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ISO 16000-3

Third edition
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Indoor air —

Part 3: Determination of formaldehyde and other carbonyl compounds in indoor 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

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 16000-3:2011), which has been technically revised.

The main changes are as follows:

— clarification of the suitability of the method for acrolein measurements.

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. 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 document 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 document.

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.

Instead of systematic IUPAC nomenclature, traditional names are used in this document. Some equivalent names are:

— 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.

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Indoor air —

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

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.

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1 Scope

This document specifies a determination of formaldehyde (HCHO) and other carbonyl compounds (aldehydes and ketones) 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 µg/m^3 to 1 mg/m^3. 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 document 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 document applies to the determination of:

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 Terms and definitions

No terms and definitions are listed in this document.

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

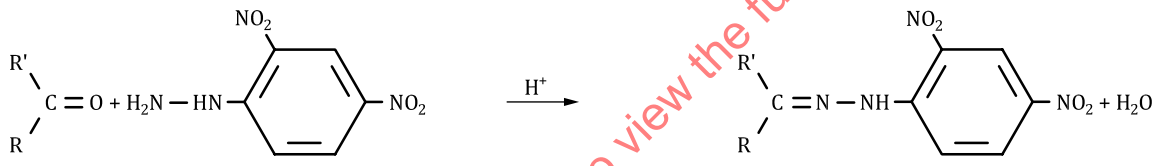
— ISO Online browsing platform: available at https://www.iso.org/obp

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

4 Principle

The method specified in this document involves 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 10.3.5.

This document 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 document. 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 can be more suitable for sampling air using battery-powered personal sampling pumps.



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

Key

R, R' H, alkyl group, aromatic 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 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).

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).

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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.

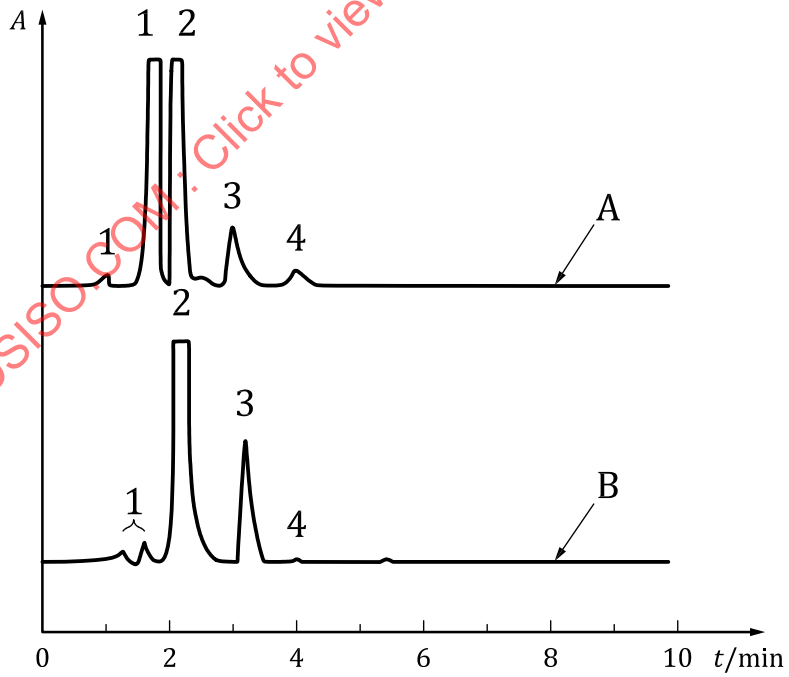
Exposure of the DNPH coated sampling cartridges to direct sunlight can produce artefacts and should be avoided[17].

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[18].

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[13][14][19].

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 exercised. Ozone has been shown to interfere negatively by reacting with both DNPH and its derivatives (hydrazones) in the cartridge[20]. 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)[19]. 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.



Key

A	relative absorbance	1	unknown
t	time	2	DNPH
A	with ozone	3	formaldehyde
B	without ozone	4	acetaldehyde

Figure 2 — Cartridge samples of formaldehyde in an air stream with and without ozone

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The most direct solution to ozone interference is to remove the ozone before the sampled air reaches the cartridge. This can be accomplished by the use of an ozone denuder or scrubber placed in front of the cartridge. Both ozone denuders and scrubber cartridges are commercially available. A denuder can be constructed of 1 m of copper tubing of outside diameter 0,64 cm and of inside diameter 0,46 cm, that is filled with a saturated solution of potassium iodide in water, allowed to stand for a few minutes (e.g. 5 min), drained and dried with a stream of clean air or nitrogen for about 1 h. The capacity of the ozone denuder as specified is about 200 µg/m^3 h. Test aldehydes (formaldehyde, acetaldehyde, propionaldehyde, benzaldehyde and *p*-tolualdehyde) that were dynamically spiked into an ambient sample air stream passed through the ozone denuder with practically no losses[21]. Commercial ozone scrubbers made from a cartridge filled with 300 mg to 500 mg of granular potassium iodide have also been found to be effective in removing ozone[22].

6 Safety measures

2,4-Dinitrophenylhydrazine is explosive in the dry state and shall be handled with extreme care. It is also toxic (in the rat, LD50 = 654 mg/kg), has been shown to be mutagenic in some tests, and is irritating to the eyes and skin.

Perchloric acid at concentrations less than 68 % mass fraction is stable and non-oxidizing at room temperature. However, it is readily dehydrated at temperatures above 160 °C and can cause explosions on contact with alcohols, wood, cellulose, and other oxidizable materials. It should be stored in a cool, dry place and used only in a chemical fume hood with caution.

7 Apparatus

Usual laboratory apparatus and in particular the following.

7.1 Sampling

7.1.1 Sampling cartridge packed with silica gel and coated with DNPH in accordance with Clause 9, or as available commercially.

The cartridge shall contain a minimum quantity of 350 mg of silica gel with a minimum DNPH loading of 0,29 % mass fraction. The ratio of the silica gel bed diameter to bed length shall not exceed 1:1. The capacity of the cartridge for formaldehyde shall be at least 75 µg and the collection efficiency at least 95 % at a sampling rate of 1,5 l/min. Sampling cartridges with very low blank levels and high performance are commercially available.

NOTE A pressure drop through the user-prepared sample cartridge of about 19 kPa at a sampling rate of 1,5 l/min has been observed. Some commercially available pre-coated cartridges exhibit lower pressure-drops, which permit the use of battery-operated personal sampling pumps.

