SLOVENSKI STANDARD
SIST ISO 16000-27:2015

01-april-2015

Notranji zrak – 27. del: Določevanje usedlih vlaken prahu na površinah z metodo štetja z elektronskim mikroskopom (direktna metoda)

Indoor air - Part 27: Determination of settled fibrous dust on surfaces by SEM (scanning electron microscopy) (direct method)

Air intérieur - Partie 27: Détermination de la poussière fibreuse déposée sur les surfaces par MEB (microscopie électronique à balayage) (méthode directe)

Ta slovenski standard je istoveten z: **ISO 16000-27:2014**

ICS:

13.040.20 Kakovost okoljskega zraka Ambient atmospheres

SIST ISO 16000-27:2015 **en,fr**

INTERNATIONAL STANDARD

ISO 16000-27

First edition
2014-06-01

Indoor air —

Part 27: Determination of settled fibrous dust on surfaces by SEM (scanning electron microscopy) (direct method)

Air intérieur —

Partie 27: Détermination de la poussière fibreuse déposée sur les surfaces par MEB (microscopie électronique à balayage) (méthode directe)

Reference number
ISO 16000-27:2014(E)

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Published in Switzerland

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is 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 in indoor air and test chamber air — Active sampling method*

— *Part 4: Determination of formaldehyde — Diffusive sampling method*

— *Part 5: Sampling strategy for volatile organic compounds (VOCs)*

— *Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS or MS-FID*

— *Part 7: Sampling strategy for determination of airborne asbestos fibre concentrations*

— *Part 8: Determination of local mean ages of air in buildings for characterizing ventilation conditions*

— *Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*

— *Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*

— *Part 11: Determination of the emission of volatile organic compounds from building products and furnishing — Sampling, storage of samples and preparation of test specimens*

— *Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)*

— *Part 13: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) — Collection on sorbent-backed filters*

— *Part 14: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDDs/PCDFs) — Extraction, clean-up and analysis by high-resolution gas chromatography and mass spectrometry*

— *Part 15: Sampling strategy for nitrogen dioxide (NO_2)*

— *Part 16: Detection and enumeration of moulds — Sampling by filtration*

— *Part 17: Detection and enumeration of moulds — Culture based method*

— *Part 18: Detection and enumeration of moulds — Sampling by impaction*

— *Part 19: Sampling strategy for moulds*

— *Part 20: Detection and enumeration of moulds — Determination of total spore count*

— *Part 21: Detection and enumeration of moulds — Sampling from materials*

— *Part 23: Performance test for evaluating the reduction of formaldehyde concentrations by sorptive building materials*

— *Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials*

— *Part 25: Determination of the emission of semi-volatile organic compounds by building products — Micro-chamber method*

— *Part 26: Sampling strategy for carbon dioxide (CO_2)*

— *Part 27: Determination of settled fibrous dust on surfaces by (SEM) scanning electron microscopy (direct method)*

— *Part 28: Determination of odour emissions from building products using test chambers*

— *Part 29: Test methods for VOC detectors*

— *Part 30: Sensory testing of indoor air*

— *Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid esters*

— *Part 32: Investigation of buildings for pollutants and other injurious factors — Inspection*

The following parts are under preparation:

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

— *Part 34: Strategies for the measurement of airborne particles (PM 2,5 fraction)*

— *Part 35: Measurement of polybrominated diphenylether, hexabromocyclododecane and hexabromobenzene*

— *Part 36: Test method for the reduction rate of airborne bacteria by air purifiers using a test chamber*

Introduction

Standardized ISO methods for measuring asbestos exposure levels using different analytical methods are available and widely used (ISO 10312, ISO 13794, ISO 14966). Standardized methods (ISO 22262-1) determining the asbestos content in bulk materials (products, etc.) are also established. This International Standard is based on the procedures described in VDI 3877 Part 1[6] and closes the remaining gap in describing a method for measuring asbestos in settled dust on surfaces.

Governmental regulations in many countries exist for asbestos exposure levels and for the asbestos content in products. The asbestos content in settled dust has been the source of widespread discussions. Regulatory efforts based on measurement results are known in only very few cases. The reasons for this have been the lack in many countries of standardized and well accepted measurement methods and the difficult and disputed judgement of the risk potential. A general accepted correlation between the asbestos content and possibly resulting airborne asbestos fibre concentration by re-entrainment of the dust is not established.

A significant difference between direct transfer samples for determining surface contamination and filter samples for air measurement is in the more common appearance of fibrous structures whose dimensions are larger than those of alveolar fibres. The analysis of air samples is performed to determine the concentration of respirable fibres; the analysis of direct transfer dust samples, in contrast, is done more according to the risk (fibre potential) to generate respirable fibres. Surface dust samples are frequently taken in connection with asbestos abatement or other events, where spreading of asbestos containing dust is expected and has to be judged.

