

# Methods for sampling and analysis of chemical pollutants in indoor air



Supplementary publication  
to the screening tool  
for assessment of health risks  
from combined exposure to  
multiple chemicals in indoor air  
in public settings for children



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## Abstract

Indoor air contains a wide variety of chemical compounds originating both from indoor and outdoor sources. The WHO Regional Office for Europe convened a literature review and expert consultations which resulted in the identification of the most common chemical pollutants of indoor air in public settings for children (schools, kindergartens and day-care centres), and the development of a screening tool for assessment of risks from combined exposure to multiple chemicals. This publication provides information on methods for sampling and analysis of the 17 chemicals included in the tool, and around 40 other pollutants of concern in indoor air that can be considered for national monitoring programmes of indoor air quality. Priority is given to methods described by the International Organization for Standardization and/or the European Committee for Standardization, followed by methods published in peer-reviewed journals.

## KEYWORDS

AIR POLLUTION, INDOOR  
HAZARDOUS SUBSTANCES  
ENVIRONMENTAL EXPOSURE

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# CONTENTS

ACKNOWLEDGEMENTS .....	iv
ABBREVIATIONS .....	v
1. INTRODUCTION .....	1
2. SELECTION OF METHODS FOR SAMPLING AND ANALYSIS OF INDOOR AIR POLLUTANTS ....	2
3. GENERAL CONSIDERATIONS FOR A SAMPLING STRATEGY .....	5
4. QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES .....	8
4.1 Replicate samples and blanks.....	8
4.2 Storage stability and sample handling.....	8
4.3 Uptake rate .....	9
4.4 Backdiffusion .....	9
4.5 Identification of compounds.....	9
4.6 Linearity.....	9
4.7 Precision.....	9
4.8 Accuracy .....	10
4.9 LOD and LOQ .....	10
5. METHODS FOR SAMPLING AND ANALYSIS OF PRIORITY CHEMICAL POLLUTANTS IN INDOOR AIR FOR THE ASSESSMENT OF RISK OF COMBINED EXPOSURE .....	12
5.1 Oxygenated VOCs – aldehydes .....	12
5.2 VOCs .....	13
5.3 SVOCs .....	14
5.4 Inorganic compounds .....	16
6. METHODS FOR SAMPLING AND ANALYSIS OF OTHER POLLUTANTS OF CONCERN IN INDOOR AIR.....	18
6.1 Particulate matter .....	18
6.2 Inorganic compounds .....	19
6.3 SVOCs .....	20
REFERENCES .....	24
ANNEX 1. LIST OF PRIORITY CHEMICALS INCLUDED IN THE SCREENING FOR ASSESSMENT OF RISK OF COMBINED EXPOSURE TO HAZARDOUS CHEMICALS.....	30
ANNEX 2. OTHER POLLUTANTS OF CONCERN IN INDOOR AIR .....	31
ANNEX 3. LIST OF RECOMMENDED METHODS.....	33
ANNEX 4. SUMMARY OF METHODS FOR SAMPLING AND ANALYSIS OF POLLUTANTS IN INDOOR AIR .....	36

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## ABBREVIATIONS

AHTN	tonalide
BDE 28	2,4,4'-tribromodiphenyl ether
BDE 47	2,2',4,4'-tetrabromodiphenyl ether
BDE 99	2,2',4,4',5-pentabromodiphenyl ether
BDE 100	2,2',4,4',6-pentabromodiphenyl ether
BDE 153	2,2',4,4',5,5'-hexabromodiphenyl ether
BDE 183	2,2',3,4,4',5',6-heptabromodiphenyl ether
BDE 209	2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether
BFR	brominated flame retardant
CO	carbon monoxide
CO <sub>2</sub>	carbon dioxide
CP	chlorinated paraffin
CRM	certified reference material
DBE-DBCH	1,2-dibromo-4-(1,2-dibromoethyl)cyclohexane
DEP	diethyl phthalate
DiBP	diisobutyl phthalate
DnBP	di-n-butyl phthalate
DNPH	2,4-dinitrophenylhydrazine
ECNI	electron capture negative ionization
EN	European normative
EPA	United States Environmental Protection Agency
FID	flame ionization detector
GC	gas chromatography
GC–MS	gas chromatography–mass spectrometry
GC–MS/MS	gas chromatography–tandem mass spectrometry
GFF	glass fibre filter
HHCB	galaxolide
HPLC	high-performance liquid chromatography
HRMS	high-resolution mass spectrometry
HVAC	heating, ventilation and air conditioning
IAQ	indoor air quality
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
LC	liquid chromatography
LCCP	long-chain chlorinated paraffins
LOAEL	lowest-observed-adverse-effect level
LOD	limit of detection

## ABBREVIATIONS contd.

LOQ	limit of quantification
LRMS	low-resolution mass spectrometry
LVS	low-volume sampler
MCCP	medium-chain chlorinated paraffins
MRM	multiple reaction monitoring
MS	mass spectrometry
MTBH	3-methyl-2-benzothiazolinone hydrazone
NOAEL	no-observed-adverse-effect level
NO <sub>2</sub>	nitrogen dioxide
O <sub>3</sub>	ozone
OPFR	organophosphate flame retardant
OSHA	Occupational Safety and Health Administration of the United States Department of Labor
Oxy-VOC	oxygenated volatile organic compound
PAH	polycyclic aromatic hydrocarbon
PAS	passive air sampler
PBDE	polybrominated diphenyl ether
PCB	polychlorinated biphenyl
PCDD/PCDF	polychlorinated dibenzo-p-dioxin/dibenzofuran
PM <sub>2.5</sub>	particulate matter with an aerodynamic diameter below 2.5 µm
PM <sub>10</sub>	particulate matter with an aerodynamic diameter below 10 µm
PTFE	polytetrafluoroethylene
PUF	polyurethane foam
QA/QC	quality assurance/quality control
QFF	quartz fibre filter
SD	solvent desorption
SINPHONIE	Schools Indoor Pollution and Health Observatory Network in Europe
SIM	selected ion monitoring
SVOC	semi-volatile organic compound
SCCP	short-chain chlorinated paraffin
TBP	tributyl phosphate
TBEP	tris(2-butoxyethyl) phosphate
TCPP	tris(1-chloropropan-2-yl) phosphate
TCEP	tris(2-chloroethyl) phosphate
TD	thermal desorption
UV-Vis	ultraviolet-visible
VOC	volatile organic compound



# 1. INTRODUCTION

Indoor air contains a wide variety of chemical compounds. Some of these compounds, including volatile organic compounds (VOCs), originate mainly from indoor sources such as paints, building products, furnishings, glues, varnishes, and consumer and household products (1). Other pollutants, such as nitrogen dioxide (NO<sub>2</sub>) and ozone (O<sub>3</sub>), are classical pollutants of outdoor air that penetrate indoor environments in different ways in addition to releases from indoor sources (2).

The importance of measuring concentrations of pollutants in indoor air has been widely discussed (3–6). Many chemicals that are hazardous to health are present in concerning concentrations (3). For example, monitoring concentrations of semi-volatile organic compounds (SVOCs) – one of the groups of chemicals emitted from building materials and consumer products – in the indoor air of school buildings is important (6,7), due to their links with adverse health effects (8–10).

Determining concentrations of indoor air pollutants is critical for many purposes, including:

- ◆ assessment of health risks;
- ◆ identification of the contribution of indoor and outdoor sources to sum concentrations in indoor air (in this case, outdoor measurements are also necessary);
- ◆ observation of temporal and spatial trends of indoor air quality;
- ◆ assessment of compliance with indoor air quality guidelines and standards; and
- ◆ development and justification of risk-reduction measures.

Within the framework of the project on the development of a screening tool for assessment of risk of combined exposure to hazardous chemicals in indoor air in public settings for children (schools, kindergartens, and day-care centres), a list of 17 chemicals for inclusion in the first version of the tool was agreed (see Annex 1). This list takes into account the likelihood of presence of chemicals in indoor air in public settings for children, the availability of toxicological information on chemicals and their potential contribution to risks in concentrations commonly observed in indoor air.

In addition to the 17 chemicals included in the tool, a literature review and expert consultation identified a range of other indoor air pollutants of concern (Annex 2). Information about the methods relevant for these chemicals and particulate matter is also included in this publication. As particulate matter with an aerodynamic diameter below 2.5 µm (PM<sub>2.5</sub>) and below 10 µm (PM<sub>10</sub>) is not chemical, it cannot be included in the calculation of combined exposure. However, these particles are important to monitor and control, given the evidence of their health effects and their presence in indoor air.

## 2. SELECTION OF METHODS FOR SAMPLING AND ANALYSIS OF INDOOR AIR POLLUTANTS

A number of methods for sampling and analysis have been used to characterize chemical pollution of indoor air. This publication explores the advantages and drawbacks of some of these methods, which were selected using the following criteria:

- ♦ most commonly used for sampling and analysis of indoor air pollutants in public settings for children (see Annex 3);
- ♦ recommended by the International Organization for Standardization (ISO) (as the first choice);
- ♦ enable quantitative analysis of the concentrations of chemicals and other pollutants typical for public settings for children; and
- ♦ with a limit of detection (LOD) lower than the threshold/no-observed-adverse-effect level (NOAEL)/lowest-observed-adverse-effect level (LOAEL) of chemicals and other pollutants.

Other considerations included:

- ♦ possibility to calculate average concentrations during the sampling period
- ♦ reliability and reproducibility of results
- ♦ sensitivity to climate conditions
- ♦ complexity of sampling and analysis
- ♦ cost
- ♦ sampling period (not longer than one month)
- ♦ possibility to be used in different types of rooms and settings
- ♦ safety of equipment and sampling procedure for children.

Sampling methods can be passive (also called diffusive) or active. Active sampling can be continuous (online measurements, for example, automatic analysers) or discontinuous (off-line measurements). In the case of discontinuous methods (passive and active sampling), pollutants are collected on a filter or sorbent tube for further analysis in a laboratory.

Laboratory analysis usually consists of extraction, clean-up (when necessary) and the analysis itself using analytical equipment. The extraction can be carried out using thermal desorption (TD) or solvent desorption (SD). Analysis can be performed by means of different analytical systems depending on the pollutant to be analysed (such as gas chromatography (GC) and liquid chromatography (LC) coupled to different detectors and an ultraviolet-visible (UV-Vis) spectrophotometer).

