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Second edition
2019-12

Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method

Air ambiant — Détermination de la concentration en nombre des particules inorganiques fibreuses — Méthode par microscopie électronique à balayage

Reference number
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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 14966:2002), which has been technically revised. It also incorporates the corrected version ISO 14699:2002/Cor 1:2007. The main changes compared to the previous edition are as follows:

- Counting rules, changed to the recommended method (membrane filter method) of the WHO (World Health Organization);
- Analytical procedure (classification), using normalized peak height ratios in addition to the method of the previous edition;
- Rule for early termination of filter evaluation (counting and analysis). A formula is given to terminate the filter evaluation, if the calculated (asbestos) fibre concentration is above a set limit value for this fibre concentration.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes a method for measurement of the numerical concentration of inorganic fibrous particles in ambient air using the scanning electron microscope. This document is based on VDI 3492[1].

The method is also suitable for determining the numerical concentrations of inorganic fibres in the interior atmospheres of buildings, for example measurement of residual airborne fibre concentrations after the removal of asbestos-containing building materials.

Biological research has shown that the fibrogenic or carcinogenic effect of a fibre is related to its length, diameter and its resistance to dissolution in a biological environment. The point at which fibres are too short, too thick or of insufficient durability to produce a fibrogenic or carcinogenic effect is uncertain. Fibres with lengths greater than 10 µm and diameters of a few tenths of 1 µm, which also have durabilities such that they remain unchanged for many years in the body, are regarded as particularly carcinogenic. Based on current knowledge, fibres shorter than 5 µm are thought to have a lower carcinogenic potential[2]–[5].

For the purposes of this document, a fibre is defined as a particle which has a minimum length to width (aspect) ratio of 3:1. Fibres with lengths greater than 5 µm and widths extending from the lower limit of visibility up to 3 µm are counted. Fibres with diameters less than 3 µm are considered to be respirable. Since the method requires recording the lengths and widths of all fibres, the data can be re-evaluated if it is required to derive concentrations for fibres with a higher minimum aspect ratio[6].

The range of concentration to be measured extends from that found in clean air environments, in which the mean value of a large number of individual measurements of asbestos fibre concentrations has been found to be generally lower than 100 fibres/m^3 (fibres longer than 5 µm), up to higher exposure scenarios in which concentrations as much as two orders of magnitude higher have been found[4][6].

This method is used to measure the numerical concentration of inorganic fibres with widths smaller than 3 µm and lengths exceeding 5 µm up to a maximum of 100 µm. Using energy-dispersive X-ray analysis (EDXA), fibres are classified as fibres with compositions consistent with those of asbestos fibres, calcium sulfate fibres and other inorganic fibres.

Calcium sulfate fibres are separated from other inorganic fibres and are not included in the final result, because on the basis of current knowledge, they do not represent any health hazard. Nevertheless, the numerical concentration of calcium sulfate fibres should be determined, since a high concentration of these fibres can negatively bias the results for probable asbestos fibres, and in some circumstances the sample may have to be rejected[7]. In addition, knowledge of the numerical concentration of calcium sulfate fibres is of importance in the interpretation of fibre concentrations in ambient atmospheres.

Detection and identification of fibres becomes progressively more uncertain as the fibre width is reduced below 0,2 µm. Identification of a fibre as a specific species is more confident if the source of emission is known or suspected, such as in a building for which bulk materials are available for analysis.

In order to facilitate the scanning electron microscope examination, organic particles collected on the filter are almost completely removed by a plasma ashing treatment.

Except in situations where fibre identification is difficult, there should be only minor differences between fibre counting results obtained by this method and those obtained using the procedures for determination of PCM-equivalent fibres in Annex E of the transmission electron microscopy method ISO 10312[8].

Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method

1 Scope

This document specifies a method using scanning electron microscopy for determination of the concentration of inorganic fibrous particles in the air. The method specifies the use of gold-coated, capillary-pore, track-etched membrane filters, through which a known volume of air has been drawn. Using energy-dispersive X-ray analysis, the method can discriminate between fibres with compositions consistent with those of the asbestos varieties (e.g. serpentine and amphibole), gypsum, and other inorganic fibres. Annex C provides a summary of fibre types which can be measured.

This document is applicable to the measurement of the concentrations of inorganic fibrous particles in ambient air. The method is also applicable for determining the numerical concentrations of inorganic fibrous particles in the interior atmospheres of buildings, for example to determine the concentration of airborne inorganic fibrous particles remaining after the removal of asbestos-containing products.

The range of concentrations for fibres with lengths greater than 5 µm, in the range of widths which can be detected under standard measurement conditions (see 7.2), is approximately 3 fibres to 200 fibres per square millimetre of filter area. The air concentrations, in fibres per cubic metre, represented by these values are a function of the volume of air sampled.

The ability of the method to detect and classify fibres with widths lower than 0,2 µm is limited. If airborne fibres in the atmosphere being sampled are predominantly <0,2 µm in width, a transmission electron microscopy method such as ISO 10312[8] can be used to determine the smaller fibres.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

3.2
amphibole
any of a group of rock-forming double-chain silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0\text{-}1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

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

Note 1 to entry: 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° and 124°.

3.3
amphibole asbestos

amphibole (3.2) in an *asbestiform* (3.5) *habit* (3.17)

3.4
analytical sensitivity

calculated airborne *fibre* (3.13) concentration equivalent to counting one fibre in the analysis

Note 1 to entry: The analytical sensitivity is expressed in fibres per cubic metre.

Note 2 to entry: 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* (3.13) and fibrils possess high tensile strength and flexibility

3.6
asbestos

any of a group of silicate minerals belonging to the serpentine and *amphibole fibres* (3.2) groups which have crystallized in the *asbestiform* (3.5) *habit* (3.17), causing them to be easily separated into long, thin, flexible, strong *fibres* (3.13) 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).

3.7
aspect ratio

ratio of length of a particle to its width

3.8
chrysotile

fibrous variety of the mineral serpentine, which has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

Note 1 to entry: Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al_3+ can occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} can also be present. Chrysotile is the most prevalent type of asbestos.

3.9
cleavage

breaking of a mineral along one of its crystallographic directions

3.10
cluster
fibrous structure in which two or more *fibres* (3.13), or *fibre bundles* (3.14) are randomly oriented in a connected grouping

3.11
countable fibre
any object longer than 5 µm, having a maximum width less than 3 µm and a minimum aspect ratio of 3:1

3.12
energy-dispersive X-ray analysis
measurement of the energies and intensities of X-rays by use of a solid-state detector and multi-channel analyser system

3.13
fibre
elongated particle which has parallel or stepped sides and a minimum aspect ratio of 3:1

3.14
fibre bundle
structure composed of apparently attached, parallel *fibres* (3.13)

Note 1 to entry: A fibre bundle can exhibit diverging fibres at one or both ends. The length is defined as equal to the maximum length of the structure, and the diameter is defined as equal to the maximum width in the compact region.

3.15
fibril
single *fibre* (3.13) of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances

3.16
fibrous structure
fibre (3.13), or connected grouping of fibres, with or without other particles

3.17
habit
the characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities

3.18
image field
the area on the filter sample which is shown on the screen

3.19
limit of detection
calculated airborne *fibre* (3.13) concentration equivalent to the upper 95 % confidence limit of 2,99 fibres predicted by the Poisson distribution for a count of zero fibres

Note 1 to entry: The limit of detection is expressed in fibres per cubic metre.

3.20
magnification
ratio of the size of the image of an object on the observation screen to the actual size of the object

Note 1 to entry: For the purposes of this document, magnification values always refer to that applicable to the observation screen.

3.21
matrix
structure in which one or more *fibres* (3.13) or *fibre bundles* (3.14) touch, are attached to, or partially concealed by a single particle or connected group of non-fibrous particle

3.22
serpentine

any of a group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

3.23
split fibre

agglomeration of *fibres* (3.13) which, at one or several points along its length, appears to be compact and undivided, whilst at other points appears to separate into separate fibres

3.24
structure

single *fibre* (3.13), *fibre bundle* (3.14), *cluster* (3.10)or matrix

4 Abbreviated terms

EDXA	Energy-dispersive X-ray analysis
FWHM	Full width, half maximum
PTFE	Polytetrafluoroethylene
SEM	Scanning electron microscope

5 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through a gold-coated, capillary pore track-etched membrane filter with a maximum nominal pore size of 0,8 µm, which is subsequently examined in the scanning electron microscope (SEM). Before analysis, the gold-coated filter is treated in a plasma asher to remove organic particles, to the extent that this is possible. The individual fibrous particles and constituent fibres in a randomly-selected area of the filter are then counted at a magnification of approximately 2 000×. If a fibre is detected at the magnification of approximately 2 000×, it is examined at a higher magnification of approximately 10 000× to measure its dimensions. At the higher magnification of approximately 10 000×, energy-dispersive X-ray analysis (EDXA) is used to classify the fibre according to the chemical composition.

The limit of detection for this method is defined as the numerical fibre concentration below which, with 95 % confidence, the actual concentration lies when no fibres are found during the SEM examination. The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by examination of a larger area of the specimen in the SEM. In practice, the lowest achievable limit of detection for a particular area of SEM specimen examined is controlled by the total suspended particulate concentration remaining after the plasma ashing step.

A limit of detection of approximately 300 fibres/m^3 is obtained if an air volume of 1 m^3 per square centimetre of filter surface area passes through the filter, and an area of 1 mm^2 of the filter area is examined in the SEM. This corresponds to an evaluated sample air volume of 0,01 m^3.

6 Apparatus and materials

6.1 Air sampling

6.1.1 Sampling head

A disposable, 3-piece, conductive plastic field monitor cassette may be used as the sampling head, provided that the design is such that significant leakage around the filter does not occur. A re-usable unit may also be used as the sampling head, consisting of a cylindrical cowl and a filter holder with backing filter. Figure 1 shows an example of a suitable sampling head. The cowl and the filter holder should be made from a corrosion-resistant material. The filter shall be clamped in such a manner that significant leaks around the filter do not occur at differential pressures up to approximately 50 kPa as described in B.4. The length of the cowl should be 0,5 to 2,5 times the effective filter diameter (the diameter of the exposed circular filter area through which the air is drawn). If it is possible that wind velocities greater than 5 m/s could occur during sampling, use a long cowl with a ratio of length to effective diameter of 2,5.

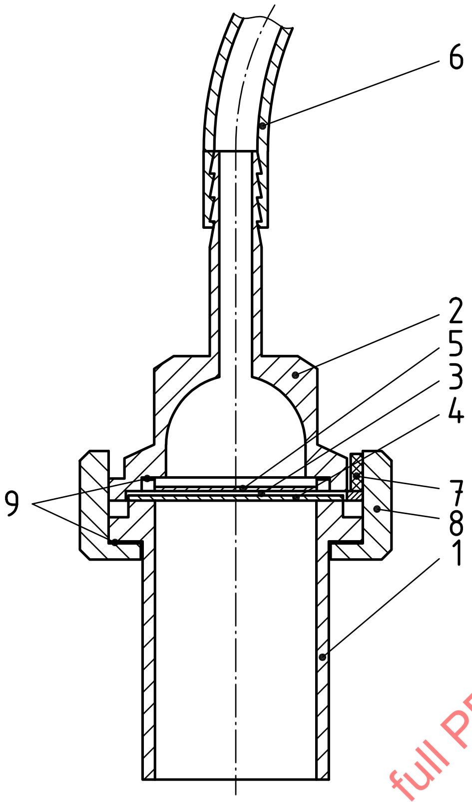
6.1.2 Sampling train

Figure 2 shows an example of a suitable sampling train. Control of the volumetric flowrate can be achieved either by the use of a throttle valve (3) or a volumetric flow controller (8) in conjunction with a regulator valve (4).

6.1.3 Sampling pump

Pulse-free or pulsation-damped, capable of maintaining, at a pressure differential across the filter of at least 50 kPa, a volumetric flow of between 8 l/min and 30 l/min, depending on the diameter of filter used.

In order to achieve the required analytical sensitivity, a flowrate of 8 l/min is required if a 25 mm diameter filter is used. This flowrate is equivalent to a filter face velocity of approximately 35 cm/s. The sampling pump shall be capable of maintaining the intended flowrate within ±10 % throughout the whole sampling period.



