ISO/FDIS 16000-33:2024(E)

ISO/TC 146/SC 6/WG 20

Secretariat: DIN

Date: 2024-02-23

Indoor air —

Part 33: Determination of phthalates with gas chromatography/mass spectrometry (GC-/MS)

Air intérieur — Partie 33: Détermination des phtalates par chromatographie en phase gazeuse/spectrométrie de masse (CPG/SM)

Second edition

Date: 2023-12-04

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2c576d3691c7/iso-fdis-16000-33

Edited DIS - MUST BE USED FOR FINAL DRAFT

FDIS stage

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

© ISO #### – All rights reserved

© ISO 20232024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: + 41 22 749 01 11
EmailE-mail: copyright@iso.org
Website: www.iso.org

Published in Switzerland

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

Contents

iTeh Standards (https://standards.iteh.ai) Document Preview ISO/FDIS 16000-33 https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ee-47a7-a1d0-2e576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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 second edition cancels and replaces the first edition (ISO 16000-33:2017), which has been technically revised.

The main change is as follows: a description of an adsorbent which can alternatively be used has been added.

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.

Field Code Changed

iTeh Standards (https://standards.iteh.ai) Document Preview
ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/sist/3576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

Introduction

Different parts of the ISO 16000 series describe the general requirements relating to the measurement of indoor air pollutants and the important conditions to be observed before or during the sampling of individual pollutants or groups of pollutants, as well as the measurement procedures themselves.

The definition of indoor environment is given by ISO 16000-1. Dwellings [living rooms, bedrooms, do-it-yourself (DIY) rooms, sports rooms and cellars, kitchens and bathrooms], workrooms or workplaces in buildings which are not subject to health and safety inspections with respect to air pollutants (e.g. offices, salesrooms), public buildings (e.g. restaurants, theatres, cinemas and other meeting rooms) and passenger cabins of motor vehicles and public transport are among the most important types of indoor environment.

Phthalates, the diesters of the ortho-phthalic acid (1,2-benzene dicarboxylic acid), are emitted into the indoor air primarily from articles of daily use made of soft polyvinyl chloride (PVC). Typically, phthalates are used as plasticizers in soft PVC. Four most frequently used phthalates are diisodecylphthalate (DiDP), diisononylphthalate (DiNP), di-2-ethylhexyl terephthalate (DOTP) and di-isononyl cyclohexane dicarboxylate (DINCH but other families of esters are available. Di(2-ethylhexyl)-phthalate (DEHP), di-n-butyl-phthalate (DBP) and benzyl-n-butyl-phthalate (BBP) were used in Europe until more recent regulatory developments placed restrictions on their use in the manufacture of new articles. However, these can still be present in articles currently in use and are subject to assessment. An overview of the most important phthalates, their acronyms and several relevant substance properties can be found in Table A.1. These phthalates can be determined in indoor environments by means of the analytical methods incorporating gas chromatography/mass spectrometry specified in this document.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

Indoor air— —

Part 33:
Determination of phthalates with gas chromatography/mass spectrometry (GC/MS)

1 Scope

This document specifies the sampling and analysis of phthalates in indoor air and describes the sampling and analysis of phthalates in house dust and in solvent wipe samples of surfaces by means of gas chromatography-mass spectrometry (GC-MS).

Two alternative sampling, sample preparation and sample introduction methods, whose comparability has been proven in an interlaboratory test, are specified for indoor air[1].[1].[1]:

— —sorbent tubes sampling with subsequent thermal desorption GC-MS, and

— —sampling by adsorption and subsequent solvent extraction and injection to GC-MS.

FurtherAdditional adsorbents, which may that can be used, are described in Annex BAnnex B.

Depending on the sampling method, the compounds dimethyl phthalate to diisoundecylphthalate can be analysed in house dust as described in Annex D[2]Annex D.[2]. The investigation of house dust samples is only appropriate as a screening method. This investigation only results in indicative values and is not acceptable for a final assessment of a potential need for action.

Dimethyl phthalate to diisoundecylphthalate can be analysed in solvent wipe samples as described in Annex CAnnex C. Solvent wipe samples are suitable for non-quantitative source identification.

