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ISO 16000-27

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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)

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Foreword

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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*

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— *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.

7.2 Measurement objectives and sampling sites

As mentioned above, the measurement objectives are often part of planning and performing asbestos abatement work, but measurements might also be of interest for documenting the status quo. The determination of man-made vitreous fibres in settled dust can also be part of the measurement objective, if in the course of construction, for instance, complaints arise regarding skin irritations which are generally caused by relatively thick fibres. The determination of the inorganic fibre content (asbestos, MMVF) of samples of settled dust principally offers answers to the following questions.

— Are inorganic fibre dusts (asbestos/MMVF) present at certain locations?

— How large is the concentration per surface area of defined fibrous structures?

— How large should the area of containment be?

— What is the size of the area requiring cleaning efforts?

The measurements are also affected by the facility and the planned changes to it. Once defined, the measurement objective determines the selection and number of sampling points. Situations can result where samples are taken not only from horizontal, but also from vertical surfaces. When analysing the results, the difference in character regarding particulate deposits on samples taken horizontally and vertically must be considered. The results cannot be directly compared with each other. Samples taken vertically have usually a lower loading both with fibrous and non-fibrous particles

It should be noted that samples collected by adhesive tape are very small in relation to the areas of the building being examined, so that the samples might not be representative.

Measurement objectives and the relevant sampling sites are summarized in Table 1.

Table 1 — Measurement objectives in the analysis of surface dusts

	Objective	Sampling site	Comments
1	Identification of damages to asbestos-containing products in the case of — improper handling and — incident, e.g. as a result of bad weather or fire.	Surfaces which were cleaned shortly before the event.	If required, analysis of samples from areas not cleaned in advance before remediation is undertaken in the building.
2	Analysis of asbestos fibre contamination, e.g. before demolition, remodelling, in particular concerning work done on areas not belonging to those used for activities.	Surfaces visibly covered with dust taking the settling of dust over time into consideration (during construction, incidents, normal building usage) — under floating screed, — in hollow spaces in walls and niches, — in suspended ceilings, — in installation areas, and — especially on electrical cables, metal surfaces, sheets, and other smooth sedimentation surfaces, where applicable with electrostatic characteristics.	
3	Containment of contaminated areas.	— at staggered distances; — in rooms adjacent to the damaged product.	Air movement must be taken into consideration regarding this.

Table 1 *(continued)*

	Objective	Sampling site	Comments
4	Determination of possible asbestos fibre contamination of products not visibly damaged or asbestos-containing products installed in hidden locations.	In older dust deposits found in uncleaned areas that provide a history of the building; e.g. — behind or on radiators, — in ventilation channels, — on top of cupboards, — on sills, — on support beams, — on machine installations at high locations, and — in reworked break-throughs.	
5	Checking cleanliness of the surfaces of furniture, tools, ventilation systems (negative pressure units, etc. following abatement.	— at a location with indications of residual dust; — at a representative clean location, if required.	
6	Assessment in areas of former asbestos usage, e.g. in factory halls and production sites in which asbestos products were typically used or manufactured.	— old dust deposits; — cracks or holes in floor; — under floor and wall coatings; — machinery installations at high locations, e.g. crane rails.	To be taken as powder samples, if required (see Annex D).
7	Spread of asbestos or asbestos containing particles outdoor. Following fires, explosions or other emission of asbestos containing dust.	— pathways; — sealed surfaces; — suitable plant surfaces.	

7.3 Number of samples

It is advantageous to organize the measurements so that a larger number of contact samples are taken than are ultimately needed for analysis. In this case there are further samples for analysis on hand for use should unforeseen knowledge be gained or doubt arise about the representative nature of the samples. The relatively minimal effort required for sampling in comparison to the analysis allows for this approach. The samples which are not analysed are to be kept as archived samples. The number of contact samples to be taken depends on the measurement objective. If areas are included as cleaned areas in terms of abatement measures, for example, then the number and spatial distribution of the contact samples are larger than in terms of random sample testing to assess cleaning procedures.

Particulate loading of a surface with dust is generally not homogenous. In particular, if sources of fibrous contamination are nearby, then very different particulate loading is to be expected depending on distance, direction of air flows, and size of the emitted particles. Different numbers of samples are needed for an assessment of the situation depending upon the question being asked (see Table 1).

The number of samples depends inter alia on the surface to be analysed. Table 2 lists the minimum number of samples to be taken in a room depending on its floor space

Table 2 — Minimum number of samples per space depending on floor area

Area m^2	Minimum number of samples
<30	3
30 to 100	5
100 to 500	7
500 to 1 000	10
>1 000	>10

NOTE In Reference [7], the number of samples needed for the comparison of two environments as a function of the anticipated difference in surface loading is calculated on a statistical basis.

7.4 Requirements on the sampled surface

Sampling can be performed only on dry surfaces. During sampling, ensure that the sampling medium comes into contact with the surface over the whole area of the sample. This method can only be used on relatively smooth surfaces, especially if rigid adhesive media are used (e.g. carbon pads on SEM sample stubs). When sampling the surfaces of construction media such as concrete, pores or cavities might occur within the sampled area. Pores and cavities should be avoided as far as possible. Rough surfaces are less suitable for sampling by this method.

7.5 Sampling procedure

7.5.1 Sample size

An area of at least 1 cm^2 must be covered by the sampling medium. It should be possible to prepare the entire adhesive surface with particulate loading for examination with SEM.

7.5.2 Method

7.5.2.1 Invisible or thin layers of dust

To analyse a dust deposit, the sampling medium is pressed with the adhesive side down on the sampling site and then removed carefully. The medium can only be pressed once on the sampling site in order to ensure a clear cross-reference to the sampling point. Pushing motions are to be avoided when taking the contact sample. If adhesive tape is used as sampling medium, a strip approximately 5 cm long is cut from the roll. After removing the protective layer of the tape, the strip is pressed on the sampling point and lifted again. During sampling care should be taken that the adhesive tape is not deformed.

The strip is then placed into the transport container. Transparent round containers with a diameter of approximately 70 mm and a height of at least 10 mm have proven to be well suited for this purpose. The strip is taped to the bottom of the container by bending the two ends so that the collection area is facing up. The tape is then pressed down so far that no contact occurs with the container lid. When using carbon pads on sample trays, transport is better carried out in one of the transport containers offered by the manufacturer. The sampling tray is usually fixed in the base of the container and then a transparent hood is placed as a protective cover above.

During transport of the samples, care shall be taken to ensure that no changes to the sample particulate loading occurs due to contact, of the sample with the transport container.

Another possibility for transport is offered by encapsulating the sample in a pocket consisting of two different components. For instance, one side can be the label, the second the component, plastic-coated paper (without adhesive). Both components are clearly larger than the adhesive tape. The tape sample is laid in the pocket so that the adhesive side is facing the non-adhesive, plastic-coated component. The pocket is subsequently closed by pressing the two components together. When following this procedure,

it is to be ensured that no noticeable portions of the dust layer for analysis sticks to the plastic-coated component.

The sample should be clearly labelled, either directly on the container or with a permanently adhesive label. The lettering must be permanent (water-proof).

