
Indoor air —

Part 3:

**Determination of formaldehyde and other
carbonyl compounds — Active sampling
method**

iTeh STANDARD PREVIEW

Air intérieur —

(standards.iteh.ai)

*Partie 3: Dosage du formaldéhyde et d'autres composés carbonyles —
Méthode par échantillonnage actif*

[ISO 16000-3:2001](#)

<https://standards.iteh.ai/catalog/standards/sist/a1966379-5579-4c9f-be26-41ca63458be5/iso-16000-3-2001>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 16000-3:2001

<https://standards.iteh.ai/catalog/standards/sist/a1966379-5579-4c9f-be26-41ca63458be5/iso-16000-3-2001>

© ISO 2001

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.

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

Printed in Switzerland

Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope.....	1
2 Normative references.....	2
3 Principle.....	2
4 Limitations and interferences.....	3
4.1 General.....	3
4.2 Ozone interference.....	4
5 Safety measures.....	5
6 Apparatus.....	5
7 Reagents.....	7
8 Preparation of reagents and cartridges.....	7
8.1 Purification of 2,4-dinitrophenylhydrazine.....	7
8.2 Preparation of DNPH-formaldehyde derivative.....	8
8.3 Preparation of DNPH-formaldehyde standards.....	8
8.4 Preparation of DNPH-coated silica gel cartridges.....	9
9 Procedure.....	10
9.1 Sample collection.....	10
9.2 Process blanks.....	11
9.3 Sample analysis.....	12
10 Calculations.....	19
11 Performance criteria and quality assurance.....	20
11.1 General.....	20
11.2 Standard Operating Procedures (SOPs).....	20
11.3 HPLC system performance.....	21
11.4 Sample loss.....	21
12 Precision and uncertainty.....	21
Annex A (informative) Precision and uncertainty.....	22
Annex B (informative) Melting points of DNPH-carbonyl derivatives.....	24
Bibliography.....	25

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 3.

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 part of ISO 16000 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 16000-3 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 1: General aspects of sampling strategy
- 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 chamber air by active sampling on TENAX TA sorbent, thermal desorption and gas chromatography using MS/FID
- Part 7: Sampling strategy for determination of airborne asbestos fibre concentrations

Annexes A and B of this part of ISO 16000 are for information only.

Introduction

This part of ISO 16000 is intended to be used for characterizing indoor air following the sampling strategy described 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 onto 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 U.S. EPA method TO-11 A [1].

Formaldehyde and certain other carbonyl compounds have a high toxic potential [2].

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 16000-3:2001](https://standards.iteh.ai/catalog/standards/sist/a1966379-5579-4c9f-be26-41ca63458be5/iso-16000-3-2001)

<https://standards.iteh.ai/catalog/standards/sist/a1966379-5579-4c9f-be26-41ca63458be5/iso-16000-3-2001>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 16000-3:2001

<https://standards.iteh.ai/catalog/standards/sist/a1966379-5579-4c9f-be26-41ca63458be5/iso-16000-3-2001>

Indoor air —

Part 3:

Determination of formaldehyd and other carbonyl compounds — Active sampling method

1 Scope

This part of ISO 16000 describes a procedure for the determination of formaldehyde (HCHO)¹⁾ and other carbonyl compounds¹⁾ (aldehydes and ketones) in air. The method is specific for formaldehyde but, with modification, at least thirteen other carbonyl compounds can be detected and quantified. It is suitable for determination of formaldehyde and other carbonyl compounds in the concentration range of approximately 1 µg/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 describes a sampling and analysis procedure for formaldehyde and other carbonyl compounds that involves collection from air onto cartridges coated with 2,4-dinitrophenylhydrazine (DNPH) and subsequent analysis by high performance liquid chromatography (HPLC) with detection by ultraviolet absorption [1, 3].

ISO 16000-3:2001

The procedures described are written specifically for the sampling and analysis of formaldehyde in air using an adsorbent cartridge and HPLC. The method also permits the determination of other aldehydes and ketones collected from air.

