# INTERNATIONAL STANDARD

ISO 16000-4

Second edition 2011-12-01

## Indoor air —

Part 4:

# Determination of formaldehyde — Diffusive sampling method

Air intérieur —

Partie 4: Dosage du formaldéhyde — Méthode par échantillonnage diffusif



ISO 16000-4:2011(E)



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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 16000-4 was prepared by Technical Committee ISO/TC 146, Air quality, Subcommittee SC 6, Indoor air.

This second edition cancels and replaces the first edition (ISO 16000-4:2004), which has been technically revised, especially 7.2.

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

- Part 1: General aspects of sampling strategy
- Part 2: Sampling strategy for formaldehyde
- Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method
- Part 4: Determination of formaldehyde Diffusive sampling method
- Part 5: Sampling strategy for volatile organic compounds (VOCs)
- Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA<sup>®</sup> sorbent, thermal desorption and gas chromatography using MS or MS-FID
- Part 7: Sampling strategy for determination of airborne asbestos fibre concentrations
- Part 8: Determination of local mean ages of air in buildings for characterizing ventilation conditions
- Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method
- Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method
- Part 11: Determination of the emission of volatile organic compounds from building products and furnishing — Sampling, storage of samples and preparation of test specimens
- Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)

- Part 13: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) — Collection on sorbent-backed filters
- Part 14: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) — Extraction, clean-up and analysis by high-resolution gas chromatography and mass spectrometry
- Part 15: Sampling strategy for nitrogen dioxide (NO<sub>2</sub>)
- Part 16: Detection and enumeration of moulds Sampling by filtration
- Part 17: Detection and enumeration of moulds Culture-based method
- Part 18: Detection and enumeration of moulds Sampling by impaction
- Part 19: Sampling strategy for moulds
- Part 23: Performance test for evaluating the reduction of formaldehyde concentrations by sorptive building materials
- Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials
- Part 25: Determination of the emission of semi-volatile organic compounds by building products Microchamber method
- Part 26: Sampling strategy for carbon dioxide (CO<sub>2</sub>)
- Part 28: Determination of odour emissions from building products using test chambers

The following parts are under preparation:

- Part 21: Detection and enumeration of moulds Sampling from materials
- Part 27: Determination of settled fibrous dust on surfaces by SEM (scanning electron microscopy) (direct method)
- Part 29: Test methods for VOC detectors
- Part 30: Sensory testing of indoor air
- Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds Phosphoric acid ester
- Part 32: Investigation of constructions on pollutants and other injurious factors Inspections

#### Introduction

Formaldehyde has a high potential toxicity, and therefore, its determination is of interest as an indoor air pollutant. Formaldehyde is an irritant that may cause allergic reactions and other health issues.

Formaldehyde is the simplest carbonyl compound, with one carbon, one oxygen and two hydrogen atoms. In its monomolecular state, it is a colourless, pungent, reactive gas. It has been used in the production of ureaformaldehyde resins, adhesives and insulating foams. Emissions from particle (chip) board and wall insulation are the major sources of formaldehyde in indoor air.

This part of ISO 16000 is intended to be used for characterizing indoor air following the sampling strategy for formaldehyde specified in ISO 16000-2. ISO 16000-1 gives general requirements for 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.

An active formaldehyde measurement procedure (reference method) is specified in ISO 16000-3.

Aspects of the determination (sampling and analysis) and the sampling strategy of specific pollutants or groups of pollutants are specified in the other parts of ISO 16000.

ISO 16017<sup>[6][7]</sup> and ISO 12219<sup>[1]–[5]</sup> also focus on volatile organic compound (VOC) measurements.

## Indoor air —

## Part 4:

## Determination of formaldehyde — Diffusive sampling method

WARNING — Persons using this part of ISO 16000 should be familiar with normal laboratory practice. This part of ISO 16000 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

#### 1 Scope

This part of ISO 16000 specifies a determination of formaldehyde in indoor air using a diffusive sampler with solvent desorption and high performance liquid chromatography (HPLC).

The test method is applicable to the measurement of formaldehyde <sup>1)</sup> in indoor air over the range from 0,001 mg/m<sup>3</sup> to 1,0 mg/m<sup>3</sup> for a sampling period of between 24 h and 72 h. For sampling periods of 24 h, the applicable concentration range is 0,003 mg/m<sup>3</sup> to 1 mg/m<sup>3</sup>, and for 72 h it is 0,001 mg/m<sup>3</sup> to 0,33 mg/m<sup>3</sup>.