7.5.2.2 Thicker layers of dust

To determine the fibre content of heavier layers of dust and to record the deposition of dust over time, a different method is necessary for sampling. Multiple pieces of the sample medium are needed for an analysis to document the dust layer in its entirety. The number of samples is oriented toward the thickness of the dust layer. Sampling is conducted repeatedly at the same point until all the dust has been collected. Each piece of medium should be briefly pressed only once on the sampling point.

7.6 Sampling record

Record the details for each sample, and include at least the following information:

— sample designation;

— exact description of the sampling point for each sample:

  — building;

  — room and room size (floor area);

  — position in room (horizontal/vertical);

  — surface type (material, structure).

If this information does not sufficiently document the details of the sample, make a supplemental diagram, take a photograph or make a note on a building plan.

Also, record

— date of sampling,

— name of person who took sample,

— measurement objective (e.g. according to Table 1), and

— reason for the selection of the sampling point(s).

If other relevant information is available for the sample, document this in the sampling record.

Sampling according to ISO 16000 Part 27

Date: _____ Name: _____

Building: _____

Measurement objective: _____

Sample number / designation: _____ Sampling point: _____

Position (horizontal/vert.): _____ Surface type: _____

Room: _____ Floor area of room: _____

Map ☐ Diagram ☐ Photo ☐

Other information: (e.g. reason for the selection of the sampling point) _____

Figure 1 — Example of a sampling record

8 Analysis (SEM)

8.1 Adjustment of the SEM

8.1.1 Magnification and accelerating voltage

Fibre structure counting is carried out using two different magnifications. The smaller magnification is in the range from 300× to 400×. The higher magnification is 1 000×.

For identification by EDXA a higher magnification (5 000× or higher) might be needed, depending on the width of the structure. The accelerating voltage for the SEM is both for counting and EDXA 15 or 20 kV.

Adjust the SEM such that fibres with a width of approximately 0,2 µm are just visible at a magnification of 1 000× (see Annex B).

The sample shall not be tilted to an angle greater than 20 ° when counting the asbestos structures.

8.2 Procedure

8.2.1 Sample preparation

When adhesive tape has been used, the particulate loading of the part of the tape used for sampling is initially examined under the stereomicroscope to determine if the sample homogeneity is satisfactory. A section with a length of at least 1 cm is chosen, if required cut out using preparation scissors and fixed

to a prepared stub. Depending on measurement objective, plasma ashing of the sample to remove any organic material can be performed if necessary. The sample is then coated with gold or carbon.

NOTE When coating samples with carbon or gold, attention must be paid that the sample is not heated such that the adhesive begins to melt. After coating, craquelure (crazing) can be partially observed on the surface. This can make analysis more difficult, but is generally without significance as long as the adhesive layer has not been noticeably altered by over-heating.

8.2.2 SEM examination

8.2.2.1 Sample area to be examined

First, at least 10 mm^2 of the sample surface (minimum of 25 image fields) is examined for fibre structures at the lower magnification (300× to 400×). Subsequently, 1 mm^2 of the sample surface is analysed at a magnification of 1 000-fold magnification. The analysis can be terminated if 60 weighted fibre structures (see Clause 9) have been found. Fibrous structures seen in addition under higher (EDX) magnification and structures <5 µm in length are not counted. The image fields to be examined shall be randomly selected over the entire surface of the contact sample (approximately 1 cm^2), avoiding overlaps of image field.

8.2.2.2 Fibre structure counting rules

— All fibre structures (containing visible fibres of the type searched for (asbestos or MMVF) are counted and divided into the four categories of individual fibre, fibre bundle, fibre cluster, and fibre matrix (see Clause 3).

— If a fibrous structure covers more than an eighth of the image field, this is to be noted in the fibre count form.

— All fibrous structures extending beyond the image field are counted.

NOTE Sizing (length/width) of the counted (asbestos) structures can be done, but is not mandatory.

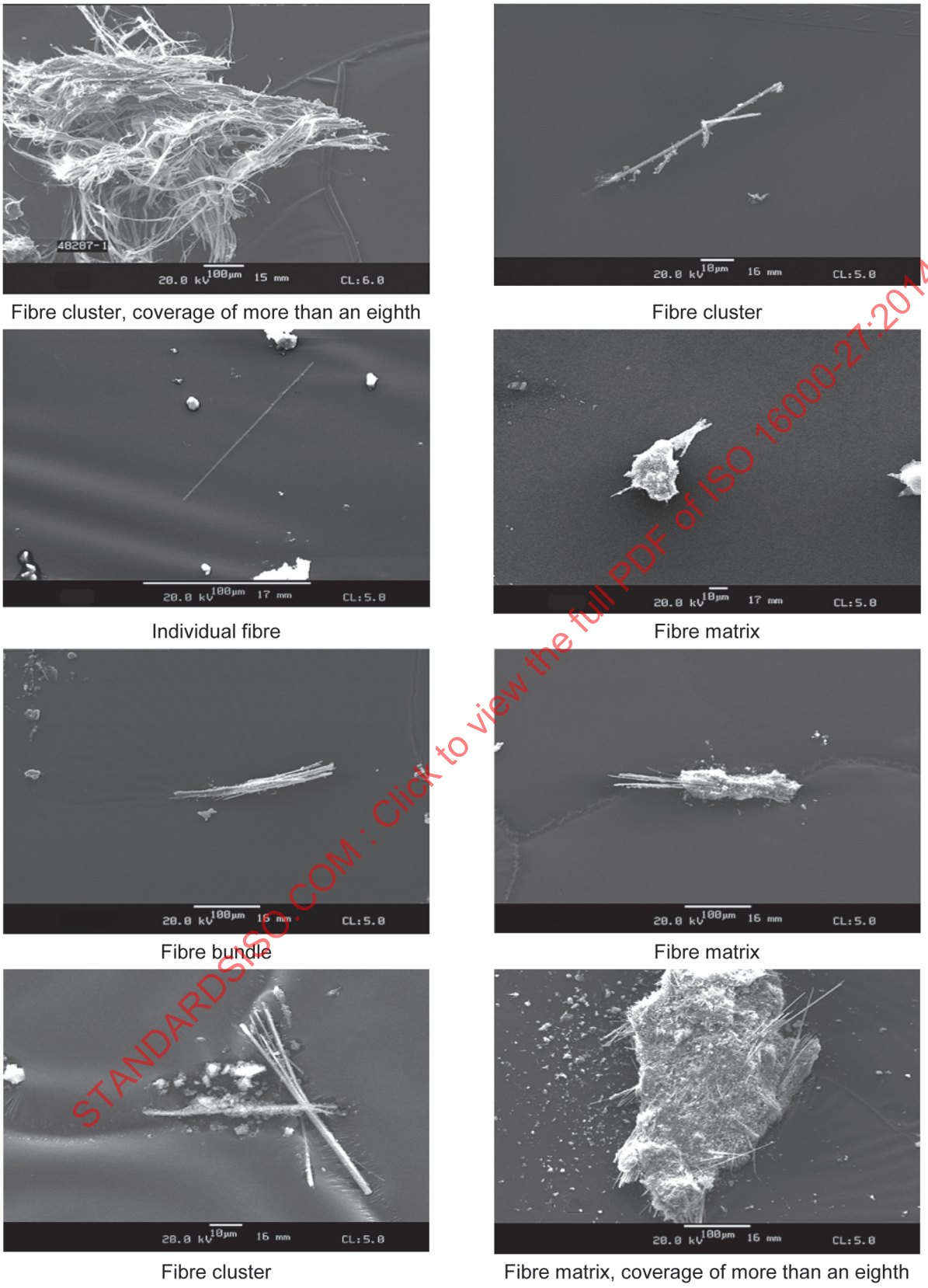


Figure 2 — Several examples for applying the fibre counting rules

8.3 Fibre classification by EDXA

8.3.1 General classification criteria

The fibres are classified by means of the EDXA spectra. Firstly, the peak heights, P, and the background level, B, are evaluated by means of the ratio:

$(P + B)/B$ and assigned to one of the three following categories:

— Category A: $(P + B)/B \geq 4$

— Category B: $2 \leq (P + B)/B < 4$

— Category C: $(P + B)/B < 2$ and significantly detected ($P > 3\sqrt{B}$)

Fibres are classified as inorganic fibres if the EDXA spectrum contains a signal of category A or B, taking account only of elements with atomic number $Z \geq 11$ (i.e. from Na onward).

NOTE Numbers given above for $(P + B)/B$ ratios are dependent from the energy resolution of the detector. For resolutions of 130 eV or less: you can use 6 instead of 4 and 3 instead of 2.

The criteria listed in Table 3 apply to the interpretation of the spectra. It is possible on the basis of these criteria to distinguish only between silicate fibres with chrysotile-like or amphibole-like spectra and the other fibre classes mentioned.

Table 3 — Criteria for interpretation of EDX spectra

Fibre class	Criteria
Chrysotile[a]	Mg and Si peaks: category A or B Fe, Mn, and Al peaks: category C (further peaks are possible depending on attached or adjacent substances, e.g. Ca, Cl)
Amphibole asbestos[b] (amosite, crocidolite)	Si and Fe peaks: category A or B Mn, Mg, Na peaks: category C possible (further peaks are possible depending on attached or adjacent substances, e.g. Ca, Cl)
Calcium sulfate (gypsum)	Distinct Ca peak, S peak present (further peaks are possible if the calcium sulfate is not chemically pure)
Other inorganic fibres	Fibres with a spectrum which does not satisfy the above criteria but contains at least one element peak of category A or B above $Z \geq 11$
Organic fibres	Fibres with a spectrum which contains no element peak of category A or B for $Z \geq 11$

[a] This criterion is also satisfied by the amphibole asbestos anthophyllite and by talc fibres which, however, are usually distinguishable from chrysotile through their lower Mg/Si ratio.

[b] If the Ca-containing amphibole asbestos types actinolite and tremolite are present in the sample, it should be noted that the Ca peak must be definitively in category A or B and that Mg (tremolite) might occur in addition to or instead of Fe (actinolite). If anthophyllite is present, in the case of an Mg-rich variant an A or B peak must be present for Si and Mg. Fe then occurs only as subsidiary component. The Mg content decreases as the Fe content increases. It should generally be noted that the amphibole asbestos variants tremolite, actinolite, and anthophyllite were used to only a small extent in industrial products. Fibres whose Mg/Si ratio is distinctly lower than chrysotile might be fibrous talc particles or fibres of the amphibole asbestos anthophyllite. It is possible to distinguish between chrysotile and talc or anthophyllite by quantitative elemental analysis. Talc and anthophyllite fibres can be reliably distinguished on the basis of differences in their crystal structure through electron diffraction using the transmission electron microscope.

It is important to recognize that, during acquisition of an EDXA spectrum from a fibre, scattering of the electron beam might result in emission of X-rays from particles attached to, or in close proximity to the

fibre being analysed. The EDXA spectrum obtained might therefore contain contributions from these particles, and the spectrum might contain X-ray peaks from elements that are not present in the asbestos varieties. In these cases, attempts should be made to acquire EDXA spectra from several positions on the fibre, as far away from adhering or adjacent particles or fibres as possible, in order to minimize the contributions from the other particles.

8.3.2 Additional classification criteria for asbestos varieties

8.3.2.1 Serpentine (chrysotile)

Classify a fibre as serpentine (chrysotile) if

a) the Mg and Si peaks are clear, with $(P + B)/B > 2$ and

b) any Fe, Mn, and Al peaks are small, with $P/B < 1$.

NOTE 1 Depending on the composition of adjacent or attached particles, other peaks such as Ca or Cl might also be visible.

NOTE 2 Anthophyllite and talc both yield EDXA spectra which conform to this specification, but the Mg/Si peak height ratio for these minerals is lower than that for serpentine. In order to avoid erroneous classification of talc or anthophyllite as serpentine, it is important to take account of the Mg/Si peak height ratio, and to calibrate the EDXA detector using known samples of serpentine and talc.

8.3.2.2 Amosite

Classify a fibre as amosite if

a) the Si and Fe peaks are clear, with $(P + B)/B > 2$ and

b) any Na, Mg, and/or Mn peaks are small.

NOTE Depending on any adjacent or attached particles, other peaks such as Ca or Cl might also be visible.

8.3.2.3 Crocidolite

Classify a fibre as crocidolite if

a) the Na, Si, and Fe peaks are clear, with $(P + B)/B > 2$ and

b) any peak from Mg is small, and any Mn peak is small with $P/B < 1$.

NOTE Depending on the adjacent or attached particles, other peaks such as Ca or Cl might also be visible.

8.3.2.4 Tremolite or actinolite

Classify a fibre as tremolite or actinolite if

a) the Mg, Si, and Ca peaks are clear, with $(P + B)/B > 2$ and

b) a peak from Fe can be present, but any Na peak is faint, with $P/B < 1$.

NOTE Depending on the adjacent or attached particles, other peaks such as Ca or Cl might also be visible.