NOTE In principle, the method is also suitable for the analysis of other phthalates, adipates and cyclohexane dicarboxylic acid esters, but this is confirmed by determination of the performance characteristics in each case.

General information on phthalates are given in Annex AAnnex A.

iTeh Standards (https://standards.iteh.ai) Document Preview
ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

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 16000- 6:2021, *Indoor air — Part 6: Determination of organic compounds (VVOC, VOC, SVOC) in indoor and test chamber air by active sampling on sorbent tubes, thermal desorption and gas chromatography using MS or MS FID*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

© ISO 2024 – All rights reserved

4 Abbreviated terms

For the purpose of this document, the following abbreviated terms apply:.

Dimethyl phthalate	DMP
Diethyl phthalate	DEP
Di-*n*-propyl phthalate	DPPbenzyl-*n*-butyl phthalate BBP
Diisobutyl phthalate	DiBPdiallyl phthalate DAIP
Di-*n*-butyl phthalate	DBPdi-*n*-butyl phthalate DBP
Benzyl-*n*-butyl phthalate	BBP
Dicyclohexyl phthalate	DCHPdicyclohexyl phthalate DCHP
Di(2-ethyl hexyl) phthalate	DEHPdi(2-ethyl hexyl) phthalate DEHP
Di(*n*-octyl) phthalate	DOPdiethyl phthalate DEP
Diphenyl phthalate	DPhPdiisobutyl phthalate DiBP
Diisononylphthalate	DiNP
Diisodecylphthalate	DiDPdiisodecylphthalate DiDP
DiNP	diisononylphthalate
Diisoundecyl phthalate	DiUPdiisoundecyl phthalate DiUP
D_4-Dimethyl phthalate	D_4-DMPdimethyl phthalate DMP
DOP	di(*n*-octyl) phthalate
DPhP	diphenyl phthalate
DPP	di-*n*-propyl phthalate
D_4-Diethyl phthalate	D_4-DEPD_4-benzyl-*n*-butyl phthalate BBP

iTeh Standards (https://standards.iteh.ai) Document Preview
ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

D_4-~~Di-*n*-butyl phthalate~~DBP	D_4-~~DBP~~di-*n*-butyl phthalate
~~D_4-Benzyl-*n*-butyl phthalate~~D_4-DEP	D_4-~~BBP~~diethyl phthalate
~~D_4-Di(2-ethyl hexyl) phthalate~~D_4-DEHP	~~D_4-DEHP~~D_4-di(2-ethyl hexyl) phthalate
D_4-DMP	D_4-dimethyl phthalate
D_4-~~Di(*n*-octyl) phthalate~~DOP	D_4-~~DOP~~di(*n*-octyl) phthalate
~~Diallylphthalate~~GC	~~DAIP~~gas chromatographic
~~4-Nonylphenol~~IS	~~4-NP~~internal standard
~~Limit of quantification~~LOQ	~~LOQ~~limit of quantification
MS	mass spectrometry
ODS	octadecyl silica
PTFE	polytetrafluoroethylene
SDB	styrene-divinylbenzene
SIM	selected ion monitoring
SVOC	semi-volatile organic compounds
TBME	tertiary butyl methyl ether
TDS	thermal desorption system
4-NP	4-nonylphenol

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

© ISO 2024 – All rights reserved

5 Sampling methods and analytical apparatus

5.1 General

Sampling of indoor air takes place either by adsorption on a thermal desorption tube filled with quartz wool and Tenax® TA[1] or on adsorbents such as Florisil®[2], octadecyl silica (ODS) and styrene–divinylbenzene (SDB) copolymer with subsequent solvent extraction.[1],[3],[22] The quantity of solvent used for solvent extraction procedures should be minimized in order to minimize blank values. All apparatus and reagents used should be clean, i.e. without detectable quantities of the compounds of interest.

The experiences from the round robininterlaboratory test have indicated that significant blank value differences can also be introduced by the solvent. Each new bottle of solvent shall therefore be tested for phthalate contamination before use.

NOTE The experiences from the round robininterlaboratory test have indicated that rinsing with clean solvent (no detectable phthalates) is sufficient to remove contamination from the apparatus and that a sterilization by heating with subsequent deactivation of the heated glass apparatus is not mandatory.