7.1.2 Air sampling pump capable of accurately and precisely sampling at a flow rate of 0,1 l/min to 1,5 l/min.

7.1.3 Flow controller: mass flow meters, mass flow controllers, or other suitable device for metering and setting air flow rates of 0,50 l/min to 1,20 l/min through the sample cartridge (see 10.1 for further information regarding the flow controller requirements).

7.1.4 Flow calibrator: device to measure the flowrate through the cartridge, accurate to within 0,1 % of the flowrate used, such as an appropriate rotameter, soap-bubble meter or wet test meter.

7.2 Sample preparation

7.2.1 Cartridge containers e.g. borosilicate glass culture tubes (20 mm x 125 mm) with polypropylene screw caps, or other suitable containers, to transport coated cartridges.

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7.2.2 Gloves: non-absorbent gloves. Polythene gloves have been found suitable.

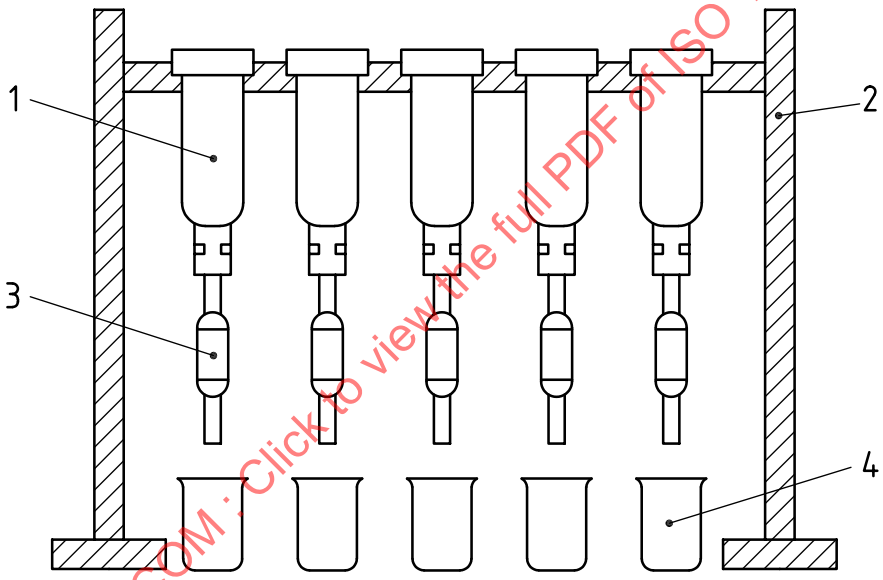
7.2.3 Transportation containers friction-top metal cans (e.g. of volume 4 l) or other suitable containers, with polyethylene air-bubble packing or other suitable padding, to hold and cushion the sealed cartridge containers.

NOTE A heat sealable foil-lined plastic pouch of the type included with some commercial pre-coated DNPH-cartridges can be used for storing a DNPH-coated cartridge after sampling, if appropriate.

7.2.4 Support for coating cartridges. A suitable inert support for holding drying cartridges shall be used, e.g. a syringe rack (see Figure 3).

7.2.5 Cartridge drying manifold such as a support with gas connectors and with multiple standard male syringe connectors (see Figure 3).

The apparatus specified in 7.2.4 and 7.2.5 is needed only if users choose to make their own DNPH-coated cartridges.



a) Rack for coating cartridges

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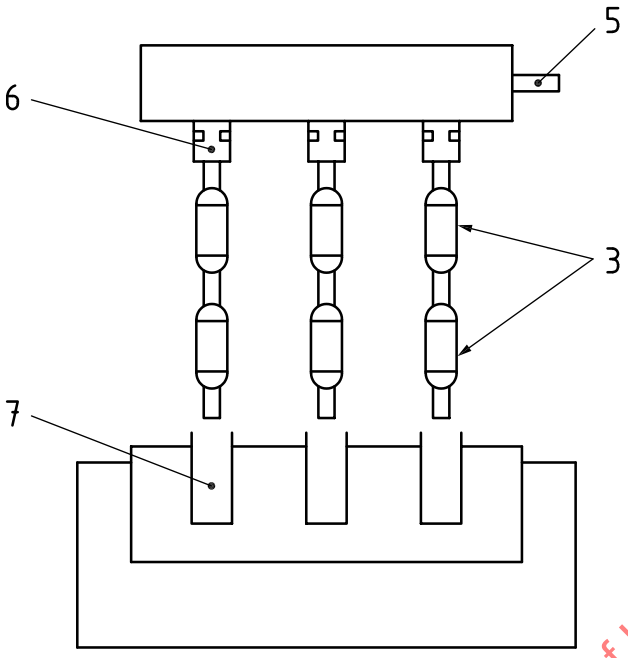

b) Rack for drying DNPH coated cartridges

Key

1	10 ml glass syringes	5	N_2 gas stream
2	test tube rack	6	syringe fitting
3	cartridges	7	waste vials
4	waste beakers		

Figure 3 — Syringe rack for coating and drying sample cartridges

7.3 Sample analysis

7.3.1 **HPLC system**, consisting of

a) a mobile phase reservoir with an outgassing device (e.g. membrane under reduced pressure);

b) a high-pressure pump;

c) an injection valve (automatic sampler with a 25 µl or other convenient loop volume);

d) a C-18 reverse phase (RP) column (e.g. 25 cm, 4,6 mm inside diameter, 5 µm particle size);

e) a UV detector or diode array detector operating at 360 nm;

f) a data recording system.

The DNPH-formaldehyde derivative is determined using isocratic reverse phase HPLC, equipped with an ultraviolet (UV) absorption detector operated at 360 nm. A blank cartridge is likewise desorbed and analysed. Formaldehyde and other carbonyl compounds in the sample are identified and quantified by comparison of their retention times and peak heights or peak areas with those of standard solutions.

NOTE 1 Most commercial HPLC analytical systems are adequate for this application.

NOTE 2 A column oven can be used to assure constant column operating temperature and improve reproducibility.

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7.3.2 Syringes and pipettes.

7.3.2.1 HPLC injection syringes with capacity at least four times the loop volume (see 7.3.1).

7.3.2.2 Syringes with a volume of 10 ml, used to prepare DNPH-coated cartridges (polypropylene syringes are adequate).

7.3.2.3 Syringe fittings and plugs to connect cartridges to the sampling system and to cap prepared cartridges.

7.3.2.4 Pipettes positive-displacement, repetitive-dispersing type, with capacities in the 0 ml to 10 ml range ISO 8655-2[1].

8 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, e.g. best quality grade, grade for chemical analysis or grade for HPLC analysis, and distilled or demineralized water or water of equivalent purity.

8.1 2,4-Dinitrophenylhydrazine, recrystallized at least twice with UV grade acetonitrile before use.