Passive sampling is the preferable method for sampling indoor air pollutants. It does not involve the use of pumping systems, does not require a power supply and does not require the involvement of professional staff. Noiseless work and low cost are other advantages. Sampling can be conducted in different places simultaneously and the sampling period can be one school week (five days).

For example, active sampling for measuring formaldehyde in public settings for children where its concentration can be low might require a longer sampling duration to determine typical formaldehyde levels in public settings for children; this can be quite expensive and time consuming. Using passive air samplers (PAS) for an averaging period of one school week is an option for measuring formaldehyde in public settings for children, although in this case it would not be possible to assess compliance with the WHO short-term (30 minutes) indoor air quality guideline values for formaldehyde 100 µg/m<sup>3</sup> (3).

PAS are exposed for a defined time period that ranges from hours to days depending on the target pollutant. For example, passive sampling for SVOCs analysis requires a long sampling period (3–4 weeks).

Driven by the concentration gradient, molecules in the gas phase pass through the diffusive surface (air) to an adsorbing surface and are trapped. Analysis of these molecules determines the average concentration of the pollutant during the sampling period. The following equation is used to calculate concentration.

$$C (\mu\text{g m}^{-3}) = \frac{m (\mu\text{g})}{Q (\text{ml min}^{-1}) \times t (\text{min})} \quad \text{Eq. 1}$$

Where:

- ♦  $m$  is the mass of the pollutant
- ♦  $t$  is the sampling time
- ♦  $Q$  is the sampling rate (known as diffusive uptake rate) that depends on the diffusion coefficient of the target pollutant, which is directly associated with the cross-sectional area of the opening of the sampler and inversely associated with the length of the diffusion zone of the device.

For each sampler model, the manufacturer determines the uptake rate by means of calibration in a standard atmosphere. Although different pollutants can be measured with the same sampler, the uptake rate can differ for each pollutant and is a function of temperature during the sampling period. Temperature must therefore be registered during sampling in order to calculate the concentration of the pollutant. Airborne gases are collected by means of a physical process such as diffusion through a static air layer or permeation through a membrane.

Active samplers involve the use of an air sampling pump to actively pull air through a collection device such as a filter or a sorbent tube.

The methods described in this publication were selected by taking into account the recommended methods in the ISO 16000 series. In the absence of ISO methods, the European normative (EN) for ambient air and methods published in peer-reviewed journals were considered as a second and third choice, respectively.

The following standards from the ISO 16000 series with specific procedures for different chemicals are included in this publication.

- ◆ ISO 16000-1:2004. Indoor air – Part 1: General aspects of sampling strategy;
- ◆ ISO 16000-2:2004. Indoor air – Part 2: Sampling strategy for formaldehyde;
- ◆ ISO 16000-4:2011. Indoor air – Part 4: Determination of formaldehyde – Diffusive sampling method;
- ◆ ISO 16000-5:2007. Indoor air – Part 5: Sampling strategy for volatile organic compounds;
- ◆ ISO 16000-6:2011. 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;
- ◆ ISO 16017-2:2003. Indoor, ambient and workplace air – Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography – Part 2: Diffusive sampling;
- ◆ ISO 16000-12:2008. Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs);
- ◆ ISO 16000-13:2008 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;
- ◆ ISO 16000-15:2008. Indoor air: Sampling strategy for nitrogen dioxide (NO<sub>2</sub>);
- ◆ ISO 16000-31:2014 Indoor air – Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds – Phosphoric acid ester;
- ◆ ISO 16000-33:2017 Indoor air – Part 33: Determination of phthalates with gas chromatography/mass spectrometry (GC/MS);
- ◆ ISO 16000-34:2018 Indoor air – Part 34: Strategies for the measurement of airborne particles; and
- ◆ ISO 16000-37:2019 Indoor air – Part 37: Measurement of PM<sub>2.5</sub> mass concentration.

### 3. GENERAL CONSIDERATIONS FOR A SAMPLING STRATEGY

With regard to monitoring strategies, ISO 16000-1 describes general considerations about the duration, period and location of sampling of indoor air pollutants, with parallel measurements of concentrations of outdoor air pollutants. The WHO Regional Office for Europe's meeting report on methods for monitoring indoor air quality in schools (11) and the Schools Indoor Pollution and Health Observatory Network in Europe (SINPHONIE) project (12) also refer to these considerations.

**Duration of sampling:** Two different approaches can be considered for sampling duration:

1. 24-hour measurements for five school days (24 h/5 days); and
2. measurements during periods when children are present indoors (5–8 hours every day of a school week) (5–8 h/5 days).

The first option (measuring non-stop for five days) can result in overestimation of exposure to certain pollutants, such as formaldehyde, which may accumulate during the night when windows are closed or mechanical systems are turned off. It can also produce biased estimates of exposure to NO<sub>2</sub> (the direction of bias would depend on contributions of indoor and outdoor sources of NO<sub>2</sub>) (11).

For example, in a study in schools in Paris, France, measurements were performed on different time intervals: eight-hour measurements for four days (Monday to Friday, excluding Wednesday when there were no classes), and 24-hour measurements for five days (Monday to Friday, including Wednesday). The results showed that by including unoccupied periods in the sampling interval, formaldehyde, acetaldehyde and tetrachloroethylene concentrations were overestimated by 14%, 16% and 51%, respectively. In contrast, toluene, ethylbenzene, styrene, xylenes, 1,2,3-trimethylbenzene and 1,4-dichlorobenzene concentrations were underestimated by 9%, 10%, 8%, 9%, 17% and 15%, respectively (13). However, in another study in Lyon, France, an underestimation of formaldehyde concentration in schools of 7% and in day-care centres of 19% was observed when unoccupied periods were included (13).

The second option (measuring during periods when children are present indoors) requires that samplers be capped at the end of each school day and uncapped the next morning. In this case the LOD is higher, and capping and uncapping manually can require additional resources if technical staff have to come every day to do so. Passive sampling over a school week is therefore recommended for the purpose of assessing combined exposure to chemicals in indoor air. The SINPHONIE project applied this strategy: it used PAS to collect chemicals (formaldehyde, benzene, trichloroethylene, tetrachloroethylene, limonene,  $\alpha$ -pinene, NO<sub>2</sub> and O<sub>3</sub>) over a five-day period from Monday morning to Friday afternoon.

The SINPHONIE project applied other approaches for different pollutants. For example, samples of particulate matter with a diameter of less than 2.5  $\mu$ m (PM<sub>2.5</sub>) were collected for gravimetric analysis for eight hours per day (08:00–16:00) due to the relationship between PM<sub>2.5</sub> and occupancy density (m<sup>2</sup>/child) and for reasons of detection limit. Carbon monoxide (CO) was continuously measured by a low-cost logger since CO causes acute health effects and a short-term assessment is more suitable. Carbon dioxide (CO<sub>2</sub>) was simultaneously

measured with CO in order to calculate ventilation rates. PM<sub>2.5</sub> and PM<sub>10</sub> were also measured continuously by optical light scattering (12).

**Sampling period:** Concentration of some pollutants can vary depending on the season when sampling is carried out. Emissions of formaldehyde from materials and products depend on several factors, such as temperature and relative humidity (ISO 16000-1:2004) and ventilation rate. Thus, analysis of indoor air pollution in the cold (heating) season (November to March in the northern hemisphere) would characterize peak exposure levels for certain compounds.

The WHO Regional Office's meeting report on methods for monitoring indoor air quality in schools recommends sampling during the heating season to detect higher levels of exposure to formaldehyde, benzene and NO<sub>2</sub> (11). To evaluate the seasonal variation of pollutant concentrations in indoor air, measurements during the warm (non-heating) season should also be conducted, especially in countries with high outdoor temperatures. Sampling during the non-heating season will depend on the geographical location of the country. In the SINPHONIE project, for example, measurements during the non-heating season were organized between August and September (depending on the start of the new school year and the climatic conditions) and between May and June.

These measurements should take place in the same school at least two months before and after the measurements taken during the heating season. Samplers must be located in the same places indoors and outdoors during both campaigns in order to achieve maximum comparability (12).

**Classroom selection criteria:** In each school, at least three classrooms (one sampler per classroom) should be selected based on the following criteria:

- ◆ how representative they are of the school building;
- ◆ how routinely and continuously they are used during school hours; and
- ◆ their location on different floors (11).

Additional criteria can be considered, such as the orientation of classrooms (whether they face a street or a yard) and how long they have been in use (for example, classrooms that have been in use for at least six months can be selected to avoid encountering emissions from new building materials) (12). In the case of classrooms with equivalent conditions, those with a higher number of pupils should be chosen (12).

**Sampler location:** An important factor for defining a sampling strategy is the effect of airflow velocity on sampling results. Diffusion samplers typically require a minimum airflow (11). ISO 16000-1 recommends placing samplers in the centre of a room. If this is not possible, the sampler should be placed 1.0–2.0 m from a wall and 1.0–1.5 m from the floor, at the breathing level of pupils. Samplers will need to be placed in such a way as to prevent people from tampering with them; this may be at a higher elevation and/or closer to a wall. In the SINPHONIE project, sampling points were established at the breathing level of pupils when seated (12). (See also recommendations for specific pollutants in Section 5 and Section 6.)

**Outdoor air sampling (optional):** Measurement of concentrations of outdoor pollutants can identify the contribution of outdoor sources to indoor air pollution. Outdoor measurement of some pollutants, such as formaldehyde and SVOCs (except PAHs), is less important, since

the main sources of these chemicals are usually indoors (11,14–17). Distance to traffic (and type of road) and/or industrial sources of emissions should be recorded for each outdoor sampling site (11). Samplers should be located close to school buildings but no closer than 1.0 m (ISO 16000-1:2004). If the building has a heating, ventilation and air conditioning (HVAC) system, ambient air should be sampled close to the air intake (12). Samplers should be protected from direct sunlight and precipitation and, when possible, from people.

**Information on activities and potential sources of emissions:** All factors that affect indoor air quality should be analysed and reported, including activities in classrooms, cleaning routines and cleaning products, HVAC systems, furnishings and fittings, and potential outdoor sources of pollutants (11,12). Recommendations about the information to be recorded during indoor air measurement can be found in ISO 16000-1.