8.3.2.5 Anthophyllite or talc

Classify a fibre as anthophyllite/talc if

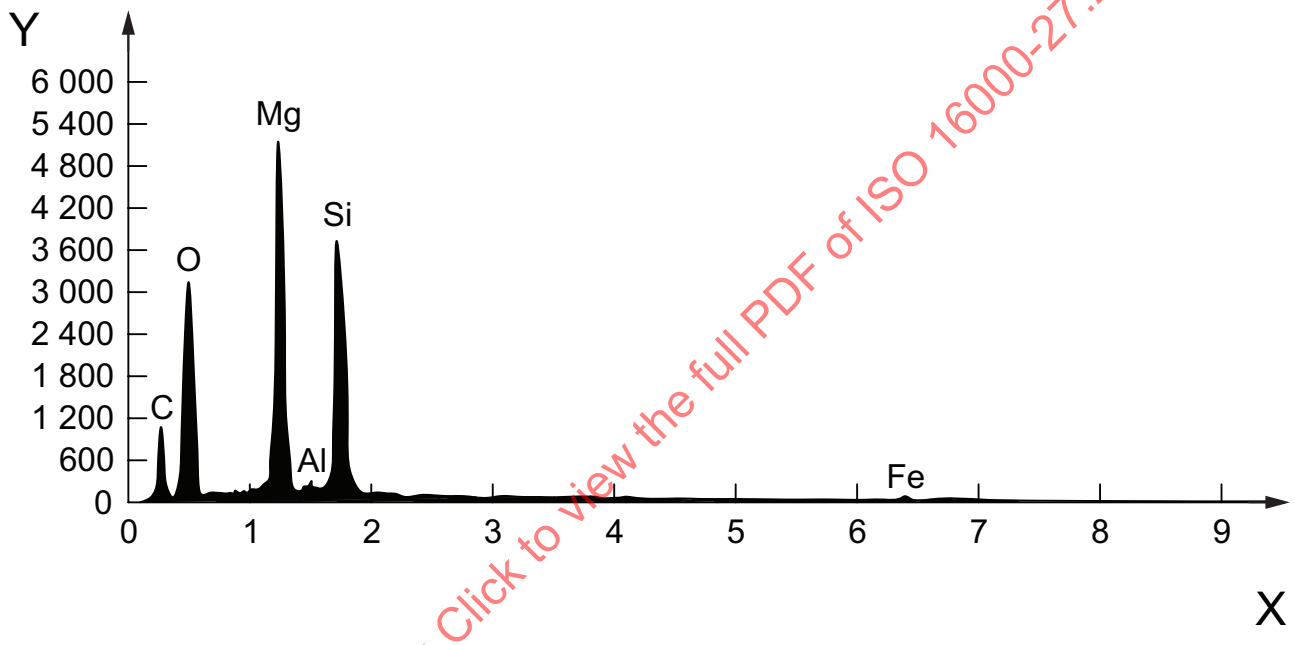
a) the Mg and Si peaks are clear, with $(P+B)/B > 2$ and

b) the Mg/Si peak height (or area) ratio is consistent with that obtained on fibres of reference anthophyllite or talc, and any peaks from Fe, and Ca are small.

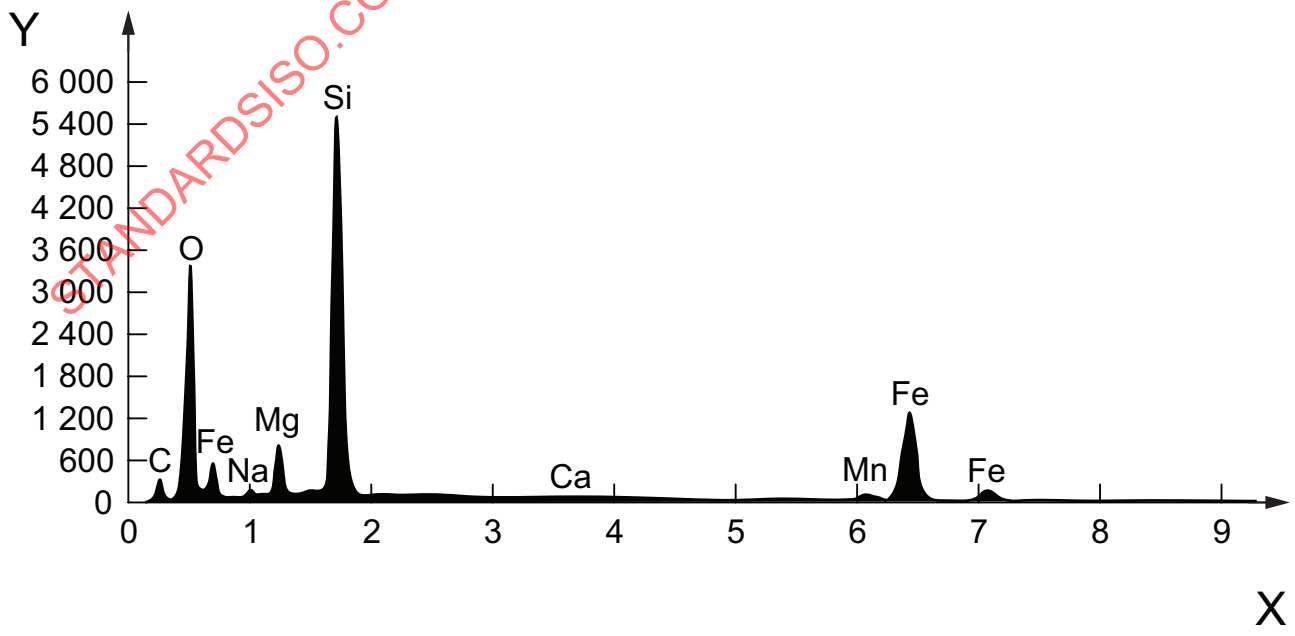
NOTE When using this additional analytical identification criteria see the remark given in Table 3 under [b].

8.3.2.6 Reference EDXA spectra from standards of the asbestos varieties

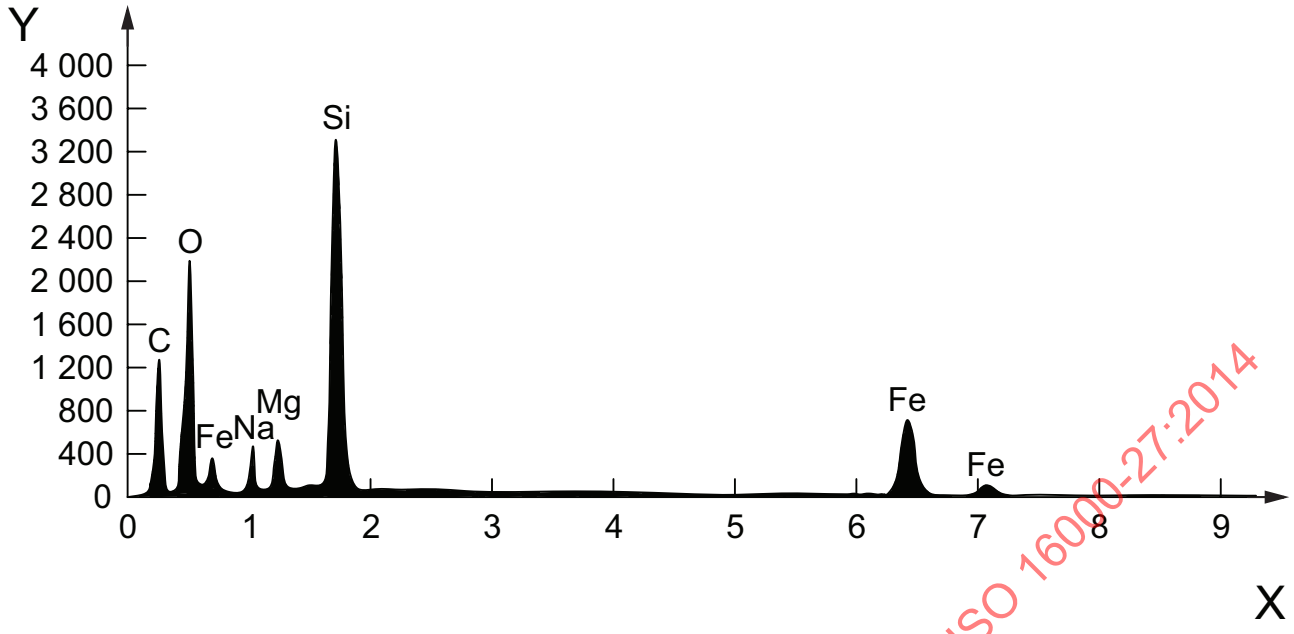
For any particular fibre, the relative heights of the peaks in the EDXA spectrum vary with the characteristics of the X-ray detector. In particular, the detection efficiency for X-ray peaks from low atomic number elements is higher for ultra-thin window detectors than it is for standard beryllium window detectors. Because each EDXA detector has different efficiency characteristics, it is necessary to obtain reference spectra for each SEM-EDXA system, using standards of the asbestos varieties. A series of such spectra, collected using an ultra-thin window detector, are shown as examples in Figure 3. These spectra are used for comparison purposes in the classification of fibres. Since the performance of the EDXA detector might change with time, new reference spectra shall be obtained at appropriate intervals, and particularly after any maintenance of the detector has been carried out.