NOTE Confirmed samplers with respect to measurement sensitivity and precision can apply to short-term sampling (less than 24 h).

The method is suitable for measurements in atmospheres with conventional indoor air relative humidity and for monitoring at air velocities as low as 0,02 m/s. The chromatographic step in the method is designed to eliminate potential interferences, including those due to the presence of other carbonyl compounds. The sampling method gives a time-weighted average result.

NOTE There are several diffusive samplers with various designs and shapes commercially available. They are all based on the reaction of 2,4-dinitrophenylhydrazine with formaldehyde. They are not specifically described in this part of ISO 16000 to avoid any impression that ISO favours one model over another.

#### 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 16000-2, Indoor air — Part 2: Sampling strategy for formaldehyde

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

ISO/IEC 17025. General requirements for the competence of testing and calibration laboratories

EN 13528-2, 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

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<sup>1)</sup> Instead of systematic IUPAC nomenclature, traditional names are used in this part of ISO 16000, e.g. "formaldehyde" is used instead of "methanal".

#### 3 **Principle**

The diffusive sampler is exposed to air for a defined time period. The rate of sampling depends on the diffusion coefficient of formaldehyde, and is proportional to the cross-sectional area of the opening of the monitor and inversely proportional to the length of the diffusion zone of the monitor. This rate is known as the diffusive uptake rate of the sampler, and has been determined by prior calibration in a standard atmosphere. The formaldehyde vapour migrates into the sampler by diffusion and is collected on a strip of cellulose paper loaded with silica gel, which has been coated with 2,4-dinitrophenylhydrazine (DNPH) and phosphoric acid. A stable hydrazone is formed which can be desorbed by acetonitrile and the solution analysed by means of a high performance liquid chromatograph (HPLC) equipped with an ultraviolet (UV) detector. The diffusive sampler may also be used for personal sampling, for which purpose it is attached to the clothing of the person being monitored.

#### 4 Reagents

During the analysis use only chemicals of recognized grade.

- 4.1 **2,4-Dinitrophenylhydrazine**, recrystallized at least twice with UV-grade acetonitrile (4.2) before use.
- Acetonitrile, HPLC UV grade (each batch of solvent should be tested before use). 4.2
- 4.3 **Concentrated hydrochloric acid**, 36,5 % to 38 % (mass fraction),  $\rho$  = 1,19 kg/l.
- 4.4 Nitrogen, high-purity grade (best source).
- 4.5 Water, HPLC grade.
- 4.6 Formaldehyde, 37 % solution (mass fraction).
- 4.7 Ethanol, 95 % (mass fraction).
- 4.8 Methanol, HPLC grade.
- 4.9 Phosphoric acid, 85 % (mass fraction).
- **4.10** Glycerin, 20 % (volume fraction).
- **4.11 Formaldehyde dinitrophenylhydrazone** (DNPH-formaldehyde).

WARNING — 2,4-Dinitrophenylhydrazine is explosive in the dry state. Handle with extreme care. It is also toxic (in the rat,  $LD_{50} = 654$  mg/kg), has been shown to be mutagenic in some tests, and is irritating to the eyes and skin.

Weigh 2 g of 2,4-dinitrophenylhydrazine (4.1) and place in a 500 ml flask. Add 10 ml of concentrated hydrochloric acid (HCI) (4.3) with stirring. Dissolve the yellow precipitate formed in 200 ml of ethanol (4.7). Filter the solution to remove undissolved hydrazine hydrochloride (DNPH HCI). To the filtered solution, add 0,8 ml of formaldehyde. Collect by filtration the yellowish DNPH-formaldehyde precipitate formed, and wash with 5 ml cold ethanol. Recrystallize the precipitate twice from hot ethanol and allow to dry in air. Check the purity of the DNPH-formaldehyde derivative by melting-point determination (166 °C) or HPLC analysis (purity >99 % mass fraction). If the impurity level is not acceptable, recrystallize the derivative in ethanol. Repeat the purity check and recrystallization, as necessary, until an acceptable level of purity (e.g. 99 % mass fraction) is achieved. The DNPH-formaldehyde derivative should be stored under refrigeration (4 °C) and protected from light. It should be stable for at least 6 months.