## 4. QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

Quality assurance and quality control (QA/QC) procedures refer to activities carried out by a laboratory to ensure the accuracy (how close to real concentrations the results reported by a laboratory are) and precision (how reproducible the results are) of data. Commonly, several analytical parameters – linearity, precision, accuracy (or recovery), LOD and limit of quantification (LOQ) – are evaluated to ensure the application of the selected method during the analysis of samples. ISO standards describe detailed QA/QC protocols for specific compounds. This chapter describes general procedures for a QA/QC programme.

For consistency, analysis of all samples in the same laboratory is recommended. However, this may not be feasible for various reasons (11). The minimum requirements of the QA/QC programme consist of an initial demonstration of laboratory capability and periodic analysis of blanks, spiked samples and certified reference materials (CRMs), as explained below, and continual monitoring of performance.

A reference laboratory in each country is commonly designated. If different laboratories are selected for the analysis of samples, they should confirm their credibility in intercomparison exercises analysing samples with known amounts of chemicals before a survey starts. Control samples must be prepared by reference laboratories (11). Statistical analysis of results can be carried out according to ISO 5725. Those laboratories that do not meet the established requirements must identify sources of errors and implement corrective actions.

The QA/QC procedures described below are designed to obtain analytical results of quality, reliability and consistency, and to minimize sampling and analysis errors with the aim of avoiding unnecessary repetition of sampling and analysis. Results of the data-quality checks should be compared with the performance requirement of the methods.

### 4.1 Replicate samples and blanks

Replicate samples and blanks must be included in each batch of samples. Sampling must include at least 10% replicate samples. A bias below or equal to 30% between the replicate samples is acceptable (12). At least one field blank sample (a sampler that is placed in a room but not open) per building should also be collected. In addition, at least one laboratory blank (a sampler that is not handled in the field and is stored in the laboratory) should be analysed per batch of samples.

Blanks are analysed following the same analytical procedure as indoor air samples. Field blanks must be prepared according to the procedures established by the laboratory and in accordance with ISO standards for the compounds to be analysed. ISO standards can also provide specific instructions regarding the level of the analytes in the blanks.

### 4.2 Storage stability and sample handling

After sampling, samples must be shipped to a laboratory as soon as possible and should be stored according to the analytical procedures established for each type of sampler. Samples



are only stable for a certain period of time and must be analysed within this period. Sections 5 and 6 describe storage requirements for each group of chemicals.

### 4.3 Uptake rate

For PAS, ISO standards describe the uptake rate for each compound, taking into account the specific dimensions of the sampler. For other specifications, it may be necessary to follow EN 838 or EN 13528-2 to calculate the uptake rate. The manufacturer of each commercial PAS will provide the uptake rate for each pollutant.

As uptake rate can be temperature dependant, the measurement of temperature during the sampling period is essential. Manufacturers provide the equation to calculate the concentration of pollutants, taking into account the correction of uptake rate with temperature.

### 4.4 Backdiffusion

Backdiffusion happens when the adsorbed mass of analyte is far from the maximum amount allowed by the adsorbing medium capacity. The extent of backdiffusion depends on concentration and exposure time. To avoid this, PAS manufacturers give instructions regarding sampling time and concentrations of pollutants.

### 4.5 Identification of compounds

The following two rules are recommended for the identification of compounds.

1. The ratio of the chromatographic retention time of the compound to that of the internal standard (relative retention time of the compound) shall correspond to that of the calibration solution at a tolerance of  $\pm 0.5\%$  for GC and  $2.5\%$  for LC. If an internal standard is not used, the retention time should be the same with a tolerance of  $5\%$  (18).
2. In GC–MS, peak identity must be confirmed by means of the ratio of the intensity of the target ion or quantification transition (Q) to the qualifier ion or confirmation transition (q) (Q/q). For that, the Q/q ratio of the sample must be compared with the Q/q ratio of the calibration standard, taking into account the tolerances allowed in European Commission decision 2002/657/EC (18).

### 4.6 Linearity

The analytical systems must be initially calibrated using a minimum of five concentrations in the range of expected concentrations in the samples in order to determine the linear response of the target analytes. The calibration curve should be verified at least every day or according to the instructions of the analytical procedure.

### 4.7 Precision

Precision is described as the degree of agreement between independent measurements obtained in well defined conditions (ISO 5725-3:1994). Analytical precision measures the variability associated with repetitive analyses of the same sample. Laboratory control samples are used to assess precision. Precision data can be obtained for the analytes using

CRMs and/or laboratory-fortified blanks with analytes at a known concentration (if CRMs are not commercially available).

Five blanks can be spiked with the method analytes and used to check instrument precision. The samples can then be extracted and analysed following the same procedure. Precision is calculated as the coefficient of variation. If the acceptance criteria are satisfied for all of the target pollutants, the laboratory can begin analysis of blanks and samples. The ongoing analysis of laboratory control samples must be carried out. The frequency should be established beforehand. For more information on precision measurement, see the ISO 5725 series of standards.

## 4.8 Accuracy

Accuracy is described as the closeness of a measurement to the true value (ISO 5725-1). It can be assessed through the calculation of recovery or desorption efficiency. The methodology to determine the accuracy of a method is the same as that for determining precision: using CRM and/or laboratory-fortified blanks. Measurement can be considered accurate when the concentration measured during the analysis has a value or presents a recovery within the laboratory's acceptable limits. Generally, the requirements for accuracy are established for each analytical method (see ISO standards).

In order to continue monitoring performance, it is recommended to prepare a table of the recoveries and the relative percent difference for each ongoing analysis of the laboratory control samples, and to prepare a graphic representation for each pollutant. For more information on accuracy measurement, see the ISO 5725 series of standards.

## 4.9 LOD and LOQ

LOD is the only parameter that presents different definitions and a large number of estimation methods (19). According to the International Union of Pure and Applied Chemistry (IUPAC), a widely accepted definition is that LOD can be derived from the smallest measure that can be detected with reasonable certainty for a given analytical procedure (20). The value of  $x_L$  is given by the following equation.

$$x_L = \bar{x}_{Bl} + ks_{Bl} \quad \text{Eq. 2}$$

Where:

- ♦  $\bar{x}_{Bl}$  is the mean of the blank measures
- ♦  $s_{Bl}$  is the standard deviation of the blank measures
- ♦  $k$  is a numerical factor chosen according to the confidence level desired (a value of 3 is recommended).

The transformation of signal of LOD in the corresponding concentration or amount is carried out with the calibration curve. For both indoor and outdoor air, LOD is usually expressed in units of mass divided by volume ( $\mu\text{g}/\text{m}^3$ ).

LOQ can be considered as the lower limit of precise quantitative measures.

Other common methods for the estimation of LOD and LOQ are:

- ♦ calculation from the signal-to-noise ratio (LOD and LOQ correspond to 3 and 10 times the noise level, respectively); or
- ♦ calculation from the calibration line at low concentrations (21).

The following equation is also commonly used to calculate LOD and LOQ.

$$LOD \text{ or } LOQ = \frac{k \times S}{b} \quad \text{Eq. 3}$$

Where:

- ♦  $k$  is a factor of 3 and 10 for LOD and LOQ, respectively
- ♦  $S$  is the standard deviation of the blank, the standard deviation of the ordinate intercept, or residual standard deviation of the linear regression
- ♦  $b$  is the slope of the regression line (in the case that  $S$  is the analytical signal and dimensionless).

In this case the level of the blank is not included.

Laboratories should provide information about the method used to estimate LOD and LOQ. Other common methods are described by the International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH) (21).

## 5. METHODS FOR SAMPLING AND ANALYSIS OF PRIORITY CHEMICAL POLLUTANTS IN INDOOR AIR FOR THE ASSESSMENT OF RISK OF COMBINED EXPOSURE

This publication prioritizes methods recommended in ISO standards for measuring chemical compounds in indoor air. However, methods accredited at the national level and/or published in peer-reviewed journals are also acceptable options.

Sampling methods used to measure concentrations of outdoor air pollutants can also be used to measure indoor air pollutants, but some limitations regarding noise, size or sampling rate should be considered. This chapter describes specific protocols for sampling and analysing the 17 pollutants included in the tool for assessment of risk of combined exposure to hazardous chemicals in indoor air (see Annex 1 and Annex 4).

### 5.1 Oxygenated VOCs – aldehydes

The aldehydes included in the list of priority chemicals are formaldehyde and acetaldehyde. A detailed sampling protocol for formaldehyde is available in ISO 16000-2, and the analytical procedure is available in ISO 16000-4. The same sampling protocol can be applied to acetaldehyde and other aldehydes.

**Location of samplers:** The position of samplers within a room can decisively influence results. Sampling in the vicinity of a suspected emission source, such as furniture made of particle board, often results in higher concentrations than those obtained at other locations in the same room. This will produce an overestimation of the formaldehyde exposure of children present in the classroom. Samplers should be placed at the centre of the classroom (or at a distance of 1.0–2.0 m from a wall) and at a height of 1.0–1.2 m, which is the average breathing zone (ISO 16000-1:2004). Locations in the sun or close to HVAC ducts where there is a noticeable stream of air should be avoided as these can influence results.

**Diffusion samplers:** Aldehyde vapour migrates into the PAS by diffusion and collects on a strip of cellulose paper coated with 2,4-dinitrophenylhydrazine (DNPH) and phosphoric acid (ISO 16000-4:2011). Aldehydes react with DNPH to form stable 2,4-dinitrophenylhydrazones. PAS that are commercially available or made in the laboratory are options if they meet performance requirements. The sampling rate must be invariant with up to 80% relative humidity and wind speeds as low as 0.02 m/s.

**Information on activities and potential sources of emissions:** In addition to the occupancy of the room and people's activities, potential sources of emissions – such as new furniture, recent renovations or the use of chemicals – need to be recorded.

**Sample handling and processing:** After sampling, samplers should be placed in a sealed protective container and stored in a freezer until analysis. Analysis should ideally take place within two weeks from sample collection, or three days if a formaldehyde-DNPH solution has already been extracted.

**Laboratory analysis:** Hydrazones are desorbed from the sampler by acetonitrile and the solution is filtered and analysed by means of a high-performance liquid chromatograph

(HPLC) equipped with an ultraviolet (UV) absorption detector operating at 360 nm. Appropriate advice on safe laboratory procedures should be included in the survey protocol. The analytical procedure and the operation conditions of the HPLC described in ISO 16000-4 and 16000-3 can be used for the determination of acetaldehyde (22). This method is suitable for the determination of these compounds in the approximate concentration range of 1 µg/m<sup>3</sup> to 1 mg/m<sup>3</sup>.

