitable pump.

6.3 Analytical reagents

6.3.1 Toluene, glass distilled, chromatographic or pesticide quality.

6.3.2 *n*-hexane, glass distilled, chromatographic or pesticide quality.

6.3.3 Dichloromethane, glass distilled, chromatographic or pesticide quality.

6.3.4 Acetone, glass distilled, chromatographic or pesticide quality.

6.3.5 *n*-nonane, glass distilled, chromatographic or pesticide quality.

6.3.6 *n*-tetradecane (keeper), glass distilled, chromatographic or pesticide quality.

6.3.7 ¹³C₁₂-labelled extraction standards, see Tables 1 and 2 for composition (see 6.4).

6.3.8 ¹³C₁₂-labelled injection standards, see Tables 1 and 2 for composition (see 6.4).

6.4 ¹³C₁₂-labelled standards

The masses of ¹³C₁₂-labelled sampling standards in 100 µl of appropriate solvent (e.g. toluene or 4 % *n*-tetradecane/*n*-nonane) that should be added to each sample at a concentration level of approximately 100 fg of TEQ/m³ for an approximately 180 m³ of sampling volume are listed in Table 1 (PCDDs/PCDFs) and Table 2 (PCBs). For TEQ and WHO-TEQ_{PCB}, see Clause C.3.

The extraction standards should be added to the various sampling media immediately after the samples are received in the laboratory. The ¹³C₁₂-labelled congeners are used for quantification because they behave exactly like the extracted native PCBs/PCDDs/PCDFs during the clean-up, due to their nearly identical chemical and physical properties. The injection standards (see Table 1) are for determining the recovery rates. The masses specified in Table 1 and Table 2 of standards to be used shall to be adjusted appropriately if considerably higher masses of native PCBs/PCDDs/PCDFs are expected in the sample. The use and handling of the extraction and recovery standards are described in ISO 16000-14.

Table 1 — ¹³C₁₂-labelled 2,3,7,8-PCDD/PCDF congeners for addition to samples before sampling, extraction and GC injection to measure approximately 100 fg TEQ/m³ and approximately 180 m³ sampling volume

Solution:	Solution to be added before:		
	Sampling (sampling standard)	Extraction (extraction standard)	GC-Injection (recovery standard)
Total volume in µl: (e.g. in toluene, <i>n</i> -nonane)	100	100	25
Congeners added	Total mass pg		
¹³ C ₁₂ -2,3,7,8-TCDF		25 ^a	
¹³ C ₁₂ -1,2,3,4-TCDD			25
¹³ C ₁₂ -2,3,7,8-TCDD		25 ^a	
¹³ C ₁₂ -1,2,3,7,8-PeCDF	25		
¹³ C ₁₂ -2,3,4,7,8-PeCDF		25 ^a	
¹³ C ₁₂ -1,2,3,7,8-PeCDD		25 ^a	
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF		25 ^a	
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF		25	
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	25		
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF		25	
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD		25	
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD		25 ^a	
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD			25
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF		50 ^a	
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	50		
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD		50 ^a	
¹³ C ₁₂ -OCDF		50	
¹³ C ₁₂ -OCDD		50	

^a These standards are used to quantify the remaining congeners of the associated groups of chlorinated homologues for which no standard was added.

Table 2 — $^{13}\text{C}_{12}$ -labelled PCBs congeners to be added to the sample at different stages of the procedure for measurement of about 0,01 ng WHO-TEQ_{PCB}/m³ assuming 180 m³ of sampling volume

Solution: Total volume in µl: (e.g. in toluene, <i>n</i> -nonane)	Total mass to be added before:		
	Sampling (sampling standard)	Extraction (extraction standard)	GC-Injection (recovery standard) ^a
	100	100	at least 10
Congeners added	Total mass pg		
$^{13}\text{C}_{12}$ -2,3,4,4'-TeCB (60)	3 600		
$^{13}\text{C}_{12}$ -3,3',4,5,5'-PeCB (127) ^b	3 600		
$^{13}\text{C}_{12}$ -2,3,3',4,5,5'-HxCB (159)	3 600		
$^{13}\text{C}_{12}$ -3,4,4',5-TeCB (81)		3 600	
$^{13}\text{C}_{12}$ -3,3',4,4'-TeCB (77)		3 600	
$^{13}\text{C}_{12}$ -3,3',4,4',5-PeCB (126)		3 600	
$^{13}\text{C}_{12}$ -3,3',4,4',5,5'-HxCB (169)		3 600	
$^{13}\text{C}_{12}$ -2,3,3',4,4'-PeCB (105) ^b		3 600	
$^{13}\text{C}_{12}$ -2,3,4,4',5-PeCB (114)		3 600	
$^{13}\text{C}_{12}$ -2,3',4,4',5-PeCB (118)		3 600	
$^{13}\text{C}_{12}$ -2',3,4,4',5-PeCB (123)		3 600	
$^{13}\text{C}_{12}$ -2,3,3',4,4',5-HxCB (156)		3 600	
$^{13}\text{C}_{12}$ -2,3,3',4,4',5,5'-HxCB (157)		3 600	
$^{13}\text{C}_{12}$ -2,3',4,4',5,5'-HxCB (167)		3 600	
$^{13}\text{C}_{12}$ -2,3,3',4,4',5,5'-HpCB (189)		3 600	
$^{13}\text{C}_{12}$ -2,3',4',5-TeCB (70)			3 600
$^{13}\text{C}_{12}$ -2,3,3',5,5'-PeCB (111)			3 600
$^{13}\text{C}_{12}$ -2,2',3,3',4,4',5-HpCB (170)			3 600

^a Table 2 shows a selection of available $^{13}\text{C}_{12}$ -labelled PCBs suitable as recovery standards. At least one shall be added for each dioxin-like PCB containing fraction.

^b Attention should be paid to possible co-elution problems of PCB 127 and PCB 105 on certain commercially available columns

7 Preparation of sampling media

Heat the particle filters before use at 250 °C (glass) or at 400 °C (quartz) for 5 h, wrap them in *n*-hexane-rinsed aluminium foil, and place them in clean, tightly-sealed containers (e.g. glass Petri dishes or wide-mouth glass jars tightly sealed with PTFE tape) for storage and transport to and from the monitoring site. Gently handle the filters with clean forceps.

Extract PUF cylinders for 24 h with acetone in a Soxhlet extractor at approximately 4 cycles/h, then for another 24 h with toluene in a Soxhlet extractor at approximately 4 cycles/h, and afterwards conduct a short extraction with pentane for easy drying. Dry in a desiccator under vacuum and a gentle stream of high-purity nitrogen or in a vacuum drying cabinet at 40 °C.

The extract from the Soxhlet extraction procedure from each batch may be analysed to determine initial cleanliness prior to certification.

For storage, place the cleaned PUF plug in *n*-hexane-rinsed aluminium foil. For sampling preparation, the PUF plug is placed into the glass sampling cartridge using polyester or latex rubber gloves and PTFE-coated forceps. The sampling cartridge is wrapped with *n*-hexane-rinsed aluminium foil, placed in a clean container, and tightly sealed for storage and transport to and from the monitoring site.

If desired, the loaded glass sampling cartridge and filter may be placed in the sampling module, which is in turn placed in a clean container, and tightly sealed for storage and transport to and from the monitoring site.

The PUF cylinders may be reused if properly cleaned with toluene after each use. The number of possible uses before significant deterioration of performance occurs has not been determined, but it should not be used more than six times without verifying that the performance is unchanged.

8 Sampling

Before sampling, the boundary conditions to be taken into account shall be defined and noted as described in ISO 16000-12.