This part of ISO 16000 is applicable to the following carbonyl compounds:

Formaldehyde	Acetaldehyde	Acetone
Benzaldehyde	Butyraldehyde	Valeraldehyde
2,5-Dimethylbenzaldehyde		Crotonaldehyde
Isovaleraldehyde	Propionaldehyde	Hexanal
<i>o</i> -Tolualdehyde	<i>p</i> -Tolualdehyde	<i>m</i> -Tolualdehyde

¹⁾ Instead of the nomenclature according to IUPAC regulations, the more common names are used in this International Standard:

formaldehyde: methanal
 acetaldehyde: ethanal
 acetone: 2-propanone
 butyraldehyde: butanal
 crotonaldehyde: 2-butenal
 isovaleraldehyde: 3-methylbutanal
 propionaldehyde: propanal
m-tolualdehyde: 3-methylbenzaldehyde
o-tolualdehyde: 2-methylbenzaldehyde
p-tolualdehyde: 4-methylbenzaldehyde
 valeraldehyde: pentanal

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 16000. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 16000 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 9000-1:1994; *Quality management and quality assurance standards — Part 1: Guidelines for selection and use.*

ISO 9000-2:1997; *Quality management and quality assurance standards — Part 2: Generic guidelines for the application of ISO 9001, ISO 9002 and ISO 9003.*

ISO 16000-1, *Indoor air — Part 1: General aspects of sampling strategy.*

ISO 16000-2, *Indoor air — Part 2: Sampling strategy for formaldehyde.*

ISO 16000-4, *Indoor air — Part 4: Determination of formaldehyde — Diffusive sampling method.*

ISO 17025:1999; *General requirements for the competence of testing and calibration laboratories.*

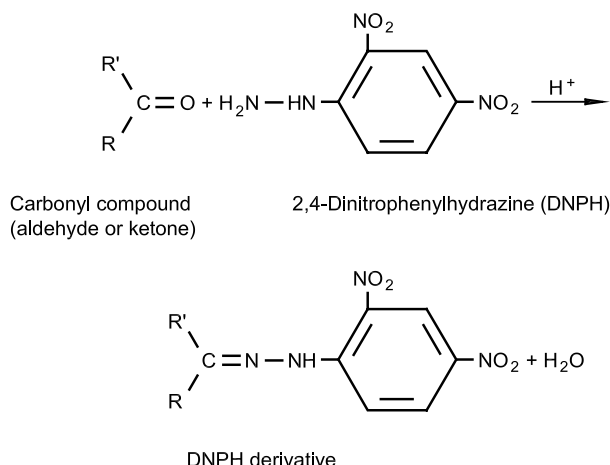
EN 45001:1989; *General criteria for the operation of testing laboratories.*

iTeh STANDARD PREVIEW (standards.iteh.ai)

3 Principle

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 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 alkyl or aromatic groups for ketones, or H for aldehydes

R' alkyl or aromatic groups, for ketones

Figure 1 — Reaction of carbonyl compounds

4 Limitations and interferences

4.1 General

The sampling flowrate, as described in this part of ISO 16000, has been validated for sampling rates up to 1,5 l/min. This flowrate 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. Organic compounds that have the same retention times and significant absorbance at 360 nm as the DNPH derivative of formaldehyde will interfere. Such interferences can often be overcome by altering the separation conditions (for example, 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. 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 may produce artefacts and should be avoided [4].

This method cannot be used for accurate quantification of acrolein in air. Inaccurate results for acrolein may result from the formation of multiple derivative peaks and the instability of the peak ratios [9].

NO₂ reacts with DNPH. High concentrations of NO₂ (for example, for gas cooking stoves) may cause problems as the retention time of the DNPH derivative may be similar to that of the DNPH formaldehyde derivative, depending on the HPLC column and the parameters [14, 15, 16].

4.2 Ozone interference

If there is suspicion that abnormally high levels of ozone may be 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 [5]. 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/m³ and 80 µg/m³, respectively) [6]. 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.

The most direct solution to ozone interference is to remove the ozone before the sampled air reaches the cartridge. This may 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 may be constructed of 1 m of 0,64 cm outside diameter by 0,46-cm inside diameter copper tubing, 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 described is about 200 µg ozone/m³ 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 [7]. 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 [8].