Interferences in this method can be caused by high NO<sub>2</sub> concentrations that could negatively affect formaldehyde sampling and measurement due to the NO<sub>2</sub>–DNPH reaction. The retention time of the NO<sub>2</sub>–DNPH derivative can be similar to that observed for the formaldehyde–DNPH derivative, depending on the parameters selected and the column.

Formaldehyde and acetaldehyde are usually collected simultaneously by means of PAS and analysed by HPLC with a UV detector (22–25).

## 5.2 VOCs

VOCs included in the priority list of chemicals (see Annex 1 and Table 4) are: aromatic hydrocarbons (benzene, ethylbenzene, o-xylene, m,p-xylenes, styrene, toluene, 1,2,3-trimethylbenzene and 1,4-dichlorobenzene), esters (butyl acetate), terpenes (limonene and α-pinene) and chlorinated hydrocarbons (tetrachloroethylene and trichloroethylene). Naphthalene is present in the gas phase and could also be collected and analysed together with the VOCs mentioned above (ISO 16000-5:2007).

A detailed sampling protocol for VOCs is available in ISO 16000-5, and the analytical procedure using TD and capillary GC (Method 1) can be found in ISO 16017-2. An advantage of Method 1 is that it does not require the manipulation of sorbent tubes or the use of hazardous solvents during analysis, specifically during chemical extraction. A different analytical procedure using SD and capillary GC (Method 2) could also be used; this method is described in EN 14662-5.

**Location of samplers:** As most VOCs are emitted from point sources, a strong source will cause a concentration gradient due to insufficient mixing. The position of a sampler can therefore influence the outcome of a measurement. The objective is to determine the concentration of VOCs where air is normally inhaled by children. Samplers should be placed 1.0–2.0 m from a wall and at a height of 1.0–1.2 m.

**Protocol for recording activities and conditions during sampling:** Temporary emissions may occur in classrooms due to people's activities or behaviours, deviations from the normal operation of HVAC systems, and/or changes in microclimate conditions. To correctly evaluate analytical results, these should be documented. Annex D of ISO 160001 provides general guidelines on the information to record.

### 5.2.1 Method 1: sampling and analysis of VOCs by sorbent tube/TD/capillary GC (ISO 16017-2)

**Diffusion samplers:** Organic vapour migrates into the PAS by diffusion and is trapped on an adsorbent (for example, Carbotrap™ B/C, Carbopack™ B/C, Carbograph™ TD-1 or Tenax® TA/TG, preferably with a mesh size of 60–80). PAS can be prepared in the laboratory following the recommendations of ISO 16017-2, or purchased. The tubes must be conditioned before

sampling. ISO 16017-2 provides sampling rates for pollutants using tubes with a 6.3 mm effective diameter, a 5 mm internal diameter and a 90 mm length, and a specific adsorbent. The tubes need to be compatible with the TD instrument.

Poly(2,6-diphenyl-p-phenylenoxid) (Tenax® TA) could be used as an adsorbent, but the sampling rates for 1,4-dichlorobenzene, limonene,  $\alpha$ -pinene and trichloroethylene are not included in ISO 16017-2. Sampling rates can be calculated following EN 838 or ISO 13528-2. However, commercially available sorbent tubes have been tested for the VOCs of interest (except naphthalene for certain commercial brands) using graphitised charcoal. Tenax® can also be used as an adsorbent if the sampling rates for all compounds of interest are provided.

**Sample handling and processing:** After sampling, the diffusive cap is changed to a store cap. The tube must be labelled without the use of paints with solvents, markers or stickers. If samplers are not going to be analysed within eight hours, they are placed in a clean, hermetically sealed metal or glass container without coating.

**Laboratory analysis:** Desorption tubes are placed in a compatible TD. With this technique, VOCs are concentrated in a cold trap prior to injection into the GC. The desorbed compounds are analysed by means of a GC coupled to an MS, a flame ionization detector (FID) or a photoionization detector (PID).

### 5.2.2 Method 2: sampling and analysis of VOCs by sorbent tube/SD/capillary GC (EN 14662-5)

If TD is not available, SD is a cost-effective alternative for the analysis of VOCs. EN 14662-5 is specific for benzene, but the SD methodology can also be used for the rest of the VOCs of interest (25).

**Diffusion samplers:** Organic vapour migrates into the PAS by diffusion and is trapped by adsorption on the adsorbent (generally activated charcoal with a particle size of 0.35–0.85 mm). Commercially available PAS are filled with activated charcoal preconditioned by the manufacturer. Suppliers of PAS used for the collection of VOCs are listed in Annex A of EN 14662-5:2005. The manufacturer determines a preliminary sampling rate.

**Laboratory analysis:** VOCs are extracted with a solvent, usually carbon disulphide (CS<sub>2</sub>). This solution is stirred for 30 minutes and analysed by GC with a FID or MS detector. An internal standard is used for quantification and can be added to the sample after CS<sub>2</sub>. The internal standard is added to correct small variations in the volume of CS<sub>2</sub> injected, and includes 2-fluorotoluene (using a FID) and benzene-d6 and toluene-d8 (using an MS). The same programme of temperature described in ISO 16017-2 is recommended to separate the VOCs in the GC.

## 5.3 SVOCs

WHO classifies SVOCs as indoor organic pollutants with boiling points ranging from 240–260 °C to 380–400 °C (26). When SVOCs are emitted in indoor air, due to their physical and chemical properties, they partition between air (gas and particulate phase) and surfaces (settled dust) (27). Two PAHs (naphthalene and benzo(a)pyrene) have been included in the list of priority chemicals (see Annex 1 and Table 4). Naphthalene has a vapour pressure of  $1.1 \times 10^{-2}$  kPa and is mainly in the gas phase; it can be sampled

together with VOCs according to ISO 16017-2 or EN 146625. Benzo(a)pyrene has a vapour pressure of  $7.3 \times 10^{-10}$  kPa and can be found in the particulate phase (ISO 12884).

The drawbacks of passive sampling for SVOCs include the need for calibration using a reference method, variability in uptake rates and uncertainty in the ability to collect particle-associated chemicals (28). Currently, the most widely used PAS for SVOCs is the polyurethane foam (PUF) disk due to its high retention capacity (29,30). Although PAS were initially developed to collect SVOCs in the gas phase, they have been widely used to report concentrations of SVOCs associated with particles (31). (Note that longer deployment times are required to achieve analytical detection.)

PUF-PAS has been widely used for sampling of outdoor air. Some studies have used them indoors after calibration for PCBs, brominated flame retardants (BFRs), polybrominated diphenyl ethers (PBDEs), organophosphates (OPs) and PAHs (16,31–35). These studies provide different sampling rates for the SVOCs of interest depending on the configuration of the PAS used: a PUF disk inside two stainless steel domes (fully sheltered) or a PUF disk inside one stainless steel dome (partially sheltered). No studies of musks in indoor air using PUF-PAS have been conducted (see Table 4), and studies of phthalates are limited (15,35).

As such, the application of PUF-PAS to all the SVOCs listed in Table 4, especially for PAHs, phthalates and polycyclic musk, is limited in indoor air, and calibrations based on reliable active air sampling are needed. Due to the limitations that the PUF-PAS presents, and because there is a specific ISO standard developed for indoor air (16000-12: 2008), active samplers are preferable for the measurement of SVOCs (especially benzo(a)pyrene) (see Annex 1) as well as other PAHs (see Annex 2).

**Location of samplers:** General rules for sampling are described in ISO 16000-1. Samplers should be placed at a distance from the wall of 1.0–2.0 m and at a height of 1.0–1.2 m.

### 5.3.1 PAHs

The method described in this section can be applied to naphthalene and benzo(a)pyrene (see Annex 1) and to the PAHs listed in Annex 2. A sampling strategy for PAHs is available in ISO 16000-12 using a low-volume sampler (LVS). The protocol for the analysis is available in ISO 12884 for the gas/particle phase and in ISO 16362 for the particle phase. According to ISO 16000-12, the same measurement protocol described in ISO 16000-13 for the determination of total PCBs and PCDDs/PCDFs must be used for the PAHs. Likewise, this measurement protocol could be extended to most of the SVOCs included in Annex 2.

**Active samplers:** Due to noise, only LVS should be used (with a flow range of 1.2–2.8 m<sup>3</sup>/h). If PM<sub>10</sub> and PM<sub>2.5</sub> will be measured (with the appropriate size-selective inlet) at the same time, the filter used to collect these particles can also be used to analyse PAHs. The method described in ISO 16000-13 incorporates a sampling procedure that collects pollutants on a fine-particle filter (for particulate matter) backed up by a sorbent trap consisting of a PUF disk (for vapour-phase fraction). Details regarding sampling can be found in ISO 16000-12.

**Sample handling and processing:** The filter and sorbent trap (PUF disk) are usually conditioned before being used. Filters are pre-baked at high temperatures for several hours, and PUF disks are cleaned with a solvent (such as dichloromethane or toluene) to remove organic compounds. After sampling, filters and sorbent traps are wrapped in aluminium foils



(previously washed with solvents), sealed in a clean and hermetically closed container, and transported to the laboratory refrigerated. The sample is stable for one month.

**Laboratory analysis:** A detailed description of the analytical procedure using GC–MS is included in ISO 12884. The filter and PUF disk can be extracted together using a Soxhlet, accelerated solvent extractor or another extraction technique after the method is validated. The extraction solvent is dichloromethane. Before extraction, recovery standards must be added to the sample. The extract is concentrated, spiked with the internal standards and stored at  $-18^{\circ}\text{C}$  prior to analysis. Information about recovery and internal standards that can be used to quantify PAHs is given in ISO 12884.

A clean-up method can be applied through column chromatography on silica gel. PAHs are analysed by means of a GC–MS operating in selected ion monitoring (SIM) mode or, if a triple quadrupole is available, operating in multiple reaction monitoring (MRM) mode. The characteristic ions for MS detection of selected PAHs as well as internal and recovery standards can be found in ISO 12884, and the transitions monitored using the MRM mode for each compound have been described previously (36,37). The recommended capillary column is 30–50 m x 0.25 mm, with 5% phenylmethylsiloxane (thickness of 0.25  $\mu\text{m}$ ).

