
**Ambient air — Determination of
asbestos fibres — Indirect-transfer
transmission electron microscopy
method**

*Air ambiant — Dosage des fibres d'amiante — Méthode par
microscopie électronique à transmission par transfert indirect*

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

ISO 13794:2019

<https://standards.iteh.ai/catalog/standards/iso/21d11543-ab95-471f-b8bf-67534bbd52f3/iso-13794-2019>



iTeh Standards
(<https://standards.itih.ai>)
Document Preview

ISO 13794:2019

<https://standards.itih.ai/catalog/standards/iso/21d11543-ab95-471f-b8bf-67534bbd52f3/iso-13794-2019>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

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
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword.....	vi
Introduction.....	vii
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Symbols and abbreviated terms.....	7
5 Type of sample.....	8
6 Range.....	8
7 Limit of detection.....	8
8 Principle.....	8
9 Reagents.....	9
10 Apparatus.....	10
10.1 Air sampling.....	10
10.1.1 Filter cassette.....	10
10.1.2 Sampling pump.....	10
10.1.3 Stand.....	10
10.1.4 Personal sampling.....	10
10.1.5 Flowmeter.....	10
10.2 Specimen preparation laboratory.....	11
10.3 Equipment for analysis.....	11
10.3.1 Transmission electron microscope.....	11
10.3.2 Energy dispersive X-ray analyser.....	13
10.3.3 Plasma asher.....	13
10.3.4 Vacuum coating unit.....	13
10.3.5 Sputter coater.....	13
10.3.6 Beakers.....	13
10.3.7 Vacuum source.....	13
10.3.8 Glass filtration apparatus.....	14
10.3.9 Solvent washer (Jaffe washer).....	14
10.3.10 Condensation washer.....	15
10.3.11 Slide warmer or oven.....	16
10.3.12 Ultrasonic bath.....	16
10.3.13 Carbon grating replica.....	16
10.3.14 Calibration specimen grids for EDXA.....	16
10.3.15 Carbon rod sharpener.....	17
10.3.16 Disposable tip micropipettes.....	17
10.3.17 Thermometer.....	17
10.3.18 Stopwatch.....	17
10.4 Consumable supplies.....	17
10.4.1 Copper or nickel electron microscope grids.....	17
10.4.2 Gold or nickel electron microscope grids.....	17
10.4.3 Aluminium foil.....	17
10.4.4 Carbon rod electrodes.....	17
10.4.5 Routine electron microscopy tools and supplies.....	18
10.4.6 Reference asbestos samples.....	18
10.4.7 Reference samples of mineral fibres other than asbestos.....	18
11 Air sample collection.....	18
11.1 Calculation of analytical sensitivity.....	18
11.2 Sample collection procedure.....	19

12	Procedure for analysis	20
12.1	General	20
12.2	Cleaning of sample cassettes	20
12.3	Preparation of analytical filters	20
12.3.1	Selection of filter area for ashing	20
12.3.2	Ashing of sample collection filters	20
12.3.3	Aqueous dispersal of residual ash from sample collection filters	21
12.3.4	Assembly of system for filtration of aqueous dispersions	21
12.3.5	Filtration of aqueous dispersions	21
12.4	Preparation of TEM specimens from PC analytical filters	22
12.4.1	Selection of filter area for carbon-coating	22
12.4.2	Carbon-coating of filter portions	22
12.4.3	Preparation of the Jaffe washer	23
12.4.4	Placing of specimens into the Jaffe washer	23
12.5	Preparation of TEM specimens from cellulose ester analytical filters	23
12.5.1	Selection of area of filter for preparation	23
12.5.2	Preparation of solution for collapsing cellulose ester filters	23
12.5.3	Filter-collapsing procedure	23
12.5.4	Plasma etching of the filter surface	23
12.5.5	Carbon-coating	24
12.5.6	Preparation of the Jaffe washer	24
12.5.7	Placing of specimens in the Jaffe washer	24
12.6	Criteria for acceptable TEM specimen grids	24
12.7	Procedure for structure counting by TEM	25
12.7.1	General	25
12.7.2	Measurement of mean grid opening area	25
12.7.3	TEM alignment and calibration procedures	26
12.7.4	Determination of criterion for termination of TEM examination	26
12.7.5	General procedure for structure counting and size analysis	26
12.7.6	Estimation of mass concentration of asbestos fibres and bundles	27
12.7.7	Magnification requirements	27
12.8	Blank and quality control determinations	29
12.9	Calculation of results	30
13	Performance characteristics	30
13.1	General	30
13.2	Interferences and limitations of fibre identification	30
13.3	Precision and accuracy	31
13.3.1	Precision	31
13.3.2	Accuracy	31
13.3.3	Inter- and intra-laboratory analyses	31
13.4	Limit of detection	32
14	Test report	32
Annex A (normative)	Determination of operating conditions for plasma asher	36
Annex B (normative)	Determination and standardization of operating conditions for ultrasonic bath	37
Annex C (normative)	Calibration procedures	39
Annex D (normative)	Structure counting criteria	42
Annex E (normative)	Fibre identification procedure	52
Annex F (normative)	Determination of the concentration of asbestos fibres and bundles longer than 5 µm and PCM equivalent asbestos fibres	68
Annex G (normative)	Calculation of results	69
Annex H (normative)	Test procedure to determine suitability of cellulose ester sample collection filters	76

