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ISO 16000-3

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# Indoor air —

## Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method

*Air intérieur —*

*Partie 3: Dosage du formaldéhyde et d'autres composés carbonylés dans l'air intérieur et dans l'air des chambres d'essai — Méthode par échantillonnage actif*

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# Foreword

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This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

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This fourth edition cancels and replaces the third edition (ISO 16000-3:2022), which has been technically revised.

The main change is as follows: Annex A has been added which provides a suitable method for the determination of acrolein.

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A list of all parts in the ISO 16000 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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# Introduction

This document is intended to be used for characterizing indoor air following the sampling strategy specified in ISO 16000-2 [1]. This document applies to 14 aldehydes and ketones. Formaldehyde is the simplest carbonyl compound, with one carbon, one oxygen and two hydrogen atoms. In its monomolecular state, formaldehyde is a colourless, pungent and reactive gas. Formaldehyde has been used in the production of urea-formaldehyde resins, adhesives and insulating foams. Emissions from particle (chip) board and wall insulation are the major sources of formaldehyde in indoor air. As formaldehyde has a high toxic potential, [2] determining its level accurately is key for air quality.

When formaldehyde is collected by passing air through a reactive medium that converts the compound to a derivative of lower vapour pressure is more efficiently retained by the sampler and its level can be easily analysed.

ISO 16017-1 [3], ISO 16017-2 [4], ISO 12219-1 [5] and ISO 12219-5 [6] describe analytical methods for a broader spectrum of volatile organic compounds (VOCs) and can be used if other groups besides carbonyl compounds are to be analyzed.

Instead of systematic International Union of Pure and Applied Chemistry (IUPAC) nomenclature, traditional names are used in this document. Some equivalent names are:

— acetaldehyde for ethanal;

— acetone for 2-propanone;

— butyraldehyde for butanal;

— capronaldehyde for hexanal;

— formaldehyde for methanal;

— isovaleraldehyde for 3-methylbutanal;

— propionaldehyde for propanal;

— m-tolualdehyde for 3-methylbenzaldehyde;

— o-tolualdehyde for 2-methylbenzaldehyde;

— p-tolualdehyde for 4-methylbenzaldehyde;

— valeraldehyde for pentanal.

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# Indoor air —

# Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method

## 1 Scope

**WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.**

This document specifies a sampling method to determine the quantity of formaldehyde (HCHO) in the air with the range of approximately 1 µg/m$^3$ to 1 mg/m$^3$ in a time-weighted average (TWA) sample, for long-term (1 h to 24 h) and short-term (5 min to 60 min) sampling. This method involves the collection of compounds from air on to adsorbent cartridges coated with 2,4-dinitrophenylhydrazine (DNPH) and subsequent analysis of the hydrazones formed by high performance liquid chromatography (HPLC) with detection by ultraviolet absorption [7][8].

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This document can also be applied for the determination of at least 12 other aromatic as well as saturated and unsaturated aliphatic carbonyl compounds (aldehydes and ketones), with modification, such as:

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— acetaldehyde;

— acetone;

— benzaldehyde;

— butyraldehyde;

— capronaldehyde;

— 2,5-dimethylbenzaldehyde;

— formaldehyde;

— isovaleraldehyde;

— propionaldehyde;

— m-tolualdehyde;

— *o*-tolualdehyde;

— *p*-tolualdehyde;

— valeraldehyde.

This document does not apply to longer chained or unsaturated carbonyl compounds such as acrolein. An alternative sampling method using sorbent tubes and analysis by thermal desorption using gas chromatography with mass spectrometry (GC-MS) is given in Annex A.

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## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

## 4 Principle

The method specified in this document involves the collection of compounds from air on to adsorbent cartridges containing silica gel coated with 2,4-DNPH. The principle of the method is based on the specific reaction of a carbonyl group with DNPH in the presence of an acid to form stable derivatives according to the reaction shown in Figure 1. The DNPH derivatives are analysed for the parent aldehydes and ketones utilizing HPLC with UV detection or diode array detection [7][8]. The detection has been extended to other carbonyl compounds that can be determined as outlined in 10.3.5.