**Sampling efficiency or dynamic retention efficiency:** Sampling efficiency is determined in order to assess the PUF retention capacity. For this, a solution of the SVOCs of interest is spiked onto a clean filter backed up with the sorbent cartridge. Air is sampled through the device at the same flow and for the same period of time as the samples.

The dynamic retention efficiency is determined by spiking the sorbent directly with a clean filter placed before the PUF disk and following the same procedure. A detailed description of the procedure is available in ISO 16000-13 and Raffy et al. (7).

## 5.4 Inorganic compounds

Inorganic compounds commonly found indoors are  $\text{NO}_2$ , CO and  $\text{O}_3$ . The most important sources of these pollutants are located outdoors.  $\text{NO}_2$ , which can be collected with a PAS, is the sole inorganic compound that was included in the tool for assessment of risk of combined exposure to hazardous chemicals in indoor air.

### 5.4.1 $\text{NO}_2$

A detailed sampling protocol for  $\text{NO}_2$  is available in ISO 16000-15.

**Location of samplers:** Generally, one sampling point per room is enough. The sampler should be placed 1.0–1.5 m (to a maximum of 2.0 m) from walls and at a height of 1.5 m. Samplers should not be placed near HVAC ducts.  $\text{NO}_2$  in indoor air are influenced by the concentration of  $\text{NO}_2$  in ambient air, especially if the building is located in the vicinity of roads with heavy traffic. If outdoor concentrations will be measured simultaneously, outdoor samplers can be placed in a protective shelter in order to avoid exposure to direct sunlight or precipitation.

**Diffusive samplers:** Several models of PAS for  $\text{NO}_2$  are commercially available. EN 13528-3 is a guide for the selection, use and maintenance of PAS. PAS based on triethanolamine



adsorbent are commonly used. Special care should be taken if relative humidity is high as triethanolamine is very hygroscopic. Additional information can be found in Hafkenscheid et al. (38).

**Sample handling and processing:** After sampling, samplers should be placed in a sealed protective container and kept in the dark at 4 °C until analysis. Although some types of PAS are stable up to four months after sampling, analysis should ideally take place within two weeks of sample collection.

**Laboratory analysis:** NO<sub>2</sub> in the air is captured and chemiadsorbed onto triethanolamine as nitrite ion. Nitrite is quantified by UV-Vis spectrophotometer at 540 nm. The analytical procedure based on the modified Griess-Saltzman method is described in ISO 6768 and EN 16339. More details are available in ISO 16000-15.

PAS with triethanolamine and analysis by Uv-Vis spectrophotometry have been widely used (25,39–42). Ozden & Dogeroglu (43) have prepared a summary of the different methods for sampling and analysis of NO<sub>2</sub>, and an example of the analytical method is available for the Radiello® sampler (44). Other types of PAS (Ogawa, SKC, etc.) are also commercially available.

## 6. METHODS FOR SAMPLING AND ANALYSIS OF OTHER POLLUTANTS OF CONCERN IN INDOOR AIR

This section describes the methods for sampling and analysis of the pollutants included in Annex 2: particulate matter, O<sub>3</sub>, CO, phthalates, musks, BFRs, organophosphate flame retardants (OPFRs) and chlorinated paraffins (CPs). These compounds are common pollutants of indoor air in public settings for children and can be measured for the control of indoor air quality. At this stage, they are not included in the tool for assessment of risk of combined exposure to hazardous chemicals, mainly due to a lack of toxicological information needed for such assessment.

### 6.1 Particulate matter

A detailed description of strategies for the measurement of airborne particles is available in ISO 16000 Part 34. ISO 16000 Part 37 describes strategies for the measurement of indoor PM<sub>2.5</sub> mass concentrations. The measurement methods and strategies described in ISO 16000-37 can also be used for determining concentrations of PM<sub>10</sub>.

There are two kinds of instruments for measuring the concentration of particulate matter: those that provide average concentrations for the sampling period and those that provide real-time monitoring of concentrations. Instruments for the measurement of concentrations in samples are generally based on a gravimetric method and are considered to be the most accurate. Optical instruments based on light scattering, light absorption or light extinction caused by particulate matter allow for real-time measurement. Microbalance methods (such as the TEOM™ sensor) also provide measurements that can be logged in near-real time (45).

ISO 16000-37 recommends the gravimetric method for the measurement of PM<sub>2.5</sub>. High temporal resolution methods are additional options. The SINPHONIE project adopted this strategy: both PM<sub>10</sub> and PM<sub>2.5</sub> were continuously measured by optical light scattering, and PM<sub>2.5</sub> levels were also measured gravimetrically after pumped sampling over a filter.

**Location of samplers:** Samplers should be positioned in the centre of the room at a height of about 1.5 m (the breathing zone of pupils) (ISO 16000-1 and ISO 16000-34).

**Active sampler and gravimetric analysis:** This is the recommended reference method in ISO 16000-37. Measurement procedures and main requirements are similar to the conditions specified in EN 12341 for PM<sub>10</sub> and PM<sub>2.5</sub>, but ISO 16000-34 allows for certain deviations from EN 12341; these are described in Section 7.1 of the document. LVS operating at a constant flow of 2.3 m<sup>3</sup>/h is required. The sampling systems are available from several commercial sources worldwide.

In order to minimize noise, sampling should be carried out with silent LVS. Another option is to place pumps in sound-insulated boxes. The reference method principle consists of collecting particulate matter on a circular filter. This must be a glass fibre filter (GFF), quartz fibre filter (QFF), polytetrafluoroethylene (PTFE) or PTFE-coated glass fibre, and must be carefully conditioned before and after collection. The filter is weighed before and after collection.

PM<sub>10</sub> or PM<sub>2.5</sub> collected in the filter (depending on the particle size-selective inlet) can be analysed to determine SVOCs as well. More details on sampling and instruments can be found in ISO 16000-37 and EN 12341.

**Sample handling and processing:** Before and after exposure, the filters must be conditioned in a weighing room. The methodology for conditioning, storing and transporting samples together with QA/QC procedures are described in detail in EN 12341:2014.

**High temporal resolution instruments:** These instruments present advantages with respect to the gravimetric method: the time resolution is in the order of minutes, it is possible to monitor peaks in concentrations, they are easy to use and they provide much more data. For all these reasons, automatic monitors are widely used for indoor air and are considered supplementary methods in ISO 16000-37. They can also be considered for QA/QC of the reference method (ISO 16000-37:2019). ISO 16000-34 includes a detailed description of the instruments that should be used.

## 6.2 Inorganic compounds

Inorganic compounds commonly found indoors are the outdoor air pollutants NO<sub>2</sub> (see Section 5.4.1), O<sub>3</sub> and CO. O<sub>3</sub> can be emitted indoor when photocopiers or laser printers are in use in addition to penetration with ambient air.

### 6.2.1 O<sub>3</sub>

An ISO standard for sampling and analysis of O<sub>3</sub> in indoor air has not yet been developed. The methods described in ISO standards for the measurement of O<sub>3</sub> in ambient air are for continuous measurement. As for NO<sub>2</sub>, several models of PAS as well as general guidance for their selection and use are commercially available.

**Location of samplers:** The protocol described for measuring NO<sub>2</sub> can generally be followed for measuring O<sub>3</sub>. Some specific recommendations for sampling are given by manufacturers.

**Diffusive samplers:** Different PAS have been developed for measuring O<sub>3</sub>. The analytical methodology varies according to the commercial sampler used. For example, the Ogawa samplers developed at Harvard University by Koutrakis et al. (46) are based on the oxidation of nitrite to nitrate by O<sub>3</sub>. The Radiello® sampler developed by Cocheo (47) at the Fondazione Salvatore Maugeri and the Passam G® sampler developed by Monn & Hangartner (48) at the Swiss Federal Institute of Technology are based on the reaction of 4,4'-dipyridylethylene with O<sub>3</sub> to yield 4-pyridylaldehyde. Later, 4-pyridylaldehyde reacts with 3-methyl-2-benzothiazolinone hydrazone (MTBH) to yield the corresponding azide. These three sampling devices have been tested successfully in indoor and outdoor air studies (12,40,49–52). More information about PAS can be found in EN 14412 and EN 13528-3.

**Sample handling and processing:** After exposure, the samplers must be stored in the dark and should be analysed within a week. Specific procedures are given by manufacturers.

**Laboratory analysis:** If O<sub>3</sub> is captured as nitrate, this ion is extracted with Milli-Q® water in an ultrasonic bath and analysed by means of ion chromatography. A similar analytical procedure to that described by Ogawa (53) is given by Occupational Safety and Health Administration (OSHA) Method ID-214.

If 4,4'-dipyridylethylene is used to collect  $O_3$ , the samples are extracted with freshly prepared MTBH solution and filtered. The absorbance of the corresponding azide is measured at 430 nm in a UV-Vis spectrophotometer. In this case, the formation of 4-pyridylaldehyde is specific for the reaction of  $O_3$ , and neither organic pollutants nor nitrogen oxides interfere. These methods are in accordance with EN 14412 and EN 13528-3.

### 6.2.2 CO

An ISO standard for the sampling of CO in indoor air has not yet been developed. Both indoor and outdoor measurements of CO have been conducted in previous studies using an electrochemical sensor. Specific automatic, portable, reasonably priced sensors are options for continuous measurement of CO. The SINPHONIE project, for example, measured CO continuously with a low-cost logger (12).

Although PAS for measuring CO have been developed, these are not yet widely used. In ambient air, the reference method is based on the non-dispersive infrared spectroscopic measuring principle, and is continuous (EN 14626:2012). Different types of automatic loggers for measuring CO,  $CO_2$ , temperature and relative humidity are commercially available. Ventilation rates in indoor air are usually calculated based on  $CO_2$  measurements. A detailed sampling protocol for  $CO_2$  is available in ISO 16000-26.

**Location of samplers:** The same protocol described for other inorganic compounds can be followed for CO measurements.

**Automatic portable indoor air quality monitors:** Several models for measuring CO are commercially available. The equipment is supplied with a factory calibration certificate, but should be checked prior to use by means of appropriate calibration kits. Several studies have used this type of monitor for indoor air in schools (54–57).