Storage under nitrogen or argon further prolongs the lifetime of the derivative. DNPH derivatives of NOTE formaldehyde for use as standards are commercially available both in the form of pure crystals and as individual or mixed stock solutions in acetonitrile.

**4.12 DNPH-formaldehyde calibration standards**. Accurately weigh about 10 mg of DNPH-formaldehyde derivative (4.11), place in a 100 ml one-mark volumetric flask, and make up to the mark with acetonitrile (4.2). Shake the flask until the crystals are dissolved. Prepare a minimum of five calibration standards in the range 1  $\mu$ g/ml to 140  $\mu$ g/ml (equivalent to 0,14  $\mu$ g/ml to 20  $\mu$ g/ml formaldehyde) by dilution of aliquots of the DNPH-formaldehyde solution with acetonitrile.

Store all standard solutions in tightly capped containers in a refrigerator and protected from light. Allow them to equilibrate to room temperature before use. They should be replaced after 4 weeks.

- **4.13 DNPH coating solution**. Weigh 900 mg of DNPH · HCl that has been recrystallized twice from HCl (c = 4 mol/l) into a flask. Add 1,7 ml of concentrated phosphoric acid (4.9), glycerin in ethanol (4.10), and 180 ml of acetonitrile (4.2).
- **4.14 Coated silica gel filter**. The uncoated filter, a cellulose paper loaded with silica gel, is commercially available in rolls from which required sizes can be cut. The size of the coated silica gel filter depends on the construction of the diffusive sampler. For example, for a sampler requiring a 20 mm × 45 mm filter, add 0,5 ml of DNPH coating solution (4.13) dropwise to the filter using a pipette. Dry the coated filters on a glass surface in an oven at 85 °C for 15 min. For other filter sizes, adjust the volume of coating solution accordingly. Place the coated filter in the diffusive sampler (5.1). An additional coated filter is enclosed in a metal gauze and this is used to absorb any formaldehyde in the air in the container used for storage of the diffusive sampler (see Clause 6). Coated silica gel filters have been found to be stable for at least 6 months when stored at 4 °C in the absence of light.

Blank coated filters prepared and stored according to this part of ISO 16000 should contain less than 0,7  $\mu$ g of DNPH-formaldehyde per filter of dimension 20 mm  $\times$  20 mm (equivalent to 0,1  $\mu$ g of formaldehyde).

NOTE This part of ISO 16000 instructs the user on how to prepare a coated filter for use in a sampler, but commercially available samplers that contain a prepared filter can have the advantage of more uniform manufacture and potentially lower blank levels. For the design of commercial samplers, see Annex A and the Bibliography.

#### 5 Apparatus

Ordinary laboratory apparatus and the following.

#### 5.1 Diffusive sampler.

Commercially available or laboratory constructed devices may be used provided they meet the performance requirements (for the design of the sampler see Figure A.1). The diffusive sampling rate shall be independent of air concentrations between 0,001 mg/m³ and 0,33 mg/m³ for 72 h and 0,003 mg/m³ and 1,0 mg/m³ for 24 h. Humidity shall have no effect on the sampling rate up to 80 % relative humidity (RH) and the sampling rate shall not be significantly influenced by ambient air velocities as low as 0,02 m/s. The sampler may require a stand or clip for positioning during sampling. When not in use, keep the sampler in a protective airtight container with a protective coated filter (encased in metal gauze). Keep the container in a refrigerator (see 7.5).

For this sampler, the coated filter is placed beneath a plastic screen within which are a number of holes. The sampler is opened by sliding a cover to expose the holes to the air and is closed by replacing the cover.

A commercially available or laboratory-constructed device may be used for periods of less than 24 h provided it meets performance requirements.

Alternative designs of samplers are presented in Figures A.2 and A.3. The diffusive sampler shall have the opening of the monitor and an appropriate length of the diffusion zone of the monitor.

NOTE Guidance on the design and performance test procedures for a diffusive sampler is given in EN 13528-2. This requires the generation of standard atmospheres in order to determine the diffusive uptake rate of the sampler under different conditions of temperature, humidity, air velocity, exposure time, and gas concentration appropriate to the intended use of the sampler. Examples of studies of the performance of a diffusive sampler for the measurement of formaldehyde in air are given in the Bibliography.