Key

1	cowl	6	suction hose
2	filter holder	7	clamping roller
3	backing filter	8	clamping ring
4	track-etched membrane filter	9	PTFE gaskets
5	supporting mesh		

Figure 1 — Example of design of sampling head

6.1.4 Needle valve

With a fine adjustment mechanism, for setting the volumetric flowrate.

6.1.5 Volumetric flowmeter (rotameter)

For measuring the volumetric flowrate.

6.1.6 Timer

For measuring the sampling time.

6.1.7 Dry type gas meter (optional)

For volumetric measurement, calibrated, designed for a maximum volumetric flowrate of 2 m^3/h.

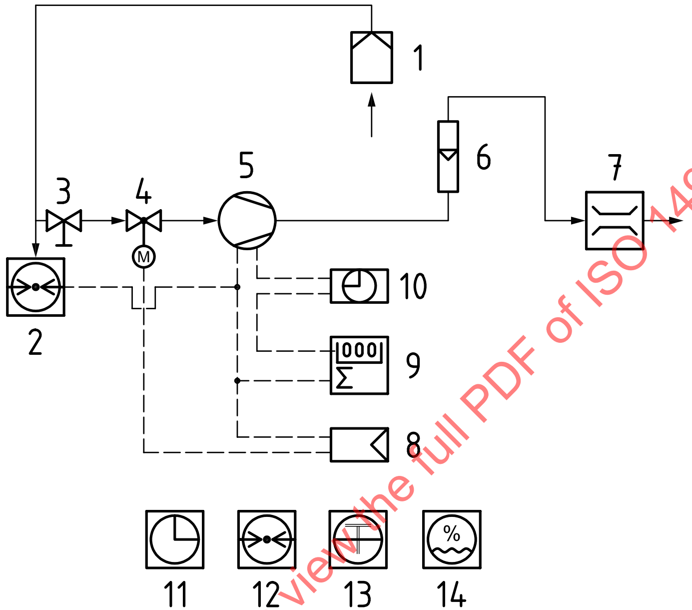
6.1.8 Meteorological instruments (optional)

For recording of meteorological conditions during sampling. Instruments such as a thermometer, a hygrometer, a barometer and a wind speed and direction recorder will be required.

6.1.9 Instruments for unattended sampling (optional)

For unattended sampling, a volumetric flow controller is required for regulation of the flowrate to within ±10 % of the nominal rate, with an automatic switch to turn off the sampling pump if the flowrate exceeds or falls below the pre-set tolerance band. The flow controller can be integrated into the suction unit.

A programmable switch is required for pre-setting the air sampling cycle. A pressure gauge which incorporates a switching contact is required to switch off the sampling pump if the pressure differential across the sampling filter increases to a pre-set value.



Key

1 sampling head or cassette
2 pressure gauge
3 throttle valve (optional)
4 regulator valve (optional)
5 pump
6 variable-area flowmeter
7 gas meter (optional) with thermometer
8 volumetric flow controller (optional)
9 sampling-time recorder (optional)
10 programmer (optional)
11 timer
12 thermometer (optional)
13 barometer (optional)
14 hygrometer (optional)

Figure 2 — Example of a suitable sampling train

6.2 Preparation of filters

6.2.1 Vacuum evaporator

Capable of producing a vacuum better than 0,013 Pa.

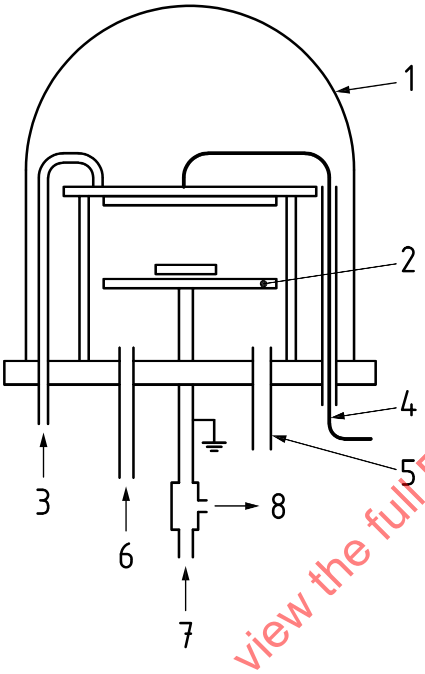
A vacuum coating unit is required for vacuum deposition of gold onto the capillary-pore membrane filters, and for carbon coating of SEM specimens if the particulate loading is such that excessive charging of the specimen occurs.

A sputter coating unit has also been found to meet the requirements for gold coating of the capillary-pore filters.

6.2.2 Plasma asher

Supplied with oxygen, to oxidize organic particles on the SEM specimen.

An example of the configuration of a suitable plasma asher is shown in Figure 3. The chamber of the plasma asher may be coupled either capacitatively or inductively. Care shall be taken not to damage the specimen during the plasma ashing process. A calibration procedure to determine suitable operating conditions for the plasma asher is described in B.3.



Key

1	bell jar
2	filter in mounting ring
3	oxygen inlet
4	power supply from plasma generator
5	connection for vacuum pump
6	air inlet
7	cooling-water inlet
8	cooling-water outlet

Figure 3 — Example of a configuration of a plasma asher

6.3 Sample analysis

6.3.1 Scanning electron microscope (SEM)

With an accelerating voltage of at least 15 kV, is required for fibre counting and identification.

6.3.2 Energy-dispersive X-ray system

For the SEM, capable of achieving a resolution better than 140 eV (FWHM) on the MnK_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. 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.

The instrumental combination shall satisfy the minimum requirements with regard to the visibility of fibres, as specified in 7.4.1, and identification of the fibres, as specified in 7.4.3.

6.3.3 Stereo-microscope

With a magnification of approximately 20×, for visual examination of the particulate deposit on the filter.

6.3.4 Gold-coated capillary-pore polycarbonate filters

Maximum nominal pore size 0,8 µm, for collection of air samples. The gold coating shall be approximately 30 nm thick applied to the shiny side of the filter. The procedure for preparation of the gold-coated filters is described in Annex A.

NOTE Optionally, a 20 nm thick layer of gold can be evaporated on to the reverse side of the filter. This coating serves to protect the filter during plasma ashing and can help to improve the contrast of fibres in the SEM image.

6.3.5 Backing filters

Cellulose ester membrane, or absorbent pads, with a porosity of approximately 5 µm to be used as diffusing filters behind the sample collection filters.

6.3.6 Disposable plastic field monitors (optional)

If disposable plastic field monitors are used, they shall consist of 25 mm to 50 mm diameter, three-piece cassettes, which conform to the requirements of 6.1.1. The cassette shall be loaded with a gold-coated, capillary-pore polycarbonate filter of maximum nominal pore size 0,8 µm, backed by a cellulose ester filter of 5 µm porosity or an absorbent pad. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly so that significant air leakage around the filter cannot occur.

Re-use of disposable plastic field monitors is not recommended.

6.3.7 Technically pure oxygen

For operation of the plasma asher.

6.3.8 Rubber connecting hoses

For connecting the sampling head to the pump, and other equipment in the sampling train.

The hose shall have a wall thickness such that it does not collapse under a vacuum of 50 kPa. Silicone rubber hose has been found to meet the requirements.

6.3.9 Filter containers

For transport and storage of filters if disposable field monitors are not used.

6.3.10 Routine electron microscopy tools and supplies

Fine point tweezers, scalpel holders and blades, double-coated adhesive tape, SEM specimen stubs and colloidal carbon paint and other routine supplies are required. If a vacuum evaporator is used for preparation of gold-coated filters, gold wire and tungsten filaments are required. For carbon evaporation, spectroscopically pure carbon rods and a means of sharpening the rods is required.

6.3.11 Sample for resolution adjustment

A gold-coated polycarbonate filter, on which chrysotile fibres with a width of ≥0,2 µm have been deposited, is required for adjustment of the operating conditions of the SEM.

6.3.12 Sample for magnification calibration

A test sample is required 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 Air sample collection and analysis

7.1 Measurement planning

When determining the spatial and temporal scope of the measurements, it is important to take into consideration the special aspects of the situation. It is therefore essential to define the objective of the measurements before samples are collected. Any available information on emission sources, meteorological conditions and the local situation should be taken into account in order to obtain the maximum information from the measurements. The number of individual measurements to be made should be selected according to the particular task. In particular, prior to collection of the samples, the required accuracy for the mean concentration of the inorganic fibres should be specified, since the error of each individual measurement needs to be taken into consideration in determining the number of samples to be collected. Measurement uncertainty is discussed in Clause 9.

7.2 Collection of air samples

Figure 2 shows an example of a sampling train. Position the sampling head approximately 1,5 m above ground level.

If a re-usable sampling head is used, place a 5 µm porosity cellulose ester backing filter on to the filter support in the sampling head. Place a gold-coated filter on top of the backing filter, with the shiny side facing into the direction of the airflow. Clamp the filters in the sampling head so that the gold-coated filter lies flush against the backing filter and is tightly fitted. Ensure that damage does not occur to the filter during clamping, and that the filter is not twisted.

Before air sampling is commenced, perform a brief test with the tube to the sampling head closed, to determine if any leaks exist in the complete sampling system. Under the conditions of this test, the flowrate indicated by the volumetric flowmeter shall be less than 10 % of the unimpeded flowrate. Open the tube only after the pump has been switched off, in order to avoid sudden pressure surges.

Leaks around the filter can also occur if the filter is inadequately sealed on the low-pressure side, or if the filter has been damaged. Observation of a lower differential pressure at the start of the air sampling indicates that a serious leak exists. If, after sampling, particulate deposits are observed around the edge of the backing filter or on the unexposed edges of the sampling filter, a leak around the filter has occurred and the sample shall be rejected.

When sampling is to be commenced, start the pump and the timer simultaneously.

Within 2 min of the start of sampling, adjust the volume flowrate to approximately 2 l/min per square centimetre of effective filter area (this value shall not vary by more than ±10 % for the period of sampling). This corresponds to a filter loading of 1 000 l per square centimetre of effective filter area over a sampling period of approximately 8 h. Sampling in an environment with high dust concentrations in the air, the loading of particulate material on the sample collection filter can be too high for adequate analysis after 8 h sampling. In such cases, it is permissible to use a shorter sampling time.

At the end of the sampling period, switch off the sampling apparatus. If a programmer was used, confirm that the sampler operated within the required parameters for the preset sampling period. Taking care not to disturb the particulate deposits on the filter surface, remove the sample collection filter and store it upright in a dust-tight sample container.

Record all sampling data which can be of significance for later interpretation. An example of a form for recording of air sampling data is shown in Figure 4. The location of the sampling apparatus shall be documented in the form of a sketch and, if possible, a photograph.

In fog, a thick coating (including calcium sulfate fibres) on the sampling filter can occur, resulting in a rapid increase in the pressure differential across the filter. Under these conditions, it will be necessary to take several sequential samples, each collected over a shorter sampling time, in order to obtain filters suitable for analysis. Annex E shows the procedure for calculation of a mean value from the results of several sequential short-term samples.

AIR SAMPLING DATA SHEET: ISO 14966	
Project: No.:	
Sample No.:	
Sampling location:	
Sampling apparatus (type):	
Sampling times:	
Start (date, time):	End (date, time):
Duration (hours, minutes):	
Sampling filter (type):	
Nominal pore size (µm):	Diameter (mm):
Effective diameter (mm):	Effective filter area (mm^2):
Sampling data	
Volumetric meter readings:	
Start (m^3):	End (m^3):
Volumetric throughput (m^3):	
Volumetric flowrate	
Start (m^3/h):	End (m^3/h):
Mean volumetric flowrate (m^3/h):	
Meteorological data (if required)	
Air temperature (°C):	Relative humidity (%):
Wind velocity (m/s):	
Weather characteristics:	
Remarks:	
Sampler (Name): Signature:____________________	Date of analysis:

Figure 4 — Example of a sampling log form for recording of sampling data

7.3 SEM specimen preparation

Before sample analysis, examine the uniformity of the particulate deposit on the filter. If the particulate deposit shows evidence of non-uniformity, reject the filter.