## 6.3 SVOCs

This section is focused on sampling and analysis methods for the SVOC families listed in Annex 2: PAHs, phthalates, musks, BFRs, OPFRs and CPs. In order to minimize costs, SVOCs could be collected using the same sampling and analytical method described in 16000-12 for PAHs, PCBs and PCDDs/PCDFs (filter + PUF disk and GC–MS). The SVOCs selected present compatibility with a multi-residue GC–MS analysis (36). However, specific protocols for some SVOCs (PAHs, phthalates and OPFRs) have been developed in the ISO 16000 series; these are described in Section 5 and below.

### 6.3.1 Phthalates

A detailed sampling protocol for phthalates is available in ISO 16000-33.

**Active samplers:** Phthalates can be collected on a sorbent tube with Tenax® TA according to ISO 16000-6 or in a sorbent tube filled with Florisil® (ISO 16000-33). Sorbent tubes are commercially available or can be prepared in the laboratory following the instructions given in 16000-33. The air is forced to pass by means of a sampling pump. Flow should be 50–20 mL/min (sampling volume of 20–70 L) for sorbent tubes filled with Tenax® TA. A flow of 2 L/min (sampling volume of 1–3 m<sup>3</sup>) is recommended for sorbent tubes filled with Florisil®.

**Laboratory analysis:** Sorbent tubes with Tenax® TA are extracted by TD, and tubes with Florisil® are extracted with a solvent and analysed by means of GC–MS. More details about the analytical method can be found in ISO 16000-33.

### 6.3.2 Musks

Neither ISO nor EN standards recommend a method for measuring musks. As such, a method published in a peer-reviewed journal can be considered (7,58–60).

**Active samplers:** Synthetic musks have most commonly been collected by active sampling with flow ranges between 2 L/min and 0.3–0.4 m<sup>3</sup>/min (sampling volume of 2–100 m<sup>3</sup>). LVS and high-volume samplers with a GFF or QFF to collect airborne particulate matter and a PUF disk to collect the gas phase have been explored for the collection of musks. For instance, Raffy et al. (7) used an active sampler consisting of a QFF fitted in front of a PUF disk placed in a glass tube. The air was pumped through the device using a personal sampling pump at 2 L/min for five consecutive days.

The flow rate must be checked before and after sampling with an external calibrator. This pump can be located in a sound-insulated box to avoid disturbing the occupants of the classroom. Another option for the collection of musks is to use the LVS described for PAHs in Section 5.3.1; this also reduces the number of samplers. Musks have been found to be dominant in the gas phase (59,61).

**Laboratory analysis:** Extraction of musks from the filter and PUF disk can be carried out by Soxhlet (or another extraction technique) using different solvent mixtures. Chen et al. (59) used a Soxhlet with dichloromethane for 72 hours while Raffy et al. (7) used pressurized liquid extraction with dichloromethane. More information about extraction and clean-up procedures can be found in previously mentioned studies (7,14,59,61).

In all of the studies of musks mentioned above, analysis was carried out by means of GC–MS. Melymuk et al. (62) provides details on the parameters of GC–MS; Raffy et al. (7) and Mercier et al. (36) include details on GC–tandem mass spectrometry (GC–MS/MS). Mass-labelled PAH compounds have been successfully used as internal standards in polycyclic musks analysis in some studies (16,62,63); d10-p-terphenyl (25 ng) could therefore be used as a recovery standard and d10-fluoranthene as an internal standard.

### 6.3.3 BFRs

Currently, there is no ISO or EN standard for the determination of BFRs in air. BFRs have been collected in indoor air by passive and active sampling. However, due to the limitations of PUF-PAS explained above, and in order to reduce the number of samplers, only the active sampler is considered in this publication.

**Active samplers:** These consist of a GFF or QFF followed by a PUF disk as explained above. In general, sample volumes in the range of a few hundred litres to less than 30 m<sup>3</sup> are enough to reach a low indoor LOD for most compounds. However, a lower LOD can be achieved if sample volumes are higher (100–385 m<sup>3</sup>) (14). The LVS described in ISO 16000-13 and specified in Section 5.3.1 for PAHs is therefore an option for BFRs as well.

Other active samplers with personal sampling pumps, such as that used by Raffy et al. (7), have been satisfactorily used to collect PBDEs (BDE 85, BDE 99, BDE 100 and BDE 119)

in schools. Sakhi et al. (64) used a similar device to measure BDE 47, BDE 85, BDE 99, BDE 100, BDE 153 and BDE 154 with a flow of 12 L/min for 24 hours.

**Laboratory analysis:** Despite its drawbacks, Soxhlet is most commonly used to extract BFRs from solvents due to its high extraction efficiency and general robustness (13). However, other extraction systems have also been used, such as pressure solvent extraction or ultrasound-assisted extraction. Typical solvents are dichloromethane, n-hexane, toluene, acetone or a mixture of these. Treatment with concentrated sulphuric acid and a variety of clean-up procedures using different sorbents (alumina, silica gel, Florisil® or a combination of these) are most common (14,64).

Separation of BFRs is generally performed by means of GC–MS. Details of the analytical method are described in Melymuk et al. (62), Lim et al. (65) and Braouezec et al. (66), and GC–MS/MS parameters can be found in Braouezec et al. (66). Other detectors coupled to a GC to quantify PBDEs are a high-resolution mass spectrometer (GC–HRMS) (65,67) and an MS in electron capture negative ionization mode (GC–ECNI–MS) (68). Commonly used internal standards of PBDEs are <sup>13</sup>C-labelled BDE 28, BDE 47, BDE 99, BDE 153, BDE 183 and BDE 209. Recovery standards are <sup>13</sup>C-labelled BDE 77 and BDE 138 (62,67).

ISO 22032 and United States Environmental Protection Agency (EPA) Method 1614A for determining PBDEs in different environmental samples might be useful for establishing the analytical procedure.

#### 6.3.4 OPFRs

ISO 16000-31 gives a detailed description of the sampling and analysis method for flame retardants based on organophosphorus compounds – phosphoric acid esters.

**Active samplers:** The recommended sampling system for OPFRs is described in detail in ISO 16000-13. OPFRs are collected from air on a fine-particle GFF and a PUF disk in an LVS with a flow of 2.7–2.8 m<sup>3</sup>/h. This sampler is also recommended for collecting PAHs in ISO 16000-12 and described in Section 5.3.1 above. The same sampling device could be considered for sampling the rest of the SVOCs (except phthalates) as well. The sampling time is one hour and the sampling volume should not exceed 10% of the air exchange rate.

**Laboratory analysis:** The GFF and PUF disk are extracted together in a Soxhlet extractor with dichloromethane, and the analysis is carried out by means of GC–HRMS or GC–LRMS. The compounds are quantified with the internal standard method.

#### 6.3.5 CPs

CP is a common term for a large group of chlorinated aliphatic substances. These can be classified into short-chain CPs (SCCPs, C<sub>10–13</sub>), medium-chain CPs (MCCPs, C<sub>14–17</sub>), and long-chain CPs (LCCPs, C<sub>18–30</sub>) according to their carbon length. Commonly applications include additives in polyvinyl chloride; flame retardants in plastics, rubbers and textiles; cooling fluids and lubricants in metal processing; and plasticizers in paints, rubbers and plastics (69).

**Active samplers:** The same sampling system described in ISO 16000-12 for PAHs (see Section 5.3.1) and in ISO 16000-31 for OPFRs is applicable for sampling CPs. CPs can be

collected from air on a fine-particle filter backed by a sorbent trap (PUF). Other LVS have also been described in the literature (15,64).

**Laboratory analysis:** Analytical methods for SCCPs and MCCPs have been described previously (15,64,70). CPs collected on a filter, or on a filter plus a PUF disk, are extracted using different techniques and cleaned on a multilayer silica gel column. The most common analytical system used is a GC–ECNI–MS, although some authors have reported successful use of a GC–MS/MS (15). More information about the procedure to sample and analyse CPs in indoor air can be found in Zhou et al. (71) and Huang et al. (17).



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## ANNEX 1. LIST OF PRIORITY CHEMICALS INCLUDED IN THE SCREENING TOOL FOR ASSESSMENT OF RISK OF COMBINED EXPOSURE TO HAZARDOUS CHEMICALS

No.	Chemical family		Substances	Chemical Abstracts Services (CAS) number
1	<b>Oxygenated volatile organic compounds (oxy-VOCs)</b>	Aldehydes	Formaldehyde	50-00-0
2			Acetaldehyde	75-07-0
3	<b>Volatile organic compounds (VOCs)</b>	Aromatic hydrocarbons	Benzene	71-43-2
4			Ethylbenzene	100-41-4
5			xylene (o-, m-, p-)	95-47-6
				108-38-3/106-42-3
6			Styrene	100-42-5
7			Toluene	108-88-3
8			1,2,3-trimethylbenzene	526-73-8
9			1,4-dichlorobenzene	106-46-7
10		Esters	Butyl acetate	123-86-4
11		Terpenes	Limonene	138-86-3
12			$\alpha$ -pinene	80-56-8
13		Chlorinated hydrocarbons	Tetrachloroethylene	127-18-4
14			Trichloroethylene	79-01-6
15		Polycyclic aromatic hydrocarbons (PAHs)	Naphthalene	91-20-3
16	<b>Semi-volatile organic compounds (SVOCs)</b>	PAHs	Benzo(a)pyrene	50-32-8
17	<b>Inorganic compounds</b>	Nitrogen dioxide (NO <sub>2</sub> )	NO <sub>2</sub>	10102-44-0