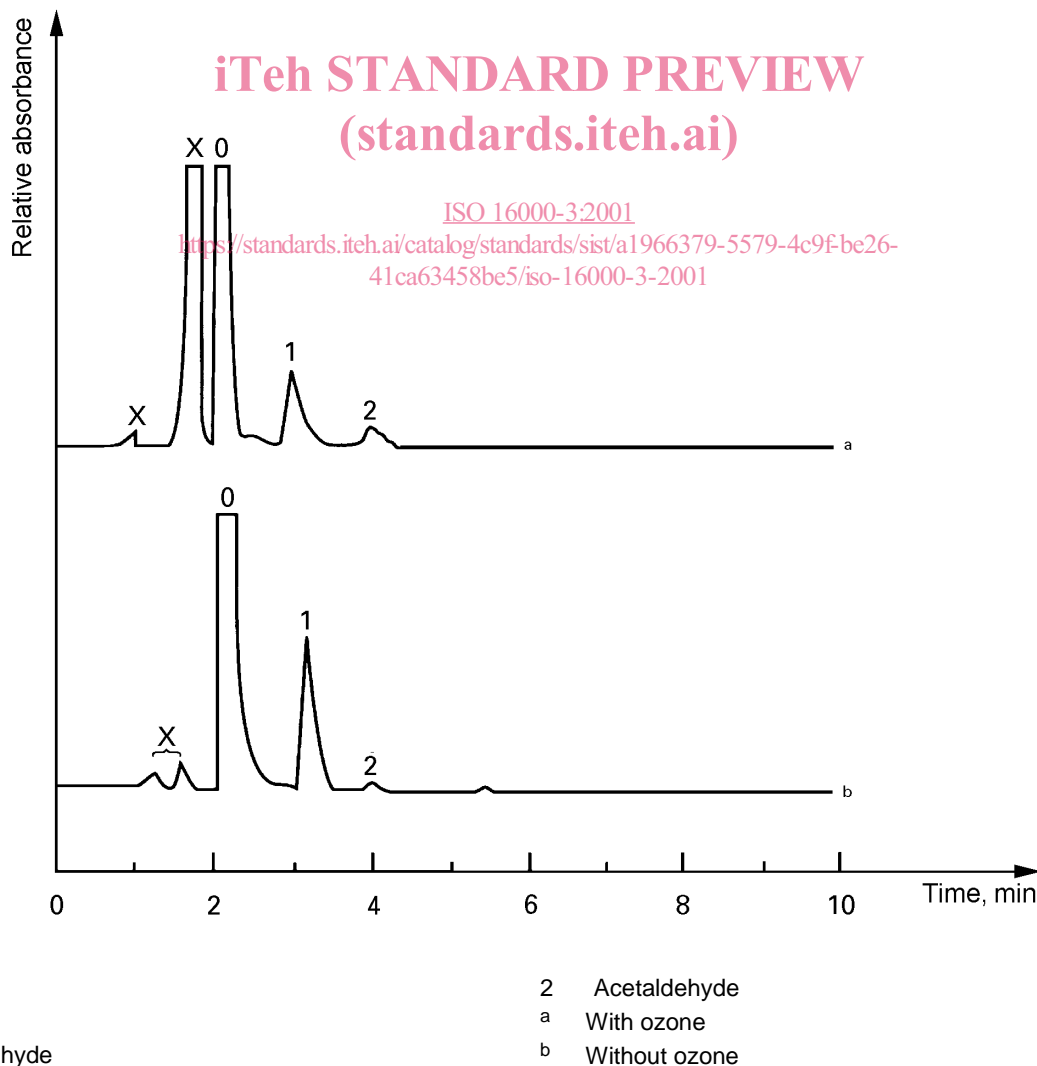


Figure 2 — Cartridge samples of formaldehyde in an air stream with and without ozone

5 Safety measures

5.1 This part of ISO 16000 does not purport to address all of 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.

5.2 2,4-Dinitrophenylhydrazine is explosive in the dry state and shall be handled with extreme care. It is also toxic (LD₅₀, rat = 654 mg/kg), has been shown to be mutagenic in some tests, and is irritating to the eyes and skin.

5.3 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.

6 Apparatus

Ordinary laboratory apparatus and the following.

6.1 Sampling

6.1.1 Sampling cartridge, packed with silica gel and coated with DNPH in accordance with clause 8, 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 may exhibit lower pressure-drops, which will permit the use of battery-operated personal sampling pumps.

6.1.2 Air sampling pump, capable of accurately and precisely sampling at a flowrate of 0,1 l/min to 1,5 l/min.

6.1.3 Flow controller, mass flowmeters and mass flow controllers, or other suitable device for metering/setting air flowrate of 0,50 l/min to 1,20 l/min through sample cartridge.

6.1.4 Flow calibrator, such as a rotameter, soap-bubble meter or wet test meter.

6.2 Sample preparation

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

6.2.2 Polyethylene gloves to handle silica gel cartridges.

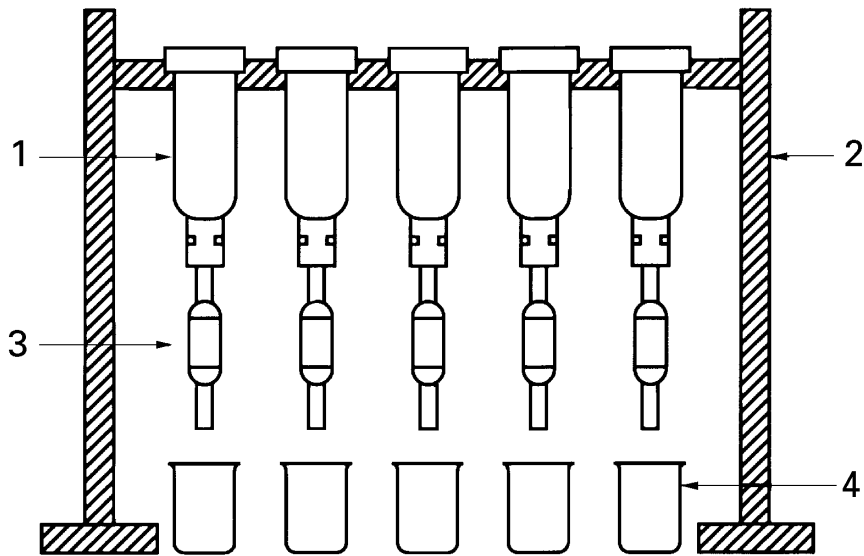
6.2.3 Transportation containers, friction-top metal cans (e.g. of volume 4 l) or other suitable containers, with polyethylene air-bubble packing or other suitable padding, to hold and cushion the sealed cartridge containers.

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

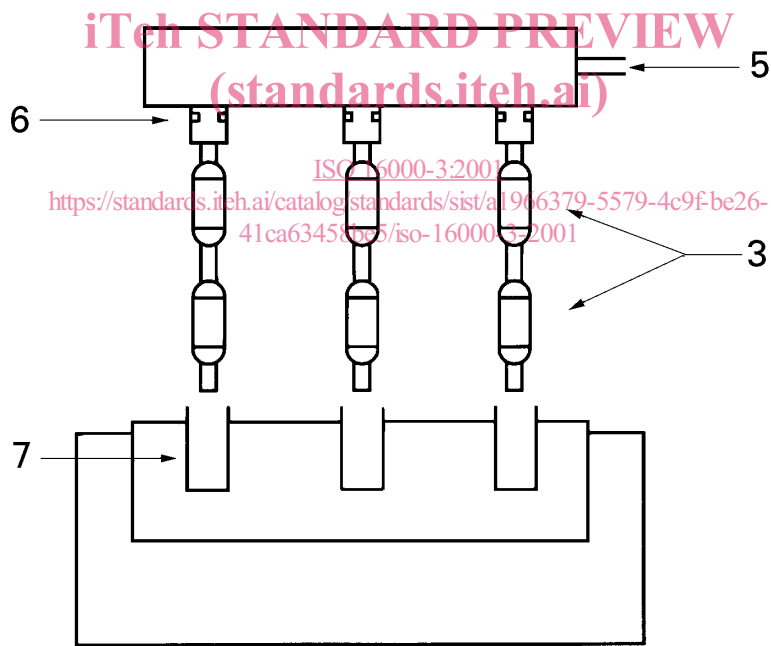
6.2.4 Support for coating cartridges.

A syringe rack, made from an aluminium plate (0,16 cm × 36 cm × 53 cm) with adjustable legs on four corners. A matrix (5 × 9) of circular holes of diameter slightly larger than the diameter of the 10 ml syringes, symmetrically drilled from the centre of the plate, to enable batch processing of 45 cartridges for cleaning, coating and/or sample elution (see Figure 3).

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



a) Rack for coating cartridges



b) Rack for drying DNPB-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

NOTE The apparatus described in 6.2.4 and 6.2.5 are needed only if the user chooses to make his own DNPB-coated cartridges.