If the particulate deposit is uniform, place the filter into the holder of the mounting ring, and position it in the plasma asher, as shown in Figure 3. The plasma ashing treatment removes the majority of the organic material on the filter, and this considerably facilitates the SEM analysis of the sample. Adjustment of the plasma asher see Annex B.3.

The rate of oxidation of the organic material on the filter by the oxygen plasma is enhanced by the electrical conductivity of the filter and the sample holder. Under the specified operating conditions, the plasma ashing treatment is generally completed after approximately 30 min. After the plasma ashing treatment, either the whole filter or a part thereof is mounted on an SEM specimen stub, without any further preparation, for SEM analysis. For adjustment of the plasma asher see Annex B.3.

NOTE 1 The portion of the filter to be analysed can be mounted on the SEM specimen stub either before or after the plasma ashing treatment.

NOTE 2 Double-sided conductive adhesive tape has been found to be an effective means of mounting the filter.

If, during SEM analysis, fibres are detected which appear to be organic, the plasma ashing treatment can be repeated to remove them.

In exceptional cases, it may be necessary to evaporate a thin film of carbon onto the SEM specimens in order to reduce localized charging, increase the contrast, and thus improve the visibility of fine fibres. This will generally be required only when the filter has a very heavy particulate loading.

7.4 Analysis in the scanning electron microscope

7.4.1 General instructions

Examine the filter sample at an accelerating voltage of 15 kV or 20 kV and an image magnification of between 2 000× and 2 500×. For fibre classification in the SEM, an accelerating voltage of 15 kV is recommended.

Adjust the SEM such that fibres with a width of approximately 0,2 µm are just visible at a magnification of 2 000×. 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 2 000× used for fibre counting. The width of this fibre is then confirmed by measuring it at a magnification of 10 000×. This adjustment shall be carried out on at least two separate fibres before the analysis is started, and it shall be repeated several times during the course of a series of analyses to ensure that the fibre visibility conditions have not changed.

Position the X-ray detector such that it subtends the largest possible solid angle at the specimen surface. The sample shall not be tilted to an angle greater than 20° when counting and sizing the fibres.

Select the operating parameters of the SEM and the X-ray detector system so that a statistically acceptable X-ray spectrum shall be acquired from a chrysotile fibre of 0,2 µm width on the test sample within a maximum period of 100 s. (For calibration and adjustment of the SEM and see B.1 and B.2).

The criteria for statistical acceptability require, for peak height, *P*, and background level, *B*:

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

with a minimum of 30 pulses in the channel corresponding to the maximum peak height for each of the magnesium and silicon peaks[9], and

$$\frac{P+B}{B} > 2 \tag{2}$$

for each of the magnesium and silicon peaks.

During analysis, each selected image field is examined for fibres of the length and width ranges specified in 7.4.2. Using EDXA, these fibres are then classified into compositional groups according to the criteria specified in 7.4.3. The sequential number of the image field, the fibre length, the fibre width, the elemental composition and the fibre classification are recorded on a fibre counting form. An example of a suitable fibre counting form is shown in Figure 5. In order to document the appearance and particulate loading of the sample, three micrographs shall be taken of each sample and attached to the fibre-counting form.

7.4.2 Fibre-counting criteria

7.4.2.1 General

Examine at least 50 image fields in order to reduce, to the extent that is possible, the effect of fluctuations in the filter loading density on the counting result. Select image fields to be evaluated in such a way that the whole area of the sample is taken into account and overlapping of the image fields does not occur. Count fibres in accordance with the specifications in 7.4.2.2 to 7.4.2.6, and the examples shown in Figure 6.

FIBRE Counting Form: ISO 14966						
Sample No.:			**Date:**		**Page No.:**	**Name:**
Calcium sulfate	**Tally list:**				**Rejected structures**	**Tally list:**
Number of image fields	**Fibre No.**	**Image field No.**	*L* µm	*D* µm	**Elemental composition**	**Fibre type**
Tally list:	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					
	21					
	22					
	23					
	24					
	25					
	26					
	27					
	28					
	29					
	30					
Totals	Chrysotile type fibres: Amphibole fibres: Other inorganic fibres: Calcium sulfate:			Number rejected: Bundles: Clusters: Matrices:		
				Number of fibres without spectrum:		
Total number of image fields: Number of image fields rejected. Calibrated magnification:				Micrograph numbers:	1: 2: 3:	

Figure 5 — Example of fibre counting form

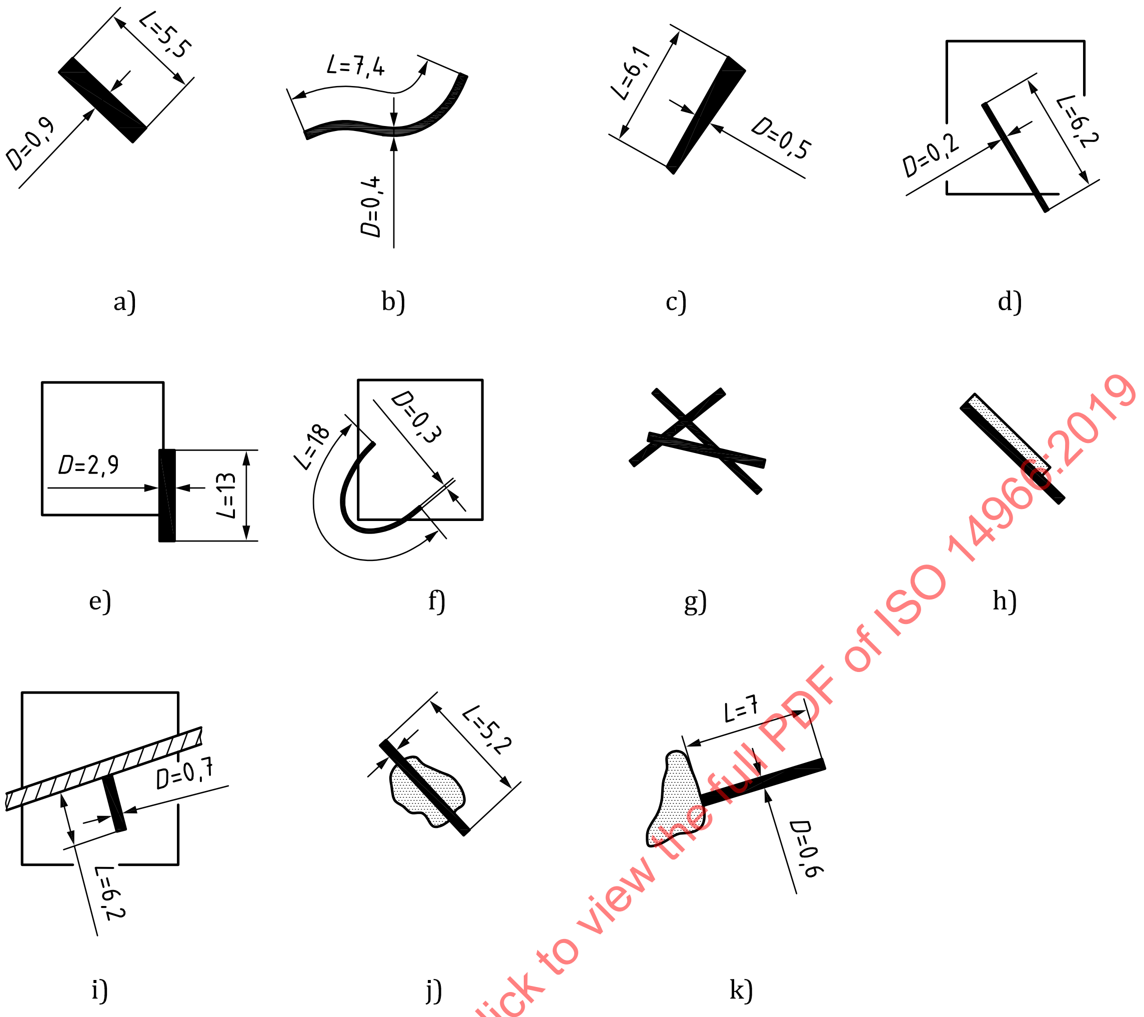


Figure 6 — Schematic examples illustrating the use of the fibre counting rules (L and D are not true to scale in the figures)

Count results:

a) 1 fibre

b) 1 fibre

c) 1 fibre

d) ½ fibre

e) 0 fibres (None of the fibre ends is to the greater part or completely within the image)

f) 1 fibre (both fibre ends within the image)

g) 3 fibres (The fibres can be distinguished and meet the *L* and *D* criteria)

h) 2 fibres (The fibres can be distinguished and meet the *L* and *D* criteria.

i) 1 fibre (The ends of one fibre are outside of the image)

j) 1 fibre (The non-fibrous particle is ignored)

k) 1 fibre (The non-fibrous particle is ignored)

7.4.2.2 Countable fibre

Elongated particle with a length L > 5 µm, a width D < 3 µm and a length/width ratio L:D > 3:1.

NOTE 1 This definition applies to particles irrespective of their origin, for example grown fibres or fibrous fragments (see Annex C).

NOTE 2 The width is regarded as being the width in the plane of the image, irrespective of whether the fibre is cylindrical or prismatic.

7.4.2.3 Bundle

Fibre agglomerate consisting of several fibres arranged in parallel and/or superimposed and/or intersecting.

7.4.2.4 Split fibre

Fibre agglomerate which appears compact and undivided at one or more places along its length but appears separated into different threads at other places.

NOTE The width is defined in the undivided part of the agglomerate.

7.4.2.5 Counting criteria relating to the image fields

- Fibres with both ends in the image field are counted.
- Fibres with only one end in the image field are counted and given the weighting factor 0,5.
- Fibres with both ends outside the image field are not counted.
- If more than approx. one eighth of the area of an image field is covered by fibres and/or particles, the image field shall be rejected due to overloading.
- If more than 10 % of the image fields of a filter sample are overloaded, the sample shall be rejected.

Figure 7 illustrates the percentage coverage of the image field with particles (approx. one eighth).

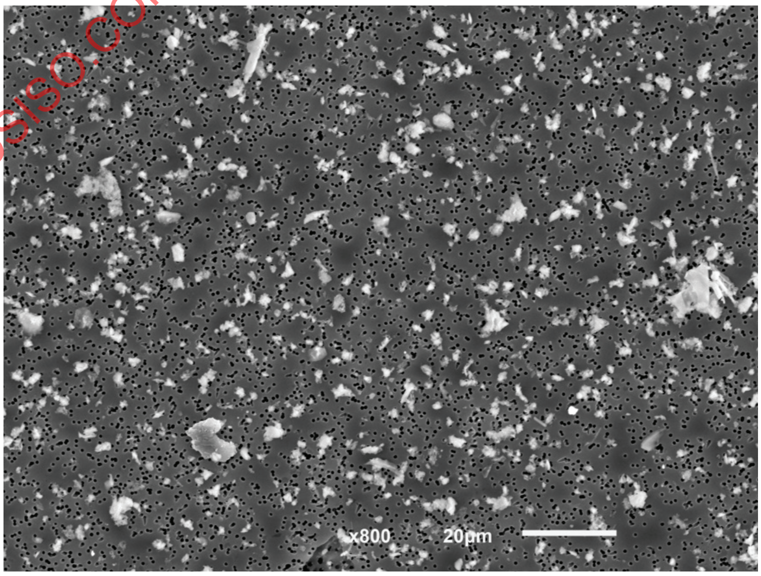


Figure 7 — Example of the coverage of an image field

7.4.2.6 Geometric and morphological counting criteria

- Fibres conforming with the definition of the countable fibre are counted.
- A split fibre is to be assessed like a fibre if it conforms with the definition of the countable fibre.
- Fibres in a fibre bundle are counted singly if they can be clearly distinguished from one another (both fibres end visible).
- A fibre bundle is to be assessed like a fibre if the individual fibres cannot be distinguished from one another, and if the complete fibre bundle conforms with the definition of the countable fibre. The presence of unresolved fibre bundles shall be noted in the fibre count form.
- Where fibres are in contact in such a way that the end of one fibre is concealed by the other fibre, only the visible part is included in the measurement.
- Where fibres are in contact with non-fibrous particles, the particles shall be ignored. If ends of fibres are concealed by the particles, only the visible parts of the fibres are measured.