The ubiquitous distribution of phthalates shall be considered during sampling of indoor air in order to avoid contamination of the sample. The measures to be considered for blank value minimization, as well as the advantages and disadvantages of the individual methods, are described in detail in 5.3.3, Clause B.2, Clause D.6 and Annex Iclause 5.3.3 and Annexes B.2, D.6 and I. Further hints to quality assurance and problems related to blank values that shall be considered are listed in Clause 11Clause 11.

5.2 Sampling by adsorption with subsequent thermal desorption

5.2.1 Apparatus, operating materials and chemicals

Use the apparatus, reagents and materials described in ISO 16000-6 with the following specific requirements.

5.2.1.1 Thermal desorption tube, stainless steel, inert-coated steel or glass tube filled with a 1 cm loosely packed plug of non-friable quartz wool backed up by a minimum of 50 mg of Tenax® TA (see ISO 16000-6).

5.2.1.2 Sampling system, in accordance with Figure 1Figure 1.

5.2.1.3 Pump, suitable for a volume flow in the range 50 ml/min to 200 ml/min under sampling conditions; recommended sampling volume of approximately 20 l to approximately 70 l.

5.2.1.4 Gas volume meter, the maximal measurement inaccuracy shall not exceed 5 %.

5.2.1.5 Laboratory sampling facilities, hygrometer, thermometer, barometer.

5.2.1.6 Internal standards, required as quality control measure of the whole analytical process including sampling; suitable examples include: the ring-deuterated compounds D_4-DMP, D_4-DEP, D_4-DBP, D_4-BBP, D_4-DEHP, D_4-DOP as well as the non-deuterated diallyl phthalate (DAIP), see Clause 6Clause 6 and Table 3Table 3. Standards shall be prepared in phthalate-free methanol, as described in ISO 16000-6, at a level such that a maximum 1 µl injection introduces approximately the same mass of analyte onto the sampling end of the tubes as is expected to be collected during sampling.

[1] Tenax® TA is the trade name of a product supplied by Buchem. This information is given for the convenience of the users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

[2] Florisil® is the trade name of product supplied by US Silica. This information is given for the convenience of the users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

© ISO 2024 – All rights reserved

5.2.1.7 Thermal desorption system, coupled to GC-MS for two-stage thermal desorption of the sorbent tubes. Transfer of desorbed vapours via a carrier gas flow into a ~~gas chromatographic (~~GC~~)~~ system, fitted with a ~~mass spectrometric (~~MS~~)~~ detector.

NOTE Deactivated (silanised) glass wool or quartz wool can also be used as adsorbent after an appropriate method validation.

5.2.2 Preparation of the thermal desorption tube

The use of a tube packed with quartz wool and Tenax® TA assumes knowledge of ISO 16000-6. Prepacked and preconditioned sorbent tubes are available commercially or can be prepared in the laboratory as follows~~:~~.

A plug of non-friable quartz wool, usually supported by a stainless steel mesh, is inserted at the sampling end of the tube. The required mass of sorbent is poured into the tube behind the quartz wool plug. The far end of the sorbent bed is typically supported by a second plug of quartz wool or a stainless steel mesh.

A minimum of 50 mg of Tenax® TA shall be used per tube in order to guarantee the sorption capacity.

NOTE Determination of the breakthrough volume is described in ISO 16017-1~~,~~:2000, Annex B. The breakthrough volumes are proportional to the dimensions and masses of the sorbents. The rule of the thumb is that the guaranteed sample volume doubles itself when the sorbent bed length is doubled (while retaining the tube diameter).

After filling of the thermal desorption tubes (e.g. with Tenax® TA), the tubes are conditioned for approximately 8 h at 280 °C followed by approximately 30 min at 300 °C in an inert gas flow (100 ml/min). The purified sorption tubes are closed and stored at room temperature and in the dark in a container that prevents sample contamination.

Analyse a representative number of conditioned tubes for blank value, using routine analytical parameters, to ensure that thermal desorption blank is sufficiently small (see ISO 16000-6:2021, Clause 9).