8.2 Acetonitrile, UV grade (each batch of solvent should be tested before use).

8.3 Perchloric acid, 60 % mass fraction, ρ = 1,51 kg/l, reagent grade (best source).

8.4 Hydrochloric acid, 36,5 % to 38 % mass fraction ρ = 1,19 kg/l, reagent grade (best source).

8.5 Hydrochloric acid, 2 mol/l reagent grade (best source).

8.6 Formaldehyde, 37 % mass fraction solution, reagent grade (best source).

8.7 Aldehydes and ketones, high purity, used for preparation of DNPH derivative standards (optional).

8.8 Ethanol or methanol, HPLC grade.

8.9 Nitrogen, high purity grade (best source).

8.10 Charcoal, granular (best source).

8.11 Helium, high purity grade (best source).

9 Preparation of reagents and cartridges

9.1 Purification of 2,4-dinitrophenylhydrazine

Formaldehyde contamination of the DNPH reagent is a frequently encountered problem. The DNPH (8.1) shall be purified by multiple recrystallizations in UV-grade acetonitrile (8.2). 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 and per individual compound.

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Prepare a supersaturated solution of DNPH by boiling excess DNPH in 200 ml of acetonitrile for approximately 1 h. After 1 h, remove and transfer the supernatant to a covered beaker on a hot plate and allow gradual cooling to 40 °C to 60 °C. Maintain the solution at this temperature (40 °C) until 95 % volume fraction of solvent has evaporated. Decant the solution to waste and rinse the remaining crystals twice with three times their apparent volume of acetonitrile. Transfer the crystals to another clean beaker, add 200 ml of acetonitrile, heat to boiling, and again let crystals grow slowly at 40 °C to 60 °C until 95 % volume fraction of the solvent has evaporated. Repeat the rinsing process as specified above. Take an aliquot of the second rinse, dilute 10 times with acetonitrile, acidify with 1 ml of 3,8 mol/l perchloric acid (8.3) per 100 ml of DNPH solution, and analyse by HPLC, in accordance with 10.3.4.

WARNING — Carry out this procedure under a properly ventilated hood and behind an explosion shield.

NOTE An acid is necessary to catalyse the reaction of carbonyl compounds with DNPH. Most strong inorganic acids such as hydrochloric, sulfuric, phosphoric or perchloric acids perform satisfactorily. In rare cases, hydrochloric and sulfuric acids cause problems.

An acceptable impurity level is <0,025 µg/ml of formaldehyde hydrazine in recrystallized DNPH reagent or 0,02 % mass fraction of the DNPH.

If the impurity level is not acceptable for the intended sampling application, repeat recrystallization. Transfer the purified crystals to an all-glass reagent bottle, add 200 ml of acetonitrile, stopper, shake gently, and allow to stand overnight. Analyse the supernatant by HPLC according to 10.3.4. If the impurity level is not satisfactory, pipette off the solution to waste, then add 25 ml of acetonitrile to the purified crystals. Repeat rinsing with 20 ml portions of acetonitrile until a satisfactorily low impurity level in the supernatant is confirmed by HPLC analysis.

If the impurity level is satisfactory, add another 25 ml of acetonitrile, stopper, shake the reagent bottle, then set aside. The saturated solution above the purified crystals is the stock DNPH reagent. Maintain only a minimum volume of saturated solution adequate for day-to-day operation. This minimizes waste of purified reagent, should it be necessary to re-rinse the crystals to decrease the level of impurity for applications requiring more stringent purity specifications. Use clean pipettes when removing saturated DNPH stock solution for any analytical applications. Do not pour the stock solution from the reagent bottle.

9.2 Preparation of DNPH formaldehyde derivative

To a portion of the recrystallized DNPH (9.1) add sufficient 2 mol/l HCl (8.5) to obtain an approximately saturated solution. Add to this solution formaldehyde (8.6) in molar excess of the DNPH. Filter the DNPH-formaldehyde precipitate, wash it with 2 mol/l HCl and water, and allow it to dry in air.

Check the purity of the DNPH-formaldehyde derivative by melting point determination (165 °C to 166 °C) or HPLC analysis. If the impurity level is not acceptable, recrystallize the derivative in ethanol (8.8). Repeat the purity check and recrystallize 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 (8.9) or argon further prolongs the lifetime of the derivative.

Melting points of DNPH derivatives of several carbonyl compounds are given in Annex A.

DNPH derivatives of formaldehyde and other carbonyls (8.7) suitable for use as standards are commercially available both in the form of pure crystals and as individual or mixed stock solutions in acetonitrile

9.3 Preparation of DNPH formaldehyde standards

Prepare a standard stock solution of the DNPH-formaldehyde derivative by dissolving accurately weighed amounts in acetonitrile (8.2). Prepare a working calibration standard mix from the standard

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stock solution. The concentration of the DNPH-formaldehyde derivative in the standard mix solutions should be adjusted to reflect the range of concentrations expected in real samples.

Individual stock solutions of approximately 100 mg/l can be prepared by dissolving 10 mg of the solid derivative in 100 ml of acetonitrile. The individual solution is used to prepare calibration standards containing the derivative of interest at concentrations of 0,5 µg/ml to 20 µg/ml, that spans the concentration of interest.

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.

9.4 Preparation of DNPH coated silica gel cartridges

9.4.1 General

This procedure shall be performed in an atmosphere with a very low aldehyde background concentration. All glassware and plasticware shall be thoroughly cleaned and rinsed with deionized water and aldehyde-free acetonitrile (8.2). Contact of reagents with laboratory air shall be minimized. Gloves shall be worn when handling the cartridges.

9.4.2 DNPH coating solution

Pipette 30 ml of saturated DNPH stock solution into a 1 000 ml volumetric flask, then add 500 ml acetonitrile (8.2). Acidify with 1,0 ml of concentrated HCl (8.5).

The atmosphere above the acidified solution should preferably be filtered through a DNPH-coated silica gel cartridge, to minimize contamination from laboratory air. Shake solution, then make up to volume with acetonitrile. Stopper the flask, invert, and shake several times until the solution is homogeneous. Transfer the acidified solution to a reagent bottle equipped with a positive-displacement dispenser of capacity in the 0 ml to 10 ml range.

Prime the dispenser and slowly dispense 10 ml to 20 ml to waste. Dispense an aliquot solution to a sample vial and check the impurity level of the acidified solution by HPLC according to 10.3.4. The impurity level of formaldehyde should be <0,025 µg/ml.

