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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

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14966 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A and B form a normative part of this International Standard. Annexes C, D and E are for information only.

Introduction

This International Standard describes a method for measurement of the numerical concentration of inorganic fibrous particles in ambient air using the scanning electron microscope. This International Standard is based on the procedures of Verein Deutscher Ingenieure (VDI) Guideline 3492 [6].

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 [7].

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. On the basis of current knowledge, fibres shorter than 5 µm are thought to have a low carcinogenic potential [8 to 11].

For the purposes of this International Standard, 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 [12].

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 [10, 12].

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 must 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 [13]. 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 10312:1995.

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

1 Scope

This International Standard 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 International Standard 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 6.2), 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.

NOTE 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 can be used to determine the smaller fibres.

2 Terms and definitions

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

2.1
acicular
shape shown by an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like

2.2
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 See references [19] and [20].

NOTE 2 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°.

2.3
amphibole asbestos
amphibole in an asbestiform habit

2.4
analytical sensitivity
calculated airborne fibre concentration equivalent to counting one fibre in the analysis

NOTE 1 It is expressed in fibres per cubic metre.

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

2.5
asbestiform
specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility

2.6
asbestos
any of 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 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).

2.7
asbestos structure
individual asbestos fibre, or any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles

2.8
aspect ratio
ratio of length of a particle to its width

2.9
blank
fibre count made on a specimen prepared from an unused filter, to determine the background measurement

2.10
chrysotile
fibrous variety of the mineral serpentine, which has the nominal composition:

$Mg_3Si_2O_5(OH)_4$

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

2.11
cleavage
breaking of a mineral along one of its crystallographic directions

2.12
cleavage fragment
fragment of a crystal that is bounded by cleavage faces

2.13
cluster
fibrous structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping

2.14
countable fibre
any object longer than 5 µm, having a maximum width less than 3 µm and a minimum aspect ratio of 3:1

2.15
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

2.16
field blank
filter cassette which has been taken to the sampling site, opened and then closed, and subsequently used to determine the background fibre count for the measurement

2.17
fibre
elongated particle which has parallel or stepped sides and a minimum aspect ratio of 3:1

2.18
fibre bundle
structure composed of apparently attached, parallel fibres

NOTE A fibre bundle may 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.

2.19
fibril
single fibre of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances

2.20
fibrous structure
fibre, or connected grouping of fibres, with or without other particles

2.21
habit
the characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities

2.22
image field
the area on the filter sample which is shown on the cathode ray tube display

2.23
limit of detection
calculated airborne fibre concentration equivalent to the upper 95 % confidence limit of 2,99 fibres predicted by the Poisson distribution for a count of zero fibres

NOTE It is expressed in fibres per cubic metre.

2.24
magnification
ratio of the size of the image of an object on the cathode ray tube screen to the actual size of the object

NOTE For the purposes of this International Standard, magnification values always refer to that applicable to the cathode ray tube display.

2.25
matrix
structure in which one or more fibres or fibre bundles touch, are attached to, or partially concealed by a single particle or connected group of non-fibrous particle

2.26
serpentine
any of a group of common rock-forming minerals having the nominal formula:

$Mg_3Si_2O_5(OH)_4$

2.27
split fibre
agglomeration of fibres 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

2.28
structure
single fibre, fibre bundle, cluster or matrix

3 Abbreviated terms

CRT Cathode ray tube

EDXA Energy-dispersive X-ray analysis

FWHM Full width, half maximum

PTFE Polytetrafluoroethylene

SEM Scanning electron microscope

UICC Union Internationale Contre le Cancer

4 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.

5 Apparatus and materials

5.1 Air sampling

5.1.1 **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 must be clamped in such a manner that significant leaks around the filter do not occur at differential pressures up to approximately 50 kPa (see 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.

5.1.2 **Sampling train**.

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

5.1.3 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, which results in a pressure differential of approximately 50 kPa. 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

5.1.4 Needle valve, with a fine adjustment mechanism, for setting the volumetric flowrate.

5.1.5 Volumetric flowmeter (rotameter), for measuring the volumetric flowrate.

5.1.6 Timer, for measuring the sampling time.

5.1.7 Dry type gas meter (optional), for volumetric measurement, calibrated, designed for a maximum volumetric flowrate of 2 m^3/h.

5.1.8 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.