The method can also be used for the determination of surface contamination of other fibrous structures like man-made mineral vitreous fibres.

Indoor air —

Part 27: Determination of settled fibrous dust on surfaces by SEM (scanning electron microscopy) (direct method)

1 Scope

This part of ISO 16000 specifies a method giving an index for the numerical concentration of fibrous structures with fibres equal or greater than 0,2 µm in diameter in settled dust on surfaces and their classification into specific substance groups (e.g. chrysotile, amphibole asbestos, other inorganic fibres). It is primarily applicable to indoor areas, but it is also suitable for certain outdoor situations. A sampling technique for collection of settled dust using adhesive tape is described. The method incorporates an analytical method for evaluation of the collected samples by scanning electron microscopy. The result can be specified in asbestos structures per unit area and/or classified into four different loading classes. The analytical sensitivity depends on the area examined and can be as low as 10 structures/cm^2.

For the purpose of this part of ISO 16000, an asbestos or fibrous structure is defined as an asbestos or (other inorganic/organic) fibre-containing particle regardless of its diameter.

The use of the sampling method described is limited, depending on the structure and type of the surface (minor roughness and curvature) and the thickness of dust layer. If the dust layer is too thick, the dust layer can be sampled by other means and eventually analysed as powder sample.[3] [4]

It is assumed that the settled dust has particle diameters mostly below 1 mm.

2 Normative references

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

ISO 22262-1, *Air quality — Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
abatement
activity undertaken to control the potential emission of asbestos fibres from an asbestos-containing building material by removing, enclosing, or encapsulating the material or by repairing damaged material

3.2
ambient sampling
air sampling to determine the airborne asbestos fibre concentration in the immediate vicinity of the building exterior

3.3
analytical sensitivity
calculated asbestos structure concentration, equivalent to counting of one asbestos structure in the analysis

3.4
asbestos
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

Note 1 to entry: 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).

3.5
asbestos (fibrous) structure
term applied to an individual asbestos, other inorganic or organic fibre, or any connected or overlapping grouping of those fibres or bundles of (asbestos) fibres, with or without other particles

3.6
aspect ratio
ratio of length to width of a particle

3.7
blank
unused adhesive tape submitted for analysis as a control

3.8
bundle
structure composed of three or more fibres in a parallel arrangement with the fibres closer than one fibre diameter to each other

3.9
cluster
structure in which two or more fibres, or bundles of fibres, are randomly oriented in a connected grouping

3.10
electron diffraction
technique in electron microscopy in which the crystal structure of a small area of a sample is examined

3.11
energy-dispersive X-ray analysis
determination of elemental composition through measurement of the energies and intensities of X-rays by use of a solid state detector and multi-channel analyser system

3.14
fibre
elongated particle with a length/diameter ratio of more than 3:1 and in this part of ISO 16000, equal or larger than 0,2 µm

3.15
indirect preparation
method in which a sample passes through one or more intermediate steps prior to final filtration; the particles are removed from the original medium and deposited on a second filter prior to analysis

3.16
limit of detection
numerical fibrous structure loading that will not be exceeded at a probability of greater than 95 % by the actual fibrous structure loading, if no asbestos structures are detected during analysis

3.17
magnification
ratio between the size of an object in a microscope image and the actual size of the object

Note 1 to entry: The magnification information refers to the monitor screen upon which the evaluation is performed.

3.18
matrix
structure in which one or more fibres, or bundles of fibres, touch, are attached to, or partially concealed by, a single particle or connected group of non-fibrous particles

3.19
process blank
adhesive tape (that has not been taken into the field) processed in accordance with the entire preparation and analytical procedure

3.20
structure
single fibre, fibre bundle, cluster or matrix

3.21
MMVF
man-made vitreous fibres, also called man-made mineral fibres (MMMF) and synthetic vitreous fibres (SVF), are a group of fibrous, non-crystalline inorganic materials, generally aluminium or calcium silicates, that are derived from rock, clay, slag, and glass

4 Symbols and abbreviations

4.1 Symbols

n	the number of structures counted
λ_U	the lower 95 % confidence limit of a structure count made by either SEM or TEM
λ_O	the upper 95 % confidence limit of a structure count made by either SEM or TEM
α	statistical significance level
B	background level of an X-ray spectrum
D	for a structure count of n, the value of the χ^2 distribution with 2 n degrees of freedom and a significance level of $(1 - \alpha/2)$
E	for a fibre count of x, the value of the $\chi 2$ distribution with $2(x + 1)$ degrees of freedom and a significance level of $\alpha/2$
A	area evaluated on the sample (adhesive tape) by SEM
P	peak height of a peak in the X-ray spectra
S_i	count result of an individual fibrous structure type i
$S_{w,I}$	weighted count result of an individual fibrous structure type i
Z	atomic number