## ANNEX 2. OTHER POLLUTANTS OF CONCERN IN INDOOR AIR

No.	Chemical family	Substances
1	<b>Particulate matter</b>	Particulate matter with an aerodynamic diameter below 10 µm (PM <sub>10</sub> )
2		Particulate matter with an aerodynamic diameter below 2.5 µm (PM <sub>2.5</sub> )
3	<b>Inorganic compounds</b>	Carbon monoxide (CO)
4		Ozone (O <sub>3</sub> )
5	<b>Phthalates</b>	Diethyl phthalate (DEP)
6		Diisobutyl phthalate (DiBP)
7		Di-n-butyl phthalate (DnBP)
8	<b>Musks</b>	Galaxolide
9		Tonalide
10	<b>PAHs</b>	Acenaphthene
11		Acenaphthylene
12		Phenanthrene
13		Anthracene
14		Benz[a]anthracene
15		Benzo[b]fluoranthene
16		Benzo[j]fluoranthene
17		Benzo[e]pyrene
18		Benzo[ghi]perylene
19		Benzo[k]fluoranthene
20		Chrysene
21		Dibenz[a,h]anthracene
22		Dibenzo[a,l]pyrene
23		Fluoranthene
24		Fluorene
25		Indeno[1,2,3-cd]pyrene
26		Pyrene
27	<b>Brominated flame retardants (BFRs)–polybrominated diphenyl ethers (PBDEs)</b>	2,4,4'-tribromodiphenyl ether (BDE 28)
28		2,2',4,4'-tetrabromodiphenyl ether (BDE 47)
29		2,2',4,4',5-pentabromodiphenyl ether (BDE 99)
30		2,2',4,4',6-pentabromodiphenyl ether (BDE 100)
31		2,2',4,4',5,5'-hexabromodiphenyl ether (BDE 153)
32		2,2',3,4,4',5,6-heptabromodiphenyl ether (BDE 183)
33		2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether (BDE 209)
34		1,2-dibromo-4-(1,2-dibromoethyl)cyclohexane (DBE-DBCH)

No.	Chemical family	Substances
35	<b>Organophosphate flame retardants (OPFRs)</b>	Tributyl phosphate (TBP)
36		Tris(2-butoxyethyl) phosphate (TBEP)
37		Tris(1-chloropropan-2-yl) phosphate (TCPP)
38		Tris(2-chloroethyl) phosphate (TCEP)
39	<b>Chlorinated paraffins (CPs)</b>	Short-chain CPs (SCCPs) (C <sub>10-13</sub> )
40		Medium-chain CPs (MCCPs) (C <sub>14-17</sub> )
41		Long-chain CPs (LCCPs) (C <sub>18-30</sub> )



## ANNEX 3. LIST OF RECOMMENDED METHODS

1. ASTM D1607-91(2005). Standard test method for nitrogen dioxide content of the atmosphere (Griess-Saltzman Reaction)
2. EPA Method TO-15, 1999. Compendium of methods for the determination of toxic organic compounds in ambient air- second edition compendium method TO-15. Determination of volatile organic compounds (VOCs) in air collected in specially prepared canisters and
3. EPA Method 1614A, 2010. Brominated diphenyl ethers in water, soil, sediment, and tissue by HRGC/HRMS
4. NIOSH 6700. Nitrogen dioxide (Diffusive sampler)
5. ISO 5725. Accuracy (trueness and precision) of measurement methods and results package
6. ISO 5725-1:1994. Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions
7. ISO 5725-3:1994. Accuracy (trueness and precision) of measurement methods and results – Part 3: Intermediate measures of the precision of a standard measurement method
8. ISO 6768:1998. Ambient air – Determination of mass concentration of nitrogen dioxide – Modified Griess-Saltzman method
9. ISO 12884:2000. Ambient air – Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons – Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analyses
10. ISO 13964:1998. Air quality – Determination of ozone in ambient air – Ultraviolet photometric method
11. ISO 16000-1:2004. Indoor air – Part 1: General aspects of sampling strategy
12. ISO 16000-2:2004. Indoor air – Part 2: Sampling strategy for formaldehyde
13. ISO 16000-3:2011. Indoor air – Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air – Active sampling method
14. ISO 16000-4:2011. Indoor air – Part 4: Determination of formaldehyde – Diffusive sampling method
15. ISO 16000-5:2007. Indoor air – Part 5: Sampling strategy for volatile organic compounds
16. ISO 16000-6:2011. Indoor air – 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

17. ISO 16000-12:2008. Indoor air – Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)
18. ISO 16000-13:2008. 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
19. ISO 16000-15:2008. Indoor air – Part 15: Sampling strategy for nitrogen dioxide (NO<sub>2</sub>)
20. ISO 16000-26:2012. Indoor air – Part 26: Sampling strategy for carbon dioxide (CO<sub>2</sub>)
21. ISO 16000-31:2014. Indoor air – Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds – Phosphoric acid ester
22. ISO 16000-33:2017. Indoor air – Part 33: Determination of phthalates with gas chromatography/mass spectrometry (GC/MS)
23. ISO 16000-34:2018. Indoor air – Part 34: Strategies for the measurement of airborne particles
24. ISO 16000-37:2019. Indoor air – Part 37: Measurement of PM<sub>2.5</sub> mass concentration
25. ISO 16017-1:2000. Indoor, ambient and workplace air – Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography – Part 1: Pumped sampling
26. ISO 16017-2:2003. Indoor, ambient and workplace air – Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography – Part 2: Diffusive sampling
27. ISO 16362:2005. Ambient air – Determination of particle-phase polycyclic aromatic hydrocarbons by high performance liquid chromatography
28. ISO 22032:2006. Water quality – Determination of selected polybrominated diphenyl ethers in sediment and sewage sludge – Method using extraction and gas chromatography/mass spectrometry
29. EN 12341:2014. Ambient air – Standard gravimetric measurement method for the determination of the PM<sub>10</sub> or PM<sub>2.5</sub> mass concentration of suspended particulate matter
30. EN 13528-2:2003. Ambient air quality – Diffusive samplers for the determination of concentrations of gases and vapours – Requirements and test methods – Part 2: Specific requirements and test methods
31. EN 13528-3:2003. Ambient air quality – Diffusive samplers for the determination of concentrations of gases and vapours. Requirements and test methods – Part 3: Guide to selection, use and maintenance
32. EN 14211:2012. Ambient air – Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence

33. EN 14412:2005. Indoor air quality – Diffusive samplers for the determination of concentrations of gases and vapours – Guide for selection, use and maintenance
34. EN 14662-2:2005. Ambient air quality – Standard method for measurement of benzene concentrations – Part 2: Pumped sampling followed by solvent desorption and gas chromatography
35. EN 14662-5:2005. Ambient air quality – Standard method for measurement of benzene concentrations – Part 5: Diffusive sampling followed by solvent desorption and gas chromatography
36. EN 838:2010. Workplace exposure – Procedures for measuring gases and vapours using diffusive samplers – Requirements and test methods
37. EN 14625:2012. Ambient air – Standard method for the measurement of the concentration of ozone by ultraviolet photometry
38. EN 14626:2013. Ambient air – Standard method for the measurement of the concentration of carbon monoxide by non-dispersive infrared spectroscopy
39. EN 16339:2013. Ambient air – Method for the determination of the concentration of nitrogen dioxide by diffusive sampling
40. OSHA Method ID-214. Ozone in workplace atmospheres (Impregnated Glass Fiber Filter) 2008.

## Annex 4. Summary of methods for sampling and analysis of pollutants in indoor air

This section provides a summary of methods for sampling and analysis of the most commonly occurring chemical pollutants in indoor air in public settings for children (schools, kindergartens and day-care centres), as identified by the WHO Regional Office for Europe. The objective is not to provide an exhaustive list of sampling and analytical methods, but rather to identify well established methods recommended by the International Organization for Standardization (ISO) or the European Committee for Standardization.

This section also presents sampling and analysis methods that have been published in peer-reviewed journals. While these methods have been widely used in studies of indoor air quality, they are not standardized; however, they have certain advantages, such as ease of handling and low cost. For example, a polyurethane foam disk on a passive air sampler (PUF-PAS) has been successfully used to collect certain compounds, such as PBDEs, in several studies.

The methods for sampling and analysis are compiled in the table 4 below. The table contains the standard methods for sampling and analysis of oxy-VOCs (aldehydes) and VOCs using passive or active sampling, thermal desorption (TD) or solvent desorption (SD) and a gas chromatography–mass spectrometry/flame ionization detector (GC–MS/FID). Reference to the standard methods for measuring benzene in ambient air as well as other VOCs in both outdoor and indoor air is also provided in the table.

For PAHs, the proposed method includes the sampling procedure recommended in ISO 16000-12 and is based on a low-volume sampler (LVS) (a filter plus a PUF disk) described in ISO 16000-13. This sampling device is also recommended for dioxin-like PCBs and PCDDs/PCDFs in ISO 16000-12 and for OPFRs in ISO 16000-31. This LVS could be used to collect the rest of the SVOCs (musks, PBDEs and CPs).

The table contains the standard methods for sampling and analysis of  $PM_{10}$  and  $PM_{2.5}$  means of LVS and gravimetric analysis including the direct reading that is widely used and included in ISO 16000-37 as a supplementary high temporal resolution method.

Finally, the table presents individual methods for sampling and analysis of inorganic compounds –  $NO_2$ , CO and  $O_3$  – in indoor air. Standard methods based on continuous measurements recommended in the European normative (EN) for ambient air and methods based on passive sampling have been also included. These are widely used for measuring concentrations of inorganic compounds in both outdoor and indoor air.

Table 4. Methods for sampling and analysis of pollutants in indoor air

Sampling and/or analytical method (title)		Sampling device	Analytical method	Application of the method (references)
<b>Aldehydes (formaldehyde and acetaldehyde) in gaseous phase</b>				
ISO 16000-2	Sampling strategy for formaldehyde	-	-	-
ISO 16000-4	Determination of formaldehyde in indoor air	Diffusive sampler (2,4-DNPH)	HPLC-UV* (360 nm)	Geiss et al., 2011 (1) Madureira et al., 2016 (2) Canha et al., 2016 (3) Villanueva et al., 2018 (4)
ISO 16000-3	Determination of formaldehyde and other carbonyl compounds	Active sampler (2,4-DNPH)	HPLC-UV/ photodiode array (360 nm)	Chiappini et al., 2011 (5)
<b>Aromatic hydrocarbons<sup>1</sup>, terpenes<sup>2</sup>, esters<sup>3</sup>, chlorinated hydrocarbons<sup>4</sup>, alkanes<sup>5</sup>, polyromantic hydrocarbon (naphthalene) in gaseous phase</b>				
ISO 16000-5	Sampling strategy for volatile organic compounds	-	-	-
ISO 16017-2	Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography. Part 2: Diffusive sampling	Diffusive sampler (Tenax, graphitised charcoal..)	TD-GC-MS/FID	Cipolla et al., 2017 Zhong et al., 2017 Faber et al., 2015 de Gennaro et al., 2014 Zhu et al., 2013 de Gennaro et al., 2013 Geiss et al., 2011
ISO 16017-1	Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography -- Part 1: Pumped sampling	Active Sampler (PSP)(Tenax, graphitised charcoal..)	TD-GC-MS/FID	-
ISO 16000-6	Determination of VOCs in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID	Active sampler (PSP)(Tenax TA(R))	TD-GC-MS/FID	Kalimeri et al., 2016 Mishra et al., 2015 Sofuoglu et al., 2011 Hoang et al., 2017

<sup>1</sup> Benzene, ethylbenzene, xylenes (o-, m-, p-), styrene, toluene, 1,2,3-trimethylbenzene, 1,4-dichlorobenzene.

