

# SLOVENSKI STANDARD oSIST ISO/DIS 10312:2018

01-september-2018

Zunanji zrak - Določevanje azbestnih vlaken - Metoda transmisijske elektronske mikroskopije z neposrednim prenosom

Ambient air - Determination of asbestos fibres - Direct transfer transmission electron microscopy method

iTeh STANDARD PREVIEW (standards.iteh.ai)

Air ambiant - Détermination des fibres d'amiante - Méthode de microscopie électronique à transmission directe

Ta slovenski standard je istoveten z: ISO/DIS 10312

ICS:

13.040.20 Kakovost okoljskega zraka Ambient atmospheres

oSIST ISO/DIS 10312:2018 en

oSIST ISO/DIS 10312:2018

# iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 10312:2019

https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019

# DRAFT INTERNATIONAL STANDARD ISO/DIS 10312

ISO/TC **146**/SC **3** Secretariat: **ANSI** 

Voting begins on: Voting terminates on:

2018-05-25 2018-08-17

# Ambient air — Determination of asbestos fibres — Direct transfer transmission electron microscopy method

Air ambiant — Détermination des fibres d'amiante — Méthode de microscopie électronique à transmission directe

ICS: 13.040.20

# iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 10312:2019
https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

This document is circulated as received from the committee secretariat.



Reference number ISO/DIS 10312:2018(E)

# iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 10312:2019
https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019



# COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

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

Cont	ents	Page
Forew	ord	vii
Introd	uction	viii
1	Scope	1
2	Normative references	
3	Terms and definitions	
4	Symbols and abbreviated terms	6
5	Type of sample	6
6	Range	6
7	Limit of detection	7
8	Principle	
	•	
9 9.1	ReagentsWater, fibre-free	
9.1 9.2	Chloroform	
9.3	1-Methyl-2-pyrrolidone	
9.4	1,2-Diaminoethane (Ethylene diamine)	
9.5	Dimethylformamide	8
9.6	Glacial acetic acid	
9.7	Acetone	
10	Apparatus	0
10.1	Air sampling – equipment and consumable supplies	
	Filter cassette	
10.1.1	Sampling pump	h7-4371-h2h4-
	Stand 6664ce22578d/sist-iso-10312-2019	
	Personal Sampling	
	Flowmeter	
10.1.3	Specimen preparation laboratory	
10.2	Equipment for analysis	
	Transmission electron microscope	
	Energy dispersive X-ray analyzer	
	Plasma asher	
	Vacuum coating unit	
	Sputter coater	
	Solvent washer (Jaffe washer)	
	Condensation washer	
	Slide warmer or oven	
	Ultrasonic bath	
	)	
	replica	0 0
10.3.1	L	
	EDXA	
10.3.12	2	Carbon rod
	sharpener	
10.3.13	3	Disposable tip
	micropipettes	
104	Consumable supplies	15

10.4.1	Copper or nickel electron microscope grids	15
10.4.2	Gold or nickel electron microscope grids	15
	Carbon rod electrodes	
10.4.4	Routine electron microscopy tools and supplies	16
	Reference asbestos samples	
	Reference samples of mineral fibres other than asbestos	
	•	
11	Air sample collection	16
12	Procedure for analysis	18
12.1	General	
12.2	Cleaning of sample cassettes	
12.3	Direct preparation of TEM specimens from polycarbonate filters	
12.3.1	Selection of filter area for carbon coating	
	Carbon coating of filter portions	
	Preparation of the Jaffe washer	
	Placing of specimens in the Jaffe washer	
	Direct preparation of TEM specimens from cellulose ester filters	
	Selection of area of filter for preparation	
	Preparation of solution for collapsing cellulose ester filters	
	Filter collapsing procedure	
	Plasma etching of the filter surfaces	
	Carbon coating	
	Preparation of the Jaffe washer	
	Placing of specimens in the Jaffe washer	
	Criteria for acceptable TEM specimen grids	
	Procedure for structure counting by TEM	
	General	
	Measurement of mean opening area	
	TEM alignment and calibration procedures	
	Determination of stopping point	
	General procedure for structure counting and size analysis	
	Magnification requirements	
	Blank and quality control determinations	
12.8	Calculation of results	26
13	Performance characteristics	26
13.1	General	
13.2	Interferences and limitations of fibre identification	
13.2	Precision and accuracy	
	Precision	
	Accuracy	
	Inter-laboratory and intra-laboratory analyses	
13.3.3 13.4	Limit of detection	
13.4		
14	Test report	28
Annov	A (normative) Determination of operating conditions for plasma asher	22
Ailliex A.1	General	
A.1 A.2	Procedure	
Annex	B (normative) Calibration procedures	33
<b>B.1</b>	Calibration of the TEM	33
B.1.1	Calibration of TEM screen magnification	33
<b>B.1.2</b>	Calibration of ED camera constant	33
<b>B.2</b>	Calibration of the EDXA system	33