A situation can arise in rare cases where fibres on a filter sample cannot be made to conform with the counting rules. Observations of this type shall be documented separately. They can normally be assessed on the basis of experience in relation to the overall situation on the filter.

If fibres with a width D < 0,2 µm are included in the fibre count, these shall be listed separately in the fibre counting form. The fibres are then divided on the basis of the width into the two categories D ≥ 0,2 µm and D < 0,2 µm.

Fibres with D < 0,2 µm shall be registered separately since the international reference method for determining the fibre number concentration is based on an optical microscopy method which is limited to the detection of fibres with D ≥ 0,2 µm. For this reason, only fibres with D ≥ 0,2 µm are included in the measurement result (see Clause 8).

Asbestos-containing objects not included in the count shall be noted in the fibre counting form.

7.4.2.7 Termination of fibre counting

Continue the examination until completion of the field in which the 50th inorganic fibre (other than calcium sulfate fibres, including asbestos fibres) occurs. If, after examination of 50 image fields, 50 fibres have not been detected, further fields shall be examined until either a total of 50 inorganic fibres has been counted or sufficient area has been examined to achieve the desired analytical sensitivity. For most applications, it is recommended that at least 1 mm^2 of the filter area be examined.

Fibre counting can be terminated early with respect to a fibre type as a function of a limit or guide value K_R (fibres per m^3). If more than N_A fibres of this type have been found fibre counting can be terminated. N_A is calculated as follows:

$$N_A = \frac{3 \cdot K_R \cdot V_s'}{F_A} \quad (3)$$

where

N_A is the fibre number of a specific type;

K_R is the benchmark or limit to be tested, in m^{-3};

V'_S is dependent from V_S the sampled volume of air per filter area:

$V'_S = 1\ \text{m}^3/\text{cm}^2$ for $V_S \leq 1\ \text{m}^3/\text{cm}^2$;

$V'_S = V_S$ for $V_S > 1\ \text{m}^3/\text{cm}^2$;

F_A is a constant (= 100 cm^{-2}).

In cases where limit or guide values do not exist, the stop criterion can be freely defined for each individual measurement in accordance with the specific measurement objective. Here it is essential that on account of the associated measurement uncertainty N_A is not set too low. N_A shall be at least 15. The analysed filter area shall not be less than 0,25 mm^2.

7.4.3 Fibre classification

7.4.3.1 General

Two different strategies can be used for fibre identification: analysis based on reference spectra and peak height ratios normalised for Si (see 7.4.4), or analysis via the signal-to-background (*S/B*) ratios in the EDX spectrum (see 7.4.3.2 to 7.4.3.8). The method based on comparison with reference spectra is recommended in the case of detectors that are suitable for analysing light elements ($Z \geq 5$), e.g. SLEW (super light element window). For detectors with beryllium windows, analysis via the signal-to-background ratio is more suitable.

Comparison with reference spectra of the relevant asbestos types, recorded under comparable conditions, is preferable to asbestos identification via the *S/B* ratios. It serves, in particular, for avoiding false positive results, which can occur more frequently with the *S/B* ratio method.

Since asbestos spectra exhibit specific differences which derive from the equipment (e.g. the detector), the instrument's age, the adjusted instrument parameters (e.g. accelerating voltage, working distance, tilt angle, beam current) or the sample's preparation (any carbon or gold coating), the comparison spectra of particular asbestos types shall be recorded with the same equipment and the same parameters as the samples' spectra.

NOTE In this method, the term “fibre classification” is used, rather than “fibre identification”, to distinguish between a definitive identification based on a combination of morphology, chemical composition and crystal structure, and the result obtained by this method in which fibres are presumed to be asbestos if they exhibit an EDXA spectrum consistent with one of the asbestos varieties. If airborne fibres detected by this method exhibit EDXA spectra consistent with bulk materials present at the site where the air samples were collected, and these bulk materials have already been identified as one of the asbestos varieties by polarized light microscopy or transmission electron microscopy, the presumption that the fibres observed are asbestos is much stronger. In addition, morphology of the fibres in the SEM can be used to strenghten the presumption.

Fibres are classified into four categories:

a) fibres with chemical compositions consistent with those of serpentine asbestos;

b) fibres with chemical compositions consistent with those of amphibole asbestos;

c) calcium sulfate fibres;

d) other inorganic fibres.

The “other inorganic fibres” category includes all fibres which cannot be classified as either asbestos or calcium sulfate, but which do exhibit spectra indicating that they are of inorganic compositions.

It is important to recognize that, during acquisition of an EDXA spectrum from a fibre, scattering of the electron beam can result in emission of X-rays from particles attached to, or in close proximity to the fibre being analysed. The EDXA spectrum obtained can therefore contain contributions from these particles, and the spectrum may 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.

For analysis via S/B ratios use the criteria given in 7.4.3.2 to 7.4.3.8 to classify the spectra.

7.4.3.2 Serpentine (chrysotile)

Classify a fibre as serpentine (chrysotile) if:

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

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 can 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.

7.4.3.3 Amosite

Classify a fibre as amosite if:

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

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

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

7.4.3.4 Crocidolite

Classify a fibre as crocidolite if:

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

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

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

7.4.3.5 Tremolite or actinolite

Classify a fibre as tremolite or actinolite if:

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

b) a peak from Fe may be present (Tremolite) or is present (Actinolite), Na peak is faint, with $P/B < 1{:}1$.

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

7.4.3.6 Anthophyllite or talc

Classify a fibre as anthophyllite or talc if:

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

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 Using this analytical method, it is not possible to discriminate routinely between anthophyllite with a low iron concentration and talc with a high iron concentration. The fibre morphology can assist in discrimination between anthophyllite and talc. Ribbon-like fibres are probably talc, whereas straight, rod-like fibres are possibly, but not necessarily, anthophyllite. If fibres of this composition are observed, it is recommended that the sample be evaluated using transmission electron microscopy.

7.4.3.7 Calcium sulfate

Classify a fibre as calcium sulfate if:

— the Ca and S peaks are clear, with $(P + B)/B > 2$.

NOTE Depending on any adjacent or attached particles, peaks from other elements can be visible.

7.4.3.8 Other inorganic fibres

Classify a fibre as an other inorganic fibre if it yields a spectrum containing any combination of elements which cannot be classified into categories 7.4.3.2 to 7.4.3.7.

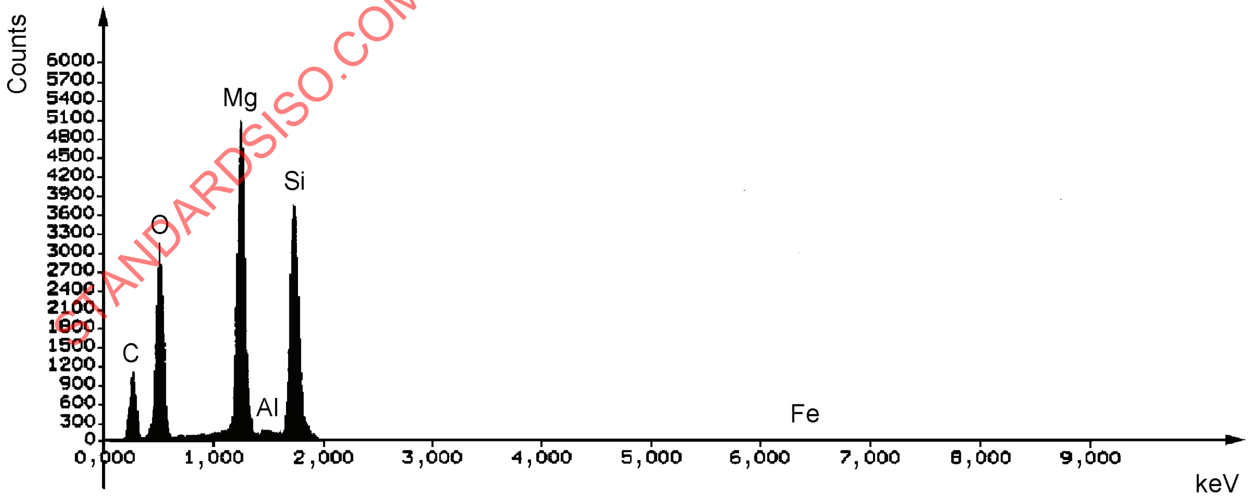
NOTE 1 On the basis of the above criteria, silicate fibres can be classified only as those exhibiting chrysotile-like or amphibole-like elemental spectra (asbestos), calcium sulfate and other inorganic fibres. This procedure can result in an over-estimation of the asbestos fibre content[6].

7.4.3.9 Fibres which exhibit no definitive X-ray peaks

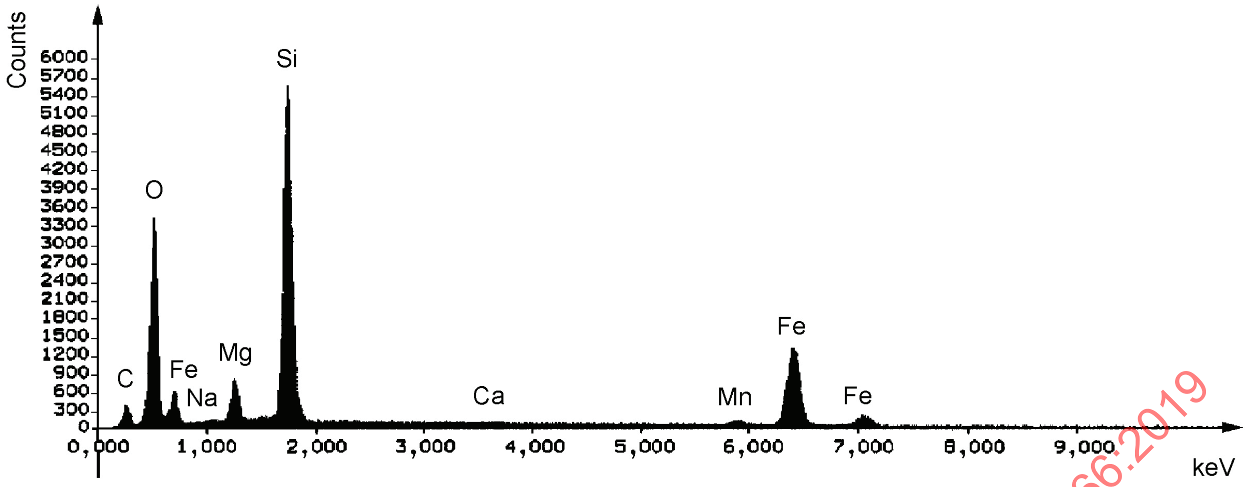
Observation of fibres which yield no definitive X-ray peaks in the EDXA spectrum can be interpreted as an indication that residual organic material is still present after ashing. Very fine inorganic fibres with widths less than 0,2 µm usually do not yield statistically significant X-ray peaks.

7.4.3.10 Reference EDXA spectra from standards of the asbestos varieties

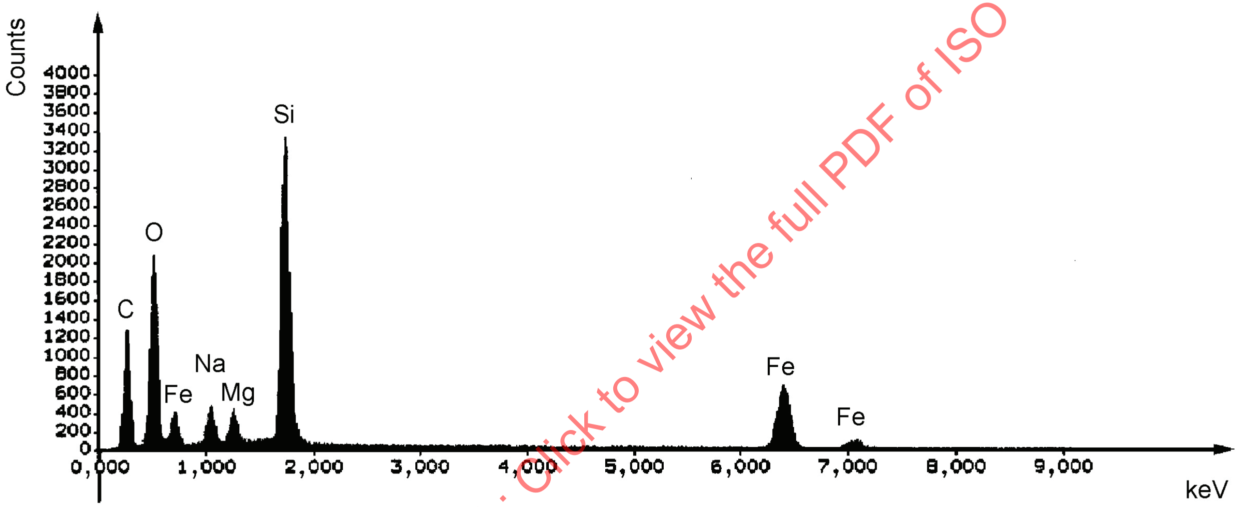
For any particular fibre, the relative heights of the peaks in the EDXA spectrum vary with the characteristics of the X-ray detector. In particular, the detection efficiency for X-ray peaks from low atomic number elements is higher for ultra-thin window detectors than it is for standard beryllium window 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 8. These spectra are used for comparison purposes in the classification of fibres. Since the performance of the EDXA detector can change with time, new reference spectra shall be obtained at appropriate intervals, and particularly after any maintenance of the detector has been carried out.