Choose a suitable pumping system and sampling module according to the measurement task (see Annex A). The hourly air flow rate of the sampling apparatus should not exceed 10 % of the air change rate of the room in question. If the air change rate is unknown, the air volume sampled per hour shall not exceed 10 % of the volume of the room. Low-volume sampling can be used for small rooms, for example children's rooms. High-volume sampling may be considered for very large indoor areas if the noise produced by the sampler is of no consequence, provided that the constraints on sampling rate or air volume specified above are observed. Conduct a leak check before and after sampling (see Clause 9). Plug the nozzle and evacuate the sampling module to the minimum pressure used during sampling: the volume flow rate shall be less than 5 % of the normal flow rate.

Take a field blank before each sampling campaign.

Confirm the efficiency of the sampler for the targeted PCBs/PCDDs/PCDFs under the conditions anticipated in the field prior to the initiation of any sampling programme. Determination of the efficiency is particularly important if sampling periods exceeding 24 h are planned. Acceptable performance may be established by determining sampling efficiency, E_s , directly or estimating it from the dynamic retention efficiency, E_r . Refer to Annex B for instructions on determining sampling efficiency.

When the sampling module is being loaded with particle filter and PUF trap, take special care to ensure that there is no contamination, e.g. by skin oils (use gloves and forceps). Take special care that PUF trap is seated properly in the module, and that channels and cavities do not form. Carefully seal the sampling module (e.g. with PTFE tape) to ensure that it is leak-proof.

Before placing the particle filter holder [see part 9 in Figure 2 b)] on the sampling module, use a micro-litre syringe to spike the surface of inlet face of the PUF sorbent with 50 pg each of $^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF, $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDF and 100 pg of $^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF as well as $^{13}\text{C}_{12}$ -2,3,4,4'-TeCB, $^{13}\text{C}_{12}$ -3,3',4,5,5'-PeCB and $^{13}\text{C}_{12}$ -2,3,3',4,5,5'-HxCB all in 100 μl of toluene containing 4 % *n*-tetradecane as keeper, and keep it cool before use. The spiking standard can also be added to the PUF sorbent in the laboratory shortly before sampling is performed (not more than 24 h before sampling), provided that the PUF media are kept refrigerated to minimize losses prior to sampling. This procedure may avoid contamination on site and safety issue that may prohibit application on site.

Monitor the airflow through the sampling system monitored by a flow-control device or devices. Conduct a multi-point calibration of the flow-control system every six months using a standard audit calibration orifice, which is temporarily attached to the inlet of the sampler. Alternatively, use a high-flow dry gas metre if it has been validated as a transfer standard.

The sampler shall be calibrated:

- a) when new;
- b) after major repairs or maintenance;
- c) whenever any audit point deviates from the calibration curve by more than 7 %; or
- d) at the frequency specified in the user's manual.

Calibration and re-calibration of the gas meter in the field is performed according to laboratory quality standards, e.g. using a calibrated orifice flow rate transfer standard.

Under the unlikely conditions of very high airborne dust loadings, the air flow rate through the filter can decrease markedly during the course of sampling, especially if the flow rate is not automatically controlled. Even with flow-controlled pumps, the filter load can become so high that the apparatus may switch off automatically. Therefore, the filter should be changed if the air flow rate changes by more than 10 % of the initial value. The new filter shall also be spiked with the sampling standards.

If it is desired to correct the sampling volume to standard conditions of pressure and temperature (1 013,25 hPa; 273,15 K), use the mean temperature and mean air pressure measured during sampling.

Take special care that the sampling system is installed so that it is not directly influenced by draughts, etc. To ensure representative sampling, take care that the air change rate in the room during the sampling period is similar to that during normal use of the room. To minimize a possible noise nuisance due to the sampling apparatus, it can be installed in a side room or outdoors. In this case, connect the sampling module to the pumping system unit using a tube connection (e.g. PVC tubular fabric) via a hose connector. Note, however, that the longer the tubing, the greater the pressure drop and burden placed on the pumping system. Smaller pumping systems may be silenced by placing them in polyurethane or polystyrene containers such as ice chests. However, care should be exercised that the systems do not overheat.

To determine a representative average value of the PCB/PCDD/PCDF concentrations in air, it may be necessary to carry out sampling over relatively long periods. For normal measurement, tasks sampling volumes shall not exceed 360 m³ (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. In special cases sampling volumes of high-volume samplers up to 1 000 m³ are validated.

After sampling, record the sampling duration, wrap the sampling module in aluminium foil and keep it cool until it is returned to the laboratory. The filter and PUF trap should be removed from the sampling module in the laboratory and placed in tightly-sealed glass or metal containers and stored at 4 °C or lower temperatures until analysis. Keep the storage time as short as possible, in no case longer than two weeks. Alternatively, if special care is taken to avoid contamination, the PUF cartridge and filter may be removed from the sampling module and placed into the storage containers at the sampling site.

Sampling periods in excess of 24 h should be avoided unless necessary to achieve the necessary limit of detection. Rather than prolonging the sampling period, the use of a sampler with a higher sampling rate should be considered, provided that its use will not upset the ventilation rate of the room or interior space being monitored or present an unacceptably intrusive situation with respect to noise production or space consumption. Modification of the determinative steps, such as carefully increasing concentration of the extract or using more sensitive MS techniques, may also enable lower detection limits.

Extraction, clean-up and analytical procedures are described in ISO 16000-14.

9 Minimum requirements for sampling

The following minimum requirements shall be fulfilled for the determination of PCB/PCDD/PCDF concentrations.

- a) A leak check shall be carried out before and after every sampling procedure. The sampling module with its intake sealed shall be evacuated to the pressure used during sampling and the volume flow rate shall be less than 5 % of the normal flow rate.
- b) A field blank shall be taken before each sampling campaign.
- c) The $^{13}\text{C}_{12}$ -labelled standards shall be used as described in Tables 1 and 2.
- d) The sampling standard solution shall contain 4 % tetradecane as a keeper.
- e) The recovery rate for each sampling standard shall be greater than 50 % calculated on the basis of the relevant extraction standard.
- f) The PCB/PCDD/PCDF concentrations in the field blank shall be more than a factor of 5 below the lowest measurement values (expressed in TEQ) assuming the same volume as for sampling. If the calculated value measured is less than the preceding field blank value, the reported result is defined as less than or equal to the blank.

Annex A (informative)

Details of samplers

A.1 General

Examples of several sampling systems that may be used to efficiently collect PCBs, PCDDs and PCDFs from indoor air are presented in this annex. All of these samplers are based on the principles described in Clause 5 and in 6.1. The sampling media in all cases consist of a glass- or quartz-fibre particle filter backed up by a PUF trap. In each case, the PUF traps are held in cylindrical glass or stainless steel sampling cartridges.

The diameter of the PUF plug should be about 2 mm to 5 mm larger than the internal diameter of the cartridge so that it is slightly compressed. If uncompressed, air may flow between the glass cylinder walls of the cartridge and the PUF, resulting in sampling errors. This can be easily done without compressing the foam lengthwise if the foam is pushed into the cylinder, then pulled back slightly.

When available, particle size-selective inlets (e.g. PM_{2,5} or PM₁₀), may be used if desired.

The user may substitute other samplers but shall meet the requirements of this part of ISO 16000.

