INTERNATIONAL STANDARD

ISO 16000-3

Second edition 2011-10-15

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



Reference number ISO 16000-3:2011(E)



© ISO 2011

<text> All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

Case postale 56 • CH-1211 Geneva 20 Tel. + 41 22 749 01 11 Fax + 41 22 749 09 47 E-mail copyright@iso.org Web www.iso.org

Published in Switzerland

Contents

Page

Fore	eword	iv
Introduction		vi
1	Scope	1
2	Normative references	2
3	Principle	2
4	Limitations and interferences	2
5	Safety measures	3
6	Apparatus	
7	Reagents and materials	7
8	Preparation of reagents and cartridges	7
9	Procedure	10
10	Calculations	19
11	Performance criteria and quality assurance	20
12	Precision and uncertainty	22
13	Test report	22
Anne	ex A (informative) Precision and uncertainty	23
Annex B (informative) Melting points of DNPH-carbonyl derivatives		25
Bibliography		

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 16000-3 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 16000-3:2001), which has been technically revised.

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[®] 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 (NO₂)
- 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_2)
- 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

12

Introduction

This part of ISO 16000 is intended to be used for characterizing indoor air following the sampling strategy specified in ISO 16000-2. It is applicable to formaldehyde and other carbonyl compounds. It has been tested for 14 aldehydes and ketones. 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 urea-formaldehyde resins, adhesives, and insulating foams. Emissions from particle (chip) board and wall insulation are the major sources of formaldehyde in indoor air.

Formaldehyde is collected by passing air through a reactive medium that converts the compound to a derivative of lower vapour pressure that is more efficiently retained by the sampler and can be easily analysed. This part of ISO 16000 determines formaldehyde and other carbonyl compounds by reaction with 2,4-dinitrophenylhydrazine coated on to a sorbent to convert them to their corresponding hydrazones, which can be recovered and measured with high sensitivity, precision, and accuracy. Other carbonyl compounds that may be emitted into air from solvents, adhesives, cosmetics, and other sources can also be determined using this part of ISO 16000.

The sampling procedure is based on US EPA method TO-11A^[12].

Formaldehyde and certain other carbonyl compounds have a high toxic potential^[15].

ISO 16017^{[7][8]} and ISO 12219^{[2]–[6]} also focus on volatile organic compound (VOC) measurements.

<text>

Indoor air —

Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active 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 (HCHO) and other carbonyl compounds (aldehydes and ketones)¹⁾ in air. The method is specific to formaldehyde but, with modification, at least 12 other aromatic as well as saturated and unsaturated aliphatic carbonyl compounds can be detected and quantified. It is suitable for determination of formaldehyde and other carbonyl compounds in the approximate concentration range 1 μ g/m³ to 1 mg/m³. The sampling method gives a time-weighted average (TWA) sample. It can be used for long-term (1 h to 24 h) or short-term (5 min to 60 min) sampling of air for formaldehyde.

This part of ISO 16000 specifies a sampling and analysis procedure for formaldehyde and other carbonyl compounds that involves collection 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^{[12],[16]}. The method is not suitable for longer chained or unsaturated carbonyl compounds.

This part of ISO 16000 applies to the determination of:

acetaldehyde acetone benzaldehyde butyraldehyde capronaldehyde 2,5-dimethylbenzaldehyde formaldehyde isovaleraldehyde propionaldehyde *m*-tolualdehyde *o*-tolualdehyde *p*-tolualdehyde valeraldehyde

acetaldehyde: ethanal acetone: 2-propanone butyraldehyde: butanal capronaldehyde: hexanal formaldehyde: methanal isovaleraldehyde: 3-methylbutanal propionaldehyde: propanal *m*-tolualdehyde: 3-methylbenzaldehyde

o-tolualdehyde: 2-methylbenzaldehyde *p*-tolualdehyde: 4-methylbenzaldehyde valeraldehyde: pentanal

¹⁾ Instead of systematic IUPAC nomenclature, traditional names are used in this part of ISO 16000. Some equivalent names are:

2 Normative references

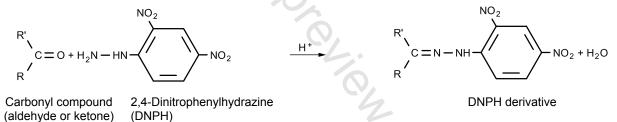
The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Principle

The method specified in this part of ISO 16000 involves drawing air through a cartridge containing silica gel coated with 2,4-dinitrophenylhydrazine (DNPH) reagent. 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 high performance liquid chromatography (HPLC) with UV detection or diode array detection. The detection has been extended to other carbonyl compounds that can be determined as outlined in 9.3.5.

This part of ISO 16000 instructs the user on how to prepare sampling cartridges from commercially available chromatographic grade silica gel cartridges by the application of acidified DNPH to each cartridge. Alternatively, pre-coated DNPH silica gel cartridges are available and are recommended since they are generally more uniform in manufacture and possess lower blank levels. However, if commercial cartridges are used, they shall be demonstrated to meet the performance criteria of this part of ISO 16000. Another advantage of commercial cartridges is that they are available with larger particle size silica gel that results in a lower pressure drop across the cartridge. These low pressure drop cartridges may be more suitable for sampling air using battery-powered personal sampling pumps.



Key

R, R' H, alkyl group, aromatic group

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

4 Limitations and interferences

4.1 General

The sampling flow rate specified in this part of ISO 16000 has been validated for sampling rates up to 1,5 l/min. This flow rate limitation is 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 μ m to 105 μ m. These cartridges are not generally compatible with battery-powered pumps used in personal sampling equipment (e.g. those used by industrial hygienists).

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 may 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).