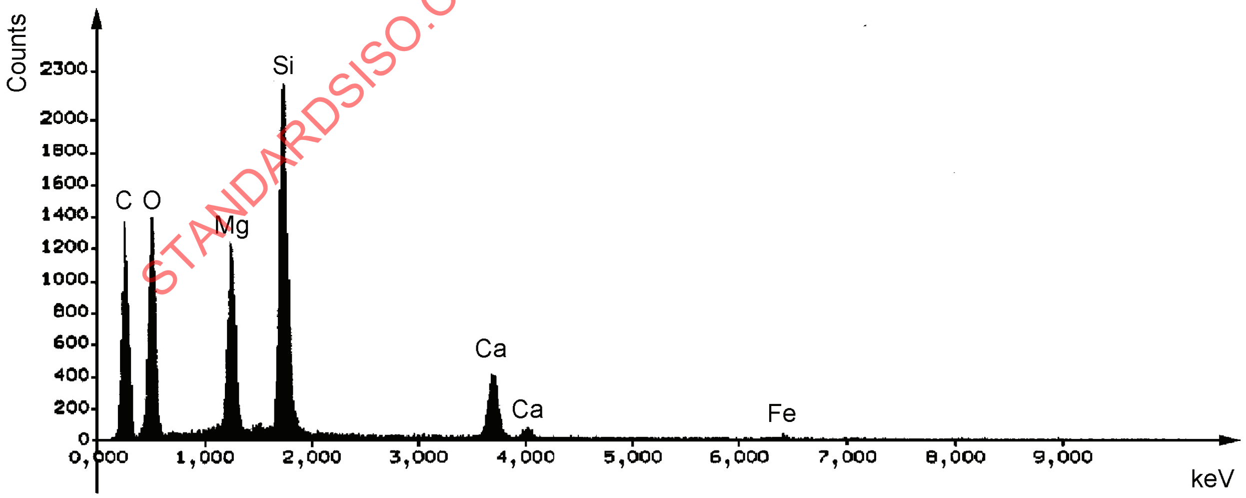
a) Fibre of reference chrysotile (no gold coating)



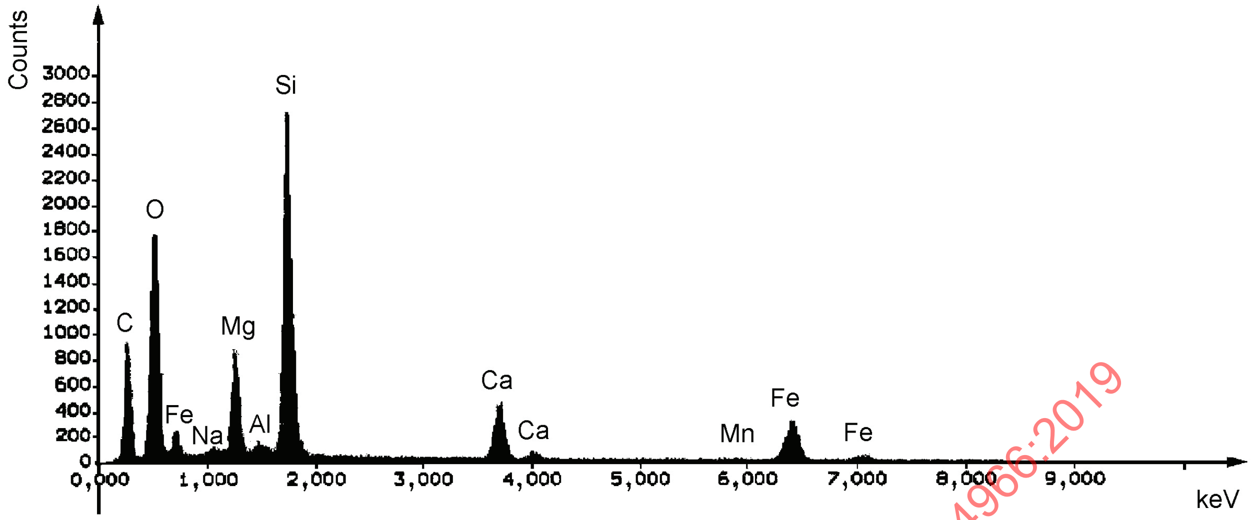
b) Fibre of reference amosite (no gold coating)



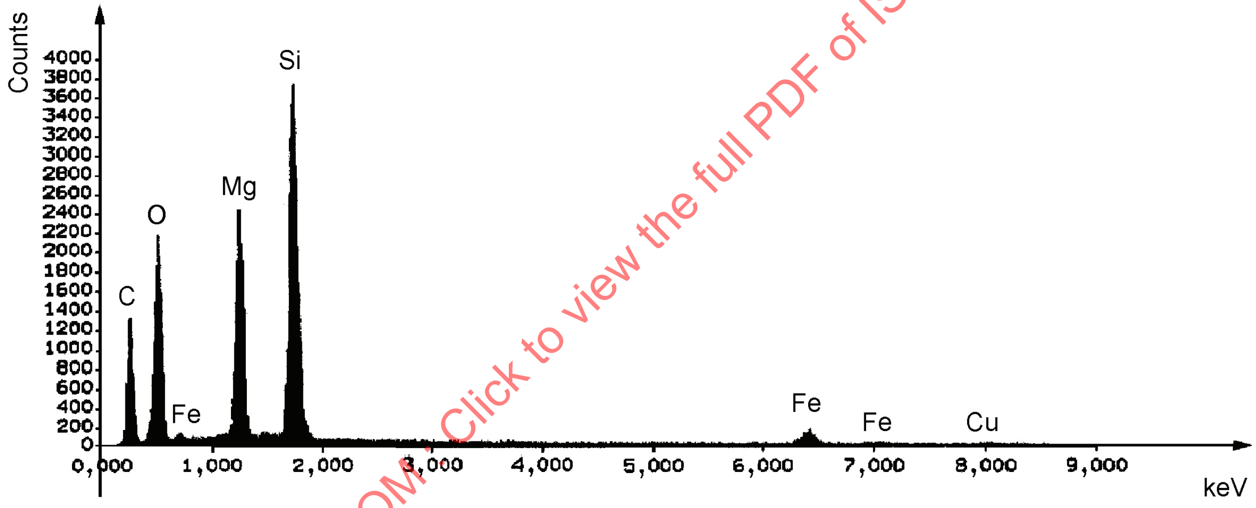
c) Fibre of reference crocidolite (no gold coating)



d) Fibre of reference tremolite (no gold coating)

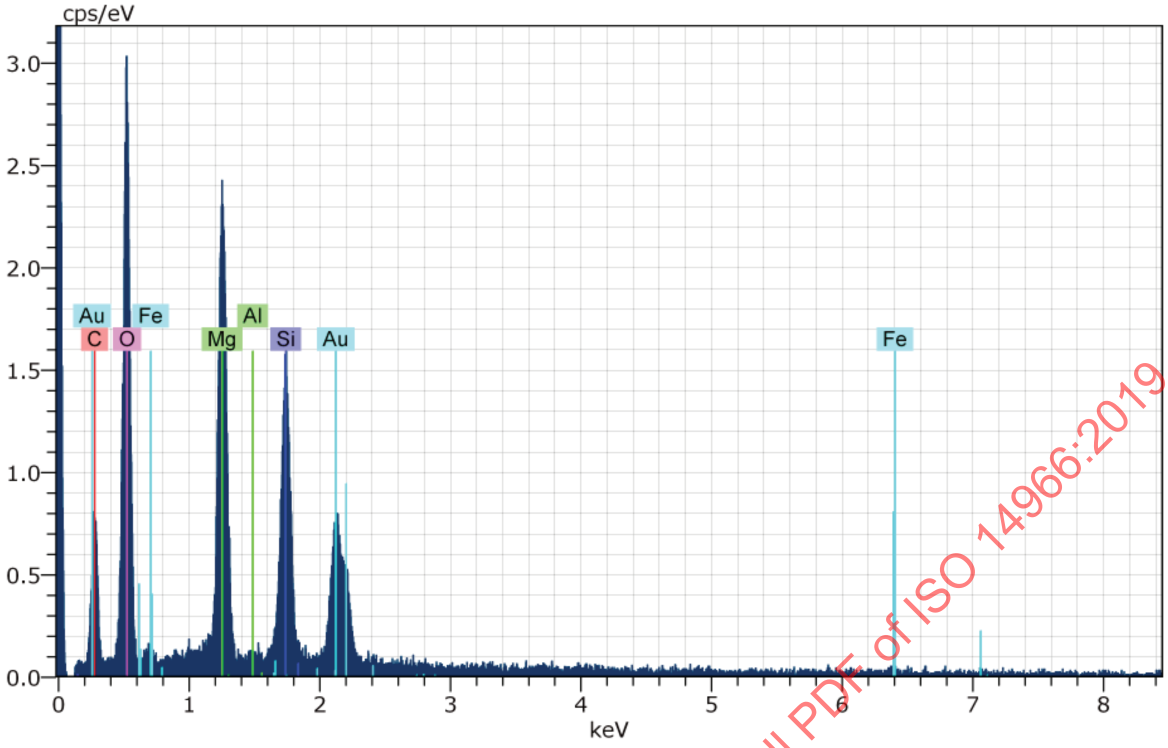


e) Fibre of reference actinolite (no gold coating)

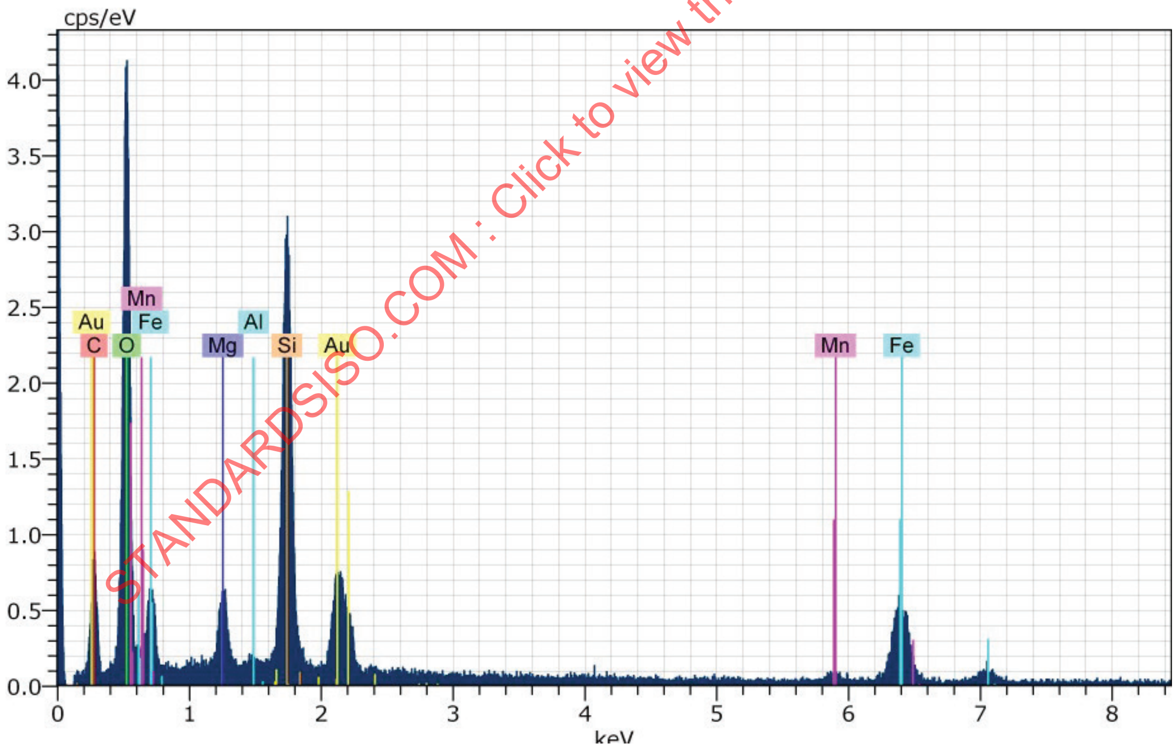


f) Fibre of reference anthophyllite (no gold coating)