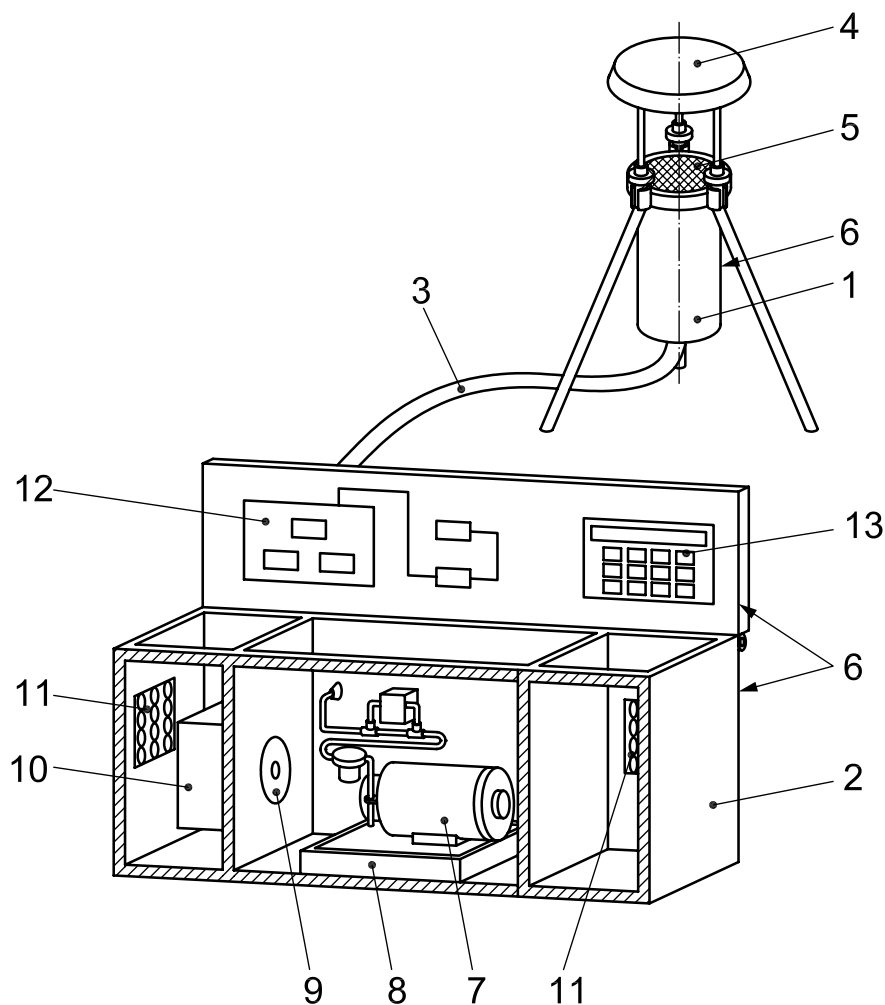
A.2 Low-volume samplers

A.2.1 US EPA method IP-7 low-volume sampler

The sampler shown in Figure A.1 is designed to operate at 1,2 m³/h to 1,6 m³/h and is quiet enough for use in occupied dwellings and offices. The sampler consists of an air pump and associated controls housed in an acoustically-insulated compartment and connected by way of a hose to a sampling module that holds a circular glass- or quartz-fibre filter backed by a glass sorbent cartridge containing a cylindrical PUF plug (see Figure A.2). It is described in References [8] and [9].

Any pump providing the prescribed air flow rate may be used, but it shall be sound-insulated for use in occupied dwellings or offices. When operated at 1,2 m³/h, the pumping system described in References [7] and [8] meets preferred noise criterion (PNC) 39 dB (less than 45 dB) (see Reference [10]), which is typical of background noise in living areas and private offices. However, other commercially available air sampling pumps that are capable of sampling at 1,2 m³/h to 1,6 m³/h are nearly as quiet, and noisier pumps may be quieted by placing them in sound-insulated boxes such as polystyrene foam coolers provided that they do not overheat.

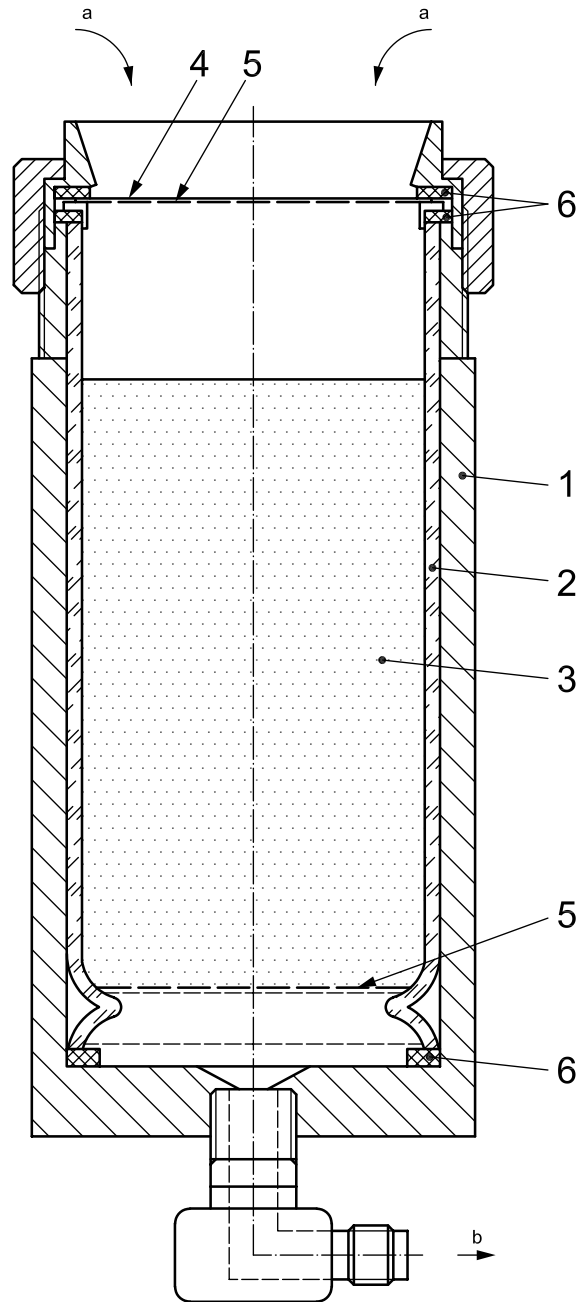
Sampling periods of up to seven days may be required to collect sufficient samples to quantify low levels of PCBs, PCDDs, and PCDFs in indoor air.



Key

- 1 sampling module (see Figure A.2)
- 2 pumping unit in acoustically-insulated box, 51 cm × 35 cm × 21 cm
- 3 sample hose line
- 4 dust cover
- 5 portable filter support screen
- 6 fibreglass acoustic insulation
- 7 holder
- 8 vacuum pump
- 9 flow transducer
- 10 fan
- 11 vents
- 12 electronic controls for flow control, etc.
- 13 seven-day programmable timer

Figure A.1 — Low-volume indoor air sampler with acoustic enclosure



Key

- 1 aluminium sampling module, cylinder, 64 mm \varnothing outer (47 mm \varnothing inner) \times 145 mm long, with screw-on filter retainer
- 2 glass cartridge for PUF sorbent, cylinder, 47 mm \varnothing outer (42 mm \varnothing inner) \times 125 mm long
- 3 PUF sorbent, open-cell polyether-type, density 22 mg/cm³, cylinder, 45 mm \varnothing \times 100 mm long
- 4 particle filter, glass- or quartz-fibre, circular, 47 mm \varnothing
- 5 support screens, circular, 1,2 mm openings (50 % open area)
- 6 inert, pliable sealing gaskets (e.g. PTFE), circular

a Air intake.

b To pump.