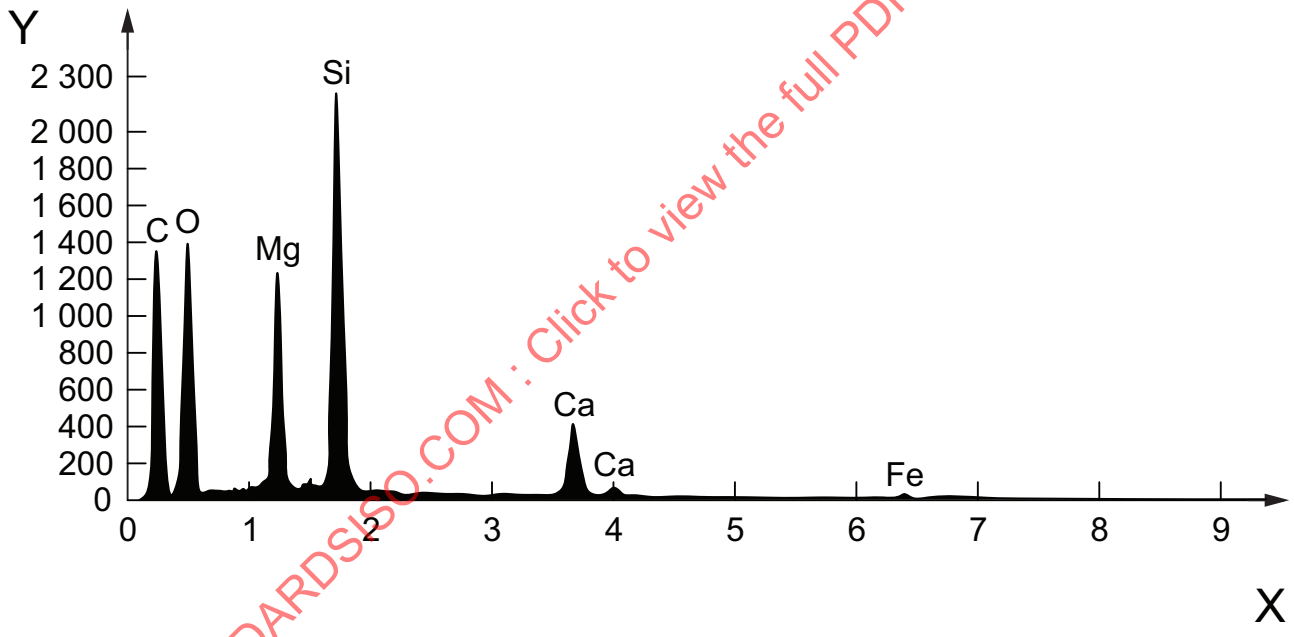
a) Chrysotile (no gold coating)



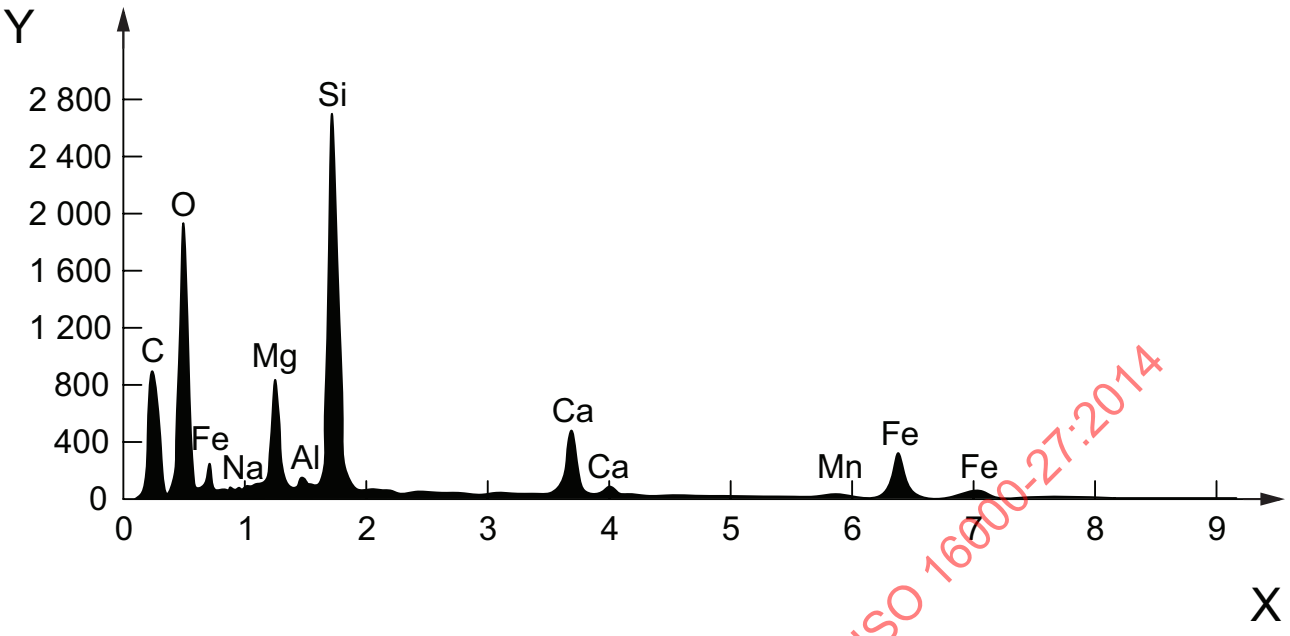
b) Amosite (no gold coating)