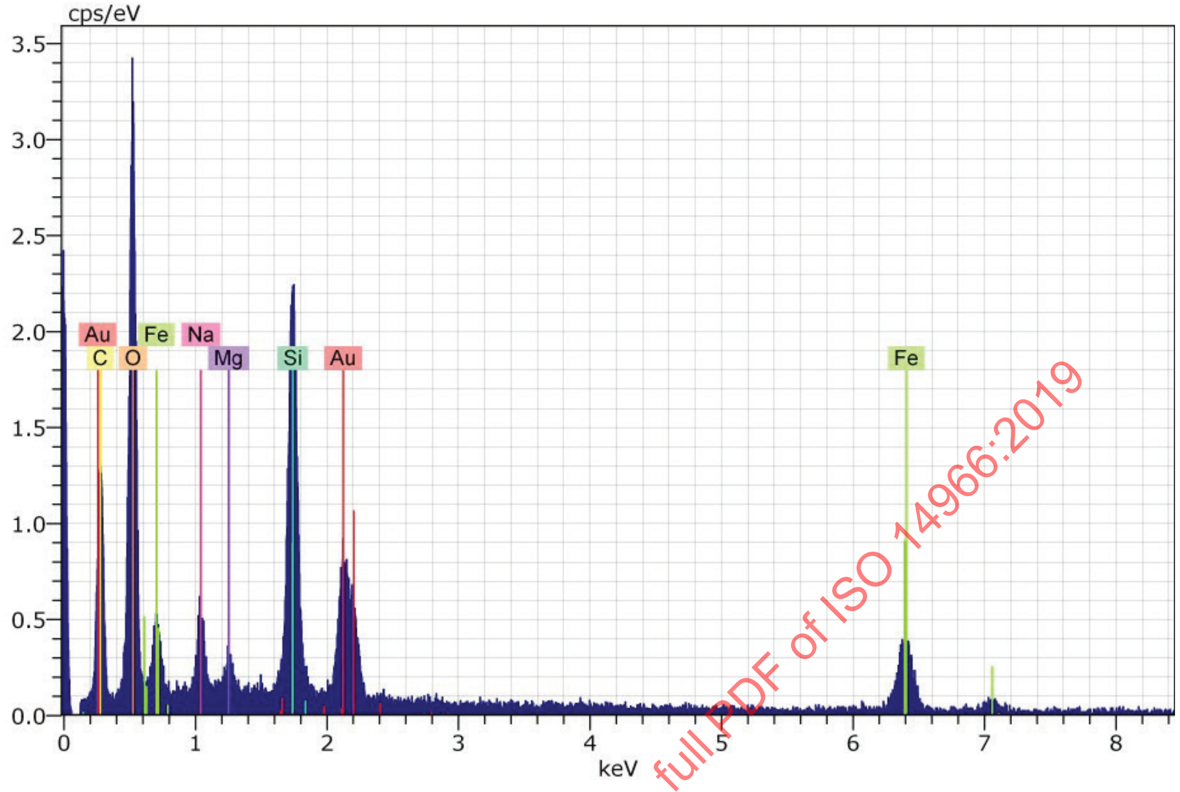
Figure 8 — EDXA spectra of fibres of reference materials



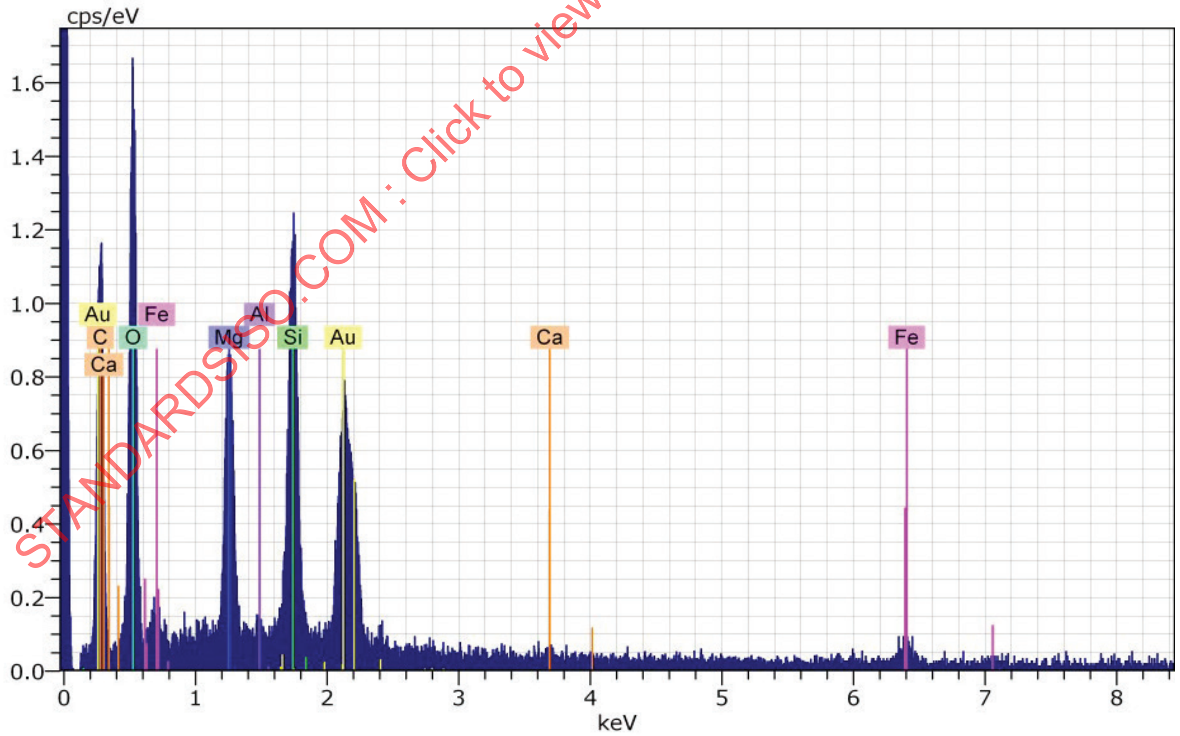
a) Fibre of chrysotile on a gold-coated filter



b) Fibre of amosite on a gold-coated filter



c) Fibre of crocidolite on a gold-coated filter



d) Fibre of Anthophyllite on a gold-coated filter

Figure 9 — Examples of EDXA spectra from asbestos fibres on gold-coated filters. The spectra were obtained from fibres (of different width) detected in actual air samples.

7.4.3.11 Precautions during acquisition of EDXA spectra

During the acquisition of EDXA spectra, care shall be taken to ensure that the electron beam is stable, that the point of incidence is on the fibre and that the beam does not drift off the fibre 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 fibre with a minimum of interference.

In some cases, it is not possible to classify a fibre 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 fibres by an asterisk and indicate the reason on the fibre counting form.

7.4.3.12 EDXA spectra collected from actual air samples

Examples of EDXA spectra collected from chrysotile, amosite and crocidolite fibres detected in actual air samples are shown in Figure 9. Peaks from gold will always be present in these spectra, but their intensities will vary depending on the size of the fibre from which the spectrum is being acquired, and other factors relating to the interaction of the electron beam with the gold-coated substrate. Other peaks present in the spectra, and variations in relative peak intensities, can be attributed to adjacent or attached particles.

7.4.4 Analysis using reference spectra and peak height ratios

This method requires the recording of reference spectra under the conditions quoted above, in order to produce a lab-specific table which in analogy with Table 1 lists the limiting criteria of the peak height ratios for identifying asbestos fibres[10]. Figure 10 illustrates these limiting criteria[11].

The range of the peak height ratios quoted in Table 1 (15 kV accelerating voltage) can serve as a template. The peak height ratio obtained from real and standard asbestos samples should be used initially as an average value. In case of doubt, the use of quantitative methods is advisable[12]. On this basis, the range should then be corrected where necessary.

In addition to the Mg/Si ratio, the Fe/Si and Ca/Si ratios play a particularly important part in the classification of asbestos types. The Na/Si ratio is important in the classification of crocidolite only.

Table 1 — Examples of criteria for the classification of asbestos varieties based on the ratio of selected peak heights

	Mg/Si	Fe/Si	Ca/Si	Na/Si
Asbestos				
Amosite	0,1 to 0,25	0,12 to 0,3	<0,08	<0,05
Anthophyllite	0,6 to 0,8	<0,04 to 0,1	<0,08	<0,05
Chrysotile	1,2 to 1,9	<0,04	<0,08	<0,05
Crocidolite	<0,25	0,1 to 0,3	<0,08	>0,08
Tremolite	0,5 to 0,9	<0,04	0,11 to 0,3	<0,05
Actinolite	0,2 to 0,7	0,04 to 0.18	0,11 to 0,3	<0,05
Mineral				
Talc	0,7 to 0,9	<0,04–0,1	<0,08	<0,05

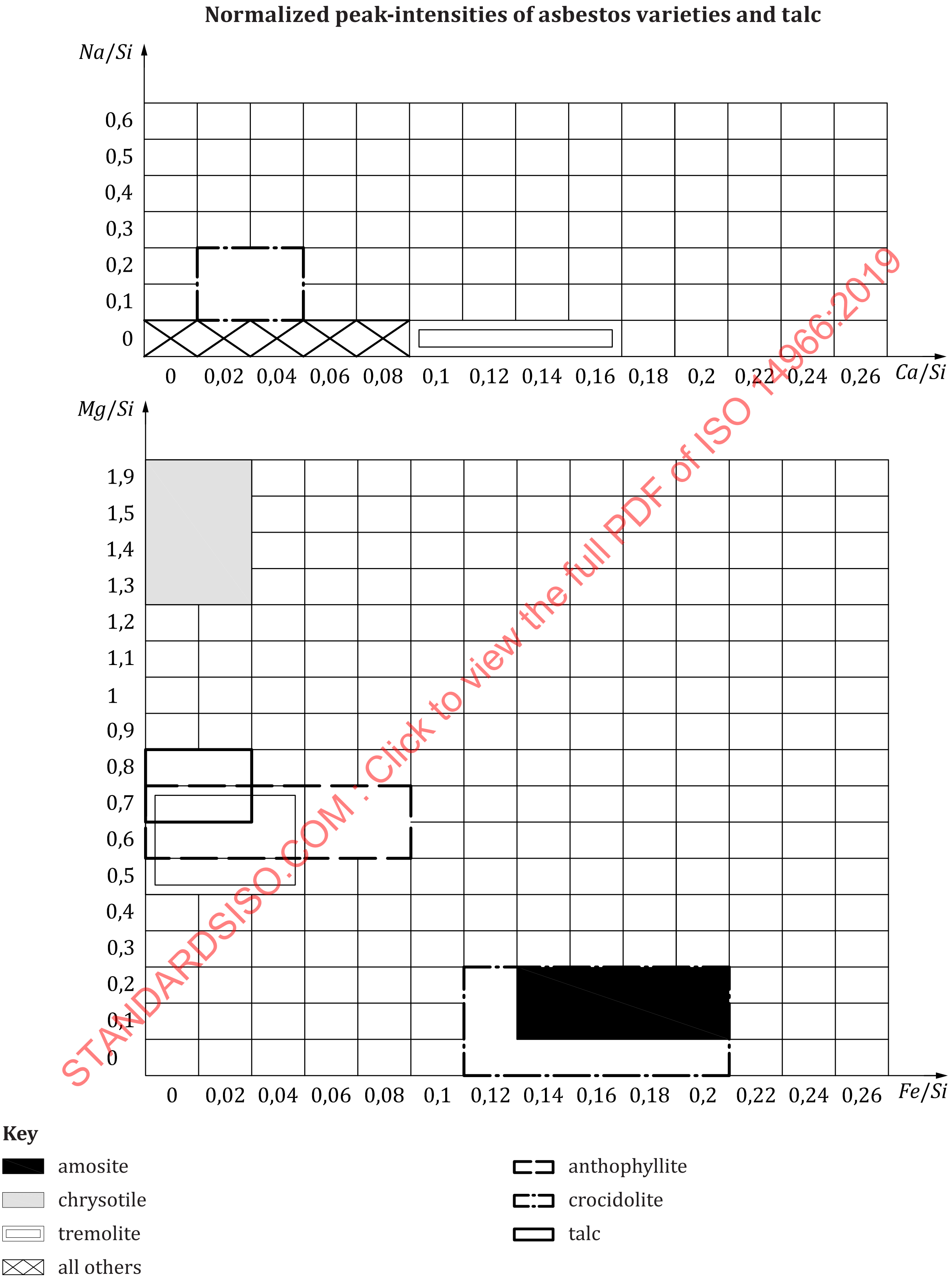


Figure 10 — Examples of limits of the element ratios for asbestos classification

Example:

In the first step, the Mg/Si ratio is examined. Depending on the outcome (usually a visual estimate, which may be confirmed or rejected where necessary by spectral readings), this is followed by considering the Fe/Si or Ca/Si ratio (the figures quoted apply only to this example) in accordance with Table 2. The Al/Si ratio shall always be lower than 0,09.

The Ca/Si ratio, and possibly also the Al/Si ratio, can be affected by the surrounding matrix. Therefore, despite careful choice of the analysis point, exceeding the figures quoted here cannot always be ruled out. Since there are statistical fluctuations of the peak heights, in the case of element ratios within the limits quoted here the measurement should be repeated (with extended measurement times if necessary) before a final result is obtained.

Table 2 — Classification of asbestos according to example

1,2 < Mg/Si < 1,9	and	Fe/Si < 0,04	and	Ca/Si < 0,08	result: chrysotile
0,5 < Mg/Si < 0,8	and	0,04 < Fe/Si < 0,11	and	Ca/Si < 0,08	result: anthophyllite or talc
0,5 < Mg/Si < 0,8	and	Fe/Si < 0,04	and	0,11 < Ca/Si < 0,3	result: tremolite
0,7 < Mg/Si < 0,9	and	Fe/Si < 0,04	and	Ca/Si < 0,08	result: talc or anthophyllite
0 < Mg/Si < 0,2	and	0,1 < Fe/Si < 0,3	and	Ca/Si < 0,08	result: amosite, if Na/Si > 0,08 then crocidolite

NOTE Element ratios in Tables 1 and 2 are given for an ultra-thin window detector and can vary depending on the window and the detector used.

7.4.5 Measurement of fibre dimensions

For measurement of the fibre dimensions, particularly the fibre width, it is recommended to increase the magnification to about 10 000× or higher. It is also recommended to acquire the EDXA spectrum at this magnification. Before moving the specimen stage and switching to a higher magnification, the position of a prominent structure in the field of view should be noted, in order to allow correct repositioning of the specimen stage after analysis of the particular fibre has been completed.

7.4.6 Recording of data on the fibre counting form

For each fibre classified as "asbestos" or "other inorganic fibres", record the image field number, fibre number, length and width, elemental composition and fibre classification. On the fibre counting form, each fibre classified as "asbestos" shall be further classified as either "chrysotile" or "amphibole". It is recommended that the "amphibole" category be further divided, to the extent possible, into categories representing compositions consistent with the different varieties of amphibole asbestos.

Count calcium sulfate fibres, but do not measure their dimensions. Enter the count of calcium sulfate fibres on the fibre counting form as a tally list, as shown in Figure 5.

8 Calculation of results

8.1 Calculation of the mean fibre concentration

The measurement obtained by this method is the numerical concentration c_i (in fibres per cubic metre for fibre type i) of inorganic fibres longer than 5 µm, less than 3 µm in maximum width, and which also have minimum aspect ratios of 3:1. Based on the EXDA results, these fibres are classified into groups according to Table 3.

Table 3 — Fibre classification

Fibre classification	**Numerical concentration c_i**
1 Fibres with compositions consistent with those of serpentine asbestos	c_1
2 Fibres with compositions consistent with those of amphibole asbestos	c_2
3 Other inorganic fibres	c_3
4 Calcium sulfate	c_4

Calculate the numerical concentration for fibre classification *i* (*i* = 1, 2, 3, 4) as follows:

$$c_i = \frac{n_i}{N \cdot V_B} \tag{4}$$

where

$$V_B = \frac{4 \cdot F_B \cdot V}{\pi \cdot d_{\mathrm{eff}}^2} \tag{5}$$

and

c_i is the numerical fibre concentration of fibre classification *i*, in fibres per cubic metre;

n_i is the number of fibres counted for fibre classification *i*;

N is the number of image fields examined;

V_B is the sampled air volume, in cubic metres, per image field;

F_B is the area of the image field, in square millimetres;

V is the sampled air volume, in cubic metres;

d_{eff} is the effective filter diameter (diameter of the exposed circular filter area), in millimetres.

The data recorded on the fibre counting form can be used to determine the size distribution for fibres (except calcium sulfate fibres) with aspect ratios greater than 3:1 within the length range from 5 µm to 100 µm, and the width range from 0,2 µm to 3 µm.

Depending on the choice of sampling equipment, the sampled air volume is calculated as the difference between the meter readings of a gas-volume meter at the start and end of sampling, or from the average volume flowrate and sampling time.

The numerical concentration of fibres with chemical compositions consistent with those of the asbestos varieties is then calculated as:

$$c = c_1 + c_2 \tag{6}$$

and the concentration for all inorganic fibres c_T (excluding calcium sulfate fibres) is then calculated as:

$$c_T = c_1 + c_2 + c_3 \tag{7}$$

8.2 Calculation of the 95 % confidence interval

For the number of fibres n_i of classification i detected in the SEM examination, obtain the values for the lower and upper 95 % confidence limits, λ_L and λ_U, from Table 4. Convert these values into numerical fibre concentrations using Formulae 8 and 9:

$$c_i^L = \frac{\lambda_L}{N \cdot V_B} \tag{8}$$

$$c_i^U = \frac{\lambda_U}{N \cdot V_B} \tag{9}$$

If n_i fibres of fibre classification i were counted during the examination, then there is a 95 % probability that the numerical fibre concentration will lie within this range.

9 Performance characteristics

9.1 General

Asbestos fibre concentrations measured in ambient air are generally of the order of less than 1 000 fibres/m^3, and mostly less than 100 fibres/m^3 (fibres longer than 5 µm). Consequently, the number of asbestos fibres counted during individual measurements is usually very low[6]. Calculation of the performance characteristics (measurement uncertainty, sampling uncertainty, analysis uncertainty) is therefore based on the total number of asbestos and other inorganic fibres.

9.2 Measurement uncertainty

9.2.1 Systematic errors

Systematic errors in the measured numerical fibre concentration can occur as a result of:

a) sampling (errors in measurement of volume flowrate);

b) SEM specimen preparation (fibre losses during handling and plasma ashing);

c) analysis (adjustment of SEM, fibre counting, measurement and identification).

The most critical items leading to systematic errors are those associated with the SEM examination. These include:

— detection and analysis of thin fibres with widths close to and lower than the calibrated visibility limit of 0,2 µm;

— subjective interpretation of aggregates comprising fibres and isometric particles during fibre counting;

— interpretation of EDXA spectra to classify fibres, particularly for spectra subject to interferences by coatings or adjacent particles.

9.2.2 Random errors

Random variation of the results occurs as a result of Poisson variability, and this is particularly important for low fibre counts. Low fibre counts are often experienced for asbestos fibres longer than 5 µm. The variability associated with counts can be estimated using the Poisson distribution, as described in 9.2.6.

Where requirements are specified with regard to the accuracy of the measuring process, this statistical uncertainty in the results should always be taken into account during the planning of the sampling process and the evaluation of the extent of the measurement process resulting therefrom.

If extreme fluctuations in the fibre concentrations in ambient air occur as a result of, for example, meteorological influences, corresponding variations in the measurements will be found. In this type of situation, the sampling period and the number of individual measurements should be selected during planning of the air sampling in order to minimize the effects of such influences.

The errors described in 9.2.1 to 9.2.4 are defined as relative variables on the basis of experience, expressed as twice the standard deviation, which is approximately equivalent to a 95 % confidence interval. All of the errors specified relate to measurements of fibres longer than 5 µm and shorter than 100 µm.

9.2.3 Errors due to sampling

The sampling error is defined as the scatter of the measured results when using side-by-side identical sampling systems. Based on comparison measurements[13], the relative standard deviation was determined to be:

$$2\sigma_P < 15\ \%$$

9.2.4 Errors associated with the SEM examination

The errors associated with the SEM examination were calculated for the sums of fibres classified as asbestos and as other inorganic mineral fibres (excluding calcium sulfate fibres) from the results of four separate series of measurements in each of which there were five different participating laboratories[8]. From these results, a relative standard deviation of:

$$2\sigma_A \leq 35\ \%$$

was obtained.

From these comparison measurements[8], the relative standard deviation due to the subjective error of the operator, using one laboratory and one sample, was found to be:

$$2\sigma_{SF\,=}\ 15\ \%$$

This shows clearly that the equipment, subjective factors and the characteristics of the preparation are major aspects which determine the total error of the measurement.

9.2.5 Total error of the measurement

Provided that the errors from different sources are independent, the standard deviation for the overall measurement is given by:

$$\sigma_T = \sqrt{\sigma_P^2 + \sigma_A^2 + \sigma_S^2} \tag{10}$$

where

σ_T is the standard deviation for the overall measurement;

σ_S is the standard deviation for the sampling errors;

σ_A is the standard deviation for the analysis errors;

σ_P is the standard deviation for the Poisson variability.

The standard deviation for the combination of sampling and analysis is calculated from σ_P and σ_A to give:

$$2\sigma_V \leq 38\ \%$$

For individual samples, the standard deviation for the Poisson variability shall be combined with these standard deviations to obtain the standard deviation for the measurement. The Poisson variability is dependent on the number of fibres counted and is estimated as in 9.2.6.

9.2.6 Random errors due to fibre counting

Assuming that the numerical fibre concentrations on the filter are low, the probability P of detecting n fibres of a given fibre class during examination of N image fields can be described using the Poisson distribution:

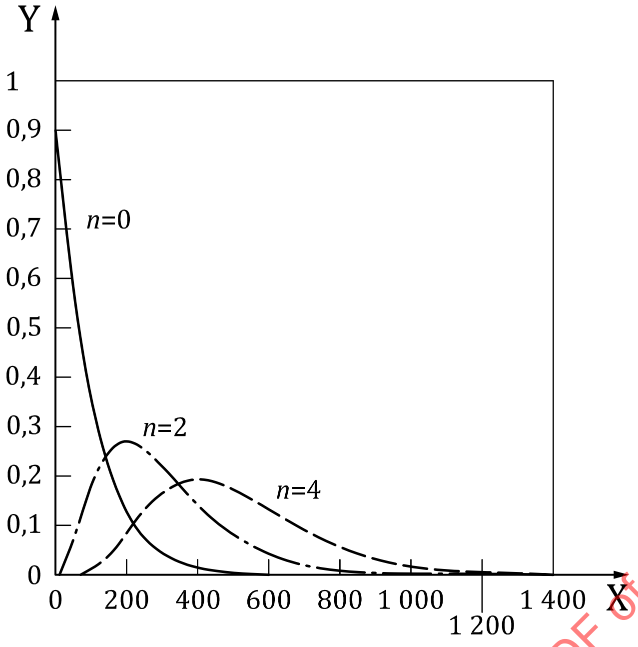
$$P(n,a) = \frac{a^n \cdot \exp(-a)}{n!} \tag{11}$$

The variable a can be regarded as the product of the probability p of finding one fibre of the corresponding fibre class in one image field, and N, the number of image fields examined. It corresponds to the expected value n of the number of fibres detected during examination of N image fields. On the basis of the Poisson distribution, using Table 2, it is possible to determine the 95 % confidence interval for the calculated fibre number concentration.