Figure A.2 — Sampling module for low-volume sampler

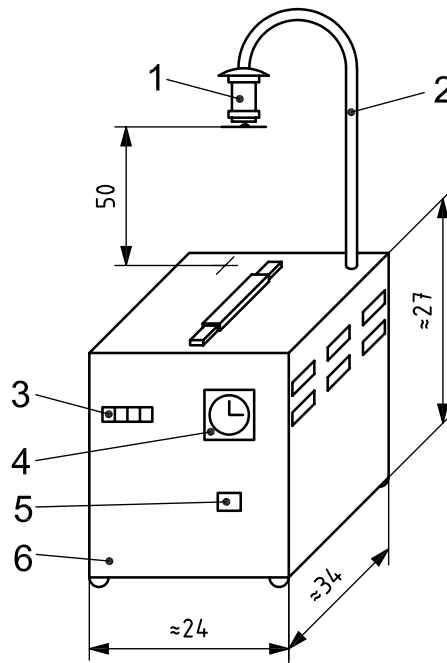
A.2.2 VDI 3498-2 low volume sampler

The sampler shown in Figure A.3 is designed to operate at 2,7 m³/h to 2,8 m³/h. The sampler consists of an air pump and associated controls housed in a compartment and connected by way of a hose to a sampling module that holds a circular glass- or quartz-fibre filter backed by a sorbent cartridge containing a cylindrical PUF plug (see Figure A.4). It is described in Reference [11]. The sampling system is available from several commercial sources worldwide. The sampling head may be changed for one with PM₁₀ or PM_{2,5} inlets.

Any pump providing the prescribed air flow rate may be used, but it shall be sound-insulated for use in occupied dwellings or offices. The pumping system described meets preferred noise criterion (PNC) 39 (less than 45 dB), which is typical of background noise in living areas and private offices. However, other commercially available air sampling pumps that are capable of sampling at 2,7 m³/h to 2,8 m³/h are nearly as quiet and noisier pumps may be quieted by placing them in sound-insulated boxes such as polystyrene foam coolers, provided that they do not overheat. Another possibility is to lengthen the outlet pipe with a hose out of the room into the exterior.

Sampling periods from 7 h up to several days are possible to collect sufficient samples to quantify low levels of PCBs, PCDDs, and PCDFs in indoor air.

Dimensions in centimetres

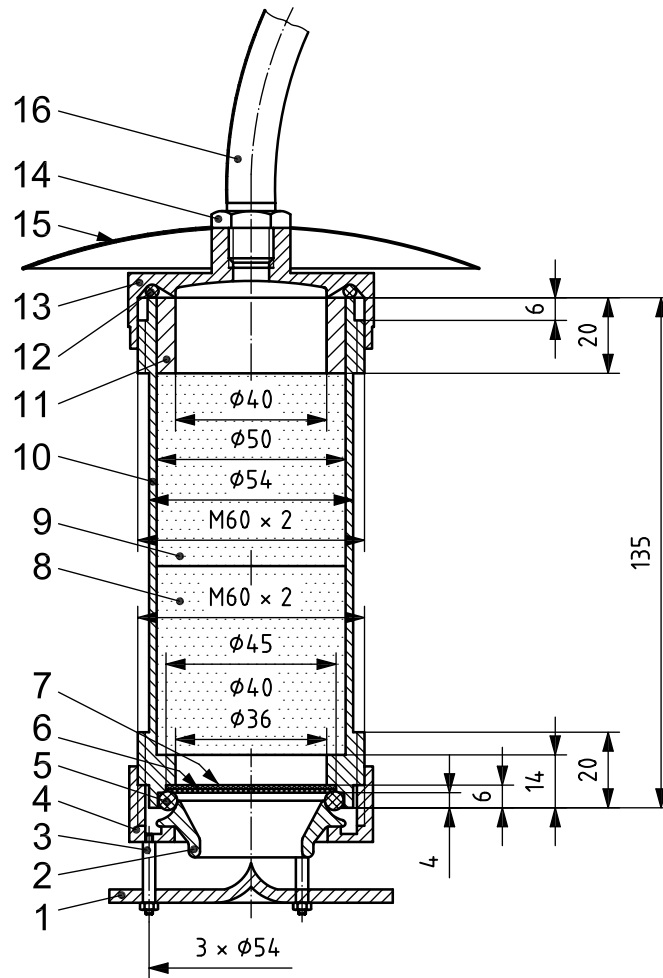


Key

- 1 sampling head
- 2 suction tube
- 3 counter
- 4 timer
- 5 switch
- 6 protective housing

Figure A.3 — Low-volume (2,7 m³/h) sampling apparatus

Dimensions in millimetres

**Key**

- 1 flow plate (stainless steel), \varnothing 75 mm, cone \varnothing 14 mm, height 3,5 mm
- 2 intake port (aluminium) hard anodized, bottom inner \varnothing 27 mm, outer \varnothing 33 mm, top inner \varnothing 39 mm, height 15 mm, outer support ring 50 mm
- 3 mounting parts (stainless steel), length 15 mm
- 4 filter holder with screw joint (polycarbonate), orifice 40,5 mm, height 20 mm, inner \varnothing 56 mm, outer \varnothing 64,5 mm
- 5 rubber gasket, inner \varnothing 39 mm, thickness 5 mm
- 6 glass-fibre filter, \varnothing 50 mm, between two flat PTFE rings outer \varnothing 50 mm, inner \varnothing 40,5 mm and outer \varnothing 50 mm, inner \varnothing 38 mm
- 7 support screen, \varnothing 40 mm to 44 mm, depending on the surface area supported
- 8 primary PUF trap, length 50 mm, \varnothing 55 mm
- 9 back-up PUF trap, length 50 mm, \varnothing 55 mm with an air space between both foams; the back-up foam shall be separated from the primary foam by an air space to prevent impedance of air flow
- 10 cartridge (stainless steel tube), total length 135 mm, inner \varnothing 50 mm, outer \varnothing 54 mm, support screen rest 40 mm to 44 mm, glass fibre or gasket rest \varnothing 50 mm
- 11 spacer (stainless steel ring), outer \varnothing 49,5 mm
- 12 rubber gasket, inner \varnothing 49 mm, thickness 3 mm to 3,5 mm
- 13 screw joint (polycarbonate), inner \varnothing 56 mm, outer \varnothing 64,5 mm, height 20 mm, orifice 8,5 mm, thread inner \varnothing 10,8 mm, metric fine thread (12 mm)
- 14 lock nut
- 15 shield, outer \varnothing 120 mm
- 16 suction tube (stainless steel), inner \varnothing 9 mm, outer \varnothing 12 mm

Figure A.4 — Sampling head

A.3 High-volume samplers

A.3.1 General

The high-volume samplers described in this clause operate at flow rates up to 16 m³/h and may be used inside large indoor enclosures such as auditoria, theatres, and open-area workplaces. They should only be used in homes and offices when it is not possible to obtain adequate sample volumes with low- or medium-volume samplers as it is likely that their use in these situations will upset the normal ventilation of the room or building. It shall be emphasized that noise produced by typical high-volume samplers may prohibit their use in occupied indoor environments. In addition, heat from the air exhaust may raise the temperature of a room to uncomfortable levels if the samplers are operated for several hours.

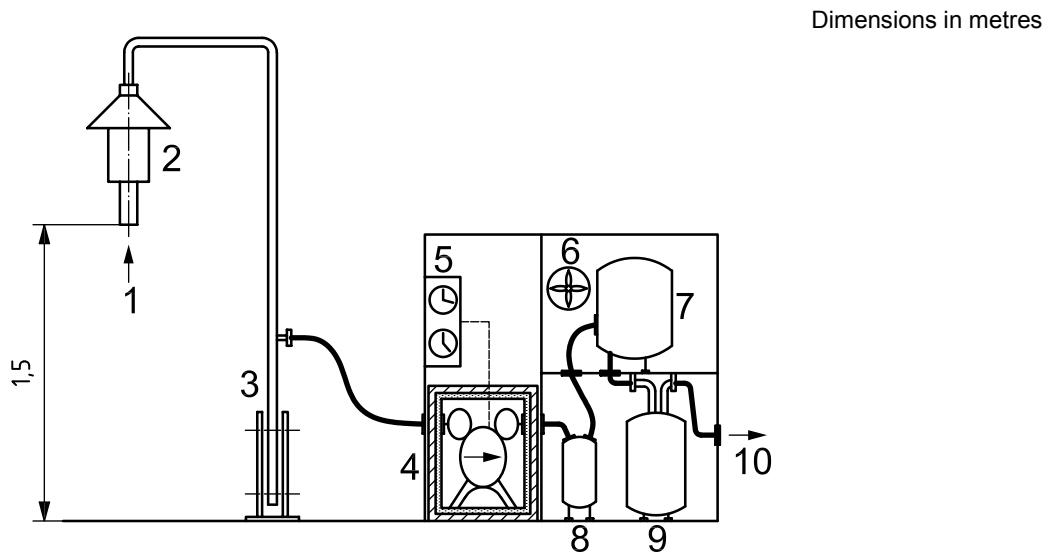
The sampler described in A.3.2 is designed so that the pumping system can be placed outdoors and connected to the sampling module located indoors by means of a sampling line (hose or metal pipe). Likewise, the sampling module may be removed from the ISO 12884 sampler described in A.3.3 and connected to the base unit by a sampling line. The sampler described in A.3.4 is extraordinarily quiet and may be used in domicile living areas and even in bedrooms while the occupants are sleeping.

