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**Indoor air —**

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

*Air intérieur —*

*Partie 4: Dosage du formaldéhyde — Méthode par échantillonnage  
diffusif*  
**(standards.iteh.ai)**

ISO 16000-4:2004

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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*.

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

- Part 2: *Sampling strategy for formaldehyde*
- Part 3: *Determination of formaldehyde and other carbonyl compounds — Active sampling method*
- Part 4: *Determination of formaldehyde — Diffusive sampling method*
- Part 6: *Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS/FID*

The following parts are in preparation

- Part 1: *General aspects of sampling strategy*
- Part 5: *Measurement strategy for volatile organic compounds (VOCs) (in preparation)*
- Part 7: *Sampling strategy for determination of airborne asbestos fibre concentrations (in preparation)*
- Part 8: *Ventilation rate measurement*
- Part 9: *Determination of the emission of volatile organic compounds — Emission test chamber method*
- Part 10: *Determination of the emission of volatile organic compounds — Emission test cell method*
- Part 11: *Determination of the emission of volatile organic compounds — Sampling, storage of samples and preparation of test specimens*

## Introduction

Formaldehyde has a high potential toxicity, and therefore its determination is of interest as an indoor air pollutant.

ISO 16000-1 gives general requirements relating to the measurement of indoor air pollutants and the important conditions to be observed before or during the sampling of individual pollutants or groups of pollutants. Aspects of the determination (sampling and analysis) and the sampling strategy of specific pollutants or groups of pollutants are described in the other parts of ISO 16000.

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## Indoor air —

### Part 4:

## Determination of formaldehyde — Diffusive sampling method

### 1 Scope

This part of ISO 16000 specifies a diffusive sampler/solvent desorption/high performance liquid chromatography (HPLC) method for the determination of formaldehyde in indoor air.

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>.

The method is suitable for measurements in atmospheres of up to 80 % relative humidity and for monitoring at air velocities as low as 0,02 m/s. Potential interferences, including those due to the presence of other carbonyl compounds, should be eliminated by the chromatographic step in the method. The sampling method gives a time-weighted average result.

### 2 Normative references

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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 16000-3:2001, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds — 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*

### 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.

<sup>1)</sup> Instead of the nomenclature according to IUPAC regulations (e.g. methanal), the more common names (e.g. formaldehyde) are used in this part of ISO 16000.

## 4 Reagents

**WARNING** — This part of ISO 16000 does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this part of ISO 16000 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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.

**4.2 Acetonitrile**, HPLC UV-grade (each batch of solvent should be tested before use).

**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 %.

**4.11 Formaldehyde dinitrophenyl hydrazone (DNPH-formaldehyde)**.

**WARNING** — 2,4-dinitrophenylhydrazine is explosive in the dry state. Handle with extreme care. It is also toxic ( $LD_{50}$  rat = 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 (HCl) (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 · HCl). To the filtered solution add 0,8 ml. 37 % 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 of > 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 (for example, 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 six months.

**NOTE** Storage under nitrogen or argon further prolongs the lifetime of the derivative. DNPH derivatives of 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 volumetric flask, and fill 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 µg/ml to 140 µg/ml (equivalent to 0,14 µg/ml to 20 µg/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 four weeks.



#### 4.13 DNPH coating solution.

Weigh 900 mg of DNPH · HCl that has been recrystallized twice from HCl ( $c = 4 \text{ mol/l}$ ) into a flask. Add 1,7 ml of concentrated phosphoric acid (4.9), 5 ml 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  $20 \text{ mm} \times 45 \text{ mm}$  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  $85 \text{ }^\circ\text{C}$  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 six months when stored at  $4 \text{ }^\circ\text{C}$  in the absence of light.

Blank coated filters prepared and stored according to this part of ISO 16000 should contain less than  $0,7 \text{ } \mu\text{g}$  of DNPH-formaldehyde per filter of dimension  $20 \text{ mm} \times 20 \text{ mm}$  (equivalent to  $0,1 \text{ } \mu\text{g}$  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 Annex A). The diffusive sampling rate shall be independent of air concentration between  $0,001 \text{ mg/m}^3$  and  $0,33 \text{ mg/m}^3$  for 72 h and  $0,003 \text{ mg/m}^3$  and  $1,0 \text{ mg/m}^3$  for 24 h. Humidity shall have no effect on sampling rate up to 80 % relative humidity (RH) and the sampling rate shall not be significantly influenced by ambient air velocities as low as  $0,02 \text{ m/s}$ . 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.

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.

**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  $360 \text{ nm}$ .

The apparatus and procedure should be capable of detecting a DNPH-formaldehyde concentration of  $0,21 \text{ } \mu\text{g/ml}$  (equivalent to  $0,03 \text{ } \mu\text{g/ml}$  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 guidance given in