Sampling should take place as soon as possible after conditioning. If sampling is not possible within approximately 14 d after conditioning, then the tube shall be reconditioned for 15 min at approximately 300 °C before sampling. Cotton gloves can be used to ~~minimise~~minimize the risk of contamination of the sorbent tubes. In addition, labelling shall be omitted.

The thermal desorption device should ensure that any contamination from external tube surfaces is excluded from the analytical sample flow path.

Tubes should be individually identifiable via etched barcodes. No adhesive labels or writing on the tube is allowed.

5.2.3 Sampling

Prior to sampling, the conditioned tubes are spiked with a maximum of 1 µl of internal standard solution in methanol (e.g. 20 ng/µl for a sampling volume of 50 l; the absolute mass of the additionally spiked standard depends on the sampling volume and the operating range of the method). The standard solution is usually applied on the sampling end of the sorbent tube.

The sampling equipment is assembled according to Figure 1~~Figure 1 and~~. The sampling equipment shall be free of leaks. The pump is connected to the non-sampling end of the sorbent tube by means of polyethylene or ~~polytetrafluoroethylene (~~PTFE~~)~~ connectors and is switched on. If the breakthrough volume of the analysed phthalates is unknown, then two sorption tubes shall be connected in series. The tubes shall be connected with a phthalate-free coupling.

The volume flow, as well as the temperature, the absolute air pressure and the relative air humidity, shall be recorded. The suitable sampling volume flows are within the range of 50 ml/min to 200 ml/min. This corresponds to a recommended sampling volume of approximately 20 l to 70 l for a sampling duration of approximately 2 h to 24 h. After sampling, the sorption tube is removed from the sampling equipment; both ends of the sorption tube shall be closed.

© ISO 2024 – All rights reserved

A duplicate sampling of the indoor air is recommended.

Sampled tubes shall be transported to the laboratory and analysed as soon as possible.

5.3 Sampling by adsorption and subsequent solvent extraction

5.3.1 Apparatus, operating materials and chemicals

5.3.1.1 Sampling system, in accordance with Figure 1~~Figure 1.~~

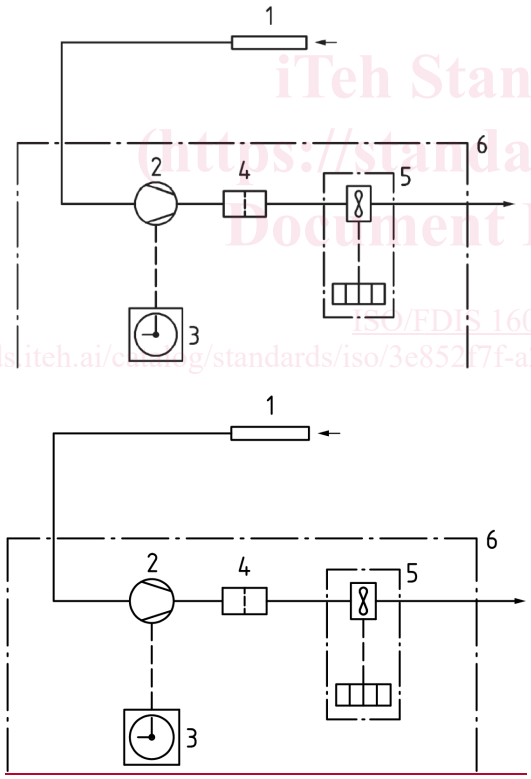
5.3.1.2 Pump, suitable for a volume of approximately 2 l/min under the conditions of the sampling, recommended sampling volume of approximately 1 m^3 to 3 m^3 in 8 h to 24 h.

5.3.1.3 Gas volume meter, the maximal measurement inaccuracy shall not exceed 5 %.

5.3.1.4 Muffle furnace.

5.3.1.5 Flat, heat resistant evaporating dish, for heating Florisil®.

5.3.1.6 Florisil®, 60 to 100 mesh.



iTeh Standards
(https://standards.iteh.ai)
Document Preview
ISO/FDIS 16000-33
https://standards.iteh.ai/catalog/standards/iso/3e852f7f-a2ce-47a7-a1d0-2d576d3691c7/iso-fdis-16000-33

Key

~~1~~ ~~sampling tube~~

~~2~~ ~~membrane vacuum pump~~

© ISO 2024 – All rights reserved