A.3.2 VDI 3498-1 sampler

The sampler shown in Figure A.5 is designed to operate at 16 m³/h and is rather noisy but is designed so that the pumping system can be placed outdoors and connected to the sampling module located indoors by means of a sampling line (hose or metal pipe). The sampler consists of an air pump and associated controls housed in a compartment and connected by way of a hose to a sampling module that holds a circular glass- or quartz-fibre filter backed by a sorbent cartridge containing a cylindrical PUF plug (see Figure A.6). It is described in Reference [12]. The sampling system is available from several commercial sources worldwide.

Any pump providing the prescribed air flow rate may be used, but it shall be sound-insulated for use in occupied dwellings or offices.

Sampling periods from 3 h up to several days are possible to collect sufficient samples to quantify low levels of PCBs, PCDDs, and PCDFs in indoor air.

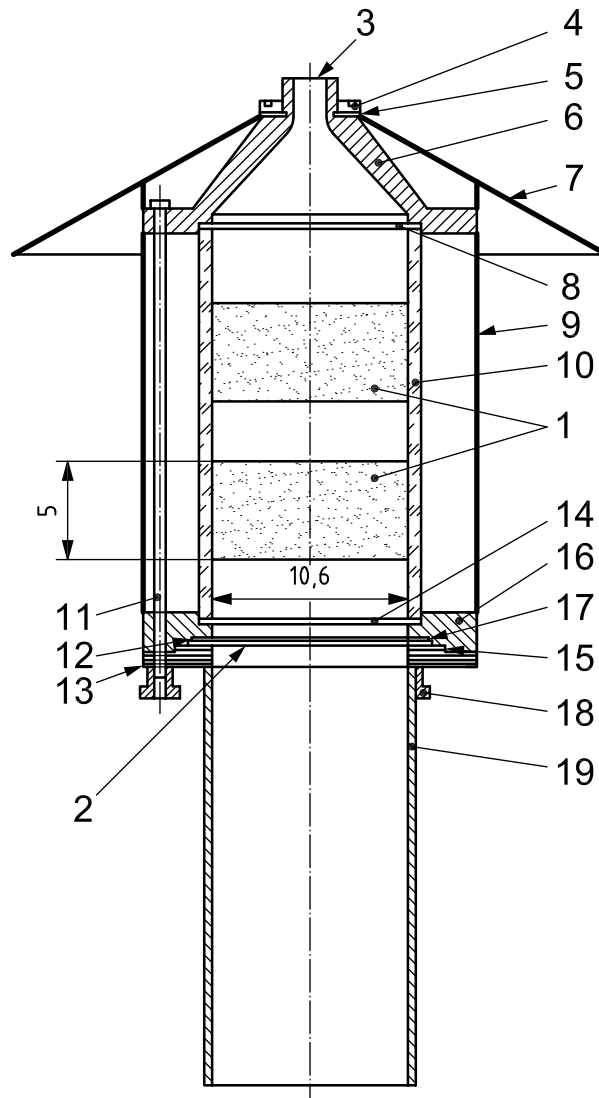


Key

- | | |
|--------------------------|---------------|
| 1 air intake | 6 fan |
| 2 sampling head | 7 air cooler |
| 3 tilting mast | 8 dust filter |
| 4 pump and two silencers | 9 gas meter |
| 5 timer | 10 exhaust |

Figure A.5 — VDI 3498-1 sampling apparatus

Dimensions in centimetres



Key

- | | |
|---|--|
| 1 polyurethane foam traps, \varnothing 110 mm \times 50 mm long | 11 thread |
| 2 round glass- or quartz-fibre filter, 120 mm | 12 filter holder (stainless steel) |
| 3 intake port | 13 aluminium flange with air intake port |
| 4 thread | 14 gasket |
| 5 seal | 15 gasket [polytetrafluoroethylene (PTFE)] |
| 6 intake head | 16 aluminium flange |
| 7 shield | 17 gasket (PTFE) |
| 8 gasket | 18 thread (brass) |
| 9 light protection (UVA) (not needed for indoor measurements) | 19 intake tube (aluminium), length 200 mm, inner diameter 100 mm |
| 10 glass cylinder | |