<sup>2</sup> Limonene,  $\alpha$ -pinene.

<sup>3</sup> Butyl acetate.

<sup>4</sup> Tetrachloroethylene, trichloroethylene.

<sup>5</sup> N-Heptane, n-decane.

\* ECNI: electron capture negative ionization; HRMS: high-resolution mass spectrometry; LRMS: low-resolution mass spectrometry; QFF: quartz fibre filter.

Table 4 contd.

Sampling and/or analytical method (title)		Sampling device	Analytical method	Application of the method (references)
<b>Aromatic hydrocarbons<sup>1</sup>, terpenes<sup>2</sup>, esters<sup>3</sup>, chlorinated hydrocarbons<sup>4</sup>, alkanes<sup>5</sup>, polyaromatic hydrocarbon (naphthalene) in gaseous phase</b>				
EN 14662-2 <sup>i</sup>	Ambient air quality - Standard method for measurement of benzene concentrations - Part 2 : Pumped sampling followed by solvent desorption and gas chromatography	Active sampler (activated charcoal)	Solvent desorption-GC-MS/FID	-
EN 14662-5 <sup>i</sup>	Ambient air quality - Standard method for measurement of benzene concentrations - Part 5: Diffusive sampling followed by solvent desorption and gas chromatography	Diffusive sampler (Activated charcoal)	Solvent desorption-GC-MS/FID	Pegas et al., 2011 (16) Norbäck et al., 2017 (17) Demirel et al., 2014 (18) Villanueva et al., 2018 (4)
EPA-TO15	Canisters and chromatographic separation	Active sampler	GC-MS	Wang et al., 2016 (19)
<b>PAHs (naphthalene and benzo(a)pyrene) in gaseous and particulate phase</b>				
ISO 16000-12	Indoor air – Part 12: Sampling strategy for PCBs, PCDDs, PCDFs and PAHs	Active sampler (LVS recommended in ISO 16000-13)	-	Oliveira et al., 2015 (20)
ISO 12884	Ambient air – Determination of total (gas and particle phase) polycyclic aromatic hydrocarbons – Collection on sorbent-backed filters with GC-MS	-	GC-MS	-
ISO 16362	Ambient air – Determination of particle-phase polycyclic aromatic hydrocarbons by HPLC	-	HPLC	-
Publication in peer-reviewed journal	Collection on QFF* and PUF disk with personal sampling pump	Active sampler (personal sampling pump)	GC-MS/MS	Raffy et al., 2017 (21)
<b>Phthalates: DEP, DiBP, DnBP mainly in gaseous phase</b>				
ISO 16000-33	Determination of phthalates with gas chromatography/mass spectrometry (GC/MS)	Active sampler	TD-GC-MS	Kang et al., 2005 (22) Sakhi et al., 2019 (23)

<sup>1</sup> Benzene, ethylbenzene, xylenes (o-, m-, p-), styrene, toluene, 1,2,3-trimethylbenzene, 1,4-dichlorobenzene.

<sup>2</sup> Limonene,  $\alpha$ -pinene.

<sup>3</sup> Butyl acetate.

<sup>4</sup> Tetrachloroethylene, trichloroethylene.

<sup>5</sup> N-Heptane, n-decane.

<sup>i</sup> The solvent desorption methodology can also be used to analyse the rest of the VOCs.

Table 4 contd.

Sampling and/or analytical method (title)		Sampling device	Analytical method	Application of the method (references)
<b>Polycyclic musk: galaxolide (HHCB) and tonalide (AHTN) in gaseous and particulate phase</b>				
ISO 16000-13 (only for sampling system) <sup>ii</sup>	Determination of total (gas and particle phase) PCBs and PCDDs/PCDFs – Collection on sorbent-backed filters	Active sampler – LVS	GC-MS/MS	Fontal et al., 2016 (24)
Publication in peer-reviewed journal	Collection on QFF and PUF disk with personal sampling pump	Active sampler	GC-MS/MS	Raffy et al., 2017 (21)
<b>BFRs–PBDEs: BDE 28, BDE 47, BDE 99, BDE 100, BDE 153, BDE 183, BDE 209, DBE-DBCH in gaseous and particulate phase</b>				
ISO 16000-13 (only for sampling system) <sup>ii</sup>	Determination of total (gas and particle phase) PCBs and PCDDs/PCDFs – Collection on sorbent-backed filters	Active sampler	GC-ECNI*-MS	Melymuk et al., 2011 (25)
Publication in peer-reviewed journal	Collection on QFF and PUF disk with personal sampling pump	Active sampler	GC-MS/MS	Sakhi et al., 2019 (23) Raffy et al., 2017 (21)
PUF-PAS	Collection on PAS with PUF disk	Diffusive sampler	GC-MS	Saini et al., 2015 (26) Wilford et al., 2004 (27) Melymuk et al., 2016 (28)
<b>OPFRs: TBP, TBEP, TCPP, TCEP in gaseous and particulate phase</b>				
ISO 16000-31	Measurement of flame retardants and plasticizers based on organophosphorus compounds – Phosphoric acid ester	Active sampler (LVS recommended in ISO 16000-13)	GC-HRMS* GC-LRMS*	-
<b>CPs: SCCPs, MCCPs, LCCPs in gaseous and particulate phase</b>				
ISO 16000-13 (only for sampling system) <sup>ii</sup>	Determination of total (gas and particle-phase) PCBs and PCDDs/PCDFs – Collection on sorbent-backed filters	-	-	Sakhi et al., 2019 (23)
Publication in peer-reviewed journal	Collection on QFF and PUF disk with personal sampling pump	-	GC-MS/MS GC-ECNI-MS	Huang et al., 2017 (29) Fridén et al., 2011 (30)

<sup>ii</sup> There is no recommended method in ISO or EN standards for musks, BFRs or CPs, but the sampling system recommended in ISO 16000-12 for PAHs and described in ISO 16000-13 could be used to collect them.

\* ECNI: electron capture negative ionization; HRMS: high-resolution mass spectrometry; LRMS: low-resolution mass spectrometry; QFF: quartz fibre filter.



Table 4 contd.

Sampling and/or analytical method (title)		Sampling device	Analytical method	Application of the method (references)
<b>Particulate matters (PM<sub>2.5</sub> and PM<sub>10</sub>)</b>				
ISO 16000-34	Strategies for the measurement of airborne particles	-	-	-
ISO 16000-37	Measurement of PM <sub>2.5</sub> mass concentration	Active sampler LVS (EN 12341)	Gravimetric- microbalance	Vicente et al., 2017 (31) Błaszczuk et al., 2017 (32) Fromme et al., 2008 (33) Alves et al., 2013 (34)
Direct reading Supplementary high temporal resolution method (ISO 16000-34)	Real-time particulate monitoring	Continuous monitoring	-	Branco et al., 2014 (35) Madureira et al., 2016 (2) Mathew et al., 2015 (36)
<b>Inorganic compounds in gaseous phase: nitrogen dioxide (NO<sub>2</sub>)</b>				
ISO 16000-15	Sampling strategy for NO <sub>2</sub>	-	-	-
ISO 6768	Determination of mass concentration of NO <sub>2</sub> – Modified Griess-Saltzman method	Diffusive sampler	UV-Vis* spectrophotometer	Demirel et al., 2014 (18)
EN 16339	Method for the determination of the concentration of NO <sub>2</sub> by diffusive sampling	Diffusive sampler	UV-Vis Spectrophotometer Ion chromatography	Villanueva et al., 2018 (4) Hafkenschheid et al., 2009 (37)
ASTM D1607-91	Standard Test Method for NO <sub>2</sub> Content of the Atmosphere (Griess-Saltzman Reaction)	Active sampler	UV-Vis spectrophotometer	-
EN 14211	Standard method for the measurement of the concentration of NO <sub>x</sub> by chemiluminescence	Automatic sampler	Chemiluminescence	Tolis et al., 2018 (38)
<b>Inorganic compounds in gaseous phase: ozone (O<sub>3</sub>)</b>				
MTBH <sup>6</sup> method According to guidelines EN 14412 and 13528-3	Ozone (O <sub>3</sub> )- 4,4'-dipyridylethylene (MTBH method)	Diffusive sampler	UV-Vis <sup>7</sup> spectrophotometer	Maugeri, 2003 (52) Ogawa & Co., 2001 (53) Sanz et al., 2007 (39) Martin et al., 2010 (40) Bozkurt et al., 2015 (41) Finco et al., 2017 (42) SINPHONIE project (43)

<sup>6</sup> 3-methyl-2-benzothiazolinone hydrazine.<sup>7</sup> ultraviolet-visible.

Table 4 contd.

Sampling and/or analytical method (title)		Sampling device	Analytical method	Application of the method (references)
<b>Inorganic compounds in gaseous phase: ozone (O<sub>3</sub>)</b>				
Nitrite method According to guidelines EN 14412 and 13528-3	Protocol for O <sub>3</sub> measurement	Diffusive sampler	Ion chromatography	Grivas et al., 2017 (44) Dimakopoulou et al., 2017 (45)
EN 14625 and ISO 13964	Ultraviolet photometric method	Automatic sampler	UV photometry	Blondeau et al., 2005 (46) Poupard et al., 2005 (47)
<b>Inorganic compounds in gaseous phase: carbon monoxide (CO)</b>				
Direct reading	Automatic portable indoor air quality monitors (electrochemical cell–continuous logger)	-	Electrochemical sensor	Pegas et al., 2012 (48) Chithra & Shiva Nagendra, 2012 (49) Yang et al., 2009 (50) Chaloulakoua & Mavroidisb, 2002 (51) SINPHONIE project (43)
EN 14626	Non-dispersive infrared analyser	Automatic sampler	Non-dispersive infrared spectroscopy	Yang et al., 2009 (50)

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