Annex I (informative) Strategies for collection of air samples	77
Bibliography	78

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

[ISO 13794:2019](https://standards.iteh.ai/catalog/standards/iso/21d11543-ab95-471f-b8bf-67534bbd52f3/iso-13794-2019)

<https://standards.iteh.ai/catalog/standards/iso/21d11543-ab95-471f-b8bf-67534bbd52f3/iso-13794-2019>

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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

This second edition cancels and replaces the first edition (ISO 13794:1999), which has been technically revised. The main changes compared to the previous edition are as follows:

- the use of electronic display systems with measurement software is permitted;
- the maximum particulate loading for TEM specimens is increased from 10 % to 25 %;
- a simplified fibre identification procedure for investigation of known sources of the regulated asbestos varieties and richterite/winchite asbestos is permitted;
- the reporting requirements have been changed to permit reporting of the concentrations of fibres and bundles longer than 5 µm and/or the concentrations of PCM equivalent fibres without the requirement to report the concentrations of structures equal to or greater than 0,5 µm;
- there is no requirement to report the 95 % confidence intervals of the fibre concentrations.

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.

Introduction

This document is applicable to the measurement of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for a detailed evaluation of any atmosphere. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre size and type are the relevant parameters for evaluation of the inhalation hazards, a fibre counting and measuring technique is the only logical approach. Most fibres in ambient atmospheres are not asbestos, and therefore there is a requirement for fibres to be identified. Many airborne asbestos fibres in ambient atmospheres have diameters below the resolution limit of the optical microscope. This document is based on transmission electron microscopy, which has adequate resolution to allow for the detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis becomes prohibitively expensive. Because of instrumental deficiencies or because of the nature of the particulate, some fibres cannot be positively identified as asbestos, even though the measurements all indicate that they could be asbestos. Subjective and instrumental factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of asbestos fibres is required.

In addition to single fibres and bundles, asbestos is often found in air samples as very complex, aggregated structures, which may or may not be also aggregated with other particles. The number of asbestos fibres and bundles incorporated in these complex structures often exceeds the number of isolated fibres and bundles observed, and many of them may be completely obscured in direct-transfer transmission electron microscope (TEM) preparations. The method defined in this document incorporates specimen preparation procedures that result in the selective concentration of asbestos fibres and the removal of organic, water-soluble and acid-soluble materials. These procedures have the effect of dispersing the majority of the complex clusters and aggregates of fibres into their component fibres and bundles so that the asbestos in the air sample can be more accurately quantified. All of the feasible specimen preparation techniques result in some modification of the airborne particulate. Even the collection of particles from a three-dimensional airborne dispersion on to a two-dimensional filter surface can be considered a modification of the particulate, and some of the particles in most samples are modified by the specimen preparation procedures. Although this method results in dispersal of complex clusters and aggregates, it minimizes other effects on the size distribution of fibres and fibre bundles.

This document requires a very detailed and logical procedure is used to reduce the subjective aspects of the measurement. The method of data recording specified in the document is designed to allow re-evaluation of the fibre counting data as new medical evidence becomes available.

This document describes the method of analysis for a single air filter. However, one of the largest potential errors in characterizing asbestos in ambient atmospheres is associated with the variability between filter samples. For this reason, it is necessary to design a replicate sampling scheme in order to determine the standard's accuracy and precision.

Comparison of results obtained using this indirect-transfer procedure with those from the direct-transfer procedure cannot be done a priori. This can only be achieved by a site-specific inter-comparison study that takes into account the fibre size and type of asbestos, and also the nature of the source of the airborne asbestos.