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**5.2 High performance liquid chromatograph (HPLC)**, consisting of one or two solvent pumps, solvent reservoir(s), a solvent mixer, a reverse-phase column, a column oven, a pump controller and data-handling system.

The column shall be linked to a UV detector able to monitor at a wavelength of 360 nm.

The apparatus and procedure should be capable of detecting a DNPH-formaldehyde concentration of  $0.21 \, \mu g/ml$  (equivalent to  $0.03 \, \mu g/ml$  of formaldehyde).

#### 6 Sampling

Immediately before sampling, the diffusive sampler (5.1) is removed from its protective bag or other suitable airtight container. For area monitoring, the sampler shall be located in accordance with the instructions given in ISO 16000-2, i.e. at least 1 m from the walls of the room at a height of 1,5 m above the floor. Areas of the room strongly influenced by local ventilation, e.g. in the proximity of windows and doors, and areas near known sources of formaldehyde, such as uncoated particleboard, should be avoided unless these are of specific interest for the study. When used for personal sampling, the sampler should be mounted on the lapel or other unobstructed part of the subjects' clothing in the breathing zone. Once the sampler has been sited, begin the sampling period by opening the closure window of the sampler. Record the time and date of opening of the sampler.

At the end of the sampling time, close the sampler. When not in use, keep the sampler in a protective airtight container along with a coated filter (identical to the filter described in 4.14) that, for ease of handling, can be enclosed in a metal gauze. Record the time and date of closure of the sampler.

Prepare sample blanks by using samplers identical to those used for sampling, and subject them to the same handling procedure as the exposed samplers, except they shall not be exposed. Alternatively, the sampler design may incorporate an unexposed portion of the coated filter that can serve as the blank. Return blank samples and exposed samplers to the laboratory for analysis.

NOTE During active sampling of ambient air under particular atmospheric conditions, it has been reported that ozone can result in negative interference by reaction with DNPH and DNPH-formaldehyde. This interference has not been observed with the diffusive sampler. Further information about this possible interference is described in ISO 16000-3.

#### 7 Procedure

#### 7.1 Desorption and sample preparation

Carry out the desorption of the DNPH-formaldehyde from the coated filter in a clean atmosphere. Remove the coated filter from the sampler with forceps. If the sampler includes an integral blank coated filter, separate it from the exposed section of the filter by cutting. For example, for a filter measuring 20 mm  $\times$  45 mm, cut into sample and blank sections and place each filter section in a 4 ml glass vial. Desorption is achieved by pipetting 3 ml of acetonitrile into each vial followed by agitation for 1 min. Remove the coated filter from the vial, then seal the vial for analysis by HPLC. Filtration of the extract may be necessary before analysis. For filters of other dimensions, the desorption procedure can be varied but it shall be shown to be >95 % efficient for desorbing DNPH-formaldehyde. Store the DNPH-formaldehyde solution in a refrigerator (4 °C). It should be analysed within 3 days.

#### 7.2 Calibration

Analyse each calibration standard (4.12) by injecting a known volume of each solution (e.g.  $10 \,\mu$ l) into the chromatograph (5.2). To avoid carry-over effects, start with the lowest concentration. The injection technique used shall be such that reproducible HPLC peak heights or areas are obtained. Prepare a calibration graph by plotting peak response against analyte concentration, in micrograms per millilitre. The normal calibration frequency is 1 month. The validity of calibration can be verified between two calibrations by analysing check points.

#### 7.3 HPLC analysis

Assemble the HPLC system and calibrate as specified in 7.2. Typical operating parameters are given in the following.

An octadecylsilane column ( $100 \text{ mm} \times 5 \text{ mm}$  internal diameter,  $10 \text{ }\mu\text{m}$  particles) with a mobile phase of 70 % volume fraction methanol + 30 % volume fraction water mixture has been found to provide sufficient resolution of the DNPH-formaldehyde and possible interfering compounds. Either a gradient or an isocratic elution programme may be employed.

Alternatively, an isocratic mobile phase of 60 % volume fraction acetonitrile + 40 % volume fraction water, as described in ISO 16000-3 has been shown to be suitable. The parameters are the following.