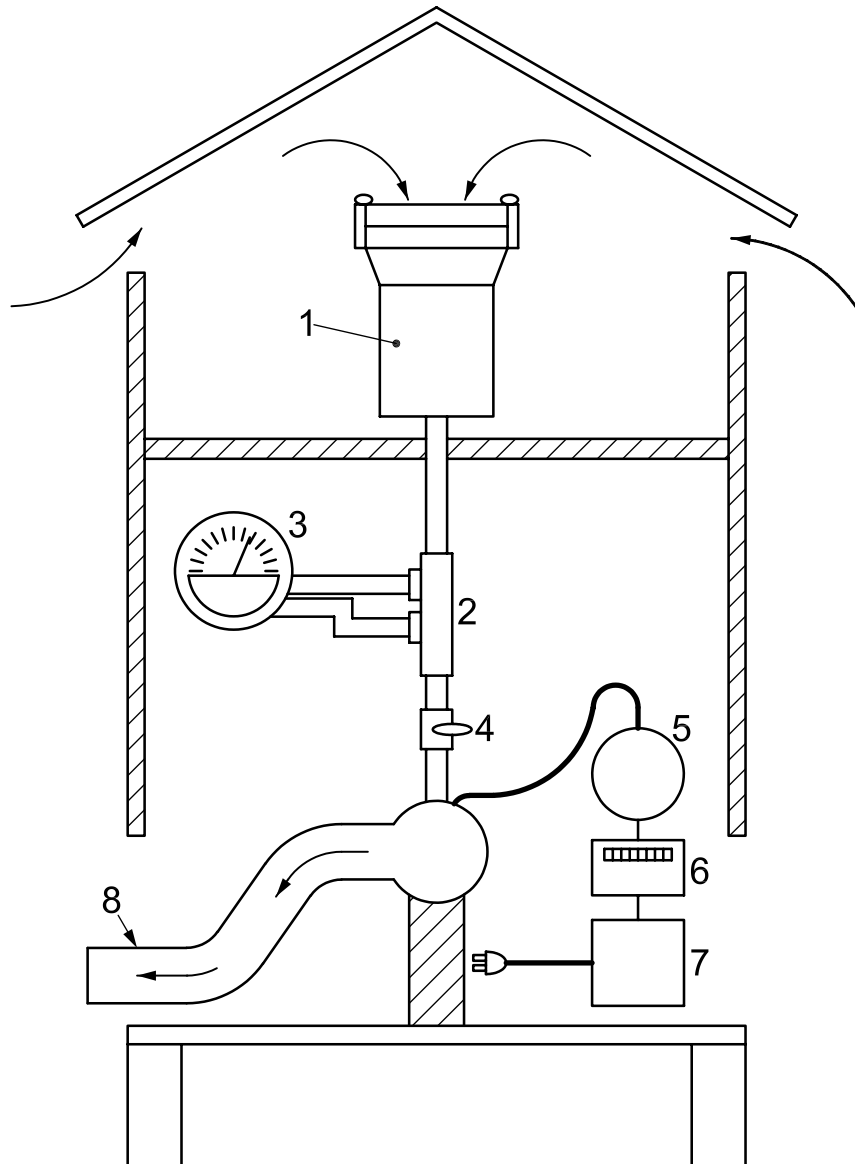
Figure A.6 — VDI 3498-1 sampling head

A.3.3 ISO 12884 sampler

The outdoor air sampler described in ISO 12884 may be adapted for use indoors for collecting PCBs, PCDDs and PCDFs from indoor air at high sampling rates (e.g. 6 m³/h to 16 m³/h). The same sampler is the basis of the United States Environmental Protection Agency Compendium Method TO-9A for polychlorinated dibenzo-*p*-dioxins and dibenzofurans in ambient air (Reference [7]). Samplers of this design are commercially available from a variety of sources worldwide. A schematic drawing of the sampler is shown in Figure A.7. It is equipped with a flow-control device, a vacuum gauge to measure pressure drop across the sampling module, an interval timer, and an exhaust hose to carry exhausted air at least 3 m away from the sampler to prevent recirculation of sampled air (Reference [13]). If feasible, the exhaust air should be vented outside of the room or building being sampled.

The sampling module for this sampler is the same as that shown in Figure 2. It consists of a metal filter holder [part 9 in Figure 2 b)] capable of holding a 104 mm circular particle filter supported by a 1,2 mm stainless-steel screen with 50 % open area and attaching to a metal cylinder [part 13 in Figure 2 b)] capable of holding a 64 mm \varnothing outer (58 mm \varnothing inner) \times 125 mm borosilicate glass sorbent cartridge. The filter holder is equipped with inert sealing gaskets (e.g. polytetrafluoroethylene) placed on either side of the filter. Likewise, inert, pliable gaskets (e.g. silicone rubber) are used to provide an air-tight seal at each end of the sorbent cartridge. The glass sorbent cartridge is indented 20 mm from the lower end to provide a support for a 1,2 mm stainless-steel screen (50 % open area) that holds the sorbent, which consists of a cylinder open-cell polyether-type PUF, density 22 mg/cm³, 62 mm \varnothing \times 100 mm long. The glass sorbent cartridge fits into part 13 [Figure 2 b)], which is screwed onto part 9 [Figure 2 b)] until the sorbent cartridge is sealed between the gaskets. The sampling module is described in References [6] and [7]. Similar sampling modules are commercially available.

The sampler is noisy, but may be used indoors in very large areas where noise is of no concern. Alternatively, the sampling module may be placed indoors and connected to the pump by means of a sampling line passed through the window or wall.



Key

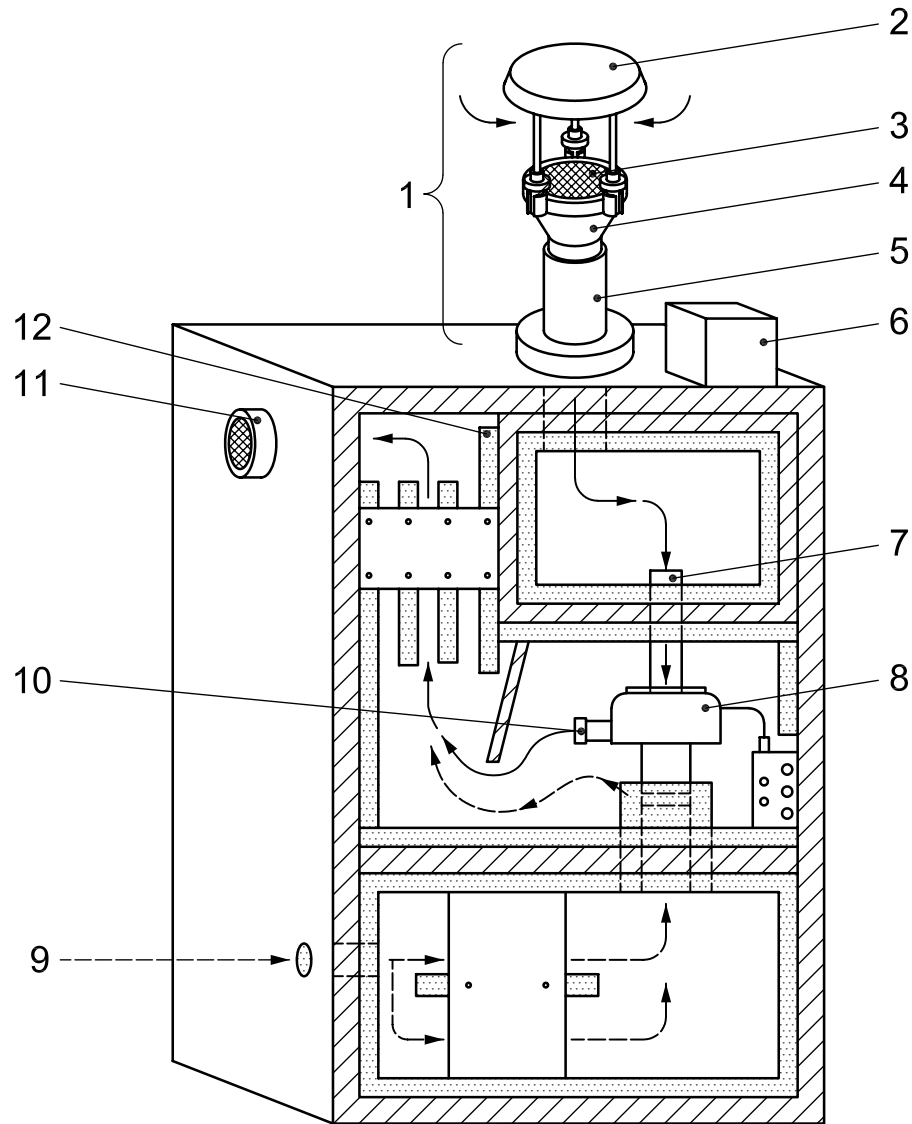
- 1 sampling module
- 2 venturi flow controller
- 3 vacuum gauge
- 4 flow control valve
- 5 voltage variator (for adjustment of flow rate)
- 6 elapsed time meter
- 7 seven-day timer
- 8 exhaust hose, 3 m to 4 m long

Figure A.7 — ISO 12884 sampler

A.3.4 Quiet high-volume sampler

The following sampler is acoustically insulated so that it can be used in occupied residences. It is described in the peer-reviewed literature (see References [9], [14] and [15]). It may be used at flow rates ranging from 6 m³/h to 16 m³/h.

The sampler is housed in a rugged, well-sealed cabinet 91 cm high × 58 cm long × 47 cm wide with good acoustic damping. The cabinet is constructed of plywood with grooved joints sealed with latex caulk. Semi-rigid fibreglass board, cut to fit the various enclosures and baffles within the cabinet, is the principal sound-absorbing medium. One side of the acoustic housing can be removed for inspection and servicing the unit's interior components, if necessary. Air flow is provided by a bypass vacuum motor that is coupled to the air inlet plenum with vibration-isolation mounts and a closed-cell foam gasket. This provides an airtight seal between the mounting flange and the inlet chamber. The sampler is designed to meet a preferred noise criterion value of NC-35, which is comparable to the background noise level in a typical bedroom. The sampler uses the same sampling module as that in Figure 2 (described in A.3.3). A schematic drawing of the sampler is shown in Figure A.8.