5.1.9 Instruments for unattended sampling (optional).

For unattended sampling, a volumetric flow controller is required for regulation of the flowrate to within $\pm$ 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

5.2 Preparation of filters

5.2.1 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.

5.2.2 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	5	Connection for vacuum pump
2	Filter in mounting ring	6	Air inlet
3	Oxygen inlet	7	Cooling-water inlet
4	Power supply from plasma generator	8	Cooling-water outlet

Figure 3 — Example of a configuration of a plasma asher

5.3 Sample analysis

5.3.1 Scanning electron microscope (SEM), with an accelerating voltage of at least 20 kV, is required for fibre counting and identification.

5.3.2 Energy-dispersive X-ray system for the SEM, capable of achieving a resolution better than 170 eV (FWHM) on the MnK_a 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 6.4.1, and identification of the fibres, as specified in 6.4.3.

5.3.3 Stereo-microscope, with a magnification of approximately 20 ×, for visual examination of the particulate deposit on the filter.

5.3.4 Gold-coated capillary-pore polycarbonate filters, of 0,8 µm maximum nominal pore size, 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 may 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.

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

5.3.6 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 5.1.1. 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. 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.

5.3.7 Technically pure oxygen, for operation of the plasma asher.

5.3.8 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.

5.3.9 Filter containers, for transport and storage of filters if disposable field monitors are not used.

5.3.10 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.

5.3.11 Sample for resolution adjustment.

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

5.3.12 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.

6 Air sample collection and analysis

6.1 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 8.

6.2 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.

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 may 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 may occur, resulting in a rapid increase in the pressure differential across the filter. Under these conditions, it is not possible to collect a satisfactory sample to represent the normal sampling period, and 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. If fog is an unusual occurrence, more representative results could be obtained by collection of air samples when the weather conditions are more typical.

6.3 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.

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.

NOTE 1 The portion of the filter to be analysed may 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.

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 (^oC):	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

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.

6.4 Analysis in the scanning electron microscope

6.4.1 General instructions

Examine the filter sample at an accelerating voltage of approximately 20 kV and an image magnification of between 2 000 × and 2 500 ×. For fibre classification in the SEM, an accelerating voltage of 20 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 20 000 ×. 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 can be acquired from a chrysotile fibre of 0,2 µm width on the test sample within a maximum period of 100 s.

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 [16], 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 6.4.2. Using EDXA, these fibres are then classified into compositional groups according to the criteria specified in 6.4.3. 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.

6.4.2 Fibre-counting criteria

6.4.2.1 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 6.4.2.2 to 6.4.2.9, and the examples shown in Figures 6 and 7.