4.2 Abbreviations

ATS	adhesive tape sampling/evaluation by SEM
ED	electron diffraction
EDXA	energy dispersive X-ray analysis
FWHM	half-width of the Mn K_{α} peak of a X-ray detector
PCM	phase contrast optical microscopy
SEM	scanning electron microscopy
TEM	transmission electron microscopy
UTW	ultra-thin window of the X-ray detector
MMVF	man-made vitreous fibres

5 Principle

Dust is collected on an adhesive medium (e.g. tape), which is pressed on to the surface being sampled. The sampling medium, or a piece of it, is prepared as a sample for examination by SEM/EDXA. The sample is examined using SEM without any modification to the collected dust. In the course of this, the fibrous structures are measured according to defined criteria on randomly selected fields of view all over the entire sample, counted, and classified according to substance. EDXA spectra are used to classify fibrous structures into compositional categories. The concentration of the fibrous dust on the surfaces is calculated from the number of counted and classified structures and the analysed sample area. After applying different weighting factors to fibrous structures according to their sizes, fibre loadings are reported as one of four loading categories.

6 Apparatus and material

6.1 Equipment and materials for adhesive tape sampling and preparation

6.1.1 Consumables for sampling.

Sampling medium:

— adhesive tape

  — aluminium or copper tape, acrylic tape (transparent) or adhesive carbon tape/backside aluminium or copper;

— carbon pads

  — diameter: 13 mm or 25 mm;

— sample container, clean, sealable used for transporting the sample into the laboratory.

NOTE Depending upon usage, the carbon pad can be taped directly onto the SEM sample tray.

6.1.2 Routine electron microscopy tools and supplies.

Tweezers, scalpel, or scissors for producing samples of suitable size for SEM, double-coated adhesive tape (carbon) or colloidal carbon paint, SEM specimen stubs, gold, or carbon suitable for coating of the sample in the specific sputter coater or evaporator.

6.1.3 Stereomicroscope, for visual examination of the settled dust in the sample, magnification approximately 20×.

6.1.4 Sputter coater or vacuum evaporator for coating with gold or carbon.

6.2 Equipment and material for analysis

6.2.1 Scanning electron microscope, with an accelerating voltage of at least 20 kV, is required for fibrous structure counting and identification.

6.2.2 SEM equipped with an energy dispersive X-ray analyser, capable of achieving a resolution better than 170 eV (FWHM) on the Mn-K_α peak. The performance of an individual combination of SEM and solid state X-ray detector is dependent on a number of geometrical factors. Accordingly, the required performance of the combination of the SEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a chrysotile fibre of width 0,2 µm, under the operating conditions used during the analysis. Some solid state X-ray detectors are least sensitive in the low energy region, and so detection of sodium in crocidolite is an additional performance criterion. An UTW (ultra-thin or windowless) detector is preferable, but not mandatory unless the analysis is to include identification of fibres with Z ≤ 11. The instrumental combination must satisfy the minimum requirements with regard to the visibility of fibres, as in Annex B.

6.2.3 Resolution test sample. Test sample on which chrysotile fibres with a width ≤0,2 µm have been deposited, is required for adjustment of the operating conditions of the SEM.

6.2.4 Magnification calibration test sample. A test sample is required in order to calibrate the magnification of the SEM. The magnification standard SRM484e (U.S. National Institute of Standards and Technology) is an example of a sample which meets the requirement.

7 Sampling

7.1 Measurement planning

In most countries the estimation of risks due to asbestos fibres is based on the determination of exposure levels. Therefore measurements of asbestos in settled dust can provide only additional information, for example, the success of cleaning efforts or the spread of asbestos contamination. The measurement planning has to be adjusted to the task to be performed. The area of sample examined is small compared to the surface area under investigation, which has to be judged. The sampling plan, including the number and distribution of the sampled areas, should be designed to minimize the statistical uncertainty in the final result. The required precision determines the number of samples. If it is required to compare the asbestos contamination on two different surfaces, statistical tests should be used.

In the measurement planning all available data (such as known sources or the results of air measurements) should be taken into account. This includes all known uses of asbestos-containing materials and the nature of the examined surface.

Furthermore, when planning the measurements, it must be taken into account that thicker dust layers cannot be examined quantitatively as described in 8.2.1 and 8.2.2.1. These might require a different sampling procedure or might need to be collected as powder samples.

The deposition of dust is influenced by a variety of factors. Also the frequency of cleaning of the sampled surface is an important factor. Different influences such as orientation of the surface, air movements in the area and others not mentioned, which might be of importance for the evaluation of the results, shall be considered and, if necessary, recorded in the sampling protocol.