Key

- 1 sampling module
- 2 dust cover
- 3 particle filter support screen
- 4 particle filter holder
- 5 PUF cartridge holder
- 6 electronic control package (flow controller, timer, etc.)
- 7 vacuum chamber and pump inlet
- 8 vacuum pump with self-cooled, bypass motor
- 9 motor cooling air inlet
- 10 vacuum pump outlet
- 11 exhaust
- 12 semi-rigid fibreglass board sound baffles

NOTE A hose should be attached to the exhaust (11) to conduct the exiting air at least three metres away from the sampler and preferably outside of the room or building being sampled.

Figure A.8 — High-volume indoor air sampler with acoustic enclosure

Annex B (normative)

Determination of sampling efficiency or dynamic retention efficiency

Sampling efficiency (E_s) is determined by spiking a solution of the compounds of interest (or a representative selection) that includes the most volatile PCB/PCDD/PCDF targeted onto a clean particle filter backed up with the sorbent cartridge, then pulling through the assembled sampling module a volume of air equivalent to the maximum volume that will be sampled.

The dynamic retention efficiency (E_r) is determined by spiking the sorbent directly, placing it behind a clean filter in the sampling module, and otherwise following the same procedure.

For E_s determinations, the spiking solution shall be added dropwise to the filter, so as to uniformly load it and avoid over-saturation. For E_r determinations, the spiking solution is carefully injected into the inlet face of the sorbent bed in a manner that will apply the solution uniformly across the face and to a depth of no more than 1 cm. The spiking solution shall be in a volatile solvent, such as *n*-hexane or dichloromethane. Spiking levels shall correspond to at least 3 times but not more than 10 times the anticipated concentrations of the targeted compounds in the air to be pulled through the sampling medium. The spiked filter or sorbent shall be allowed to dry for about one hour in a clean, light-protected area prior to pulling air through the system.

The sampling rate and sampling period shall be the same as that planned for the programme. Ambient temperatures during the test shall also approximate those expected in the sampling period.

For determination of sampling efficiency, the sorbent and spiked filter shall be analysed separately and any residue retained by the filter subtracted from the initial spike quantity for calculation of sampling efficiency. For determination of dynamic retention efficiency, only the sorbent is analysed.

The sampling efficiency for a given analyte, in percent, is calculated using Equation (B.1):

$$E_s = \frac{m}{m_0 - m_R} \times 100 \quad (\text{B.1})$$

where

m is the mass of analyte extracted from the sorbent after air is pulled through it, in picograms (pg);

m_0 is the mass of analyte initially applied to the filter, in picograms (pg);

m_R is the mass of analyte remaining on the filter after air is pulled through it, in picograms (pg).

Sampling efficiencies shall fall between 75 % and 125 %. In no case shall sampling efficiencies below 50 % or above 150 % be accepted.

The dynamic retention efficiency, in percent, is calculated from Equation (B.2):

$$E_r = \frac{m}{m_0} \times 100 \quad (\text{B.2})$$

where m_0 is the mass of analyte initially applied to the sorbent bed, in picograms (pg).

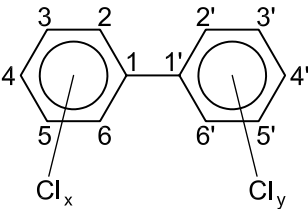
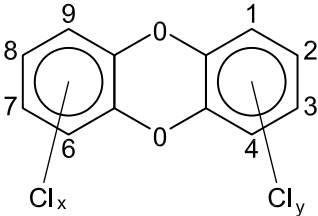
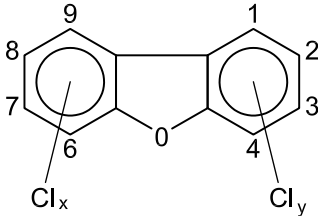
The % E_r has generally been found to be approximately equal to or slightly lower than the % E_s for semi-volatile organic compounds. The same range of acceptability applies to % E_r as to % E_s .

Annex C (informative)

Structure, toxicity and calculation of toxicity equivalents

C.1 Generic structures of PCBs, PCDDs and PCDFs

Table C.1 — Structures of PCBs, PCDDs and PCDFs and number of possible isomers

			
PCB Polychlorinated biphenyls	PCDD Polychlorinated dibenzodioxins	PCDF Polychlorinated dibenzofurans	
Number of chlorine atoms	Number of PCB isomers	Number of CDD isomers	Number of CDF isomers
1	3	2	4
2	12	10	16
3	24	14	28
4	42	22	38
5	46	14	28
6	42	10	16
7	24	2	4
8	12	1	1
9	3	—	—
10	1	—	—
Total	209	75	135

C.2 Structures of selected coplanar PCBs

Table C.2 — Structures of 12 selected coplanar polychlorinated biphenyls

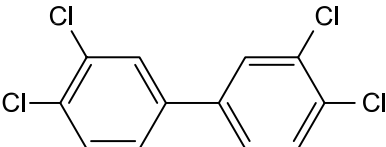
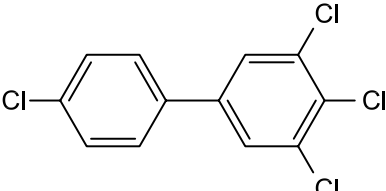
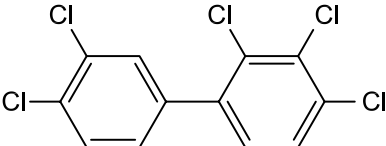
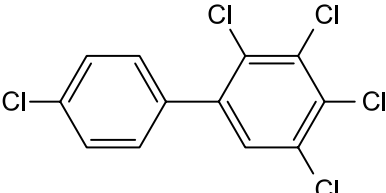
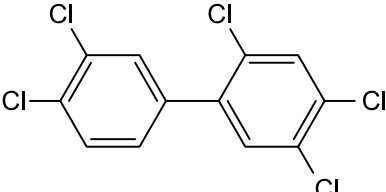
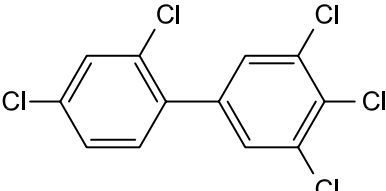
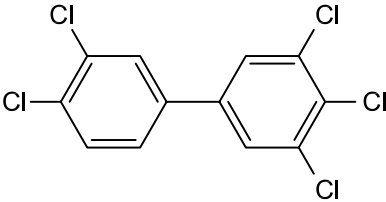
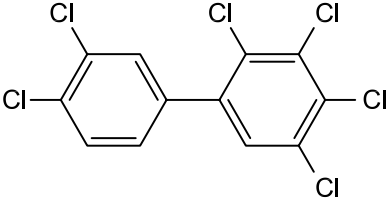
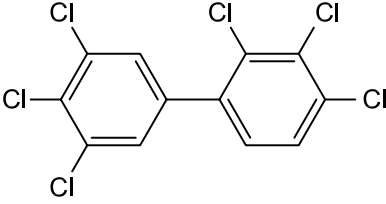
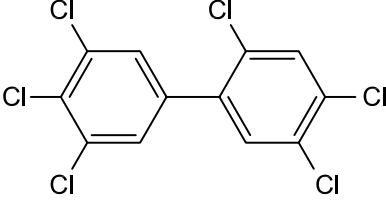
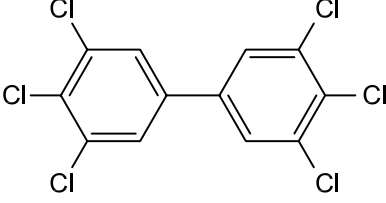
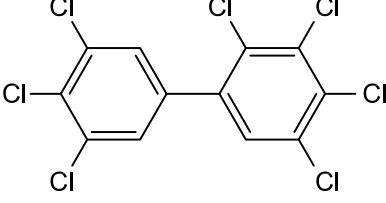
IUPAC names and synonyms	Empirical formula	Molar mass g/mol	Structural formula
3,3',4,4'-Tetrachlorobiphenyl (3,3',4,4'-TeCB) PCB-77	$C_{12}H_6Cl_4$	291,9	
3,4,4',5-tetrachlorobiphenyl (3,4,4',5-TeCB) PCB-81	$C_{12}H_6Cl_4$	291,9	
2,3,3',4,4'-pentachlorobiphenyl (2,3,3',4,4'-PeCB) PCB-105	$C_{12}H_5Cl_5$	326,4	
2,3,4,4',5-pentachlorobiphenyl (2,3,4,4',5-PeCB) PCB-114	$C_{12}H_5Cl_5$	326,4	
2,3',4,4',5-pentachlorobiphenyl (2,3',4,4',5-PeCB) PCB-118	$C_{12}H_5Cl_5$	326,4	
2',3,4,4',5-pentachlorobiphenyl (2',3,4,4',5-PeCB) PCB-123	$C_{12}H_5Cl_5$	326,4	