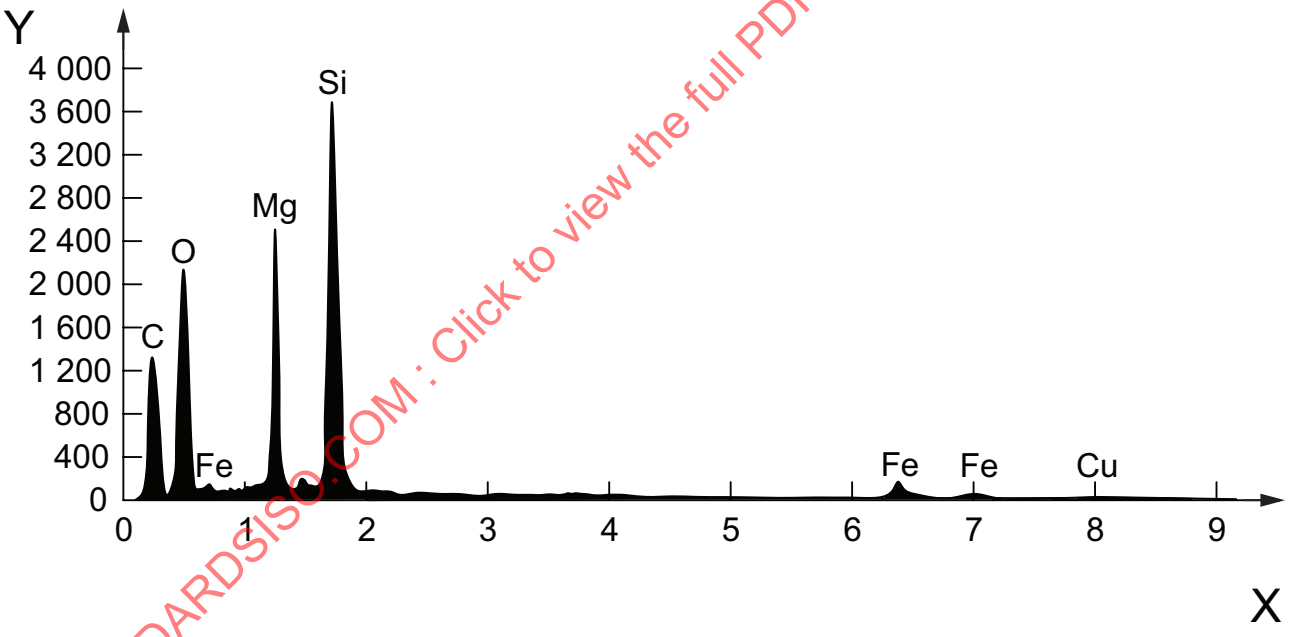
c) Crocidolite (no gold coating)



d) Tremolite (no gold coating)



e) Actinolite (no gold coating)



f) Anthophyllite (no gold coating)

Key

X keV

Y counts

Figure 3 — EDXA spectrum from fibre of reference asbestos varieties

8.3.2.7 Additional precautions during acquisition of EDXA spectra

During the acquisition of EDXA spectra, care must be taken to ensure that the electron beam is stable, that the point of incidence is on the structure and that the beam does not drift off the structure during the analysis. It is also necessary to ensure that the point of incidence of the electron beam is as far as possible from any attached or adjacent fibres and/or particles, in order to obtain a spectrum from

the structure with a minimum of interference. In some cases it is not possible to classify a structure unambiguously. This could be because of interference by other particles or fibres, or because the peak to background ratios are insufficient. When this occurs, annotate the data for these structures by an asterisk and indicate the reason on the structure counting form.

8.3.3 Differential classification of other fibres

8.3.3.1 Inorganic product fibres

Fibres can be classified as product fibres only if the origin of these fibres can be attributed to a product which is present on the vicinity of the sampling location. For this, it is necessary for the spectra of the fibres on the filter to agree substantially with those of the product.

In order to provide evidence a bulk material sample shall be taken according to ISO 22262-1. The EDX spectra of several thin fibres taken from different places from this bulk sample shall be compared with each other. If relatively uniform spectra with coinciding element peaks and intensity patterns are detected, a list of characteristic elements can be derived from these spectra. The obtained list of characteristic elements found in the material fibres can in principle be used to classify the fibres on the sample collection filter.

For assignment as product fibres, the element spectra are recorded for 10 thin fibres in a dispersed specimen of each sample of material. An element is included in the list of elements in the product in category A if it is found in more than eight of these 10 fibres with a signal/background ratio of ≥3:1. If it is detected in more than six of the fibres in category A or B, i.e. with a signal/background ratio ≥1:1, but does not satisfy the condition for inclusion as an A element, it is regarded as a B element.

Concerning the comparison with the spectra on the sample collection filters, the following procedure is recommended: In the element list of the product fibres every element peak is classified in one of the categories A, B, or C in dependence on the relation $(P + B)/B$. Based on this classification scheme fibres originating from the air sample and attributed to the class “other inorganic fibres” can be classified as product fibres if the EDX spectrum complies with the following conditions.

a) Elements, the peaks of which are attributed to category A in the reference spectrum, must be detected with an intensity pattern according to category B.

b) Of the elements which are attributed to category B in the reference spectrum at least on third must be detected. If less than three elements are detected in the bulk material reference spectrum, the fibre is classified as a product fibre even if none of these can be attributed to category B. However, at least one element shall meet the category C criterion. For example, the EDX spectrum of a glass fibre from the bulk sample might indicate the elements Na, Mg, Al, Si, K, and Ca, whereas the Na peak or also the Al or Mg peak might be missing. In any case the distinct peaks of Si and Ca are detected.

The specification of the above criteria for the classification of “other inorganic fibres” as product fibres is a convention which might result in both an overestimation and underestimation of the actual product fibre concentration.

8.3.3.2 Organic fibres

Differentiation between different types of organic fibres is not possible. Prior information is necessary for this. It can be helpful in some circumstances if the known original substances are available so that morphological aspects features which can facilitate the assignment to defined types of fibres are to be found in Reference [8].

The following points must be complied with to detect organic fibres.

— No cold ashing of the samples must be carried out.

— The permissible accelerating voltage shall be tested on the actual sample. It shall be ensured in this case that fibres with a width of 0.2 µm or, if fibres as thin as this are not present in the sample, the thinnest fibres present can be identified.

— Detectors without windows or with ultrathin windows should be used so that it is possible to detect light elements from Z = 6 (carbon) upward. It should be noted in this connection that some light element detectors greatly attenuate the nitrogen peak.

— Fibres are classified as organic fibres if the main peaks in the EDX spectrum exhibit elements with Z < 11, especially C (except carbon fibres), O, and possibly N and F, Cl, or S might also be present.

9 Calculation and presentation of results

Along with a purely qualitative analysis (fibres of a certain category present: yes/no), the count results in connection with the analysed filter area can be calculated as fibrous structures per cm^2. To take the higher potential of bundles, clusters, and matrices to release fibres, these structures are weighted more heavily (see Table 4).

Table 4 — Weighting factors

Fibrous structure type	Weighting factor
Individual fibre	1
Fibre bundle	5 (10[a])
Fibre cluster	5 (10[a])
Fibre matrix	5 (10[a])
[a] With coverage of one eighth of the image field area by the fibrous structure at 300 to 400-fold magnification. One structure is weighted only once: either 5 or 10.	

The weighted fibrous structures are calculated as follows:

$$Z_W = \frac{\sum_{i=1}^{4} S_{W,i}}{A} \quad (1)$$

where

Z_W is the weighted count result, in cm^{-2};

$S_{W,i}$ is the weighted count result of an individual fibrous structure type i;

A is the analysed sample area, in cm^2.