9.4.3 Coating of silica gel cartridges

Open the cartridge package, connect the short end of the silica gel cartridge (7.1.1) to a 10 ml syringe, and place it in the syringe rack (7.2.4) as illustrated in Figure 3. Using a positive-displacement repetitive pipette (7.3.2.4), add 10 ml of acetonitrile (8.2) to each of the syringes. Let liquid drain to waste by gravity.

Remove any air bubbles that may be trapped between the syringe and the silica cartridge by displacing them with the acetonitrile in the syringe.

Set the repetitive dispenser, containing the acidified DNPH coating solution, to dispense 7 ml into the cartridges. Once the effluent flow at the outlet of the cartridge has stopped, dispense 7 ml of the coating reagent into each of the syringes. Let the coating reagent drain by gravity through the cartridge until flow at the other end of the cartridge stops. Wipe away the excess liquid at the outlet of each of the cartridges with clean tissue paper.

Assemble a drying manifold as shown in Figure 3 b). This contains a previously prepared DNPH-coated cartridge at each of the exit ports (e.g. scrubber or “guard cartridges”). These “guard cartridges” serve to remove traces of formaldehyde that may be present in the nitrogen (8.9) gas supply. They can be prepared by drying a few of the newly coated cartridges in accordance with the instructions below and “sacrificing” these few to ensure the purity of the rest.

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Insert cartridge connectors [flared at both ends, 0,64 cm × 2,5 cm outside diameter polytetrafluoroethylene (PTFE) tubing with inside diameter slightly smaller than the outside diameter of the cartridge port] on to the long end of the scrubber cartridges.

Remove the cartridges from the syringes and connect the short ends of the cartridges to the open end of the cartridge connectors already attached to the scrubber cartridges.

Pass nitrogen (8.9) through each of the cartridges at about 300 ml/min to 400 ml/min. Rinse the exterior surfaces and outlet end of the cartridges with acetonitrile using a Pasteur pipette. After 15 min, stop the flow of nitrogen, wipe the cartridge exterior free of acetonitrile and remove the dried cartridges. Plug both ends of the coated cartridge with standard polypropylene male syringe plugs and place the plugged cartridge in a borosilicate glass culture tube with polypropylene screw caps (7.2.1).

Put a serial number and a lot number label on each of the individual cartridge glass storage containers and refrigerate the prepared lot until use.

Sampling cartridges have been found to be stable for at least 6 months when stored at 4 °C in the absence of light.

10 Procedure

10.1 Sample collection

Assemble the sampling system and ensure that the pump (7.1.2) is capable of constant flow rate throughout the sampling period. The sampling cartridges (7.1.1) can be safely used for sampling air when the temperature is above 10 °C. If required, add an ozone denuder or scrubber (see 5.2).

Before sample collection, check the system for leaks. Plug the inlet of the cartridge so no flow is indicated at the outlet end of the pump. The flow meter should not indicate any air flow through the sampling apparatus.

For unattended or extended sampling periods, a mass flow controller (7.1.3) or, as appropriate, a compensated personal sampling pump is highly recommended to maintain constant flow. The flow controller should be set at least 20 % below the fixed maximum air flow rate through the cartridge.

NOTE 1 The silica gel is held in the cartridge between two fine-porosity filter frits. Air flow during sampling can change as airborne particulates deposit on the front frit. The flow change can be significant when sampling particulate-laden atmospheres.

Install the entire assembly (including a “dummy” sampling cartridge) and check the flow rate at a value near the desired rate. Flow rates of 0,5 l/min to 1,2 l/min shall be employed. The total number of moles of carbonyl in the volume of air sampled shall not exceed that of the DNPH (2 mg or 0,01 mol/cartridge; 1 mg to 2 mg/cartridge for commercially available pre-coated cartridges). In general, a safe estimate of the sample size should be lower than 75 % of the DNPH mass loading of the cartridge [100 µg to 200 µg as HCHO, with respect to interferences to be taken into account (see Clause 5)]. Generally, calibration can be accomplished using a soap-bubble flow meter or calibrated wet test meter (7.1.4) connected to the flow exit, assuming the system is leak tight.

NOTE 2 ISO 13137[10] specifies an appropriate calibration scheme that does not require a sealed flow system downstream of the pump.

Measure and record the sampling flow rate at the beginning and end of the sampling period to determine sample volume. If the sampling period exceeds 2 h, the flow rate should be measured at intermediate points during the sampling period. Include a rotameter to allow observation of the flow rate without interruption of the sampling process. Alternatively, a sampling pump which directly measures and continuously records the flow rate can be used.

Before sampling, remove the cartridge container from the friction-top metal can or other suitable container. Let the cartridge warm to a temperature above 10 °C in the glass tube before connecting it to the sampling train.

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With a commercial pre-coated DNPH cartridge, also let the cartridge warm to a temperature above 10 °C before connecting to the sampling train.

Using gloves (7.2.2), remove the syringe plugs and connect the cartridge to the sampling system with a syringe adapter fitting. Connect the cartridge to the sampling train so that the short end becomes the sample inlet.

With commercial pre-coated DNPH cartridges, follow the manufacturer's instructions.

Turn the sampler on and adjust the flow to the desired rate. A typical flow rate through one cartridge is 1,0 l/min and 0,8 l/min for two cartridges in tandem. Operate the sampler for the desired period, with periodic recording of the sampling variables.

If the ambient air temperature during sampling is below 10 °C, the sampling cartridge should be kept in a warmer environment. No significant effects of relative humidity have been observed for sampling under various weather conditions — cold, wet and dry winter months as well as hot and humid summer months.

At the end of the sampling period, check the flow rate just before stopping the flow. If the flow rates at the beginning and end of the sampling period differ by more than 10 %, the sample should be marked as suspect.

If there is the need to convert the concentrations to standard conditions (temperature and pressure) by calculation, temperature and pressure have to be measured during sampling.

Immediately after sampling, remove the cartridge (using gloves) from the sampling system, cap with the original end plugs, and place it back in the original labelled container. Seal with PTFE tape, and place in a friction-top can (7.2.3) containing 2 cm to 5 cm depth of granular charcoal (8.10) or in another suitable container with appropriate padding. If appropriate, a heat-sealable foil-lined plastic pouch may be used for storing the exposed cartridge. Refrigerate the exposed sample cartridge until analysis. The refrigeration period prior to analysis should not exceed 30 days.

If samples are to be transported to a central laboratory for analysis, the duration of the non-refrigerated period should be kept to a minimum, preferably less than 2 days.

Calculate the average sample flow rate from Formula (1)

$$\bar{q}_V = \frac{q_1 + q_2 + \ldots + q_n}{n} \tag{1}$$

where

$\bar{q}_V$ is the average flow rate, in millimetres per minute;

$q_1, q_2 \ldots q_n$ are the flow rates determined at beginning, end and intermediate points during sampling;

n is the number of points averaged.