- Column: C<sub>18</sub> reverse phase.
- Mobile phase: 60 % volume fraction acetonitrile + 40 % volume fraction water.
- Detector: ultraviolet, at 360 nm.
- Flow rate: 1 ml/min.
- Retention time: ~7 min for formaldehyde, 13 min for formaldehyde with two C<sub>18</sub> columns.
- Sample injection volume: 25 μl.

Before each analysis, check the detector baseline to ensure stable conditions.

Prepare the HPLC mobile phase by mixing 600 ml of acetonitrile and 400 ml of water, or set the parameters on the gradient elution HPLC appropriately. Filter this mixture through a 0,22 µm polyester membrane filter in an all-glass and polytetrafluoroethene (PTFE) suction filtration apparatus. Degas the filtered mobile phase by purging with helium for 10 min to 15 min (100 ml/min) or by heating to 60 °C for 5 min to 10 min in an Erlenmeyer flask covered with a watchglass or by the degassing of solvents with ultrasound. A constant backpressure restrictor (350 kPa) or a short length (15 cm to 30 cm) of 0,25 mm (inside diameter) PTFE tubing should be placed after the detector to eliminate further mobile-phase out-gassing.

Place the mobile phase in the HPLC solvent reservoir and set the pump at a flow rate of 1,0 ml/min. Allow it to pump for 20 min to 30 min before the first analysis. Switch on the detector at least 30 min before the first analysis. Display the detector output on a strip chart recorder or similar output device.

For manual injection systems, draw at least 100  $\mu$ l of the sample into a clean HPLC injection syringe. Fill the HPLC loop (load position of valve) by adding excess sample via the syringe. Turn the valve to the "inject" position to start the run. Activate the data system simultaneously with the injection and mark the point of injection on the strip chart recorder. After approximately 1 min, return the injection valve to the load position and rinse or flush the syringe and valve with the acetonitrile + water mixture in preparation for the next sample analysis.

Do not syringe solvent through the HPLC loop while the valve is in the inject position.

After elution of the DNPH-formaldehyde derivative, terminate data acquisition and calculate the component concentrations. Once a stable baseline is achieved, the system can be used for further sample analysis as specified in the preceding.

NOTE After several analyses, build-up on the column (indicated, for example, by increasing pressure from run to run at a given flow and solvent composition) can be removed by flushing with several column volumes of 100 % acetonitrile. The same protection can be achieved if precolumns are used.

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If the concentration of analyte exceeds the linear range of the instrument, the sample should be diluted with mobile phase or a smaller volume injected into the HPLC. If the retention time found in earlier runs is not duplicated (±10 %), the ratio of acetonitrile to water may be increased or decreased to obtain the correct elution time. If the elution time is too long, increase the ratio; if it is too short, decrease the ratio. If a solvent change is necessary, always recalibrate before running samples.

Analysts are advised to experiment with their HPLC system to optimize chromatographic conditions for their particular analytical needs. HPLC systems with automated injection and start of data acquisition may also be used.

Examine the chromatogram for evidence of ozone interference in accordance with ISO 16000-3:2011, 4.2.

#### 7.4 **Determination of sample concentration**

Analyse the solutions produced by desorption of the sample and blank coated filters (7.1) in the same way as the calibration standards (7.2). Determine the peak responses and read the concentrations of DNPHformaldehyde from the calibration graph.

An unexposed blank coated filter that has been taken to the sampling site for the duration of the sampling period is likely to have a higher DNPH-formaldehyde content than a freshly prepared coated filter.

#### 7.5 Storage

When sealed in a protective container and stored in a freezer (-18 °C) prior to use, the prepared filter has a shelf-life of at least 6 months. After exposure, the DNPH-formaldehyde analyte formed should be stable for at least 2 weeks if stored in a refrigerator prior to desorption and analysis.

#### **Determination of desorption efficiency** 7.6

For procedures that differ from those specified in 7.1, the desorption efficiency should be determined by spiking samplers with a known mass of DNPH-formaldehyde. This is achieved by exposing samplers to atmospheres having a constant and known concentration of formaldehyde. At least two tests should be undertaken to assess desorption efficiencies from samplers exposed at the lower and higher ends of the concentration range to be monitored. The mass of DNPH-formaldehyde on the coated filter is calculated on the bases of the exposure time anticipated and the uptake rate of the sampler, verified in accordance with EN 13528-2. The analysis of the samplers is undertaken and the recovery of DNPH-formaldehyde is determined. This should be at least 85 % efficient.