Along with the weighted count result, the number of the fibrous structures actually found and the total analysed image field area should also be given. This is helpful for further (e.g. statistical) analyses.

Due to the substantial variability of fibre width, which can be caused, for example, by the inhomogeneity of fibre distribution on the surface connected with unavoidable, purely coincidental deviations in the analysis, four classes characterized by on weighted count result are implemented (see Table 5). The analysis and presentation of results are separated according to fibre types (asbestos, MMVF).

NOTE The numbers of (weighted) structures found at the two magnifications are added as well as the areas examined.

Table 5 — Division into classes (fibre type indicates asbestos, MMVF, other fibres)

Analysis	Weighted count result Z_W cm^{-2}	Class
No fibre type detected	0	0
Fibre type detected	>0 to 100	1
Surface clearly loaded with fibre type	>100 to 500	2
Surface heavily loaded with fibre type	>500	3

It is advisable to use the above classification instead of "precise" structure density figures, due to the considerable scattering of the measured structure densities, partially stochastically influenced or due to inhomogeneities in the dust surface loading. The weighting of the structures is a convention and takes into account, that a multifibre structure (especially in the case of asbestos is bearing a risk) for generating a larger number of fibres, if further split up for example by mechanical influences.

10 Record of analysis

The record of analysis must contain the following information as a minimum:

— the two magnifications set for the SEM (e.g. 300× and 1 000×);

— the sample area analysed at the particular magnification;

— image field numbers of the fibrous structures found and the associated identification characteristics (fibrous structure, amphibole asbestos, chrysotile, MMVF).

NOTE If in a fibrous structure different types of asbestos are found, this structure is to be either recorded as only one asbestos type or an appropriate note is to be made in the column for remarks. This prevents the fibrous structure from being counted twice.

An example for a counting protocol is given in Annex A.

11 Measurement uncertainty

11.1 General

Experience shows that the examination of surfaces in rooms or buildings in which no asbestos-containing products are or have been used yields the result "no asbestos detected" in the majority of cases when the measurement method described here is applied and the ambient air concentration of asbestos is not influenced by emissions sources in the vicinity of the building. The reason for this is the low asbestos concentration normally present in ambient air, so that even surfaces which have not been cleaned for long periods of time do not demonstrate any detectable asbestos contamination as a result.

The measured value is determined, along with the random sample related deviation (11.2), through a series of influential factors whose variables are not always known with precision and which generally lead to a deviation of too low values. Thus, with some surfaces, e.g. concrete, it is to be expected that micro-roughness, cavities, and pores will lead to inhomogeneous particulate loading of the samples. Likewise, it is to be assumed that possibly not all fibres on the surface were removed with the adhesive.

Despite this, a positive detection of fibres according to this guideline is a very sensitive indication of surface contamination. Both effect-directed indicators (alveolar fibres) and factors, which are useful as indicators of future fibre release and the effectiveness of cleaning regimes are of interest. Therefore a weighting for "multiple fibrous structures" (fibre bundle, cluster, matrix) has been introduced. This approach makes analysis significantly easier and ensures that the direct advantage of contact samples, namely the simplicity of sampling, is not heavily compromised by the necessity of great effort in the

analysis. In this case priority is given to a larger scope of random samples (more individual samples) than to the greater precision of the individual sample.

11.2 Random sample related deviation

The deposit of fibrous structures on the surface is subject to random distribution. The resulting unavoidable random sample related deviation is identical in its structure to the expected deviation concerning air measurements.

If a small area section is observed (for instance, the area to be analysed later) and the time sequence of the deposition is divided into very small periods of time t_i, then beginning from a point in time t_0 (begin of deposition), a structure is deposited at a time point t_n and at a later time point t_{n+x} another structure is deposited, and so on. After a time T (time of adhesive sampling), it can be observed that the number of fibrous structures shows a Poisson distribution, if we compare the results from same sized areas (our sample size) within the larger surface area (having a homogenous deposition) to be judged. If it is assumed that the contact sample has removed the fibrous structures from the surface without causing any substantial changes, then correspondingly unweighted count result will also show Poisson distribution according to this guideline (see also ISO 14966). Parameters influencing the random sample related deviation and the calculation of confidence intervals are summarized in Annex C.

11.3 Deviations in analysis and weighting of the count results

Smaller counting deviations can be expected compared to fiber analysis due to the relatively simple counting rules for detecting fibrous structures (there are no length or average limits and no separation of multi-fibrous structures into individual fibres and sizing). Deviations are however caused by the use of two magnifications. With the smaller magnification (300× to 400×), it is to be expected that individual fibres with diameters of less than approximately 0,6 µm are not detected. Bigger asbestos containing structures (d > 0,6 µm) are counted more efficiently due to the low magnifications used. This leads to a more mass oriented value, which is underlined if weighting factors are used.

The use of two different magnifications regarding differentiated counting of clusters would lead to deviations due to the better resolution at higher magnification, since the individual fibres can be differentiated more from each other. The uniform weighting factors, among other things, counteract this. The weighting factors are a convention. The resulting deviations cannot be quantified exactly as a result. This restriction is taken into account through the general classification into only four classes for the degree of the surface contamination.

On a set of four samples the following results have been achieved by four laboratories from three different countries (see Table 6) using this method.

Table 6 — Comparison of results / weighted counting results (asbestos)/ division into classes

	Weighted fibre concentration (cm^{-2})/class			
Sample	**Laboratory 1**	**Laboratory 2**	**Laboratory 3**	**Laboratory 4**
1	ND/0	ND/0	ND/0	ND/0
2	2 300/3	1 240/3	1 482/3	2 250/3
3	45/1	63/1	82/1	92/1
4	18/1	ND/0	ND/0	ND/0
ND = Not detected.				

Deviations might vary depending from fibre type and particle loading of the sample.

11.4 Detection limit

Detection limit for the purposes of this guideline is understood as the fibrous structure density (cm^{-2}) below which, with 95 % probability, the actual density lies when no fibrous structures are detected

during SEM examination. The detection limit depends on the examined sample area *F*. Provided that the instructions specified in this guideline are observed, the detection limit is determined as follows: For $n = 0$ or $S_W = 0$ (no fibrous structures found), an upper limit is rounded up to a whole number $\lambda_o = 4$ for the 97,5 % confidence interval.

NOTE In contrast to ISO 14966, here calculation is made for the value 0 with a unilateral 97,5 % confidence interval and rounded up. This method takes several influential variables that are quantifiable only with difficulty into account.

From this detection limit, *D*, results:

$$D = \frac{4}{F} \quad (2)$$

where

F is examined sample area, in cm^2.

For a sample area of 0,11 cm^2 analysed according to this guideline, a detection limit of 36 cm^{-2} exists for the fibrous structures.

12 Applications and instructions for use

12.1 General

In practice, a series of questions arise which are only partially addressed in the following. The transition from the previously most used approaches to the method described in this guideline can possibly lead to other results. This applies not only to the classification and analysis of the end result, but also to the way the count result is arrived at. Previously the sample was often initially examined at varying magnifications and then the more precise analysis of the visibly loaded areas was performed at higher magnification. This has the consequence that the lower fibre concentration (<100 cm^{-2}) is generally estimated to be higher than the concentrations detected using this guideline. However, depending on method and criteria for analysis, the process of examination was often prematurely terminated once the relevant number of structures was found, possibly leading to lower assessments in the upper range as is the case when following this guideline.