The total flow is then calculated using Formula (2):

$$V_m = \frac{(t_2 - t_1) \times \bar{q}_V}{1\ 000} \tag{2}$$

where

V_m is the total volume, in litres, sampled at the measured temperature and pressure;

t_2 is the stop time;

t_1 is the start time;

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$t_2 - t_1$ is the total sampling duration, in minutes;

$\bar{q}_V$ is the average flow rate, in millilitres per minute.

10.2 Process blanks

At least one field blank shall be analysed with each set of samples. For sets larger than 10 samples, a minimum of 10 % of the samples analysed shall be field blanks. The number of samples within a group or time frame, or both, should be recorded so that a specified percentage of field blanks is obtained for a given number of air samples. The field blank is treated identically to the samples except that no air is drawn through the cartridge. The performance criteria specified in 10.1 should be met for process blanks. It is desirable to analyse blank cartridges retained in the laboratory (lab blanks) as well, to distinguish between possible field and laboratory contamination.

10.3 Sample analysis

10.3.1 Sample preparation

Return the samples to the laboratory in a suitable external container with 2 cm to 5 cm of granular charcoal (8.10) and store them in a refrigerator until analysis. Alternatively, the samples may also be stored alone in their individual containers. The time between sampling and analysis should not exceed 30 days.

10.3.2 Sample desorption

Connect the sample cartridge (inlet or short end during sampling) to a clean syringe.

The liquid flow during desorption should be in the same direction as the air flow during sampling, to prevent insoluble particulates from getting into the eluate. Reverse desorption may be performed if the eluate is filtered prior to HPLC analysis. A filtered blank extract shall be analysed with each batch of samples to confirm that no contamination is being introduced by the filter.

Place the cartridge and syringe in the syringe rack. Desorb the DNPH derivatives of the carbonyls and the unreacted DNPH from the cartridge (gravity feed) by passing 5 ml of acetonitrile (8.2) from the syringe through the cartridge to a graduated test tube or to a 5 ml volumetric flask. Other volumes of acetonitrile may be appropriate, depending on the sampling cartridge used.

NOTE A dry cartridge has an acetonitrile holdup volume slightly greater than 1 ml. The eluate flow can stop before the acetonitrile in the syringe is completely drained into the cartridge because of air trapped between the cartridge filter and the syringe adapter tip. If this happens, displace the trapped air with the acetonitrile in the syringe using a long-tip disposable Pasteur pipette.

Make up to the 5 ml mark with acetonitrile. Label the flask with sample identification. Pipette an aliquot into a sample vial with a PTFE-lined septum. Analyse the aliquot for the carbonyl derivatives by HPLC. As a backup, a second aliquot may be taken and stored under refrigeration until the results of the analysis of the first aliquot are complete and validated. The second aliquot can be used for confirmatory analysis, if necessary.

For glass sealed DNPH sampling tubes that contain two sorbent beds, uncap the end of the tube closest to the second sorbent layer (exit end). Carefully remove the spring (snap ring) and plug of glass wool holding the sorbent layer in place. Empty the sorbent into a clean 4 ml glass vial with a PTFE-lined septum or cap. Mark this as the backup sampling section. Carefully remove the next plug of glass wool and empty the remaining sorbent into another 4 ml vial. Mark this as the primary sampling section. Into each vial, carefully pipette 3 ml acetonitrile into each vial, cap the vials and allow to stand for 30 min with occasional agitation.

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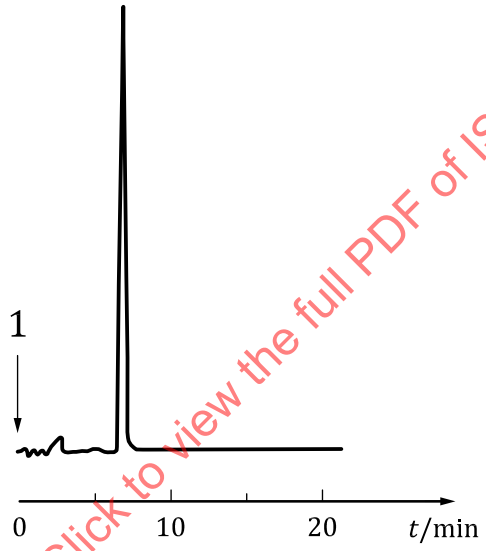
10.3.3 HPLC calibration

Prepare calibration standards from prepared or commercial stock solutions in acetonitrile (9.2) from the DNPH-formaldehyde derivative (see 9.3). Individual stock solutions of 100 mg/l are prepared by dissolving 10 mg of solid derivative in 100 ml of mobile phase.

Analyse each calibration standard (at least five levels) twice and tabulate area response against the mass injected (or, more conveniently, versus the DNPH-formaldehyde injected, for a fixed loop volume; see Figures 4 and 5). Perform all calibration runs as specified for sample analysis in 10.3.4. To avoid carry-over effects, start with the lower concentration. Using the UV detector or the diode array detector, a linear response range of approximately 0,05 µg/ml to 20 µg/ml should be achieved for 25 µl injection volumes. The results can be used to prepare a calibration curve, as illustrated in Figure 6. Linear response is indicated where a correlation coefficient of at least 0,999 for a linear least-squares fit of the data (concentration versus area response) is obtained. The retention times for each analyte should agree within 2 %.



Key

t time, in min

1 injection

Operating parameters, HPLC

Column: C-18 reverse phase

Mobile phase: 60 % volume fraction acetonitrile + 40 % volume fraction water

Detector: ultraviolet, at 360 nm

Flow rate: 1 ml/min

Retention time: about 7 min for formaldehyde

Sample injection volume: 25 µl

Figure 4 — Example chromatogram of DNPH formaldehyde derivative

Once linear response has been documented, an intermediate concentration standard near the anticipated levels of each component, but at least 10 times the detection limit, should be chosen for daily calibration. The day-to-day response for the various components should be within 10 % for analyte concentrations of 1 µg/ml or greater, and below 20 % for analyte concentrations near 0,5 µg/ml. If greater variability is observed, recalibrate or establish a new calibration curve from fresh standards.