The preparation of sampling cartridges from commercially available chromatographic grade silica gel cartridges by the application of acidified DNPH to each cartridge is covered in this method. If pre-coated DNPH silica gel cartridges are available, they should be used since they are generally more uniform in manufacture and possess lower blank levels. However, if commercial cartridges are used, demonstrate that they meet the performance criteria of this document. An advantage of commercial cartridges is that they are available with larger particle size silica gel – this results in a lower pressure drop across the cartridge. These low pressure drop cartridges can be more suitable for sampling air using battery-powered personal sampling pumps.

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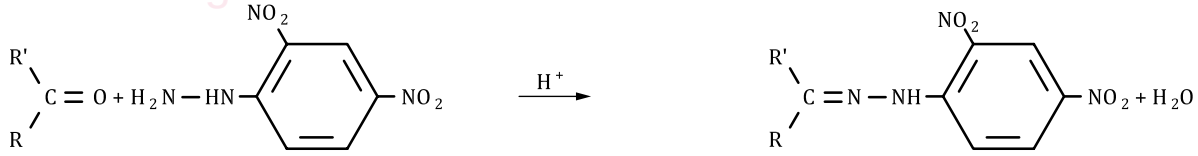


Carbonyl compound (aldehyde or ketone) | 2,4-DNPH | DNPH derivative

**Key**

| | |
|---|---|
| R | alkyl group |
| R' | aldehyde or ketone (alkyl group) |

**Figure 1 — Reaction of carbonyl compounds to form 2,4-dinitrophenylhydrazones**

## 5 Limitations and interferences

### 5.1 General

The sampling flow rate specified in this document has been validated for sampling rates up to 1,5 l/min. This flow rate limitation is set principally due to the high pressure drop (>8 kPa at 1,0 l/min) across the user-prepared silica gel cartridges, which have particle sizes of 55 µm to 105 µm. These cartridges are not generally compatible with battery-powered pumps used in personal sampling equipment (e.g. those used by industrial hygienists).

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The solid-sorbent sampling procedure is specific for sampling and analysis of formaldehyde. Interferences in this method are caused by certain isomeric aldehydes or ketones that can be unresolved by the HPLC system when analysing for other aldehydes and ketones. Any organic compounds that have the same retention times and significant absorbance at 360 nm as the DNPH derivative of formaldehyde interfere. Such interferences can often be overcome by altering the separation conditions (e.g. using alternative HPLC columns or mobile phase compositions).

Formaldehyde contamination of the DNPH reagent is a frequently encountered problem. The DNPH shall be purified by multiple recrystallizations in UV-grade acetonitrile (ACN). Recrystallization is accomplished, at 40 °C to 60 °C, by slow evaporation of the solvent to maximize crystal size. Impurity levels of carbonyl compounds in the DNPH are determined prior to use by HPLC and should be less than 0,15 µg per cartridge.

Exposure of the DNPH coated sampling cartridges to direct sunlight can produce artefacts and should be avoided [9].

Acrolein and crotonaldehyde cannot be accurately quantified by the method. Inaccurate results for these compounds can result from the formation of multiple derivative peaks and the instability of the peak ratios[10].

Nitrogen dioxide reacts with DNPH. High concentrations of $NO_2$ (e.g. for gas cooking stoves) can cause problems as the retention time of the DNPH derivative can be similar to that of the DNPH formaldehyde derivative, depending on the HPLC column and the parameters (see VDI 3862 [11], VDI 3862 [12] and Reference [13]).

## 5.2 Ozone interference

If there is suspicion that abnormally high levels of ozone are present in the area being sampled (e.g. from office copiers), special care should be taken. Ozone has been shown to interfere negatively by reacting with both DNPH and its derivatives (hydrazones) in the cartridge [14]. The extent of interference depends on the temporal variations of both the ozone and the carbonyl compounds and the duration of sampling. Significant negative interference from ozone has been observed even at concentrations of formaldehyde and ozone typical of clean ambient air (2 µg/m$^3$ and 80 µg/m$^3$, respectively) [13]. The presence of ozone in the sample is readily inferred upon analysis by the appearance of new compounds with retention times shorter than that of the hydrazone of formaldehyde. Figure 2 shows chromatograms of samples of a formaldehyde-spiked air stream with and without ozone.

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