#### Calculation 8

#### Mass of DNPH-formaldehyde on the filter

Calculate the mass of DNPH-formaldehyde desorbed from the exposed sample or blank sample by multiplying the concentration in the solution as read from the calibration curve by the volume of solution used for sample desorption.

#### 8.2 Concentration of formaldehyde in air

Calculate the mass concentration,  $\rho$ , of formaldehyde in air, in micrograms per metre cubed, by Equation (1):

$$\rho = \frac{(m - m_{\text{blank}}) \times 0.143 \times 10^6}{q_V t} \frac{d}{100}$$
 (1)

where

*m* is the mass, in micrograms, of DNPH-formaldehyde in the exposed sampler;

 $m_{\rm blank}$  is the mass, in micrograms, of DNPH-formaldehyde in the blank sampler;

 $q_V$  is the uptake rate, in centimetres cubed per minute, of the sampler;

*t* is the exposure time, in minutes;

d is the desorption efficiency, as a percentage (see 7.6);

0,143 is the quotient of the ratio M (formaldehyde)/M (DNPH-formaldehyde), in which M is the molecular mass.

NOTE The use of parts per billion or parts per million is deprecated. However, for the convenience of certain users, the following equation is provided for calculating the concentration of formaldehyde,  $\rho'$ , in the sampled air in terms of parts per billion by volume (ppb) (volume fraction):

$$\rho' = \rho \left( \frac{22,4}{M} \frac{101,3}{p_{\text{amb}}} \frac{T}{273,2} \right) \frac{d}{100}$$
 (2)

where

 $p_{\text{amb}}$  is the average ambient pressure, in kilopascals, during the sampling period (or normal pressure, 101,3 kPa, if desired);

T is the average temperature, in kelvins, during the sampling period (or normal temperature, 298 K, if desired);

M is the molecular mass (30,03 g/mol) of formaldehyde;

d is the desorption efficiency, as a percentage (see 7.6).

#### 9 Precision and uncertainty of the method

The intralaboratory precision of replicate measurements should be less than 10 %, expressed as the coefficient of variation.

NOTE From field experiments in which six samplers at a time were exposed in three indoor environments to concentrations of formaldehyde of 0,027 mg/m³ to 1,1 mg/m³, the precision of replicate measurements was determined to be 1 % to 9 %, expressed as a coefficient of variation [9]. Replicate measurements in bedrooms of three homes having air concentrations of 0,007 mg/m³ to 0,01 mg/m³ yielded coefficients of variation of 6,2 % to 8,7 %[10][11]. When four samplers were placed in two locations in each of five rooms with measured formaldehyde concentrations of 0,007 mg/m³ to 0,048 mg/m³, the coefficient of variations ranged from 0,8 % to 8,3 %, except at one location where it was 10,9 %[12]. Comparisons of the results of diffusive sampling using simultaneous sampling with an independent active sampling method have been reported to show good agreement[8]–[12].

### 10 Quality assurance/quality control

The user shall adhere to the requirements of ISO/IEC 17025.

Field blank and replicate samples shall be taken as part of each investigation or batch of samplers. These should equal at least 10 % of the total number of samplers used and be a minimum of one for small studies.

It is also recommended that:

- for internal quality control, diffusive sampling rates should be checked routinely, at least once during large surveys — this should be done by exposing samplers in laboratory standard atmospheres or by laboratory or field comparisons with an independent (e.g. pumped) method;
- for external quality assurance, diffusive sampling rates should be checked routinely, at least once during large surveys — this should done by laboratory or field intercomparisons (one sampler, various laboratories), which should be conducted by experienced institutes.

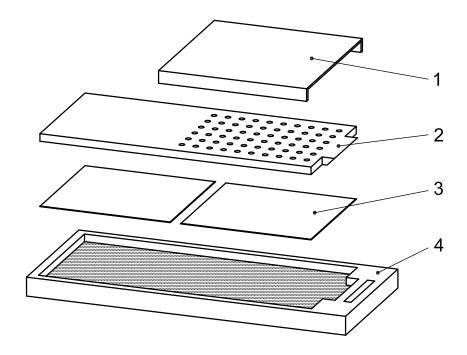
### 11 Test report

The test report shall contain at least the following information:

- complete identification of the sample;
- reference to this part of ISO 16000 (ISO 16000-4);
- the place and period of sampling; C)
- environmental conditions and occupant activity during sampling;
- the test result, in micrograms per metre cubed; e)
- any unusual features noted during the determination; f)
- any operation not included in this part of ISO 16000 or to which reference is made or which is regarded g) as optional.