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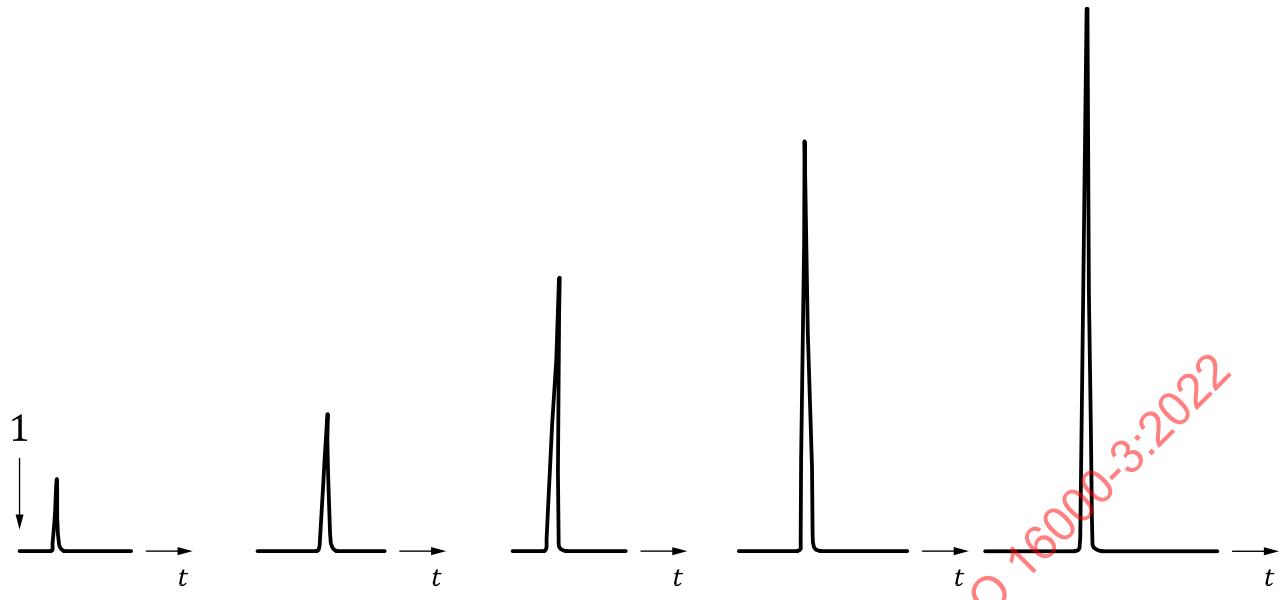

a) 0,61 µg/ml **b)** 1,23 µg/ml **c)** 6,16 µg/ml **d)** 12,32 µg/ml **e)** 18,48 µg/ml

Key

t time

1 injection

Operating parameters, HPLC

Column: C-18 reverse phase (4,6 mm inside diameter x 25 cm or equivalent)

Mobile phase: 60 % volume fraction acetonitrile + 40 % volume fraction water

Column temperature: 25 °C

Detector: ultraviolet, at 360 nm

Flow rate: 1 ml/min

Retention time: about 7 min for formaldehyde

Sample injection volume: 25 µl

Concentration µg/ml	**Number of area counts**
0,61	226 541
1,23	452 166
6,16	2 257 271
12,32	4 711 408
18,48	6 953 812

Figure 5 — Examples of HPLC chromatograms of varying concentrations of DNPH-formaldehyde derivative

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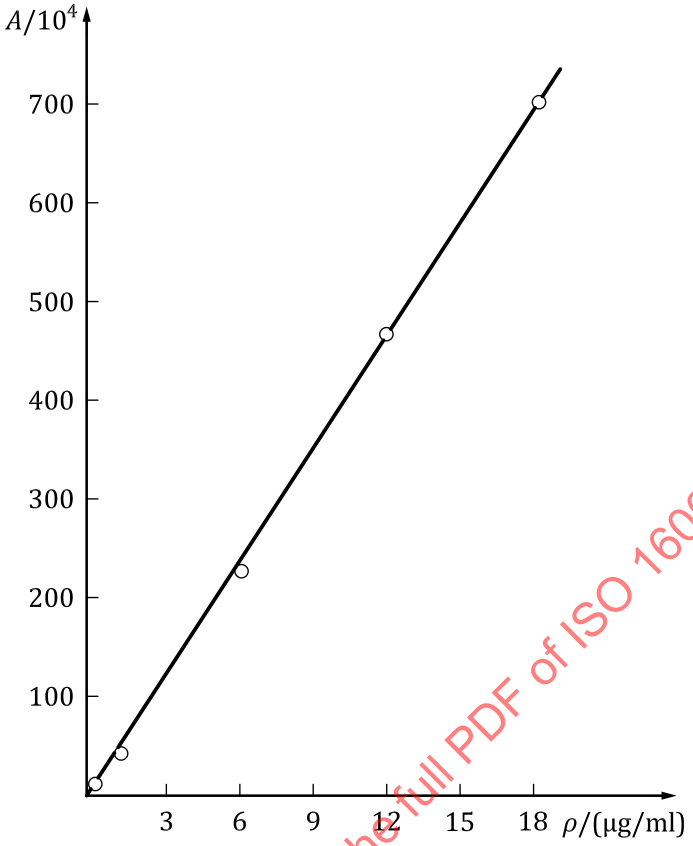

Key

A number of area counts

ρ mass concentration of DNPH-formaldehyde derivative

Operating parameters, HPLC

Correlation coefficient: 0,999 9

Column: C-18 reverse phase

Mobile phase: 60 % volume fraction acetonitrile + 40 % volume fraction water

Detector: ultraviolet, at 360 nm

Flow rate: 1 ml/min

Retention time: about 7 min for formaldehyde

Sample injection volume: 25 µl

Figure 6 — Example calibration curve for formaldehyde

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10.3.4 HPLC analysis for formaldehyde

Assemble the HPLC system and calibrate as specified in 10.3.3. Typical operating parameters are given in the following.

An octadecylsilane column (100 mm × 5 mm inside diameter, 10 µm particles) with a mobile phase of a 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 (see ISO 16000-4).

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Alternatively, an isocratic mobile phase of a 60 % volume fraction acetonitrile + 40 % volume fraction water mixture has been shown to be suitable. The parameters are as follows:

— column: C-18 (4,6 mm inside diameter x 25 cm, or equivalent); a column oven may be used for more accurate temperature control, if needed

— mobile phase: 60 % volume fraction acetonitrile +40 % volume fraction water, isocratic, bath temperature 40 °C

— detector: Ultraviolet, operating at 360 nm

— flow rate: 1,0 ml/min

— retention time: 7 min for formaldehyde with one C-18 column, 13 min for formaldehyde with two C-18 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 or PTFE suction filtration apparatus. Degas the filtered mobile phase by purging with helium (8.11) 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 watch glass. A constant back-pressure restrictor (350 kPa) or 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.

NOTE 1 Instead of degassing the mobile phase (acetonitrile and water mixture) by helium purging or heating, common laboratory practice is to employ ultrasonic deaeration or on-line deaeration through membrane under reduced pressure.