For illustration, Figure 11 shows the probability density for detection of 0, 2 and 4 fibres in 165 image fields. The abscissa scale has been converted to fibre concentration in fibres per cubic metre, assuming an evaluated air volume of 0,01 m^3. Annex D shows the relation between random error, area of filter analysed and fibre concentration.

Table 4 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Fibre count	Lower λ_L	Upper λ_U	Fibre count	Lower λ_L	Upper λ_U	Fibre count	Lower λ_L	Upper λ_U
0	0	3,689*	20,5	12,607	31,495	41	29,422	55,621
0,5	0,002	4,674	21	13,000	32,101	41,5	29,846	56,197
1	0,025	5,572	21,5	13,393	32,705	42	30,270	56,772
1,5	0,108	6,416	22	13,788	33,309	42,5	30,694	57,346
2	0,242	7,225	22,5	14,183	33,910	43	31,119	57,921
2,5	0,416	8,006	23	14,581	34,512	43,5	31,545	58,495
3	0,619	8,767	23,5	14,978	35,111	44	31,970	59,068
3,5	0,815	9,511	24	15,378	35,711	44,5	32,397	59,641
4	1,010	10,242	24,5	15,777	36,308	45	32,823	60,214
4,5	1,350	10,960	25	16,178	36,905	45,5	33,250	60,786
5	1,624	11,669	25,5	16,581	37,501	46	33,678	61,358
5,5	1,908	12,368	26	16,983	38,097	46,5	34,106	61,929
6	2,202	13,060	26,5	17,388	38,690	47	34,534	62,500
6,5	2,505	13,744	27	17,793	39,284	47,5	34,962	63,071
7	2,914	14,423	27,5	18,199	39,876	48	35,391	63,641
7,5	3,131	15,096	28	18,606	40,468	48,5	35,821	64,211
8	3,454	15,864	28,5	19,013	41,059	49	36,250	64,781
8,5	3,782	16,426	29	19,422	41,649	49,5	36,681	65,350
9	4,115	17,085	29,5	19,831	42,238	50	37,111	65,919
9,5	4,453	17,739	30	20,241	42,827	51	37,973	67,056
10	4,795	18,391	30,5	20,652	43,415	52	38,836	68,191
10,5	5,141	19,038	31	21,063	44,002	53	39,701	69,325
11	5,491	19,683	31,5	21,475	44,589	54	40,566	70,458
11,5	5,844	20,323	32	21,888	45,174	55	41,434	71,590
12	6,201	20,962	32,5	22,301	45,760	56	42,302	72,721
12,5	6,560	21,597	33	22,716	46,344	57	43,171	73,850
13	6,922	22,231	33,5	23,130	46,928	58	44,042	74,978
13,5	7,287	22,861	34	23,546	47,512	59	44,914	76,106
14	7,554	23,490	34,5	23,962	48,094	60	45,786	77,232
14,5	8,024	24,116	35	24,379	48,676	62	47,54	79,48
15	8,396	24,741	35,5	24,796	49,258	64	49,29	81,73
15,5	8,769	25,363	36	25,214	49,839	66	51,04	83,97
16	9,146	25,983	36,5	25,632	50,420	68	52,80	86,21
16,5	9,523	26,602	37	26,051	51,000	70	54,57	88,44
17	9,904	27,219	37,5	26,471	51,579	75	58,99	94,01
17,5	10,285	27,834	38	26,891	52,158	80	63,44	99,57
18	10,668	28,448	38,5	27,312	52,736	85	67,89	105,10
18,5	11,053	29,060	39	27,733	53,314	90	72,37	110,63
19	11,440	29,671	39,5	28,154	53,892	95	76,86	116,13
19,5	11,827	30,280	40	28,577	54,469	100	81,36	121,63
20	12,217	30,889	40,5	28,999	55,045	400	361,76	441,19
* The one-sided upper 95% confidence limit for zero fibres is 2,99								



Key

X fibre concentration, fibres/m^3

Y probability density

Figure 11 — Distribution of the probability density of the fibre concentration for 0, 2 and 4 fibres detected in an analysed air volume of 0,01 m^3

9.3 Limit of detection

The limit of detection is defined as the numerical fibre concentration below which, with 95 % confidence, the actual fibre concentration lies when no fibres are detected during the SEM examination.

The detection limit depends on:

a) the volume of air which passed through the filter during the period of sampling;

b) the effective area of the filter;

c) the area of filter examined.

If no fibres are detected during the SEM examination, Table 2 shows that the upper 95 % confidence limit is 2,99 fibres. The detection limit, E, for the measurement is given in Formula 12:

$$E = \frac{2{,}99}{N \cdot V_B} \tag{12}$$

A limit of detection of approximately 300 fibres/m^3 is obtained with a sampled air volume of 1 m^3 per square centimetre of filter surface area, if an area of 1 mm^2 is examined in the SEM. This corresponds to evaluation of an air volume of 0,01 m^3

Theoretically, the limit of detection can be reduced indefinitely by increasing the area of filter examined in the SEM. For example, the limit of detection can be reduced to approximately 150 fibres per cubic metre if, with a sampled air volume of 1 m^3 per square centimetre of filter area, the SEM examination is extended to an area of 2 mm^2.

Any background contamination by fibres which can exist on unused filters is not taken into account in the quoted detection limit. Experience has shown, however, that background contamination levels of unused filters are negligible compared with the above detection limit.

The limit of detection can also be reduced by increasing the volume of air sampled. However, the extent to which the sampled air volume can be increased can be limited by the concentration of non-fibrous particles in the ambient air. This can result in an increased formation of agglomerates on the filter and consequent obscuration of fibres, or the pores of the filter can become blocked during sampling, leading to an unacceptable increase in the differential pressure across the filter.

The choice of an appropriate sampling time and the necessary extent of the SEM examination are therefore aspects of the measurement which shall be optimized at the planning stage for each particular application.

NOTE The detection limit of a measurement method is usually understood as the signal level that lies significantly above the 'noise level' of the background. As a rule, the significance limit is taken to be 3 times the standard deviation of the background.

This definition, however, cannot be used for the measurement method described in this Standard, since for the visible fibres ($D > 0{,}2\ \mu m$) there is no background or noise level: each individual fibre (or fibre structure) detected and identified as asbestos is unambiguously a signal. The detection limit is, therefore, defined as described above.

10 Test report

The test report shall include at least the following information:

a) reference to this document;

b) identification of the sample;

c) the date, time and location of the air sample collection, and all necessary sampling data, including the volume of air sampled, the sampling time and the effective diameter of the sample collection filter;

d) the analysed air volume;

e) the analytical sensitivity;

f) the number of fibres in each of the fibre classifications detected during the SEM examination, and their calculated numerical concentrations.

It is recommended that the upper and lower 95 % confidence limits be reported for the fibres classified as asbestos and those classified as other inorganic fibres. An example of a test report is shown in Figure 12.

TEST REPORT

"Project title"

Sampling	
Date: "*Date of sample collection*"	Sample volume: 3,8 m^3
Measurement number: "*Number*"	Sampling duration: 8 h
Sampling apparatus: "*Type*"	Effective filter area: 380 mm^2
Type of measurement: "........"	
Location: "*Description of the sampling location*"	
Air sampling by:	

Analysis in the SEM			
Scanning electron microscope: "*Type of SEM*"			
Analysis system: "*Type of EDXA*"		Magnification: 2 000 ×	
Filter area analysed: 1 mm^2		Number of image fields examined: 100	
Image field area: 0,01 mm^2		Number of image fields rejected: 5	
Number of rejected:	Bundles: 1	Clusters: 0	Matrices: 2
Number of fibres without spectrum: 4		Micrograph Numbers: 456, 457, 458	

Results			
Fibre classification	**Number of fibres counted**	**Fibre concentration (fibres/m^3)**	
		Mean	**Poissonian 95 % confidence interval**
Amphibole	6	600	220 to 1 310
Chrysotile	3	300	60 to 880
Total asbestos	9	900	410 to 1 710
Other inorganic	15	1 500	840 to 2 470
Calcium sulfate	8	800	— —

Sampling and analysis performed in accordance with ISO 14966.

Detection of one fibre corresponds to a concentration of 100 fibres/m^3.

The volume of air analysed was 0,01 m^3.

Figure 12 — Example of a test report

Annex A
(normative)

Preparation of filters for air sampling

The membrane filters of a new batch shall be examined in the SEM to establish that any background level of inorganic fibres is sufficiently low that it does not significantly affect the reported results, and that the filters have a uniform pore distribution.

Before sampling, it is necessary to coat the surface of the membrane filters with a gold layer. The gold coating protects the filter during plasma ashing and allows the filter to be examined in the SEM without the increase in the geometric dimensions of the inorganic fibres, which would result if an evaporated film were applied after sampling. The gold coating is applied using either a vacuum evaporator or a sputter-coating unit.

The thickness of the gold coating applied to the side of the filter on which particles are to be collected during air sampling (the smooth and more strongly reflecting side) shall be approximately 30 nm. A uniform thickness of gold coating is required in order to minimize variations in contrast in the SEM image. Optional vacuum deposition of approximately 20 nm thickness of gold onto the other side of the filter protects the sampling filter during ashing and can help to improve the contrast of fibres in the SEM image. If a means of measuring the thickness of the gold coating is not available in the vacuum evaporator, the gold coating can be assumed to be satisfactory when the filter loses its initial dark colour during the course of the evaporation and takes on a typical metallic gold lustre. If, in addition, a coated filter appears to have a green colour when observed in transmitted light, it can be assumed that the thickness of the gold coating is within the required limits.

The filter coating thickness can also be checked easily using the SEM. A number of membrane filters are initially weighed, after which they are coated with gold and weighed again. The mass of the gold coating can be calculated by subtraction, and the thickness can be determined from the area of the filters and the density of gold. Using a constant beam current in the SEM, the height or peak integral for one of the gold X-ray peaks produced by these reference filters can then be compared with the corresponding gold peak produced on a filter prepared for air sampling. The thickness of the gold coating on the sampling filter can be calculated, assuming a linear relationship between the gold coating thickness and the size of the gold peak. After subtracting the background signal, the integral under the gold peak, or the height of this peak, is a direct measure of the thickness of the gold coating.

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 kV to 20 kV and a magnification of between 2 000× and 2 500×. For fibre identification in the SEM, an accelerating voltage of 15 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 (observation screen). The SEM examination is performed directly on the viewing screen, and the magnification calibration shall relate to the viewing screen.

Adjust the SEM in such a way that chrysotile fibres with a width of 0,2 µm and lengths between 5 µm and 10 µm are visible at the counting magnification of approximately 2 000×. This adjustment is performed by selecting a fibre, either on the prepared sample or on a test sample, which is just visible at the magnification used for counting. The width of this fibre is then determined at a magnification of approximately 20 000×. This adjustment shall be carried out on at least two separate fibres before starting the analysis, and shall be repeated several times during the course of a series of analyses.

NOTE 1 On a 30 cm observation screen display, 100 image fields at a magnification of approximately 2 000× correspond to an area of approximately 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 0,2 µm, no severe image degradation relevant to resolution of a 0,2 µm wide fibre longer than 5 µm is observed. With currently-available observation screen display sizes and nominal line numbers of about 500 to 800 at magnifications of 2 000× or 2 500×, the above conditions are usually met.

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:

$$P > 3 \cdot \sqrt{B} \qquad \text{(B.1)}$$

with a minimum of 30 pulses in the channel corresponding to the maximum peak height for each of the magnesium and silicon peaks[11]; and

$$\frac{P + B}{B} > 2 \qquad \text{(B.2)}$$

for each of the magnesium and silicon peaks.