Table C.2 (continued)

IUPAC names and synonyms	Empirical formula	Molar mass g/mol	Structural formula
3,3',4,4',5-pentachlorobiphenyl (3,3',4,4',5-PeCB) PCB-126	$C_{12}H_5Cl_5$	326,4	
2,3,3',4,4',5-hexachlorobiphenyl (2,3,3',4,4',5-HxCB) PCB-156	$C_{12}H_4Cl_6$	360,9	
2,3,3',4,4',5'-hexachlorobiphenyl (2,3,3',4,4',5'-HxCB) PCB-157	$C_{12}H_4Cl_6$	360,9	
2,3',4,4',5,5'-hexachlorobiphenyl (2,3',4,4',5,5'-HxCB) PCB-167	$C_{12}H_4Cl_6$	360,9	
3,3',4,4',5,5'-hexachlorobiphenyl (3,3',4,4',5,5'-HxCB) PCB-169	$C_{12}H_4Cl_6$	360,9	
2,3,3',4,4',5,5'-heptachlorobiphenyl (2,3,3',4,4',5,5'-HpCB) PCB-189	$C_{12}H_3Cl_7$	396,3	

C.3 Toxicity and calculation of the toxic equivalents for PCDDs/PCDFs and PCBs

In the environment, PCDDs/PCDFs practically never appear as single compounds but always as a complex mixture associated with other structurally related (“dioxin-like”) compounds such as PCBs.

The TEQ system uses 2,3,7,8-TCDD as the standard to which the toxicity of the other compounds is weighted as toxic equivalents (TEQs). This normalization is based on the assumption that PCDDs/PCDFs and dioxin-like compounds act through the same mechanism of action. The toxic effects are assessed through subchronic toxicity studies and from certain biochemical properties such as Ah receptor binding capacity.

The toxic potential of a single congener is indicated through its toxic equivalence factor (TEF) describing the individual toxicity relative to the toxic effect of 2,3,7,8-TCDD. For the TEQ calculation, the amount or concentration of each relevant congener is multiplied with the corresponding TEF (see Table C.3). When all congeners are given as “equivalents of 2,3,7,8-TCDD”, they can simply be added up and the resulting TEQ represent the total toxicity of the mixture.

Even if there still remain uncertainties concerning the toxicity of PCDDs/PCDFs, there have been established international toxic equivalency factors (I-TEF) by NATO/CCMS (Reference [16]) (see Table C.3) and recently by WHO (Reference [5]) standardizing the toxicity of 2,3,7,8-Cl substituted dioxins and furans and recently certain dioxin-like PCBs. For all other congeners which may be present in a sample, a TEQ value of zero is assigned.

The NATO/CCMS scheme has been adopted internationally as a basis for the TEQ determination. During recent years, the toxicity of PCDDs/PCDFs has been reported mainly in I-TEQ.

The most recent TEQ scheme, developed by the World Health Organization (WHO) and the International Programme on Chemical Safety (IPCS), standardizes the toxicity of 17 dioxin and furan congeners and includes for the first time 12 dioxin-like PCBs (Reference [5]). The PCBs listed in the WHO proposal are considered to show comparable toxicity as 2,3,7,8-chlorine-substituted PCDDs/PCDFs. It reflects the present knowledge about toxic effects of PCDDs/PCDFs and dioxin-like PCBs (see Table C.3).

The WHO-TEQ approach is linked to a WHO recommendation concerning a Tolerable Daily Intake (TDI) for humans of 1 pg to 4 pg WHO-TEQ/kg body weight (including PCBs) that should not be exceeded. The TDI was recommended on the basis of critical effects (including developmental, reproductive, hormonal, immune system and neuro-behavioural effects), dose-response relationships and quantitative risk extrapolation.

In the sense of an international harmonized risk assessment, which should be based on the most current knowledge, it appears to be reasonable to accept the WHO-TEQ_{PCB} system and to discuss the WHO-TDI as an assessment scale for future risk assessment at the international level.

The calculation of the PCDD/PCDF TEQ results is normally performed by using the international toxic equivalency factors (I-TEF) of NATO/CCMS (see above). The calculation of the PCDDs/PCDFs and dioxin-like PCBs (coplanar and non-ortho PCBs) TEQ is only possible using the toxic equivalency factors of WHO (WHO-TEQ_{PCB}). Both calculation schemes are not compatible (see Table C.3). Due to the situation that in several countries the calculation according to NATO/CCMS (I-TEQ) and in others the calculation according to WHO (WHO-TEQ_{PCB}) are mandatory, the results and the calculation scheme have to be reported.

Table C.3 — WHO toxic equivalency factors (WHO-TEFs) (Reference [5]) and international toxic equivalency factors (I-TEFs) (Reference [16])

Congener	WHO-TEF	I-TEF
2,3,7,8-TCDD	1	1
1,2,3,7,8-PeCDD	1	0,5
1,2,3,4,7,8-HxCDD	0,1	0,1
1,2,3,6,7,8-HxCDD	0,1	0,1
1,2,3,7,8,9-HxCDD	0,1	0,1
1,2,3,4,6,7,8-HpCDD	0,01	0,01
OCDD	0,000 3	0,001
2,3,7,8-TCDF	0,1	0,1
1,2,3,7,8-PeCDF	0,03	0,05
2,3,4,7,8-PeCDF	0,3	0,5
1,2,3,4,7,8-HxCDF	0,1	0,1
1,2,3,6,7,8-HxCDF	0,1	0,1
1,2,3,7,8,9-HxCDF	0,1	0,1
2,3,4,6,7,8-HxCDF	0,1	0,1
1,2,3,4,6,7,8-HpCDF	0,01	0,01
1,2,3,4,7,8,9-HpCDF	0,01	0,01
OCDF	0,000 3	0,001
Non-ortho PCB		
3,4,4',5-TeCB (81)	0,000 1	—
3,3',4,4'-TeCB(77)	0,000 3	—
3,3',4,4',5-PeCB (126)	0,1	—
3,3',4,4',5,5'-HxCB (169)	0,03	—
Mono-ortho PCB		
2,3,3',4,4'-PeCB (105)	0,000 03	—
2,3,4,4',5-PeCB (114)	0,000 03	—
2,3',4,4',5-PeCB (118)	0,000 03	—
2',3,4,4',5-PeCB (123)	0,000 03	—
2,3,3',4,4',5-HxCB (156)	0,000 03	—
2,3,3',4,4',5'-HxCB (157)	0,000 03	—
2,3',4,4',5,5'-HxCB (167)	0,000 03	—
2,3,3',4,4',5,5'-HpCB (189)	0,000 03	—

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