Place the mobile phase in the HPLC solvent reservoir and set the pump at a flow rate of 1,0 ml/min. The bath temperature is set to 40 °C. Allow it to pump for 20 min to 30 min before the first analysis. Switch the detector on 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 µl 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 and 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 (see Figure 4), terminate data acquisition and calculate the component concentrations as specified in Clause 11. After a stable baseline is achieved, the system can be used for further sample analysis as specified in the preceding.

NOTE 2 After several cartridge analyses, build up on the column (if indicated, e.g. 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 % volume fraction acetonitrile. The same protection can be achieved if pre-columns are used.

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 to within ±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,

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decrease the ratio. If a solvent change is necessary, always recalibrate (see 10.3.3) before running samples.

NOTE 3 The chromatographic conditions specified here have been optimized for the detection of formaldehyde. 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 can also be used.

Examine the chromatogram for evidence of ozone interference in accordance with 5.2 and Figure 2.

10.3.5 HPLC analysis of other aldehydes and ketones

10.3.5.1 General

Optimizing chromatographic conditions, by using two C-18 columns in series and varying the mobile phase composition through a gradient programme, enables the analysis of other aldehydes and ketones collected from air. In particular, chromatographic conditions can be optimized to separate acetone, propionaldehyde and some higher molecular mass aldehydes within an analysis time of about 1 h.

The linear gradient programme varies the mobile phase composition periodically to achieve maximum resolution of the C_3, C_4, and benzaldehyde region of the chromatogram.

EXAMPLE The following gradient programme was found to be adequate to achieve this goal: upon sample injection, linear gradient in volume fraction from 60 % volume fraction acetonitrile (ACN) + 40 % volume fraction water to 75 % volume fraction ACN + 25 % volume fraction water in 36 min; to 100 % volume fraction ACN in 20 min; 100 % ACN for 5 min; reverse linear gradient from 100 % ACN to 60 % volume fraction ACN + 40 % volume fraction water in 1 min; maintain at 60 % volume fraction ACN + 40 % volume fraction water for 15 min.

10.3.5.2 Sample analysis for other carbonyl compounds

Assemble and calibrate the HPLC system as specified in 9.3.3. The operating parameters are:

Column: C-18, two columns in series

Mobile phase: acetonitrile + water, linear gradient

Detector: ultraviolet, operating at 360 nm

Flow rate: 1,0 ml/min

Programme: see 10.3.4

The chromatographic conditions described herein have been optimized for a gradient HPLC system equipped with a UV detector or a diode array detector, an automatic sampler with a 25 µl loop injector and two C-18 columns (4,6 mm ´ 250 mm), and a recorder or electronic integrator. Analysts are advised to experiment with their HPLC systems to optimize chromatographic conditions for their particular analytical needs. To ensure a safe quantification of acetone and propionaldehyde, the separation of these two substances from the interfering acrolein peak(s) should be a minimum goal of the optimization.

NOTE Column manufacturers usually recommend optimal conditions for the separation of DNPH derivatives with their reverse phase columns. These recommendations can eliminate the need for dual columns without compromising the resolution of the carbonyl compounds.

The carbonyl compounds in the sample are identified and quantified by comparing their retention times and number of area counts with those of standard DNPH derivatives. Formaldehyde, acetaldehyde, acetone, propionaldehyde, benzaldehyde and *o*-, *m*-, *p*-tolualdehydes can be identified with a high degree of confidence. The identification of butyraldehyde is less certain, because it coelutes with isobutyraldehyde and methyl ethyl ketone under the stated chromatographic conditions. Quantification of the unsaturated aldehydes acrolein and crotonaldehyde is not possible with this method due to instability of the derivatives and uncontrollable further reactions with DNPH. Figure 7 illustrates a typical chromatogram obtained with the gradient HPLC system.

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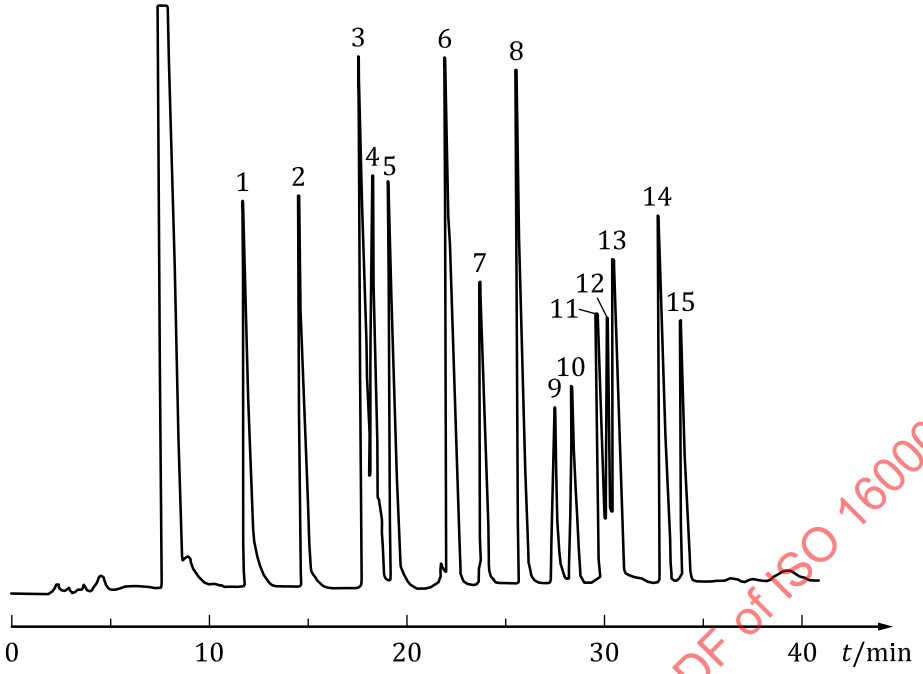
The concentrations of individual carbonyl compounds are determined as specified in 10.3.4.



Key

t time

Peak identification		
Number	**Compound**	**Concentration** µg/ml
1	Formaldehyde	1,140
2	Acetaldehyde	1,000
3	Acrolein	1,000
4	Acetone	1,000
5	Propionaldehyde	1,000
6	Crotonaldehyde	1,000
7	Butyraldehyde[a]	1,000
8	Benzaldehyde	1,000
9	Isovaleraldehyde	0,450
10	Valeraldehyde	0,450
11	*o*-Tolualdehyde	0,515
12	*m*-Tolualdehyde	0,505
13	*p*-Tolualdehyde	0,510
14	Capronaldehyde	1,000
15	2,5-Dimethylbenzaldehyde	0,510

[a] The identification of butyraldehyde is less certain, because it coelutes with isobutyraldehyde and methyl ethyl ketone (2-butanone) under the stated chromatographic conditions. Therefore, special care should be exercised if this programme is to be used to determine butyraldehyde in the presence of either of the latter potentially interfering compounds.

Figure 7 — Example of chromatographic separation of DNPH hydrazones of 15 carbonyl standards

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11 Calculations

Calculate the corrected total mass, in micrograms, of analyte (DNPH derivative) extracted from the cartridge, m_d, for each sample using Formula (3):

$$m_d = m_s - m_b \tag{3}$$

where m_s is the uncorrected mass of the DNPH derivative, in micrograms, on the sample cartridge;

$$m_s = A_s \frac{\gamma_{std}}{A_{std}} V_s d_s \tag{4}$$

where m_b is the analyte mass, in micrograms, in the blank cartridge;

$$m_b = A_b \frac{\gamma_{std}}{A_{std}} V_b d_b \tag{5}$$

where

A_s is the number of area counts, eluate from sample cartridge;

A_b is the number of area counts, eluate from blank cartridge;

A_{std} is the number of area counts, standard;

γ_{std} is the concentration, in micrograms per millilitre, of analyte in the daily calibration standard;

V_s is the total volume, in millilitres, of the sample cartridge eluate;

V_b is the total volume, in millilitres, of the blank cartridge eluate;

d_s is the dilution factor for the sample cartridge eluate:

= 1 if sample was not rediluted;

= V_d/V_a if sample was rediluted to bring the detector response within linear range;

where

V_d is the redilution volume, in millilitres;

V_a is the aliquot used for redilution, in millilitres;

d_b is the dilution factor for the blank cartridge eluate = 1,0.

Calculate the concentration, in nanograms per litre, of the carbonyl compound in the original sample, γ_A, from Formula (6):

$$\gamma_A = m_d \frac{M_c}{M_{der}} * \frac{1\ 000}{V_m} \tag{6}$$

where

V_m is the total air sample volume, in litres, under indoor conditions, from 10.1;

M_c is the molar mass of carbonyl compound (for formaldehyde = 30 g/mol);

M_{der} is the molar mass of the DNPH derivative (for formaldehyde = 210 g/mol).

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NOTE The use of parts per billion or parts per million is discouraged. However, for the convenience of certain users, the following information is provided.

To convert the carbonyl compound concentration, γ_A, to parts per billion (10^{-9}) as a volume fraction, use Formula (7):

$$\varphi_{As} = \frac{\gamma_{As} * 24,3}{M_c} \tag{7}$$

where

φ_{As} is the concentration under standard conditions, in parts per billion (10^{-9}) as a volume fraction, of carbonyl compound;

γ_{As} is the concentration, in nanograms per litre, of carbonyl compound in the original sample, calculated using the air volume corrected to 23 °C and 101,3 kPa (V_s);

24,3 is the ideal gas volume, corrected to 23 °C, in nanolitres.

The corrected air total sample volume, in litres, at 23 °C and 101,3 kPa, V_s, is calculated from Formula (8):

$$V_s = \frac{V_m \bar{p}}{101,3} * \frac{296,15}{273,15+\bar{T}} \tag{8}$$

where

V_m is the total sample volume, in litres, under indoor conditions;

$\bar{p}$ is the average indoor pressure, in kilopascals;

$\bar{T}$ is the average indoor temperature, in degrees Celsius.

If it is desired to obtain a concentration result in terms of parts per million (10^{-6}) (23 °C and 101,3 kPa) to compare with standards stated in these terms, the volume sampled is not corrected for temperature and pressure.

12 Performance criteria and quality assurance

12.1 General

This clause summarizes required quality assurance measures and provides guidance concerning performance criteria that should be achieved within each laboratory.

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

12.2 Standard operating procedures (SOPs)

Users should generate SOPs describing the following activities in their laboratory: assembly, calibration, and operation of the sampling system, with the manufacturer and model of equipment used; preparation, purification, storage, and handling of sampling reagent and samples; assembly, calibration, and operation of the HPLC system, with make and model of equipment used; and all aspects of data recording and processing, including lists of computer hardware and software used.

The SOPs should provide specific stepwise instructions and should be readily available to and understood by the laboratory personnel conducting the work. The SOPs shall be consistent with this document.

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12.3 HPLC system performance

Calculate the column efficiency (theoretical plates), η, of the HPLC system according to Formula (9):

$$\eta = 5{,}54\left(\frac{t_r}{b_{1/2}}\right)^2 \tag{9}$$

where

t_r is the retention time, in seconds, of analyte;

$b_{1/2}$ is the width of the component peak at half-height, in seconds.

A column efficiency of >5 000 theoretical plates should be obtained.

The coefficient of variation (CV) (standard deviation) of the response for replicate HPLC injections should be ±10 % or less, day to day, for analyte calibration standards at 1 µg/ml or greater levels. At the 0,5 µg/ml level and below, the precision of replicate analyses can vary by up to 20 % for some carbonyl compounds. Precision of retention times should be better than ±7 % on any given day.

12.4 Sample loss

Sample loss can occur when the capacity of the sorbent is exceeded, or when the flow rate exceeds the maximum compatible with complete collection. This possibility can be guarded against by setting two sampling cartridges in series, and analysing the contents of each, or by analysing both sections of a two-section sorbent cartridge. Should the quantity of collected analyte in the back-up section exceed 15 % of the analyte collected by the primary sampling section, breakthrough can be assumed to have occurred and may have compromised the accuracy of the results.

12.5 Measurement plan

The measurement plan shall specify the measures to be taken to meet the quality requirements specified by the client.

It is advisable to carry out replicate sampling. One or more of the samples may be archived for later analysis, if desired. The recovery rates shall be documented.

Important criteria to be taken into account including those for selection of a contractor or laboratory to perform the measurements should address included the following questions.

— Does the measuring laboratory have a documented quality assurance system (e.g. in accordance with ISO/IEC 17025)?

— What calibration procedures are used, how often and how extensively?

— Which methods are to be used to identify formaldehyde and other carbonyl compounds?

— Are collocated measurements necessary?

— How are the uncertainties defined (e.g. in accordance with ISO/IEC Guide 98-3[9])?

— Does the laboratory participate in interlaboratory tests?

13 Precision and uncertainty

As is the case for other compounds, the precision and uncertainty of the determination of formaldehyde in indoor air is influenced by two parameters, the reproducibility of the analytical procedure and the variation over time of the analyte concentration in the air. It is reasonable to assume that, generally, the

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