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

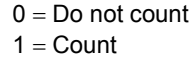

Figure 6 — Examples of fibres extending outside the image field

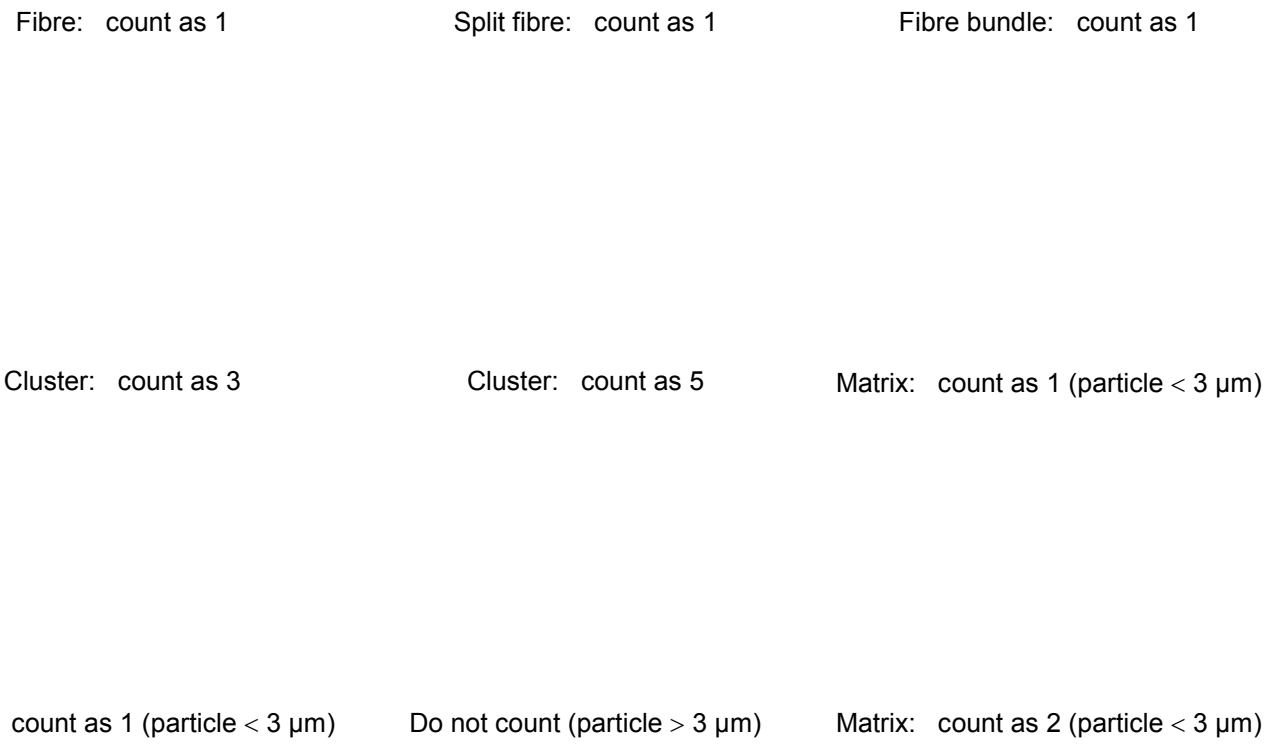

Figure 7 — Examples of the interpretation of the counting rules

6.4.2.2 Countable fibre

A countable fibre is defined in 2.14 as any object longer than 5 µm, a maximum width less than 3 µm, and a minimum aspect ratio of 3:1.

6.4.2.3 Bundle

A fibre bundle or a split fibre is counted as a single fibre if the overall dimensions of the bundle or the split fibre conform to those of the countable fibre definition as given in 2.14. The diameter of a fibre bundle or split fibre is defined as the maximum width in the compact region.

6.4.2.4 Cluster

Each countable fibre, fibre bundle or split fibre within a cluster is counted individually if both ends of the fibre, bundle or split fibre can be separately located and its length and maximum width measured. For a cluster in which no individual countable fibres are visible, count the cluster as a single fibre if the overall dimensions of the cluster conform to those of the countable fibre definition in 2.14.

6.4.2.5 Matrix

Each countable fibre, fibre bundle or split fibre within a matrix is counted individually if both ends of the fibre, bundle or split fibre can be separately located and its length and maximum width measured. For a matrix in which no individual countable fibres are visible, count the matrix as a single fibre if overall dimensions of the matrix conform to those of the fibre definition in 2.14.

6.4.2.6 Fibres extending outside of the image field

For each image field, count all countable fibres except those which extend over the right-hand or the bottom edge of the image field. Do not count fibres which have no terminations within the image field. Figure 6 shows examples of the counting criteria.

6.4.2.7 Rejection of overloaded image fields

Reject the image field if more than one eighth of an image field is covered by aggregates of fibres and/or particles. Record each rejection of an image field on the fibre counting form. If more than 10 % of the image fields are rejected in accordance with this criterion, the sample is overloaded and shall be rejected.

6.4.2.8 Recording of fibrous particles not meeting the specified definitions

Observations of fibrous particles which do not meet the specified definitions, for example, fibres with adherent particles (matrices), or fibre bundles and clusters with widths greater than 3 µm, shall be recorded on the fibre counting form.

6.4.2.9 Termination of fibre counting

Continue the examination until completion of the field in which the 100th inorganic fibre (other than calcium sulfate fibres) occurs. If, after examination of 50 image fields, 100 fibres have not been detected, further fields shall be examined until either a total of 100 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.

6.4.3 Fibre classification

6.4.3.1 General

A simple fibre classification procedure is used in which each fibre is classified as chrysotile asbestos, amphibole asbestos, calcium sulfate or other type of inorganic fibre on the basis of a semi-quantitative analysis of the elemental spectrum. The fibres are classified into the above categories on the basis of their X-ray emission 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.

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 may result in emission of X-rays from particles attached to, or in close proximity to the fibre being analysed. The EDXA spectrum obtained may 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.

Use the criteria given in 6.4.3.2 to 6.4.3.8 to classify the spectra:

6.4.3.2 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 may 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.

6.4.3.3 Amosite

Classify a fibre as amosite if:

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

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 may also be visible.

6.4.3.4 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 may also be visible.

6.4.3.5 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, but any Na peak is faint, with $P/B < 1{:}1$.

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

6.4.3.6 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 may 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.

6.4.3.7 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 may be visible.

6.4.3.8 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 6.4.3.2 to 6.4.3.7.

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 [12].

NOTE 2 In the above criteria, amosite and crocidolite have been classified separately. However, some X-ray detectors may not be sufficiently sensitive to detect the sodium peak from crocidolite and, when using these types of detector, routine discrimination between amosite and crocidolite may not be possible. In this case, amosite and crocidolite fibres are classified into the same category.

6.4.3.9 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.

6.4.3.10 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 may change with time, new reference spectra must 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)

Figure 8 — EDXA spectra of fibres of reference materials

c) Fibre of reference crocidolite (no gold coating)

d) Fibre of reference tremolite (no gold coating)

Figure 8 (*continued*)

e) Fibre of reference actinolite (no gold coating)

f) Fibre of reference anthophyllite (no gold coating)

Figure 8 (*continued*)

a) Fibre of chrysotile on a gold-coated filter with other particles

b) Fibre of amosite on a gold-coated filter with other particles

Figure 9 — Examples of EDXA spectra from asbestos fibres on gold-coated filters

c) Fibre of crocidolite on a gold-coated filter with other particles

NOTE The spectra were obtained from fibres detected in actual air samples.

Figure 9 (*continued*)

6.4.3.11 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.

6.4.3.12 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.

6.4.4 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.

6.4.5 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.

7 Calculation of results

7.1 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 1.

Table 1 — Fibre classifications

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:

$$c_i = \frac{n_i}{N \cdot V_B} \tag{3}$$

where

$$V_B = \frac{4 \cdot F_B \cdot V \times 10^6}{\pi \cdot d_{\text{eff}}^2} \tag{4}$$

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:

$$c = c_1 + c_2 \tag{5}$$

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

$$c_T = c_1 + c_2 + c_3 \tag{6}$$

7.2 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 2. Convert these values into numerical fibre concentrations using the equations:

$$c_i^L = \frac{\lambda_L}{N \cdot V_B} \tag{7}$$

$$c_i^U = \frac{\lambda_U}{N \cdot V_B} \tag{8}$$

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.

8 Performance characteristics

8.1 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 [12]. Calculation of the performance characteristics (measurement uncertainty, sampling uncertainty, analysis uncertainty) is therefore based on the total number of asbestos and other inorganic fibres.

8.2 Measurement uncertainty

8.2.1 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.

8.2.2 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 particular fibre counts can be estimated using the Poisson distribution, as described in 8.2.6.

Where particular 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 8.2.1 to 8.2.4 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.

8.2.3 Errors due to sampling

The sampling error is defined as the scatter of the measured results when using side-by-side identical sampling systems. On the basis of comparison measurements [17], the relative standard deviation was determined to be:

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

8.2.4 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 [17]. From these results, a relative standard deviation of:

$$2\,\sigma_A \leqslant 35\ \%$$

was obtained.

From these comparison measurements [17], 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.

8.2.5 Total error of the measurement

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

$$\sigma_T = \sqrt{\sigma_P^2 + \sigma_A^2 + \sigma_S^2} \tag{9}$$

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:

$$2\sigma_V \leqslant 38\ \%$$

For individual samples, the standard deviation for the Poisson variability must 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 8.2.6.

8.2.6 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:

$$P(n,a) = \frac{a^n \cdot \exp(-a)}{n!} \tag{10}$$

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 10 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 a sampled air volume of 0,01 m^3 per image field.

Table 2 — 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 [a]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,000	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,315	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

[a] The one-sided upper 95 % confidence limit for zero structures is 2,99.

Figure 10 — 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

8.3 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 by:

$$E = \frac{2{,}99}{N \cdot V_B} \tag{11}$$

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 may 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 may also be reduced by increasing the volume of air sampled. However, the extent to which the sampled air volume can be increased may 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 may 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.

9 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

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 11.

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 11 — 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 20 kV and a magnification of between 2 000 × and 2 500 ×. For fibre identification in the SEM, an accelerating voltage of 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 [cathode ray tube (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 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 a 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 CRT 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 CRT display sizes and nominal line numbers of about 500 to 700 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:

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

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

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

for each of the magnesium and silicon peaks.

B.3 Adjustment of the plasma asher

The operating conditions of the plasma asher shall be adjusted so that the time for removal of organic materials from sample filters can be predicted. Place a burning candle which produces smoke at a distance of approximately 30 cm from a sampling head equipped with a gold-coated filter in accordance with annex A. Using the nominal volume flowrate, collect smoke on the filter for approximately 30 s. Remove the filter from the sampling head (it should be greyish or black at the surface) and place it in the plasma asher. Cover approximately half of the filter surface with a glass microscope slide.

If the ashing conditions are correctly adjusted, the smoke particles from the unprotected half of the filter will be removed in approximately 20 min. The colour of this filter area should then appear the same as that of the unused filter. The filter material itself should appear unchanged.

B.4 Detection of leaks in the sampling head

The method for collecting smoke as described in B.3 can also be used to determine whether there are leaks around the filter when it is installed in the sampling head. After exposure of the sampling head to the smoke, a leak will result in colouration around the edge of the backing filter or around the edge of the gold-coated filter. If this occurs, improve the fitting of the filter in the sampling head, so that an airtight seal is obtained.

Annex C
(informative)

Characteristics and chemical composition of inorganic fibres

C.1 General

For the purposes of this International Standard, particles with a minimum ratio of length to width of 3:1 are defined as fibres.

All fibrous particles can be classified according to their chemical composition (inorganic/organic), according to their physical structure (amorphous/crystalline) and according to their origin (natural/synthetic) [18]. The term “synthetic inorganic fibres” is often used as a synonym for “artificial mineral fibres”, or “man-made mineral fibres”.

The development of fibrous morphology in a material can be attributed to a number of causes.

a) The lattice structure of a crystalline material may contain privileged directions such as lattice planes, lattice constants, or zones, which demand a fibrous habit. The chain or band silicates, such as pyroxenes, amphiboles and wollastonite, or the sheet silicates such as chrysotile, which have Si_4O_{10} sheets with a polar structure whose free valences point in the same direction, are examples of these types of structure [18 to 20].

b) The physical and chemical conditions during formation, such as pressure, temperature and the chemical species in solution, can lead to growth in a fibrous habit:

   1) monocrystalline “whiskers”, such as tin, alumina, silicon carbide and potassium titanate, can be formed by crystallization from solution;

   2) quartz and aragonite can be formed by crystallization from a gel phase;

   3) alkaline sulfates can be produced during the formation of druses and pores in bricks, and;

   4) chrysotile asbestos, amosite, crocidolite and fibrous gypsum can be formed during metamorphosis of rock [19, 20].

c) Amorphous fibres can be manufactured from numerous inorganic mixtures. In these processes, the material is solidified from a melt or a solution process, using procedures such as spinning or stretching to form fibres such as glass fibres. After formation, the properties or crystallinity are sometimes modified by thermal treatment.

C.2 Asbestos fibres

Asbestos is a term used to designate a group of naturally occurring inorganic crystalline silicates when they occur in a fibrous habit. They occur as individual fibres, or bundles of fibres, in rocks. During transformation of the rock, growth of fibres is favoured by conditions in fissures and gaps in the rock [19, 20].

The asbestos types can be classified mineralogically into serpentine asbestos (chrysotile asbestos) and amphibole asbestos [19 to 23]. Serpentine asbestos forms during hydrothermal transformation of ultra-basic rocks containing magnesium and aluminium, such as dunites, gabbros and basalts, at temperatures lower than 360°C. The starting minerals are olivine and lower-ranking pyroxenes and amphiboles. If whole rock complexes are involved in this transformation process (serpentinization), the final product is referred to as serpentine. During further hydrothermal mobilization, chrysotile fibre bundles may precipitate in fissures and gaps of the serpentine, with an intermediate gel phase.

Although chrysotile is a sheet silicate mineral, stresses in the lattice structure cause curvature in the sheet, which results in the formation of spiral scrolls and a fibrous habit. The diameter of these scrolls (fibrils) is variable and in the range of 0,02 µm to 0,05 µm. It is therefore possible to separate macroscopic chrysotile fibre bundles into fundamental fibrils of these diameters.

Asbestos varieties of the amphibole minerals also result from rock metamorphoses [19 to 23]. The amphiboles are chain silicates which exhibit a preferred crystallographic direction, which is apparent from the crystal morphology. Amphibole minerals appear to be columnar, acicular or fibrous, and they exhibit prominent cleavage parallel to the longitudinal axis of the fibre.

The ranges of composition for the various asbestos types are shown in Table C.1. The compositions include oxides of elements which do not necessarily appear in the nominal formulae. Elements other than those of the nominal formulae can occupy certain lattice points in the structure. Aluminium, for example, can substitute silicon in some lattice positions, and iron, manganese, titanium, nickel, chromium, lithium or zinc can substitute calcium or magnesium. Substitution of iron and magnesium by other elements is common.

Table C.1 — Chemical composition of various asbestos types (% mass fraction)

	Chrysotile	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
SiO_2	36 to 44	49 to 53	49 to 56	53 to 60	55 to 60	51 to 56
MgO	38 to 42	1 to 7	0 to 3	17 to 34	20 to 26	12 to 20
FeO	0 to 3	34 to 44	13 to 21	0 to 20	0 to 5	5 to 15
Fe_2O_3	0 to 5	0 to 5	13 to 20	0 to 5	0 to 5	0 to 5
Al_2O_3	0 to 2	0 to 1	0 to 1	0 to 3	0 to 3	0 to 3
CaO	0 to 2	0 to 2	0 to 3	0 to 3	10 to 15	10 to 13
K_2O	0 to 1	0 to 1	0 to 1	0 to 1	0 to 1	0 to 1
Na_2O	0 to 1	0 to 1	4 to 9	0 to 1	0 to 2	0 to 2
H_2O	12 to 14	2 to 5	2 to 5	1 to 6	1 to 3	1 to 3

C.3 Other fibrous minerals

C.3.1 General

A large number of natural minerals exist which, due to their lattice structure or the particular conditions of formation, crystallize in a fibrous habit. A number of these minerals which also occur in technical processes and as constituents in their products are listed here. For the many other such minerals, reference should be made to the relevant mineralogical and petrographical literature [19, 20, 22].

C.3.2 Silicates

Mullite $Al_6Si_2O_{13}$ and willemite Zn_2SiO_4 occur widely as fibrous, acicular or columnar individual crystals or as felt-like, partly spherulitic crystallite bundles in mineral phases in stoneware clays, porcelain, chamotte, and in silica and high alumina refractory bricks after use in zinc smelting plants and open-hearth furnaces.

Sillimanite Al_2SiO_5 and dumortierite $Al_7O_3(BO_3)(SiO_4)_3$, as fibrous varieties, are found only in the raw materials used for the production of high alumina refractory bricks [19, 24].

Pseudo-wollastonite $CaSiO_3$ is found as a columnar fibrous form in ceramic clays, iron blast furnace slags, welding electrodes and as devitrifying elements in soda-lime glass [19, 24].

Numerous members of the zeolite group, particularly wide-meshed tectosilicates with ring systems of SiO_4, or AlO_4 tetrahedra linked by channel-like elements, are fibrous. The channels in the zeolite crystal structures give rise to reversible ion exchange, molecular sieve and hydration properties. They are used as catalysts in cracking of petroleum, and in isomerization.

Important fibrous zeolites include the mixed crystals natrolite and scolecite $Na_2Al_2Si_3O_{10}{\cdot}2H_2O$ to $CaAl_2Si_3O_{10}{\cdot}3H_2O$. The cubic zeolites faujasite $(Na_2,Ca)Al_2Si_4O_{12}{\cdot}6H_2O$, erionite $(Ca,Na_2,K_2)_{1,5}$–$(Al_9Si_{27}O_{72}){\cdot}27H_2O$ and mordenite $(Ca,K_2,Na_2)Al_2Si_{10}O_{24}{\cdot}7H_2O$ frequently exhibit a fibrous habit. Industrially synthesized zeolites are usually used, since it is then possible to control the material properties more precisely [19].

C.3.3 Sulfates

Fibrous sulfates are common. Anhydrite $CaSO_4$ is found in mortar, in concrete after exposure to sulfuric acid and sulfate solutions, and in fly ash. Gypsum $CaSO_4{\cdot}2H_2O$ is found in plaster of Paris and sculptor's plaster after it is mixed with water, and it is also found in other building materials. Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}{\cdot}24H_2O$ occurs as a binding agent in high-alumina cements. Hydrated tricalcium aluminate in these cements reacts with sulfate solutions to form bundles of acicular ettringite fibres, as new formations in hydrated brown coal flyash and as satin white filler pigments in paper.

Epsomite $MgSO_4{\cdot}7H_2O$ and mirabilite $Na_2SO_4{\cdot}10H_2O$ occur as “blooms” in soil, and the latter also in masonry.

C.3.4 Industrial crystalline fibres

The demand for fibre-reinforced high-performance composite materials is growing continuously. Since the properties of natural fibres do not satisfy industrial requirements, a large number of synthetic inorganic fibres have been developed.

These fibres are either polycrystalline, each consisting of a large number of smaller fibrous crystals oriented in the same direction, or each is single crystal whisker which has grown into a fibre as a result of a privileged direction, generally a helical dislocation.

The polycrystalline fibres generally exhibit a columnar form with a finely structured surface similar to that of the source material used for manufacture. Monocrystalline fibres (whiskers) have a primarily plane form with a polygonal cross-section. Examples of synthetic inorganic crystalline fibres are shown in Table C.2 [25 to 27].

C.3.5 Amorphous fibres

Amorphous inorganic fibres, also referred to as “man-made vitreous fibres”, are produced by a melting process. The primary constituents of the melt are silicates, as shown by the compositional ranges in Table C.3. Table C.4 shows typical fibre diameters and a classification according to the type of raw materials used [25, 27].

The strands of melted material, which are cooled at various rates, solidify to form glassy fibres. The fibres generally exhibit a circular cross-section and a structureless, smooth surface.

The mechanical drawing process used for production of textile glass fibres generally yields fibres which have a constant diameter over their full length. Non-textile glass fibres, manufactured by processes such as centrifugal blowing, can have irregular fibre thicknesses with droplet-like thickenings, but these materials may also contain very fine fibres with diameters lower than 1 µm. Non-textile glass fibres include glass wool, cinder wool, rock wool and ceramic wools [25].

Table C.2 — Synthetic inorganic crystalline fibres

	Fibre diameter μm	Morphology	Method of manufacture
Carbon/graphite	5 to 10	columnar	microfibrillation of oriented polyacrylonitrile or cellulose fibres by carbonization or graphitization
Boron	100	columnar	precipitation of boron as a “corn cob” structure on 12 μm diameter tungsten core
Boron carbide (B_4C)	1 to 25	planar	reaction of primary carbon fibre with boron trichloride and hydrogen
Boron nitride (BN)	4 to 8		reaction of primary B_2O_3 fibre with ammonia
Alumina (α-Al_2O_3)	3 to 9		formation of α-Al_2O_3 from a polymer gel phase in nitrogen at 100°C
Silicon carbide (SiC)	2 to 8		SiO_2-impregnated cellulose fibres are pyrolysed and converted to SiC in a protective gas atmosphere
Zirconia (ZrO_2)	3 to 6	columnar	cellulose fibres impregnated with a zirconium salt are pyrolysed and the carbon is removed
Tungsten	12		primarily sintering processes
Steel (austenitic)	12		bundle drawing process with ductile matrix (copper) or melt-drawing process in a glass matrix
α-Alumina (α-Al_2O_3)	0,5 to 10	planar	precipitation from a gas phase
α-Alumina (α-Al_2O_3)	50 to 100	columnar	melt-drawing process
α-SiC	0,5 to 10		precipitation from the vapour phase onto finely dispersed lanthanum
β-SiC	0,5 to 10		decomposition of methyltrichlorsilane in hydrogen and precipitation onto carbon
Potassium hexatitanate $K_2Ti_6O_{13}$	0,2 to 1		melt crystallization

Table C.3 — Compositions of synthetic inorganic amorphous fibres

Component	Textile glass	Insulating glass	Rock fibres	Slag fibres	Ceramic fibres
SiO_2	54 to 74	48 to 63	45 to 53	≈ 41	45 to 52
Al_2O_3	0 to 25	3 to 9	6 to 13	≈ 12	42 to 51
B_2O_3	0 to 22	0 to 6			0 to 1
Fe_2O_3	0 to 5	3 to 14	5 to 8		1 to 16
FeO		0 to 4	1 to 7	≈ 1	
CaO	0 to 17	7 to 28	11 to 30	37 to 40	0 to 3
MgO	0 to 6	32 to 38	3 to 10	≈ 4	0 to 5
BaO	0 to 1	0 to 25			
ZrO_2	0 to 16				0 to 3
TiO_2	0 to 2	0 to 2	5 to 2	≈ 0,4	0 to 6
MnO		0 to 5	6 to 5	≈ 0,5	
P_2O_5		0 to 6	0 to 1	≈ 0,3	
CaS				0 to 1	
Li_2O	0 to 1				
K_2O	0 to 8	8 to 36	≈ 1,3	≈ 0,4	0 to 3
Na_2O	0 to 13	4 to 14	≈ 2,4	2 to 14	0 to 7
S				≈ 0,4	
F				≈ 0,4	

Table C.4 — Raw materials used in the manufacture of synthetic inorganic, amorphous fibres

Fibre	Diameter µm	Source
Glass fibres	0,15 to 35	Natural minerals or rocks
Stone fibres	0,3 to 14	Natural minerals or rocks
Slag fibres	0,4 to 5	Metallurgical or non-metallurgical slags
Ceramic fibres	0,2 to 10	Melts primarily of alumina and silica
Quartz fibres	1 to 10	Pure quartz melts
Silica glass fibres	< 5 to 10	Leaching and thermal treatment of aluminium borosilicate glass

Annex D
(informative)

Poisson variability as a function of fibre density on sampling filter and area of filter analysed

Figure D.1 shows the relationship between the fibre density on the filter, the area of filter examined by the analyst, and the resulting unavoidable statistical fluctuations in the number of fibres counted. Recognition of these limitations are important in design of the sampling. The standard deviations shown in the graphs are calculated on the assumption that the distribution of fibres on the filter is Poissonian, for which the standard deviation of a fibre count is the square root of the fibre count. The variability is shown as twice the relative standard deviation, σ_s. The 95 % confidence intervals for the Poisson distribution are asymmetric about the mean, particularly for low numbers of fibres. The 95 % confidence intervals are shown in Table 2.

The curves illustrate the requirement to consider variability of fibre counting in design of strategies for sample collection. For example, assuming that the fibres are distributed on the filter according to a Poisson distribution, for a fibre density of 20 fibres per square millimetre of filter area the variability of the fibre count, expressed as $2\sigma_s$ after the examination of a filter area of 0,7 mm^2, is shown to be approximately 50 %. For lower fibre densities, analysis of larger areas of filter is necessary in order to achieve a comparable variability of approximately 50 %. Correspondingly, if higher fibre densities can be obtained on the filters, either because a higher airborne fibre concentration exists or because the suspended particulate is sufficiently low that the sampling time can be extended, the area of filter examined can be reduced, while still maintaining an acceptable variability for the measurement. In some cases, a combination of the two approaches can be an expedient means of improving the precision of the measurements.

Figure D.1 — Poisson variability of fibre counts as a function of fibre density on the sampling filter and area of filter analysed

Annex E
(informative)

Combination of the results from multiple samples

For some applications, for example to reduce the error in the measured result, it may be desirable to calculate a mean value from several samples. A mean air concentration for the same location, but over a longer period of time, can also be derived by combining the results from a number of air samples collected sequentially over the required time period.

The measured fibre concentration is determined by two parameters: the number of fibres counted during the SEM examination and the volume of air examined by the analyst. The detection limit is determined solely by the volume of air examined by the analyst.

A mean concentration, $\overline{c_i}$, for fibres of type i is derived by summation of the fibres from all contributing samples and dividing by the summation of the individual volumes analysed in each sample.

$$\overline{c_i} = \frac{\sum n_i}{\sum V_P} \tag{E.1}$$

The best estimate for the mean concentration is given by:

$$\langle \overline{c_i} \rangle = \frac{\sum n_i + 1}{\sum V_P} \tag{E.2}$$

The detection limit, E, for the mean concentration is:

$$E = \frac{2{,}99}{\sum V_{\mathrm{P}}} \tag{E.3}$$

for the mean fibre concentration, the 95 % confidence interval due to Poisson variability is calculated on the basis of n_i and V_{P}.

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