Annex	c C (normative) Structure counting criteria	36
<b>C.1</b>	General	36
<b>C.2</b>	Structure definitions and treatment	36
C.2.1	Fibre	36
<b>C.2.2</b>	Bundle	
C.2.3	Cluster	37
C.2.4	Matrix	
C.2.5	Asbestos structure larger than 5 µm	
C.2.6	Asbestos fibre or bundle longer than 5 μm	
C.2.7	PCM equivalent structure	
C.2.8	PCM equivalent fibre	
<b>C.3</b>	Other structure counting criteria	
C.3.1	Structures which intersect grid bars	
C.3.2	Fibres which extend outside the field of view	
<b>C.4</b>	Procedure for data recording	
C.4.1	General	
C.4.2	Fibres	
C.4.3	Bundles	
C.4.4	Disperse clusters (type D)	
C.4.5	Compact clusters (type C)	
C.4.6	Disperse matrices (type D)	
C.4.7	Compact matrices (type C)	
C.4.8	Procedure for recording of partially obscured fibres and bundles	
C.5	Special considerations for counting of PCM equivalent fibres	
	D (normative) Fibre identification procedure	
D.1	General	
D.2	ED and EDXA techniques	
D.2.1	General SIST SO 10312:2019	
D.2.2	ED techniques and site hai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-	
D.2.3	EDXA measurements 66446225784/sist-iso-10312-2019	
D.3	Interpretation of fibre analysis data	
D.3.1	Chrysotile	
D.3.2	Amphiboles	
D.4	Fibre classification categories	
D.4.1	Procedure for classification of fibres with tubular morphology suspected to be	
	chrysotile	51
D.4.2	Procedure for classification of fibres without tubular morphology, suspected to be	
_	amphibole	53
D.5	Routine fibre identification for investigation of known sources of the regulated	
	asbestos varieties	56
Annex	E (normative) Determination of the concentration of asbestos fibres and bundles	
	longer than 5 µm, and PCM equivalent asbestos fibres	63
	• • •	
	x F (normative) Calculation of results	
F.1	General	
F.2	Test for uniformity of distribution of fibrous structures on TEM grids	
F.3	Calculation of the analytical sensitivity	
F.4	Calculation of the mean and confidence interval of the structure concentration	
F.4.1	Calculation of the mean structure concentration	
F.4.2	Calculation of confidence intervals	
F.4.3	Example of calculation of Poissonian 95 % confidence intervals	
F.4.4	Example of calculation of Gaussian 95 % confidence intervals	66

F.4.5	Summary of procedure for calculation of results	66
F.5	Calculation of structure length, width, and aspect ratio distributions	67
F.5.1	General	67
F.5.2	Calculation of structure length cumulative number distribution	67
F.5.3	Calculation of structure width cumulative number distribution	67
F.5.4	Calculation of structure aspect ratio cumulative number distribution	68
Anne	x G (informative) Strategies for collection of air samples	70
G.1	General	70
<b>G.2</b>	Air sample collection in the outdoors environment	70
<b>G.3</b>	Air sample collection inside buildings	70
Anne	x H (informative) Methods for removal of gypsum fibres fibres	71
Biblic	ography	72

# iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>SIST ISO 10312:2019</u> https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019

# **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 on 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.

The committee responsible for this document is ISO/XXX 0e224c3e-1fb7-4371-b2b4-

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

ISO XXXX consists of the following parts. [Add information as necessary.]

# Introduction

This International Standard is applicable to the determination of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos structures are likely to be present. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre sizes are the relevant parameters for evaluation of the inhalation hazards, a fibre counting 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 International Standard is based on transmission electron microscopy, which has adequate resolution to allow detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. Airborne asbestos is often found as a mixture of single fibres and more complex, aggregated structures which may or may not be also aggregated with other particles. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if a sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis would become 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 factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of, asbestos fibres is required. The method specified in this International Standard is designed to provide the best description possible of the nature, numerical concentration, and sizes of asbestos-containing particles found in an air sample. This International Standard requires that a very detailed and logical procedure be used to reduce the subjective aspects of the measurement. The method of data recording specified in this International Standard is designed to allow re-evaluation of the structure counting data as new medical evidence becomes available. 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 onto 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. However, the procedures specified in this International Standard are designed to minimize the disturbance of the collected particulate material, and the effect of those disturbances that do occur can be evaluated.

This International Standard 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 this International Standard's accuracy and precision.

# iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 10312:2019 https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019 oSIST ISO/DIS 10312:2018

# iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 10312:2019
https://standards.iteh.ai/catalog/standards/sist/0e224c3e-1fb7-4371-b2b4-6664ce22578d/sist-iso-10312-2019

# Ambient air — Determination of asbestos fibres — Directtransfer transmission electron microscopy method

# 1 Scope

This International Standard specifies a reference method using transmission electron microscopy for the determination of airborne asbestos fibres and structures in in a wide range of ambient air situations, including the interior atmospheres of buildings, and for a detailed evaluation for asbestos structures in any atmosphere. The method allows determination of the type(s) of asbestos fibres present and also includes measurement of the lengths, widths and aspect ratios of the asbestos structures. The method cannot discriminate between individual fibres of asbestos and elongate fragments from non-asbestos analogues of the same amphibole mineral.

# 2 Normative references

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

ISO 4225:1994, Air Quality — General aspects — Vocabulary

ISO 4226:2007, Air quality — General aspects — Units of measurement

ISO 13794:1999, Ambient air — Determination of asbestos fibres — Indirect-transfer transmission electron microscopy method

# 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply (see also ISO 4225).

## 3.1

### acicular

shape shown by an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like. [SOURCE: ISO 10312:1995, definition 3.1]

# 3,2

# amphibole

group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:

 $A_{0\text{-}1}B_{2}C_{5}T_{8}O_{22}\text{(OH,F,Cl)}_{2}$ 

where: A = K, Na;

 $B = Fe^{2+}$ , Mn, Mg, Ca, Na;

 $C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};$ 

T = Si, Al, Cr,  $Fe^{3+}$ , Ti.

[SOURCE: ISO 10312:1995, definition 3.2

NOTE In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about  $56^{\circ}$  and  $124^{\circ}$ .

## 3.3

# amphibole asbestos

amphibole in an asbestiform habit. [SOURCE: ISO 10312:1995, definition 3.3]

#### 3.4

# analytical sensitivity

calculated airborne asbestos structure concentration in structures/litre, equivalent to counting of one asbestos structure in the analysis. [SOURCE: ISO 10312:1995, definition 3.4]

NOTE 1 It is expressed in structures/litre.

NOTE 2 This method does not specify a unique analytical sensitivity. The analytical sensitivity is determined by the needs of the measurement and the conditions found on the prepared sample.

### 3.5

# asbestiform

specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility. [SOURCE: ISO 10312:1995, definition 3.5]

# 3.6

### asbestos

SIST ISO 10312:2019

term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, flexible, strong fibres when crushed or processed. [SOURCE: ISO 10312:1995, definition 3.6]

NOTE The Chemical Abstracts Service Registry Numbers of the **most common** asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grunerite asbestos (Amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4). Other varieties of asbestiform amphibole, such as richterite asbestos and winchite asbestos [12], are also found in some products such as vermiculite and talc.

# 3.7

#### asbestos structure

term applied to an individual fibre, or any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles. [SOURCE: ISO 10312:1995, definition 3.7]

#### 3.8

# aspect ratio

ratio of length to width of a particle. [SOURCE: ISO 10312:1995, definition 3.8]

# 3.9

# blank

structure count made on TEM specimens prepared from an unused filter, to determine the background measurement. [SOURCE: ISO 10312:1995, definition 3.9]

# 3.10

# camera length

equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action. [SOURCE: ISO 10312:1995, definition 3.10]

# 3.11

# chrysotile

fibrous mineral of the serpentine group which has the nominal composition:  $Mg_3Si_2O_5(OH)_4$  [SOURCE: ISO 10312:1995, definition 3.11]

NOTE Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by  $Al^{3+}$  may occur. Minor substitution of magnesium by  $Al^{3+}$ ,  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Ni^{2+}$ ,  $Mn^{2+}$  and  $Co^{2+}$  may also be present. Chrysotile is the most prevalent type of asbestos.

# 3.12

# cleavage

breaking of a mineral along one of its crystallographic directions. [SOURCE: ISO 10312:1995, definition 3.12]

#### 3.13

# cleavage fragment

fragment of a crystal that is bounded by cleavage faces. [SOURCE: ISO 10312:1995, definition 3.13]

NOTE Crushing of non-asbestiform amphibole generally yields elongated fragments that conform to the definition of a fibre, but rarely have aspect ratios exceeding 30:1.

# 3.14

# cluster

structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping. [SOURCE: ISO 10312:1995, definition 3.14]

## 3.15

# d-spacing

The distance between identical adjacent and parallel planes of atoms in a crystal. [SOURCE: ISO 10312:1995, definition 3.15]

# 3.16

# electron diffraction

A technique in electron microscopy by which the crystal structure of a specimen is examined. [SOURCE: ISO 10312:1995, definition 3.16]

# 3.17

# electron scattering power

The extent to which a thin layer of substance scatters electrons from their original directions. [SOURCE: ISO 10312:1995, definition 3.17]

# 3.18

## energy dispersive X-ray analysis

Measurement of the energies and intensities of X-rays by use of a solid state detector and multi-channel analyzer system. [SOURCE: ISO 10312:1995, definition 3.18]