# **Annex A** (informative)

# Typical designs of diffusive samplers

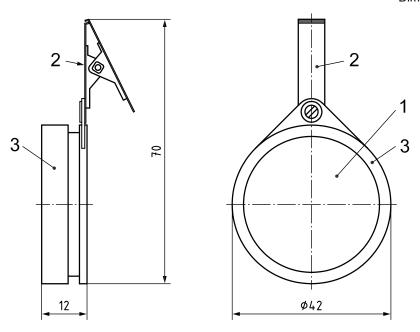


#### Key

- 1 sliding cover
- 2 diffusion screen
- 3 DNPH-coated filters
- 4 sampler housing

Figure A.1 — Typical design of the diffusive sampler for formaldehyde<sup>[9]</sup>

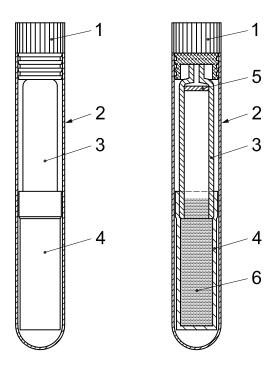
Dimensions in millimetres



#### Key

- filter
- badge clip
- 3 frame (cap)

Figure A.2 — Example of a badge-type sampler for formaldehyde



#### Key

- diffusive filter сар
- 5 2 storage tube frit
  - elution tube DNPH-coated silica gel

Figure A.3 — Example of a tube-type sampler for formaldehyde

## **Bibliography**

- [1] ISO 12219-1, Indoor air of road vehicles Part 1: Whole vehicle test chamber Specification and method for the determination of volatile organic compounds in cabin interiors
- [2] ISO 12219-2, Indoor air of road vehicles Part 2: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials Bag method
- [3] ISO 12219-3, Indoor air of road vehicles Part 3: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials Micro-chamber method
- [4] ISO 12219-4<sup>2)</sup>, Indoor air of road vehicles Part 4: Determination of the emissions of volatile organic compounds from car trim components Small chamber method
- [5] ISO 12219-5<sup>2)</sup>, Indoor air of road vehicles Part 5: Screening method for the determination of emissions of volatile organic compounds (VOC) from car trim components Static chamber method
- [6] ISO 16017-1, Indoor, ambient and workplace air Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography Part 1: Pumped sampling
- [7] ISO 16017-2, Indoor, ambient and workplace air Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography Part 2: Diffusive sampling
- [8] MDHS 78:1994, Formaldehyde in air Laboratory method using a diffusive sampler, solvent desorption and high performance liquid chromatography. Available (viewed 2011-11-18) at: <a href="http://www.hse.gov.uk/pubns/mdhs/pdfs/mdhs78.pdf">http://www.hse.gov.uk/pubns/mdhs/pdfs/mdhs78.pdf</a>
- [9] LEVIN J.-O., LINDAHL R., ANDERSSON K. High-performance liquid-chromatographic determination of formaldehyde in air in the ppb to ppm range using diffusive sampling and hydrazone formation. *Environ. Technol. Lett.* 1988, **9**, pp. 1423-1430
- [10] BERRY, R.W., BROWN, V.M., COWARD, S.K.D. *Indoor air quality in homes; the Building Research Establishment Indoor Environment Study*, Parts 1 and 2. Watford: Building Research Establishment, 1996. (Reports BR 299 and BR 300)
- [11] BROWN V.M., CRUMP D.R., GAVIN M.A., GARDINER D. Aldehydes in the non-industrial indoor environment. In: BROWN, R.H., editor. *Clean air at work: New trends in assessment and measurement for the 1990s*, Proceedings of an International Symposium, Luxembourg, 1991-09-09/13, pp. 365-367. Cambridge, UK: Royal Society of Chemistry, 1992. (Special Publication No. 108.)
- [12] GAVIN M., CRUMP D.R., BROWN V.M. Appropriate sampling strategies for the measurement of formaldehyde in indoor air. *Environ. Technol.* 1995, **16**, pp. 579-586

<sup>2)</sup> Under preparation.



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