The clear distinction between clusters, bundles, and matrices is not always possible. In these cases the analyst must decide which assignment is to be made. The assignment is, however without importance to the final result because both structures are weighted equally.

The counting rules and the corresponding definitions have been intentionally kept simple in order to not compromise the actual advantage of contact samples, namely the ease of sampling, with a too high demand for precision, as mentioned in 11.1 above. It is to be assumed that the deviation arising from random sampling in the final result is dominant in all cases and therefore a very detailed and complex analysis of the individual sample is not useful.

12.2 Sample requirements

The sample area to be analysed according to this guideline is 1 cm^2. This size has been chosen because the SEM stub on some devices only allows a sample size of slightly more than 1 cm^2. This sample size has the advantage, however, that within such small area a more uniform presence of particles is to be expected which leads to smaller variance of the single results. If sampling is performed using adhesive tape, the total sample area available is clearly greater. The possibility exists here to create two or three individual samples out of this. Whether this is useful, depends on the particular question to be answered.

If statistically-valid results are required for a project, the sampled surface should not incorporate any large defects such as cavities or pores within the area of the collected sample, and the surface should be

smooth, as tapes might vary in their collection, the same tape type must be used for the same site or if comparisons between sites are made.

When using rigid media as adhesive material (e.g. carbon pads mounted on SEM stubs), it is especially important for a valid result that in the sampled area there are no large, macroscopically evident particles on the surface. This generally results not only in these particles adhering to the surface of the contact sample, but also in a large portion of the sampled surface not coming into contact with the adhesive medium.

12.3 Summary of the results for multiple individual samples

Determination of an average value across multiple samples of one surface area is appropriate if the contamination of two surfaces is to be compared. The requirement for this is that the contamination within the particular areas can be assumed to be homogenous.

Should multiple individual results from one surface area be summarized, first the unweighted counted structures S_i are to be added together and applied to the total analysed area. This corresponds to the average value S_M for this area.

$$S_M = \frac{\sum S_i}{\sum F_i} \tag{3}$$

where

F is examined sample area, in cm^2.

In doing this, it makes sense to initially add the individual fibre structures separately from the multiple fibre structures so that the final result can also be weighted later, if required.

Should the average values of two areas be compared with each other, significance tests are to be done based on the sum of the total found structures ΣS_i (see 11.2) and not in the basis of the average fibrous structure density S_M calculated according to Formula (3).

If the areas analysed in the samples from the two areas are different sizes, the sum of the structures $\Sigma S_{i,1}$ from the area F_1 with the larger analysed area is to be accordingly converted and rounded to a whole number:

$$\sum S'_{i,1} = \text{int}\left(\sum S_{i,1} \frac{F_2}{F_1}\right) \tag{4}$$

where

$\Sigma S_{i,1}$ is the sum of the structures from area F_1;

$\Sigma S'_{i,1}$ is the normalized sum of structures;

F_1 is area 1;

F_2 is area 2.

Annex A
(informative)

Example of SEM structure counting form

				Page: of:
Sample No.:	Date:	Name:		
Magnification:		**Image field area (mm^2):**		
Structure no.	Image field no.	Type	Structure	Weight
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
Summary: structures counted/weighted result Σ				

Keys to abbreviations		
Type (asbestos fibres):	**Structure:**	**Remarks:**
C = chrysotile	F = fibre	
A = amosite	B = bundle	
CR = crocidolite	C = cluster	
AC = actinolite	M = matrix	
TR = tremolite		
AN = anthophyllite		
Other fibres specify type and label:		

Annex B
(normative)

Procedures for calibration and adjustment of the SEM

B.1 Calibration of the scanning electron microscope

The SEM specimen is examined at an accelerating voltage of approximately 15-20 kV and a magnification of 300× to 400× or 1 000×. For fibre identification in the SEM, an accelerating voltage of 15-20 kV is recommended.

The magnification on the screen shall be calibrated using a certified commercially-available magnification standard. It is important to recognize that the magnification value displayed on some models of SEM is that applicable to micrographs produced by the recording system, and not to the viewing screen (CRT display). The SEM examination is performed directly on the viewing screen, and the magnification calibration must relate to the viewing screen.

Adjust the SEM such that fibres with a width of approximately 0,2 µm are just visible at a magnification of 1 000×.

This adjustment is performed by selecting a fibre on the prepared sample, or on a test sample, which is just visible at the magnification of approximately 1 000× used for structure counting. The width of this fibre is then confirmed by measuring it at a magnification of 10 000×. Carry out this adjustment routinely before each series of analyses.

Position the X-ray detector such that it subtends the largest possible solid angle at the specimen surface.

NOTE 1 On a 25 cm × 16 cm CRT display, 25 image fields at a magnification of 1 000 correspond to an area of 1 mm^2 on the specimen.

NOTE 2 The width of the scan line (or pixel width for an SEM with digital imaging) on the sample and the diameter of the electron beam are the factors which determine the resolution in the SEM. Provided that the scan line width or the pixel width does not exceed 0,25 µm, no severe image degradation relevant to resolution of a 0,2 µm thick fibre longer than 5 µm is observed.

B.2 Adjustment of the EDXA system

The largest possible solid angle of the EDXA detector system should be used. The operating parameters of the SEM and the X-ray detector system shall be selected so that a statistically-acceptable X-ray spectrum can be acquired from a 0,2 µm width chrysotile fibre on the test sample within a maximum period of 100 s.

The criterion for statistical acceptability requires, for peak height P and background level B:

$$P > 3\sqrt{B} \tag{B.1}$$

with a minimum of 30 pulses in the channel corresponding to the maximum peak height for each of the magnesium and silicon peaks and,

$$(P + B) / B > 2 \tag{B.2}$$

for each of the magnesium and silicon peaks.

Annex C
(informative)

Random sample related deviation

The random sample deviation can be described by a Poisson distribution:

$$W\left(\langle n\rangle, n\right) = \frac{1}{n!} \cdot \langle n\rangle^{n} \cdot \mathrm{e}^{-\langle n\rangle} \tag{C.1}$$

where

W is the probability;

$\langle n\rangle$ is the average value of the number of fibrous structures on the surface area under consideration;

n is the count result (number of counted structures).

For a count result n, the 95 % confidence interval of the average value leading to this count result is then calculated as follows:

$$\frac{\int_{\lambda u}^{\lambda o} \langle n\rangle^{n} \cdot \exp\{-\langle n\rangle\} \cdot \mathrm{d}\langle n\rangle}{\int_{0}^{\infty} \langle n\rangle^{n} \cdot \exp\{-\langle n\rangle\} \cdot \mathrm{d}\langle n\rangle} = 0{,}95 \tag{C.2}$$

where

λ_o and λ_u are the upper and lower limits of the 95 % confidence interval of the average value $\langle n\rangle$.

The confidence limits can be calculated using the χ^2 distribution: