

## **Method for the separate determination of concentrations of inorganic fibres in work areas – Scanning electron microscopic method –**

Method tested and recommended by the German Social Accident Insurance for the determination of airborne inorganic fibres with a length of  $L > 5 \mu\text{m}$ , a width of  $0.2 \mu\text{m} \leq D < 3 \mu\text{m}$  and a length-to-width-ratio (aspect ratio) of  $L/D > 3:1$  (criteria according to WHO [1]) in work areas.

Both personal and stationary sampling can be conducted for the assessment of work areas.

Sampling is carried out by separation of particles in air drawn by means of a pump and collected onto a gold coated capillary-pore membrane filter.

Analysis is performed by scanning electron microscopy (SEM) using energy dispersive X-ray microanalysis (EDXA).

This method uses the principle of determination described in the VDI-Richtlinie (guidelines) 3492 [2]. It supplements the phase-contrast optical microscopy method (PCM) BGI 505-31 [3] for the cases in which

1. different types of inorganic fibres are present, which have to be distinguished from each other and from organic fibres;
2. the limit of detection of the phase-contrast microscopic method is not sufficient to monitor the compliance of given threshold limit values and trigger thresholds.

This method permits the detection and identification of asbestos, calcium sulfate and other inorganic fibres having a width of  $D \geq 0.2 \mu\text{m}$ . Fibrous particles with  $D < 0.2 \mu\text{m}$  are not taken into account for the calculation of the measuring result because the method here described may be used instead of the phase-contrast optical microscopy method BGI 505-31 [3], if in addition to quantification, identification of fibrous particles is required. Using the method BGI 505-31, fibres thinner than approximately  $0.2 \mu\text{m}$  cannot be detected. Furthermore, identification of fibres being as thin as that usually is not possible when using EDXA.

Infrared spectroscopic method (BGI 505-30 [4]) in combination with the phase-contrast microscopy permits a directly quantitative determination of asbestos weight fractions in fine dust or after respective preparation in material samples and on request can deliver useful information in addition to the methods BGI 505-31 and BGI 505-46.

Measurements within the meaning of the country's guidelines "Asbest-Richtlinie der Länder" [5] are carried out according to [2]. However, they are not object of this method.

## Summary

This method permits the determination of concentrations of inorganic fibres having a length of  $L > 5 \mu\text{m}$ , a width of  $0.2 \mu\text{m} \leq D < 3 \mu\text{m}$  and a length-to-width-ratio (aspect ratio) of  $L/D > 3 : 1$  [1] in work areas averaged over the sampling time after personal or stationary sampling.

**Principle:** A pump is used to draw a measured volume of air through a gold coated capillary-pore membrane filter. The fibres collected are counted and analysed by means of scanning electron microscopy and energy dispersive X-ray microanalysis.

### Technical data:

**Limit of detection:** The limit of detection depends above all on the sample volume. For a sample volume of 40 litres per  $\text{cm}^2$  filter area the statistic limit of detection is in accordance with standard analysis conditions 15 000 fibres/ $\text{m}^3$ .

**Selectivity:** Discrimination between chrysotile, amphibole asbestos, calcium sulfate, and also product fibres (see Section 2.2.2) and other inorganic fibres possible.

**Advantages:** The analysis is fibre-specific (morphology) and fibre type specific (material).

**Disadvantages:** Extensive equipment and high evaluation expenditure.

**Apparatus:** Appliance for gold coating (e.g. sputter coating unit)<sup>1</sup>,  
Sampling apparatus,  
Plasma asher,  
Scanning electron microscope,  
Energy-dispersive X-ray microanalytical system (EDXA).

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<sup>1</sup> Not necessary, if commercially available capillary-pore membrane filters sputter coated with gold are used for the measurement.

## Detailed description of the method

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## 1 Equipment, consumables and accessories

For the procedure of a measurement according to the method here described the equipment, consumables, and accessories as given in Section 1.1 to 1.5 are required. It must be safeguarded that they are clean and free of fibres before being used. Monitoring occurs in random tests by means of laboratory blank samples. For this the entire analytical procedure is applied to an unloaded sample collection filter.

## 1.1 Apparatus for sampling

### *Sampling head:*

It mainly consists of a cylindrical cowl, filter holder with sample collection filter and an intake tube (see Figure 1).

Cowl and filter holder must be manufactured from non-corrosive material. Airtight fit of the inserted sample collection filter must be guaranteed. The length of the cowl before the filter must be 1.5 to 3.0 times the exposed (effective) filter diameter  $d_{eff}$  (diameter of the circular filter area being exposed) [1].

For fibre measurements in work areas the sampling heads usually used in phase-contrast microscopy analysis are suitable [3]. Figure 1 shows the schematic diagram of a suitable sampling head with a cassette serving as filter holder for the sample collection filter located on a backing filter. After sampling the cassette is sealed with covers and used as a transport vessel (filter case) (see also sampling system FAP-BIA in Figure 2).

### *Sampling pump:*

For sampling a pump is used which is able to draw at least volumes of air between 0.24 L/min and 0.3 L/min per  $\text{cm}^2$  through the filter. Higher flow rates may be used for work areas with low dust concentrations.

The volumetric flow must be free of pulsation so that a secure flow rate measurement is possible. In case of a battery-driven pump is used, capacity of the battery must be sufficient for a continuous use throughout the entire sampling time chosen.

### *Flow meter:*

Suitable measuring apparatus, permitting the measurement of the flow rate with a precision of better than 5%. Monitoring is carried out using a calibrated volumetric flow meter (e.g. soap bubble flow meter, variable area flow meter).

*Chronometer:* Stop watch

## 1.2 Apparatus for filter preparation

Appliance for coating the capillary-pore membrane filter with gold<sup>2</sup>, e.g. from GaLa-Instrumente, D-65307 Bad Schwalbach.

Plasma asher (oxygen plasma), e.g. from GaLa-Instrumente, for removal of organic components on the loaded sample collection filter. Plasma ashers are commercially available for cold or low temperature incineration with capacitive or inductive coupling of the plasma. For inductive coupling it must be observed that the filter is not located within the field of the induction coils.

<sup>2</sup> Not necessary, if commercially available capillary-pore membrane filters sputter coated with gold are used for the measurement.

### 1.3 Apparatus for analysis

Scanning electron microscope, e.g. from Carl Zeiss NTS GmbH, D-73447 Oberkochen with EDXA, e.g. from Thermo Electron, D-63303 Dreieich for fibre counting and identification. The instrument must comply with the minimum requirements regarding detectability and identification (refer to Section 4.1).

### 1.4 Consumables

Sample collection filter:	Capillary-pore membrane filter made of polycarbonate. Filter diameter each according to system of sampling (commonly used: 25 mm and 37 mm), nominal pore size 0.4 µm or 0.8 µm, coated with gold (front side approx. 40 nm, rear side approx. 20 nm), e.g. from alto tec GmbH, D-22763 Hamburg.
Backing filter:	Membrane filter made of cellulose ester having an average pore size of >3 µm (no fibrous material, such as e.g. glass fibre filters or cardboard).
Oxygen:	Technically pure, for operating the plasma asher.
Gold target:	For capillary-pore membrane filter coating.
Argon:	Technically pure, for operating the gold-coating unit <sup>3</sup> .
Colloidal carbon paint or double coated adhesive tape or the like:	For electrically conductive fixing of the filter to be analysed on the sample holder of the SEM.
Liquid nitrogen:	For cooling the EDX detector and the field effect transistor (FET) of the EDX analytical system.

### 1.5 Accessories

Filter container:	The sample collection filters mounted in the filter holder are transported dust-tight in a vessel (filter cassette).
Tweezers:	With rounded points to handle the filters.
Scalpel:	With a crooked blade for cutting loaded filters to pieces.
Stereo microscope:	For visual inspection of filter loading or detection of signs of damage, magnification approx. 20 times.

<sup>3</sup> Not necessary, if commercially available capillary-pore membrane filters sputter coated with gold are used for the measurement.

Test sample:	Gold-coated capillary-pore membrane filter loaded with chrysotile fibres from which a part has a width of $\leq 0.2 \mu\text{m}$ in order to check visibility and the EDX spectrum on the SEM (preparation like in Section 3.1).
Calibration standard:	For calibration of the magnification of the SEM.
Test object (element standard), particularly Cu:	For energy calibration of the EDX analytical system.
Reference material:	According to the defined task (e. g. for determination of product fibres; asbestos reference material, available e. g. at the Institute of Occupational Medicine (IOM), Edinburgh, Scotland).

## 2 Sampling

### 2.1 Preparation of sample collection filters

Prior to sampling, the capillary-pore membrane filters must be coated with gold e.g. by means of a sputter coating unit. Suitable capillary-pore membrane filters sputter coated with gold are also commercially available. The coating with gold is required for an SEM image free of charge and has the following advantages compared to a subsequent coating:

- Geometrical dimensions of the fibres remain unchanged.
- The element peaks are not weakened.
- The filter remains stable during plasma ashing.

In order to minimize contrast variations an even gold layer thickness is necessary. A sufficient layer thickness can be assumed when the filter surface loses its dark colour during coating and takes on the typical metallic golden lustre and appears to have a green gleam when viewed in transmitted light.

The gold layer thickness shall be approx. 40 nm on the side of the sample collection filter to be loaded (stronger reflecting, shining side). Coating the rear side of the filter with a gold layer of approx. 20 nm serves for stabilization of the sample collection filter and may result in improvement of the contrast ratio. In case no integrated layer thickness measuring system is available, layer thickness may be easily monitored in the SEM by means of the EDX facility. For this purpose filters of a known diameter are coated with gold and the weights of gold determined by differential weighing. When comparing the gold peaks for these test filters at a constant stream with the weights a linear relationship can be deduced, from which the layer thickness can be determined [2].

Prior to further usage, capillary-pore membrane filters of a new batch are checked on the SEM for impurities resulting from inorganic fibres. Using the SEM/EDXA,  $0.5 \text{ mm}^2$  for each of two gold-coated filters of the batch to be tested are analysed according to the conditions described in Section 4. In this process, a maximum of one inorganic fibre having a length of  $L > 5 \mu\text{m}$  may be found.

The sample collection filter prepared for sampling is placed into the filter holder such that it lies plane on the backing filter, it is not damaged when inserted and an airtight fit is guaranteed. Contact with the filter surface with naked fingers is to be avoided. Already in the laboratory the filter cassettes are equipped with sample collection filters and fibre-free backing filters and sealed. In case a backing grid is to be placed below the backing filter, it may be problematic to keep the sampling head free of leakage. The sample collection filter must not rest directly upon the backing grid.

## 2.2 Procedure of sampling

### 2.2.1 Air samples

Touching the filters with naked fingers during manipulation is to be avoided. The sampling head with inserted sample collection filter (e.g. 25 mm diameter) is opened right before starting sampling. When using filter cassettes (e.g. 37 mm diameter) these are opened right before starting sampling and placed into the sampling head; contact with filter surface is to be avoided. Sampling takes place with a cowl oriented downwards. An example of a sampling head with filter cassette is shown in Figure 2.

Before starting sampling the flow rate is to be adjusted such that for each  $\text{cm}^2$  of exposed (effective) filter area an air volume of 0.24 to 0.3 L/min (respectively 4 cm/s to 5 cm/s filter flow velocity) is delivered. For instance, if a filter holder with an exposed diameter of 30 mm is used, a flow rate of 1.7 L/min to 2.1 L/min is required. The specific flow rate shall not fall short of 0.24 L/( $\text{cm}^2 \cdot \text{min}$ ). For individual cases (e.g. for high dust concentrations) the flow velocity may be decreased to 2 cm/s. Only if no coarse airborne dust particles can be expected in the work area, it can be recommended to increase the specific flow rate up to approx. 1.2 L/( $\text{cm}^2 \cdot \text{min}$ ) (respectively up to approx. 20 cm/s flow velocity). Measurement of the flow rate is carried out for the complete sampling system (sampling head equipped with sample collection filter and backing filter, hose with intake tube and pump) by means of a suitable and calibrated flow meter (e.g. variable area flow meter).

At the end of the sampling the flow rate must not deviate by more than 10% from the initial flow rate. For the calculation of the air sample volume the mean value calculated from initial and final flow rate is used. For the flow rate given, the duration of sampling is dependent on the dust concentration. High dust loading must be avoided in any case. The golden shining filter surface must still be visible with naked eyes. Immediately after completion of sampling the sampling apparatus is switched off, the sampling time is recorded and the filter cassette is removed together with the loaded sample collection filter and sealed dust-tight. Place, time and duration of sampling are to be selected such that the exposition is collected representatively [7].

As a rule, a sampling time from 2 to 3 hours for a filter flow velocity of 5 cm/s delivers evaluable filter loadings. In case of reduced dust concentrations without coarse dust particles a sampling time of 8 hours or even longer or a higher flow velocity of up to 20 cm/s is possible as well. In case of doubt, a set of filters with graded sampling time (e.g. 1 hour, 2 hours, 4 hours, etc.) may be loaded, so that at least one evaluable sample



may be expected among them. In exceptional cases it may be inevitable to select a sampling time of less than 1 hour, e.g. for high dust concentrations or a large amount of coarse dust particles. The multiple application of the same sample collection filter for short-term expositions during various short-term phases has proved reliable in order to achieve sufficient limits of detection. Even if only one single short-term exposition appears, a sampling time of at least one hour is recommended.

Table 1 shows the relationship between the achievable limit of detection and the specific sample volume for the analysis of a filter area of  $0.5 \text{ mm}^2$ . By increase of the examined area the limit of detection may be decreased (e.g. to  $300 \text{ fibres/m}^3$  for  $1 \text{ mm}^2$  examined area and  $1000 \text{ L/cm}^2$ ). Figure 3 shows the relationship between the limit of detection and the sample volume.

**Table 1.** Dependence of the limit of detection on the specific sample volume for an examined area of  $0.5 \text{ mm}^2$ .

Limit of detection fibres/ $\text{m}^3$	Specific sample volume $\text{L/cm}^2$	Sampling time (h) for $5 \text{ cm/s}$	Sampling time (h) for $10 \text{ cm/s}$	Sampling time (h) for $20 \text{ cm/s}$
15000	40	2.2	1.1	0.6 *)
10000	60	3.3	1.7	0.8 *)
7500	80	4.4	2.2	1.1
6000	100	5.6	2.8	1.4
4000	150	8.3	4.2	2.1
1000	600	33	17	8.3
600	1000	56	28	14

\*) Sampling time should not fall short of 1 hour

### 2.2.2 Product fibres

Product fibres are fibres allocated to materials which are used in the individual operational process or which are present in the environment of the sampling location. In order to determine the concentration of product fibres of critical dimensions ("WHO fibres") in the work area, also samples of the materials in consideration must be taken in addition to the filter samples and both made available to the evaluating laboratory as reference samples. Each of the samples shall have at least a volume of  $1 \text{ cm}^3$  and be representative for the material – also with regard to contamination present or composite fibres (composite material). If possible, copies of safety data sheets, at least existing product information (manufacturer, product name, identification, etc.) are to be made available to the laboratory.

The analytical possibilities of the method described here for the delimitation of product fibres are restricted to inorganic fibres. Discrimination of different types of organic fibres is usually not possible.

### 3 Sample preparation

#### 3.1 Preparation of loaded filter

Using a stereo microscope and/or SEM at a minor magnification, the loaded sample collection filter is checked by observation at least of the filter edge and the center of the filter for homogeneous particle loading of the filter. In case an inhomogeneous loading is detected, the filter is not suitable for quantitative analysis. It is recommended to also examine the edge which the filter was pressed to for possible leakage during sampling by means of the stereo microscope. In addition, the backing filter must be checked for possible discolouration which is also a sign for leakage. Furthermore, the filter surface of the preparation must be checked macroscopically for signs of smudges, finger prints or damage using glancing illumination (e.g. by means of a halogen lamp). Then the loaded filter is mounted electrically conductive to the SEM sample holder with the dust side up and – as long as no organic fibres shall be determined – transferred to the plasma asher. By plasma ashing the organic material on the filter is almost completely removed. This facilitates the analysis of the sample in the SEM to a great extent.

Due to the electrical conductivity of filter and sample holder, corroding activity of the O<sub>2</sub>-plasma on the organic material located on the filter is highly effective. For equipment with capacitive probe-to-specimen contact of the plasma, power consumption must be controlled in a way that no sparkovers appear that result in holes in the gold coating of the filter. After approx. 30 to 60 minutes incineration is usually completed.

The filter sample prepared in this way is now ready for examination in the SEM (particularly without any further secondary coating of the sample).

In case too many interfering organic particles are found in the beginning of the analysis by the SEM, plasma ashing should be repeated.

It is recommended to mount the whole filter for the SEM analysis. If it is necessary to cut the filter, it should be carried out in a rolling manner by means of a sharp scalpel with a crooked blade (scissors should not be used). At this it is to be observed that the dust loading on the filter surface is not affected. If a piece of the filter is used, it must be cut in a way that edge and center area of the filter are contained (e.g. sector). When using carbon tabs for assembly on the sample holder, modifications might appear in the condition of the filter surface after some time so that it is recommended to store a section of the filter as a retain sample.

#### 3.2 Preparation of reference samples

Scatter preparations are prepared from the reference samples (material samples). Solid materials are reduced to small pieces (e.g. by crushing, cutting, sawing, rasping, scraping); using tweezers some material is taken from fibre mats or similar products. The small pieces obtained in this way as well as powders are deposited on the carbon tabs, then gently pressed to the tabs by a spatula, and loose residue removed carefully (tap or blow off under the hood). The carbon tabs are fastened to the SEM sample holders.

## 4 Analysis by scanning electron microscopy

### 4.1 General directions

#### *Magnification:*

Magnification is the ratio between the length of an object on the image to be analysed and the actual length of the same object. Therefore, the actual magnification is concerned and not the nominal magnification indicated on the instrument. Fibre counting is performed at an actual magnification of at least 2000:1 and should not exceed 2500:1. Deviations may be permissible if equivalent results can be achieved.

At regular intervals and following maintenance and repair work on the SEM, magnification must be verified by means of a suitable test sample (e.g. graticule).

#### *Accelerating voltage:*

Accelerating voltage should be at least 15 kV. Both, the Mg peak of chrysotile and the Fe peak of amphibolic asbestos fibres, must be able to be detected securely. For a stable image of organic fibres it may be necessary to operate with an accelerating voltage of 10 kV or less.

#### *Tilt angle:*

The sample must not be tilted during counting (tilt angle 0°).

#### *Visibility of thin fibres:*

All SEM parameters (especially magnification, accelerating voltage, beam diameter, working distance, and scan time) must be selected in a way that also very thin fibres and fibres poor in contrast are still visible. For this purpose, a fibre just visible at the magnification selected for counting is chosen on a test sample with chrysotile fibres according to Section 1.5. The width  $D$  of this fibre is then determined at a magnification of at least 10 000:1. If the width is  $\leq 0.2 \mu\text{m}$ , the minimum requirement regarding visibility of fibrous particles is met. In case the minimum requirement has not been met after various tests, the SEM parameters must be adapted correspondingly and the visibility test repeated<sup>4</sup>. This test is to be carried out once per working week as well as after maintenance and repair work on the SEM.

#### *EDX-Analysis:*

It is recommended to use a light element detector. The adjustments of the operational parameters are to be selected in a way that a chrysotile fibre having a diameter of  $\leq 0.2 \mu\text{m}$  delivers a sufficiently distinctive X-ray emission spectrum within a maximum measuring time of 100 s (compare with Section 4.3). This means that for the Mg and Si lines for chrysotile a ratio of intensities  $(S+U)/U$  ( $S+U$  = peak height,  $U$  = background signal at peak position) greater than 2:1 is to be achieved. At the same time the required boundary condition  $S > 3 \cdot \sqrt{U}$  for the respective peak position shall be met [2]. As a rule, a magnification of  $\geq 5000:1$  must be adjusted on the SEM for EDX analysis.

<sup>4</sup> It is not recommended to use the TV mode.

For EDX analysis the largest possible solid angle of the detector system is desirable. Identification of fibres having a width of approximately  $0.2\ \mu\text{m}$  is only guaranteed for solid angles of  $>10^{-3}$  sr.

Inspection and, if required, calibration of the energy system and the peak intensity ratio must be performed at regular intervals and after maintenance and repair work.

*Analysis procedure:*

The parameters determined after performance of the test for the visibility of thin fibres and EDX analysis (see above) are to be maintained for the analysis of the filter samples. A magnification larger than the one used for fibre counting may be applied for EDX analysis and for the determination of fibre dimensions.

The element peaks are assigned to categories A, B and C, which are defined as follows [2]:

- Category A:  $(S + U)/U \leq 4$
- Category B:  $2 \leq (S + U)/U < 4$
- Category C:  $(S + U)/U < 2$  and  $S > 3 \cdot \sqrt{U}$  (element significantly detected)

Where:

$S$  = peak height

$U$  = background signal

## 4.2 Fibre counting rules

Basis for fibre counting is the criteria according to the WHO [1].

- According to these rules each object is considered to be a fibre, showing a length of  $L > 5\ \mu\text{m}$ , a width of  $D < 3\ \mu\text{m}$  and an aspect ratio of  $L/D > 3:1$ . The length applies to the rectified length, the width to the average width (see Figure 4). In case fibres demonstrating a width of  $D < 0.2\ \mu\text{m}$  are counted, they must be recorded separately in the analysis protocol.
- Convexities that can ensue from e.g. resins or binders in synthetic mineral fibres (SMF) are ignored. In case of doubt  $D < 3\ \mu\text{m}$  is assumed [1].
- Fibres adjoining non-fibrous particles or seem to adjoin them, are treated as if the non-fibrous particles were not present. However, only the visible length of fibres is taken into account, unless the fibres penetrate the particles and do not appear to be interrupted.
- Fibres whose both ends are lying within the counting field are counted as 1 fibre. Fibres having one end within the counting field are counted as 1/2 fibre. Fibres whose both ends are lying outside of the field are not counted.
- Fibre agglomerates that appear to be compact and not separated in one or more sections but, however, seem to be separated in individual fibres in other sections are considered to be split fibres. All other agglomerates with fibres touching or crossing are considered to be bundles.
- Split fibres are counted as 1 fibre, as long as the above mentioned criteria are fulfilled. Their width is measured in the section not split. Overlapping (crossing) fibres (fibre bundles) are counted individually, if possible.

- In case too many fibres are overlapping so that they cannot be counted individually (fibre bundle), the fibre bundle is only counted as one fibre as long as its total dimensions meet the above mentioned criteria for length, width and aspect ratio. Otherwise the fibre bundle is not taken into account.
- If more than 1/8 of the counting field area is covered by fibre or particle agglomerates this field is not taken into account.
- At least an area of 0.15 mm<sup>2</sup> is to be examined.
- At least a total of 50 fibres (without calcium sulfate fibres) are to be counted and identified. In case 50 fibres (without calcium sulfate fibres) could not have been detected yet on a filter surface of 0.15 mm<sup>2</sup>, further image fields must be analysed until the fibre number is achieved. If the required number of fibres is not achieved after examination of at least 0.5 mm<sup>2</sup> filter surface, the analysis may be terminated.
- Image fields to be analysed are selected in a way that the complete area of the filter section is taken into account uniformly without preference of the margins or the centre of the filter area and overlappings of the image fields to be counted will not occur. It is recommended to distribute the image fields in zigzag fashion over the entire exposed filter area.

Schematic examples for the application of the fibre counting rules are shown in Figure 5.

### 4.3 Fibre identification

This method permits the discrimination of chrysotile, amphibole asbestos, calcium sulfate fibres, inorganic product fibres, if applicable, and other inorganic fibres. All those fibres are assigned to group “other inorganic fibres” which cannot be identified as asbestos, calcium sulfate or product fibres, however, yield an EDX spectrum with elements of ordinal number  $Z \geq 11$  (exception: carbon fibres). For organic fibres see Section 4.5.

Fibre identification, in which chrysotile, amphibole asbestos, calcium sulfate, and product fibres and other inorganic fibres can be discriminated, is carried out by semi-quantitative assessment of element spectra. Examples for spectra of inorganic fibres are shown in the Figures given in Appendix 1 (the gold peaks are a result of the gold-coating of the capillary-pore membrane filter). Since the peak height is dependent on the detector and the instrument parameters used, separate fibre reference spectra must be established under the conditions described, e.g. by means of standard samples.

During practical performance of the analysis it is to be observed that the electron beam is focussed onto the fibre without drifting and potentially attached or adjacent particles are located as far away as possible from its focal point.

For identification of fibres the criteria given in Tables 2 and 3 are to be applied.

Fibres which cannot be assigned to the types of fibres given in Tables 2 and 3 are not taken into account for the calculation of the analytical result.

Identification according to the criteria described here is not clear for every case [8]. In case of doubt, knowledge about material used in the work area is to be taken into ac-

**Table 2.** Criteria of identification for fibrous dusts (S: signal, U: background).

Type of fibre	Criteria of identification	Remarks
Chrysotile	<ul style="list-style-type: none"> <li>– Mg and Si peak significant, ratio <math>(S + U)/U &gt; 2</math> (Cat. A or B)</li> <li>– Fe and Mn peak possible, ratio <math>S/U &lt; 1</math> (Cat. C)</li> <li>– Al peak missing or very small (Cat. C)</li> </ul>	<i>Chrysotile from asbestos cement:</i> Additional Ca peak possible due to residues of binders <i>Chrysotile from magnesium plaster floor:</i> Additional Cl peak possible
Amphibole asbestos	See Table 3 and analytical notes in [8]	Since amphibole asbestos is a group of minerals, the spectra are different regarding detected elements and their peak heights
Calcium sulfate	<ul style="list-style-type: none"> <li>– Ca peak significant</li> <li>– S peak significant (partly overlapped by M line of the Au)</li> </ul>	
Product fibres	Compare with Section 4.4	For used product fibres other element peaks, e.g. Fe, might appear in addition to the characteristic peaks
Other inorganic fibres	All fibres yielding a different element spectrum with a ratio of $(S + U)/U > 2$ for at least one element with $Z \geq 11$ (exception: carbon fibres)	Main characteristic elements can be: Na, Mg, Al, Si, K, Ca, Fe Furthermore Ti, Mn, Ba, Zr, and B might appear

**Table 3.** Distinguishing characteristics for amphibole asbestos.

Amphibole asbestos	Criteria of identification	Remarks
Actinolite	<ul style="list-style-type: none"> <li>– Si, Fe and Ca peak significant</li> <li>– Mg peak weak to significant</li> <li>– Na peak possible</li> <li>– Al peak missing or very small</li> </ul>	Transitions to tremolite present
Amosite	<ul style="list-style-type: none"> <li>– Si and Fe peak significant, ratio <math>(S + U)/U &gt; 2</math></li> <li>– Mg peak and Ca peak possible</li> <li>– Al peak missing or very small</li> </ul>	
Anthophyllite	<ul style="list-style-type: none"> <li>– Si, Mg peak significant</li> <li>– Fe peak weak to significant</li> <li>– Al peak missing or very small</li> </ul>	Delimitation to talc containing Fe is not possible with this method
Crocidolite	<ul style="list-style-type: none"> <li>– Si, Fe peak significant</li> <li>– Na peak weak</li> <li>– Al peak missing or very small</li> </ul>	
Tremolite	<ul style="list-style-type: none"> <li>– Si, Mg, Ca peak significant</li> <li>– Al peak missing or very small</li> </ul>	Transitions to actinolite present

(Note: In case an exact discrimination is necessary, the supplementary identification criteria [10] are to be applied and the EDXA must be calibrated with suitable standards for quantitative element determination)

count. For all cases the M and L lines of the gold-coating of the filter are additionally present in the spectrum and lead to overlappings e. g. with the K line of sulfur.

Criteria of identification are to be modified, if:

- the SEM-EDX spectra of asbestos standards demonstrate deviating peak heights and/or signal-to-background ratios due to the instrument used,
- the asbestos fibres present in the particular product to be tested deviate from the standards regarding their elemental composition (e. g. significant fraction of an element which is not characteristic, also refer to Figure A1.1 in the Appendix).

In case peaks are missing in the element spectrum for elements with  $Z \geq 11$  (excluding gold peaks), organic material might be present as a residue after plasma ashing. Even in the presence of very thin inorganic fibres ( $D < 0.2 \mu\text{m}$ ) non-evaluable or incomplete element signals must be expected.

#### 4.4 Inorganic product fibres

For classification of fibres as inorganic product fibres, the spectra of the fibres on the sample collection filter must coincide largely (according to criteria given in the following) with those present in the product. Therefore, reference samples are prepared for the considered product according to Section 3.2 and element spectra of thin fibres recorded from different sections as reference spectra. These reference spectra must be taken – possibly, with exception of the magnification used – under the same instrument conditions as used for the analysis of the sample collection filter.

For characterization of these reference spectra, for each material sample element spectra are recorded for each of ten thin fibres [2]. In this process, the product

- is assigned an element peak of category A, if for at least 9 of 10 fibres correspond to the criteria described for category A (refer to Section 4.1).
- is assigned an element peak of category B, if for at least 7 of 10 fibres correspond to the criteria described for category A or B, and for the product the conditions for a classification into A are not met.

When comparing the spectra found for fibres on the sample collection filter with the reference spectra

- element peaks of category A must reappear on the sample collection filter in the reference spectrum at least in category B and
- at least one third of the fibre elements appearing in element peaks of category B in the reference spectrum must reappear in the filter sample (here: not necessarily as a category B peak),
- at least one element peak of category B must be found for the fibre on the sample collection filter in the reference spectrum as category B or C, if the reference spectrum shows less than three peaks of category B,

in order to interpret the fibre found as product fibre. For product fibres from synthetic mineral fibres morphology (parallel edges) must in addition be compatible with the one present for the fibres in the product.

## 4.5 Organic fibres

In case also organic fibres shall be taken into account, the sample must not be treated by plasma ashing. In order to achieve a stable image, the optimal accelerating voltage must be determined by means of the sample (sometimes less than 10 kV). It must be secured, that fibres having a width of 0.2  $\mu\text{m}$ , or, if fibres as thin as that do not appear in the sample, the thinnest fibres present can be securely detected. Windowless detectors or detectors with ultra thin windows shall be used, so that light elements starting from  $Z = 6$  (carbon) can be detected. Fibres are classed as organic fibres, if the main peaks can be assigned to elements with  $Z < 11$  (exception: carbon fibres). In special cases chlorine or sulfur might also be observed.

This method does not permit discrimination of various types of organic fibres. Possibly, morphological characteristics may deliver valuable information [11].

## 4.6 Calculation of the analytical result

As analytical result this method delivers the numerical fibre concentration  $C_i$  (fibres per  $\text{m}^3$  air, fibre class  $i$ ) of airborne inorganic fibres in work areas fulfilling the criteria  $0.2 \mu\text{m} \leq D < 3 \mu\text{m}$ ,  $L > 5 \mu\text{m}$ , and  $L/D > 3 : 1$ .

$C_1$ : Chrysotile fibres

$C_2$ : Amphibole asbestos fibres

$C_3$ : Calcium sulfate fibres

$C_4$ : Product fibres (if measured)

$C_5$ : Other inorganic fibres

The concentration of the number of fibres for fibre class  $i$  ( $i = 1$  to  $5$ ) is calculated according to equation (1):

$$C_i = \frac{n_i \cdot A}{N \cdot a \cdot V} \quad (1)$$

Where:

$C_i$  is the numerical fibre concentration of the fibre class  $i$  in fibres/ $\text{m}^3$

$n_i$  is the number of fibres determined according to the counting rules (Section 4.2) for fibre class  $i$

$A$  is the exposed (effective) filter area in  $\text{mm}^2$

$N$  is the number of counting fields analysed

$a$  is the area of a counting field in  $\text{mm}^2$

$V$  is the air sample volume in  $\text{m}^3$  with  $V = Q \cdot t$

( $Q$ : sample flow rate in  $\text{m}^3/\text{h}$ ,  $t$ : sampling time in hours)

If found, fibres having a width of  $D < 0.2 \mu\text{m}$ , are listed separately without being assigned to a special type of fibre.



## 4.7 Analytical report

During analysis it is recommended to record a protocol (fibre counting form) following that in [2]. This is the basis for the compilation of the analytical report.

The analytical report contains at least:

- Name and address of the analysing laboratory,
- Name and address of the customer,
- Unequivocal sample identification,
- Data for sampling,
- Date of analysis,
- Identification of the SEM/EDXA systems used,
- Information regarding the analytical method used (here: BGI 505-46),
- Name of analyst,
- Analytical result, consisting of the numerical fibre concentrations and the number of fibres found, subdivided by the type of fibre (chrysotile, amphibole asbestos, calcium sulfate, product fibres, if applicable, as well as other inorganic fibres and, if desired, organic fibres), area and number of analysed image fields, image field magnification, remarks (e.g. regarding fibres having a width of  $D < 0.2 \mu\text{m}$ , fibre bundles and fibres of  $D > 3 \mu\text{m}$ ). In addition, illustrations and spectra of the fibres analysed may be useful.

## 5 Performance characteristics

### 5.1 Uncertainty of measurements

Deviations of the measuring value (numerical fibre concentration) from the true value may appear in addition to sampling during:

- Sample preparation (when handling and cutting filters, during plasma ashing),
- Analysis (instrument adjustment, selection of counting fields, fibre counting and identification),
- Collection and elemental analysis of thin fibres with  $D < 0.2 \mu\text{m}$ ,
- Assessment of fibre agglomerates and isometric particles during fibre counting,
- Interpretation of EDX spectra.
- Random variations of measurement results due to counting statistics are estimated by Poisson distribution [9]; compare Appendix 2.

The optimal amount of fibres on the sample collection filter lies within a range of around 100 to 1000 fibres/ $\text{mm}^2$ . Also high fractions of non-fibrous particles affect analysis, as they can interfere and cover fibres. Thus, identification as well as elemental analysis are inhibited.

The 95% confidence interval for the numerical fibre concentration due to the counting statistics may be estimated by Poisson distribution (see Table in Appendix 2). For calculation of the confidence range the limits of confidence  $\lambda_u$  and  $\lambda_o$  associated with the determined fibre number  $n_i$  are inserted into the equation given in Section 4.6.

## 5.2 Limit of detection

The limit of detection of the counting method falls short of, if no fibre has been found in the SEM analysis. Using Poisson distribution, a limit of detection according to equation (1) given in Section 4.6 is yielded from the upper limit of confidence of the 95% confidence interval for  $x = 0$  with  $\lambda_o \approx 3.0$ . Thus, the limit of detection is dependent on the sample used as well as on the analysis expenditure and amounts to approx. 15 000 fibres/m<sup>3</sup> for e.g. a specific sample volume of 40 L/cm<sup>2</sup> and an examined area of 0.5 mm<sup>2</sup>. For a specific sample volume of 1 m<sup>3</sup>/cm<sup>2</sup> only delivering analysable samples under optimal conditions at very low dust concentrations, the limit of detection would be approx. 300 fibres/m<sup>3</sup> for 1 mm<sup>2</sup> examined area and approx. 600 fibres/m<sup>3</sup> for 0.5 mm<sup>2</sup> examined area. Under the sampling conditions described above (flow velocity approximately 5 cm/s) sampling would take around 56 hours.

## 5.3 Selectivity

The method is selective according to the criteria described in Sections 4.2 and 4.3 for chrysotile fibres, amphibole asbestos fibres, calcium sulfate fibres, product fibres, if applicable, and other inorganic fibres.

Misinterpretations, particularly for the identification of asbestos fibres are possible,

- if silicate fibres of an elemental composition similar to asbestos are used in work areas;
- if the fibres are contaminated (e.g. for mortar, colours and paints, asbestos cement, magnesium plaster floor and thus result in additional peaks);
- if non-fibrous particles are lying within direct neighbourhood of fibres (high loading of the sample collection filter, coarse dust particles, chainlike smoke particles, especially welding fumes, tobacco smoke);
- due to non-uniform loading of particles on the filter (as a result of e.g. high air humidity during sampling, presence of mists or aerosol droplets, respectively, in the air sample).

## 6 References

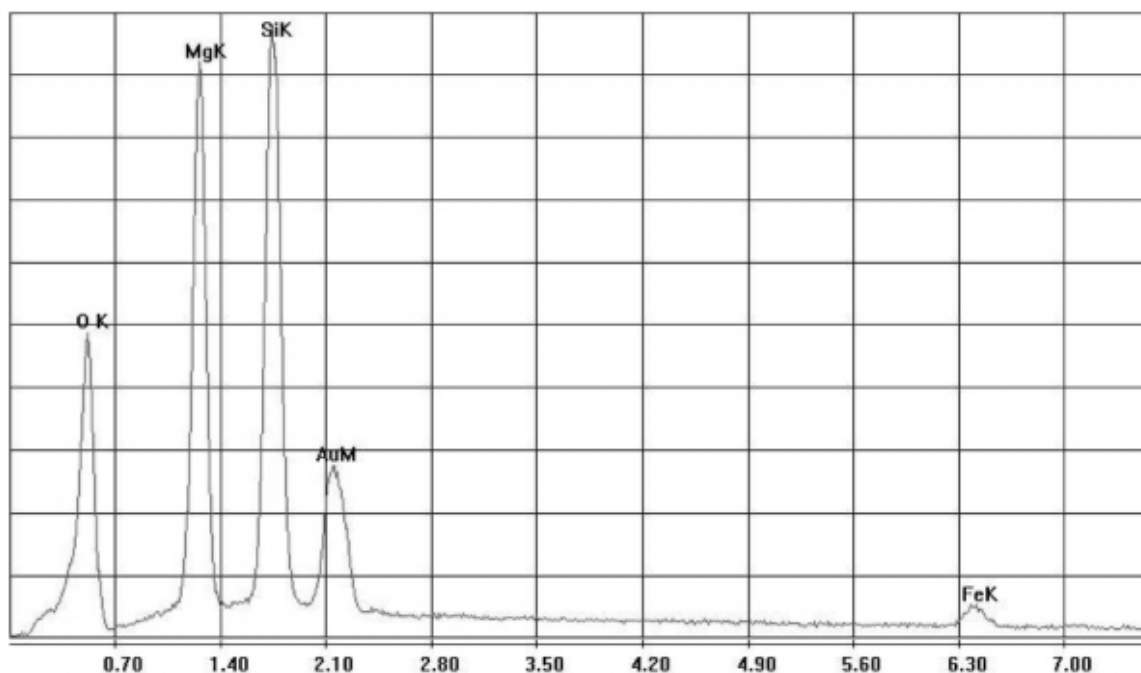
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Authors: *G. Binde, M. Mattenklott, G. Riediger, K. Rödelberger* †

## Appendix 1: Examples for inorganic fibre spectra

All spectra: – Abscissae in keV  
 – Filter gold-coated  
 – 20 kV accelerating voltage



**Fig. A 1.1.** Chrysotile spectrum (here: example of chrysotile containing Fe, S/U ratio approx. 1 for Fe)

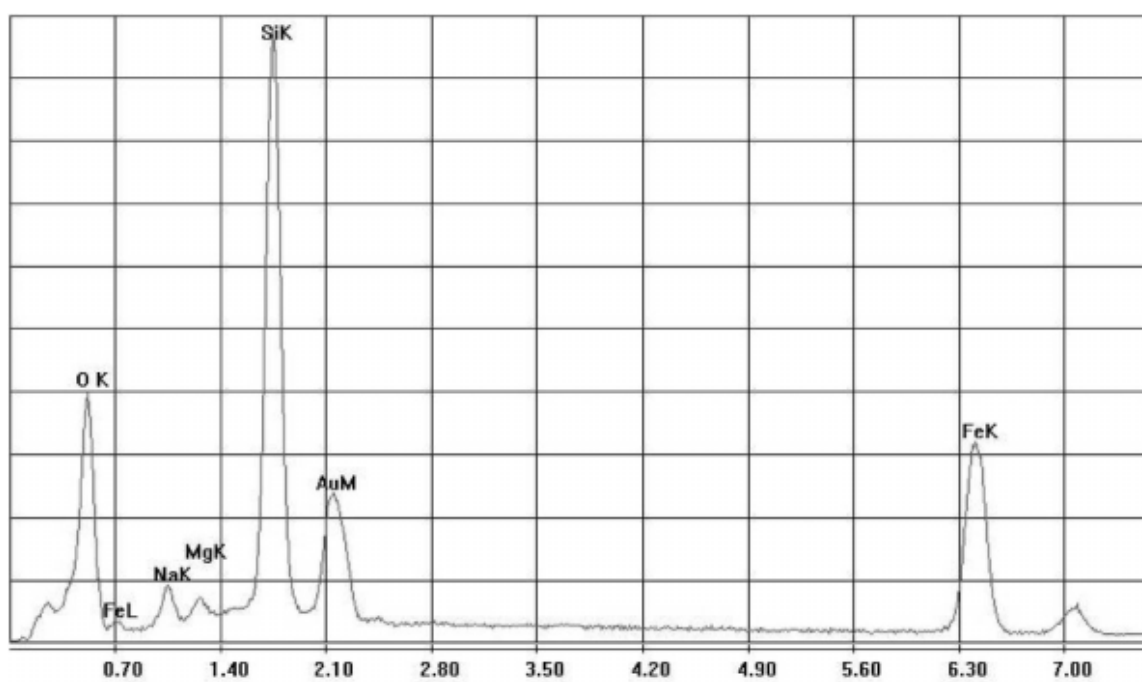


Fig. A 1.2. Crocidolite spectrum

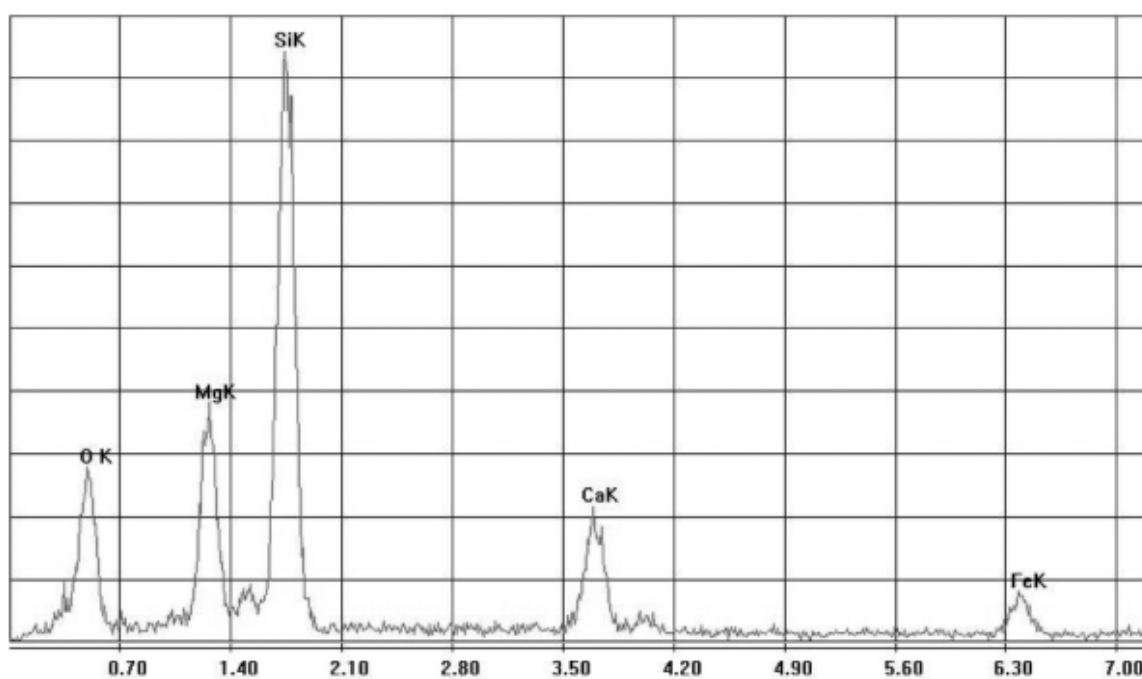


Fig. A 1.3. Actinolite spectrum

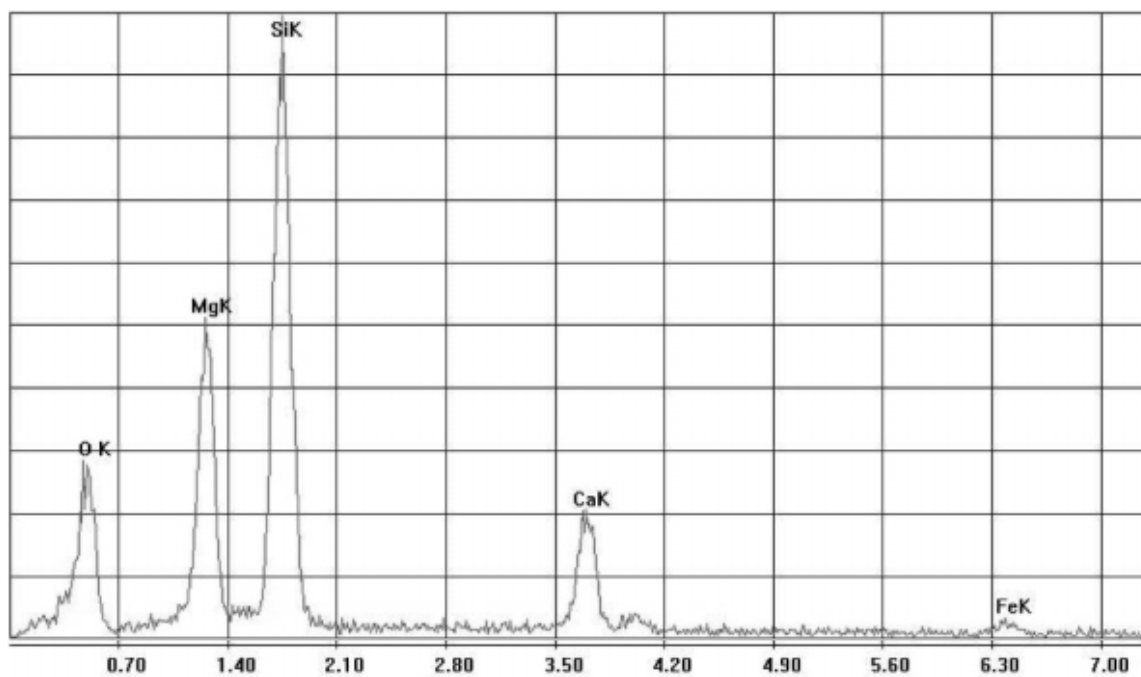


Fig. A 1.4. Tremolite spectrum

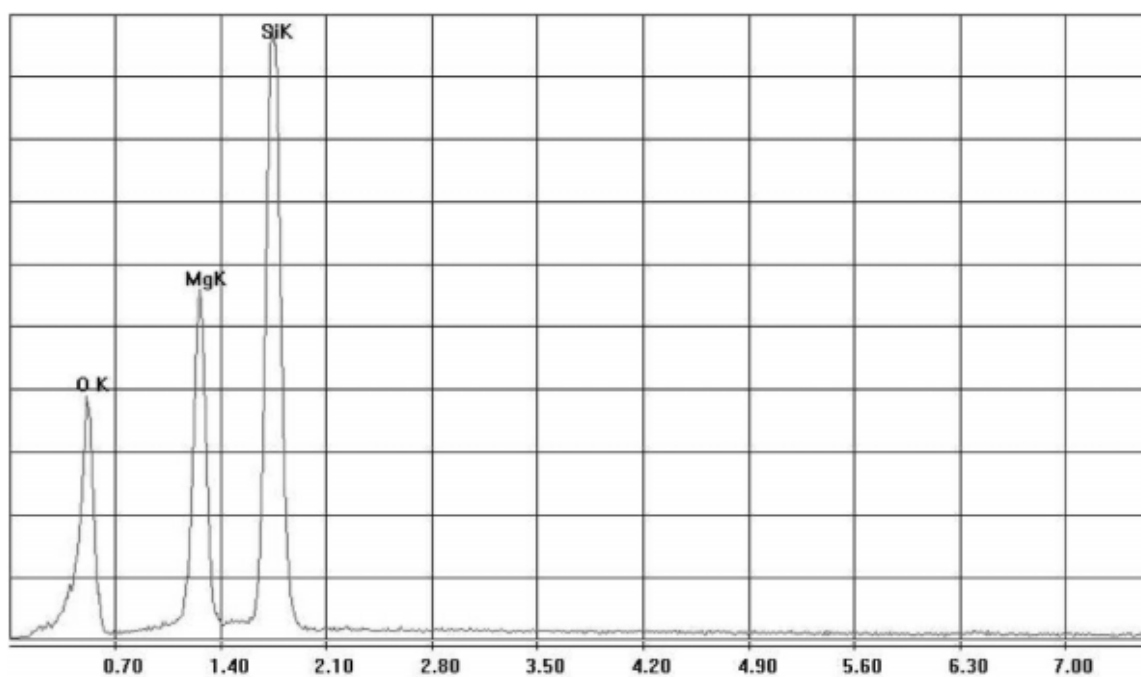


Fig. A 1.5. Talc spectrum

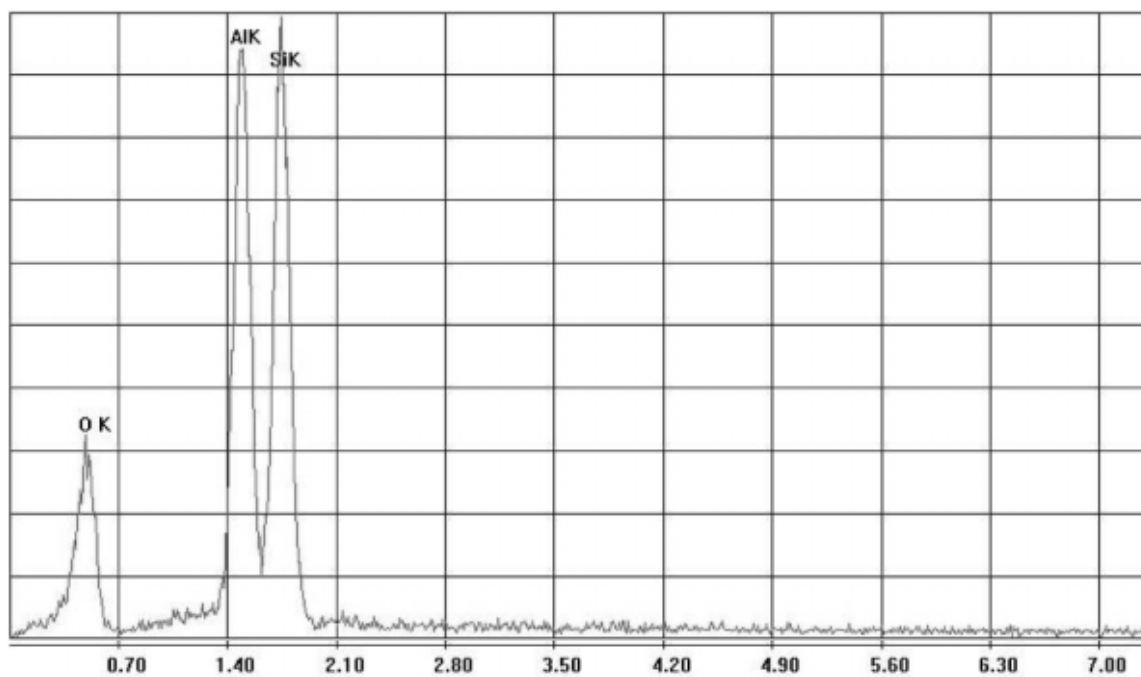


Fig. A 1.6. Ceramic fibre spectrum

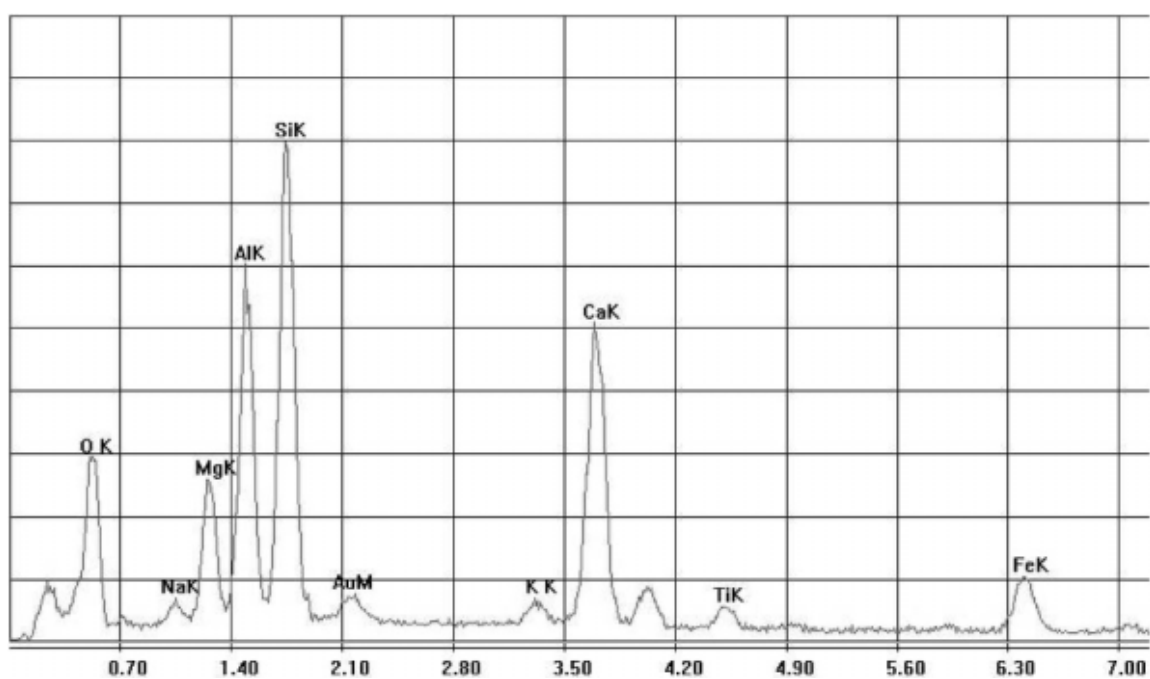


Fig. A 1.7. Rock wool fibre spectrum

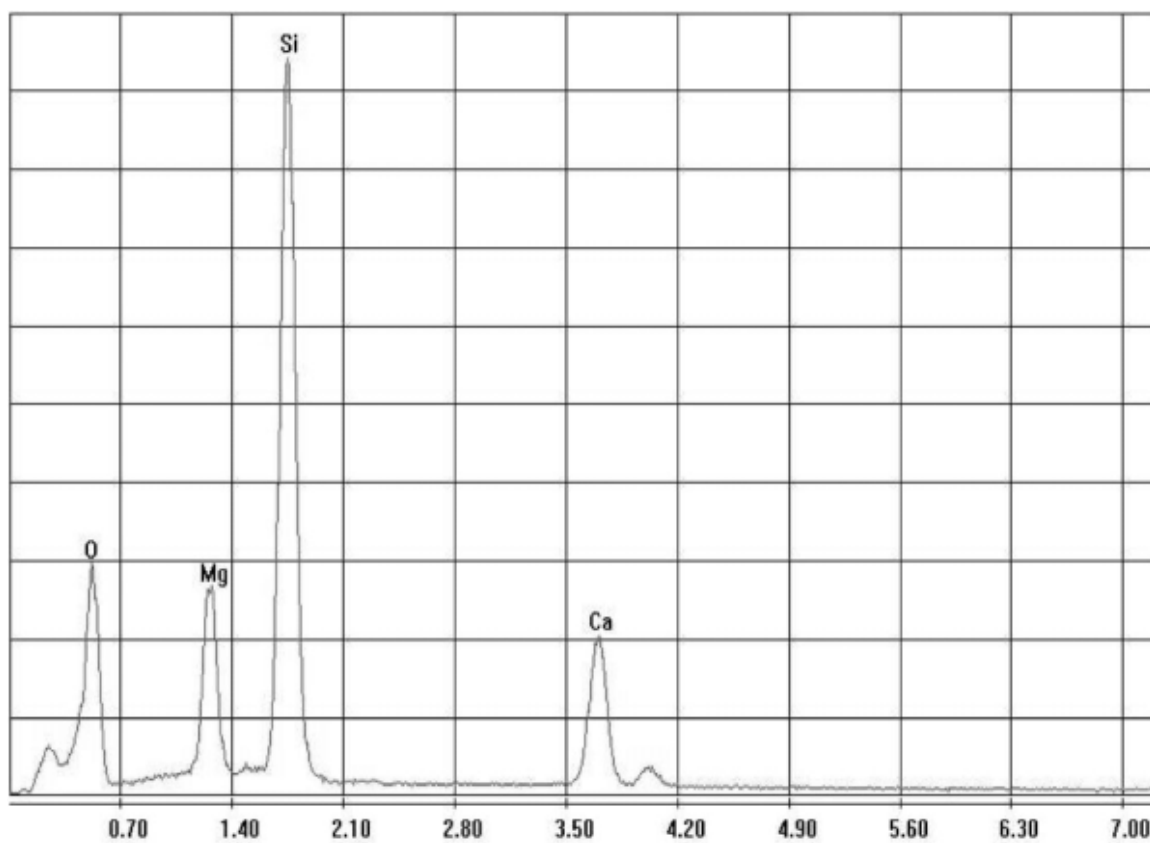


Fig. A 1.8 High-temperature glass fibre spectrum

## Appendix 2: Limits of confidence range for the counting result

**Table A.** Lower and upper limits  $\lambda_u$  and  $\lambda_o$  of the 95% confidence interval of a counting result  $x$  using Poisson distribution.

$x$	$\lambda_u$	$\lambda_o$	$x$	$\lambda_u$	$\lambda_o$
0.5	0.000	4.674	7	2.814	14.423
1	0.025	5.572	7.5	3.131	15.095
1.5	0.108	6.416	8	3.454	15.763
2	0.242	7.225	8.5	3.782	16.426
2.5	0.416	8.006	9	4.115	17.085
3	0.619	8.767	9.5	4.453	17.739
3.5	0.845	9.511	10	4.795	18.390
4	1.090	10.242	10.5	5.141	19.038
4.5	1.350	10.960	11	5.491	19.682
5	1.623	11.668	11.5	5.844	20.323
5.5	1.908	12.368	12	6.201	20.962
6	2.202	13.059	12.5	6.560	21.597
6.5	2.504	13.744	13	6.922	22.230

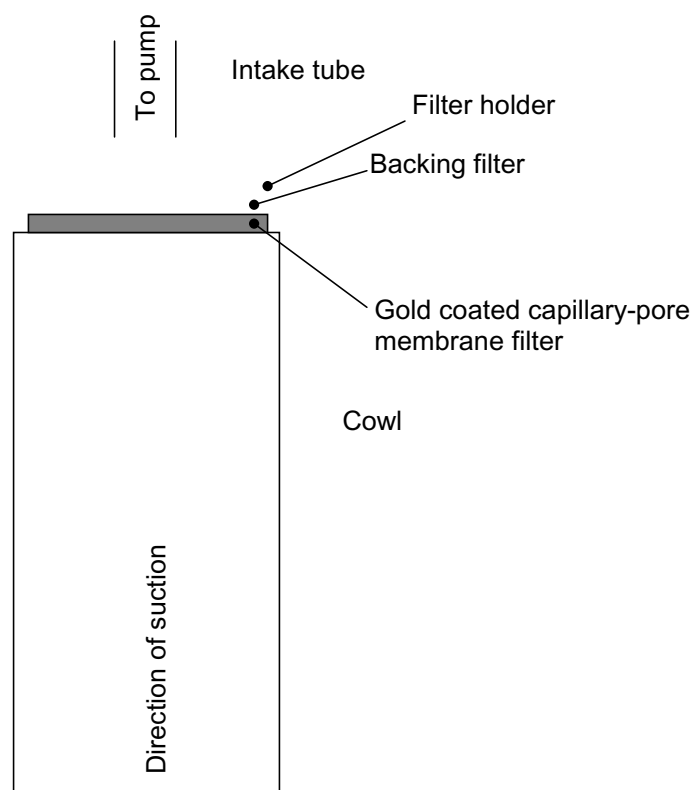
Table A: (continued)

x	$\lambda_u$	$\lambda_o$	x	$\lambda_u$	$\lambda_o$
13.5	7.287	22.861	36	25.214	49.839
14	7.654	23.490	36.5	25.632	50.420
14.5	8.024	24.116	37	26.051	51.000
15	8.395	24.740	37.5	26.471	51.579
15.5	8.769	25.363	38	26.891	52.158
16	9.145	25.983	38.5	27.312	52.736
16.5	9.523	26.602	39	27.733	53.314
17	9.903	27.219	39.5	28.154	53.892
17.5	10.285	27.834	40	28.577	54.469
18	10.668	28.448	40.5	28.999	55.045
18.5	11.053	29.060	41	29.422	55.621
19	11.439	29.671	41.5	29.846	56.197
19.5	11.827	30.280	42	30.270	56.772
20	12.217	30.888	42.5	30.694	57.346
20.5	12.607	31.495	43	31.119	57.921
21	12.999	32.101	43.5	31.545	58.495
21.5	13.393	32.705	44	31.970	59.068
22	13.787	33.308	44.5	32.397	59.641
22.5	14.183	33.910	45	32.823	60.214
23	14.580	34.511	45.5	33.250	60.786
23.5	14.978	35.111	46	33.678	61.358
24	15.377	35.710	46.5	34.106	61.929
24.5	15.777	36.308	47	34.534	62.500
25	16.179	36.905	47.5	34.962	63.071
25.5	16.581	37.501	48	35.391	63.641
26	16.984	38.096	48.5	35.821	64.211
26.5	17.388	38.690	49	36.250	64.781
27	17.793	39.284	49.5	36.681	65.350
27.5	18.199	39.876	50	37.111	65.919
28	18.606	40.468	50.5	37.54	66.49
28.5	19.013	41.059	51	37.97	67.06
29	19.422	41.649	51.5	38.40	67.62
29.5	19.831	42.238	52	38.84	68.19
30	20.241	42.827	52.5	39.27	68.76
30.5	20.652	43.415	53	39.70	69.33
31	21.063	44.002	53.5	40.13	69.89
31.5	21.475	44.589	54	40.57	70.46
32	21.888	45.174	54.5	41.00	71.02
32.5	22.301	45.760	55	41.43	71.59
33	22.716	46.344	55.5	41.87	72.16
33.5	23.130	46.928	56	42.30	72.72
34	23.546	47.512	56.5	42.74	73.29
34.5	23.962	48.094	57	43.17	73.85
35	24.379	48.676	57.5	43.61	74.41
35.5	24.796	49.258	58	44.04	74.98

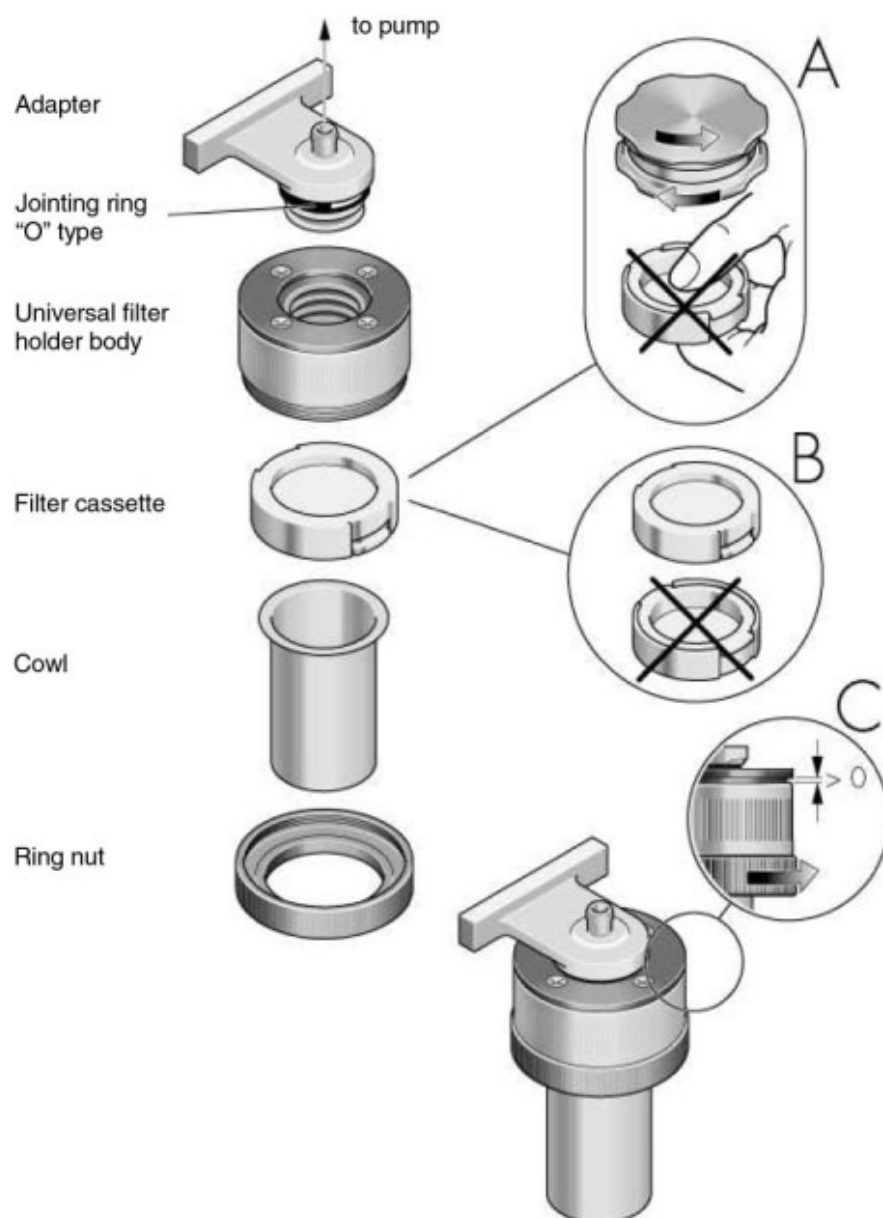


Table A: (continued)

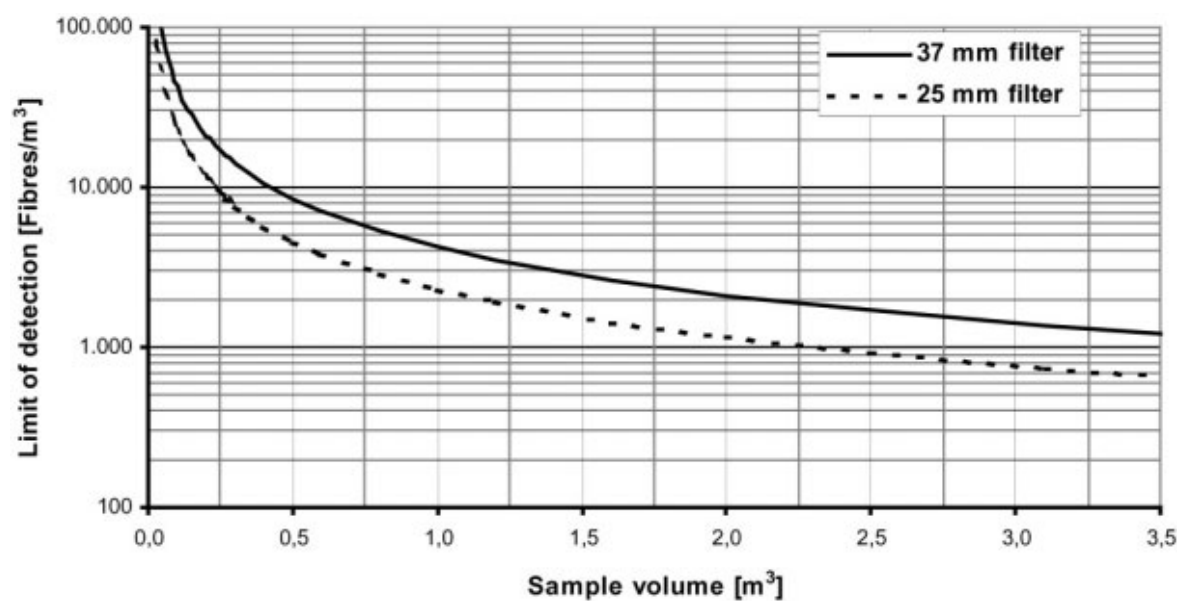
x	$\lambda_u$	$\lambda_o$	x	$\lambda_u$	$\lambda_o$
58.5	44.48	75.54	79.5	62.99	99.01
59	44.91	76.11	80	63.44	99.57
59.5	45.35	76.67	80.5	63.88	100.12
60	45.79	77.23	81	64.33	100.68
60.5	46.22	77.79	81.5	64.77	101.23
61	46.66	78.36	82	65.22	101.78
61.5	47.10	78.92	82.5	65.66	102.34
62	47.54	79.48	83	66.11	102.89
62.5	47.97	80.04	83.5	66.56	103.44
63	48.41	80.60	84	67.00	104.00
63.5	48.85	81.17	84.5	67.45	104.55
64	49.29	81.73	85	67.89	105.10
64.5	49.73	82.29	85.5	68.34	105.66
65	50.17	82.85	86	68.79	106.21
65.5	50.60	83.41	86.5	69.24	106.76
66	51.04	83.97	87	69.68	107.31
66.5	51.48	84.53	87.5	70.13	107.87
67	51.92	85.09	88	70.58	108.42
67.5	52.36	85.65	88.5	71.03	108.97
68	52.80	86.21	89	71.47	109.52
68.5	53.25	86.77	89.5	71.92	110.07
69	53.69	87.32	90	72.37	110.63
69.5	54.13	87.88	90.5	72.82	111.18
70	54.57	88.44	91	73.27	111.73
70.5	55.01	89.00	91.5	73.72	112.28
71	55.45	89.56	92	74.16	112.83
71.5	55.89	90.11	92.5	74.61	113.38
72	56.34	90.67	93	75.06	113.93
72.5	56.78	91.23	93.5	75.51	114.48
73	57.22	91.79	94	75.96	115.03
73.5	57.66	92.34	94.5	76.41	115.58
74	58.11	92.90	95	76.86	116.13
74.5	58.55	93.46	95.5	77.31	116.68
75	58.99	94.01	96	77.76	117.23
75.5	59.44	94.57	96.5	78.21	117.78
76	59.88	95.13	97	78.66	118.33
76.5	60.32	95.68	97.5	79.11	118.88
77	60.77	96.24	98	79.56	119.43
77.5	61.21	96.79	98.5	80.01	119.98
78	61.66	97.35	99	80.46	120.53
78.5	62.10	97.90	99.5	80.91	121.08
79	62.55	98.46	100	81.36	121.63



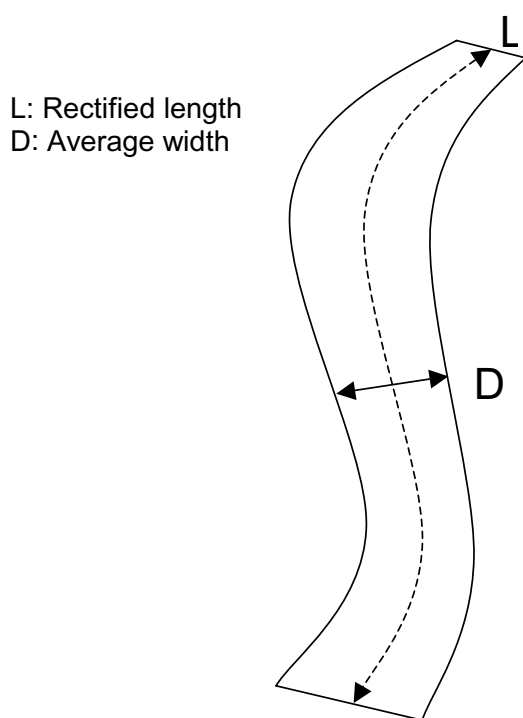
**Fig. 1.** Schematic diagram showing a suitable sampling head.



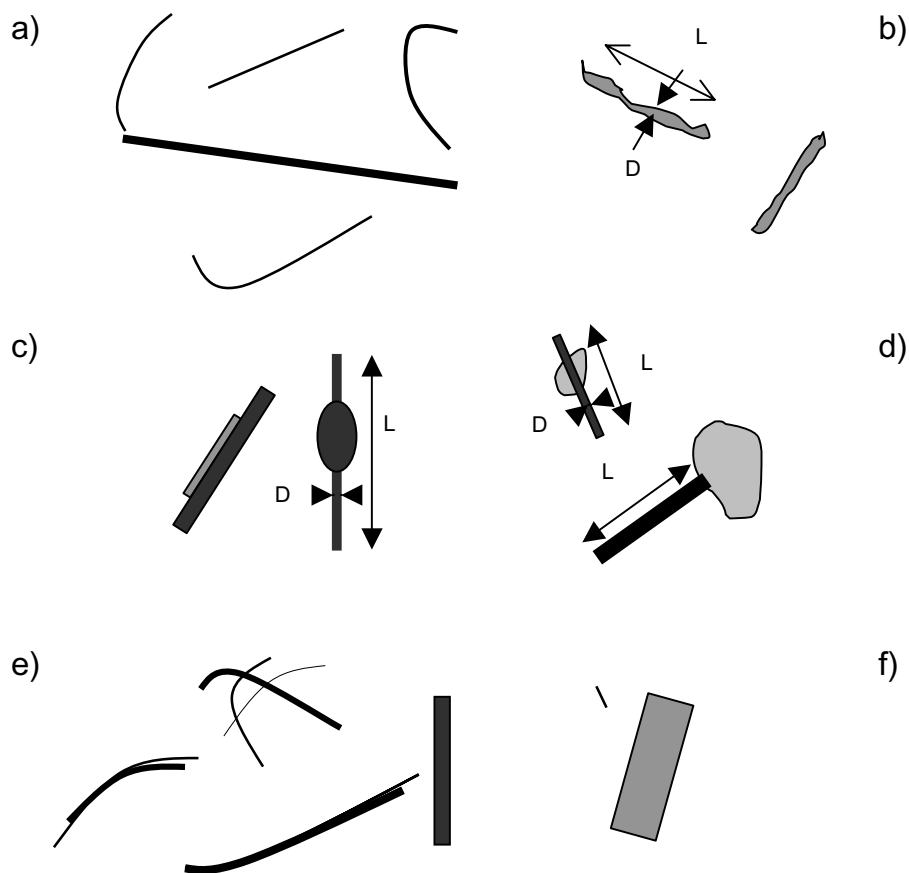
**Fig 2.** Sampling head with filter cassette – system BGIA [6].



**Fig. 3.** Limit of detection in  $F/m^3$  in dependence on the air sample volume in  $m^3$  for 37 mm and 25 mm filters for an analysis of  $0.5 \mu m^2$  and an assumed exposed filter area of  $707 \mu m^2$  and  $380 \mu m^2$ , respectively. See Sections 4.6 and 5.2.



**Fig. 4.** Determination of fibre length and width.



a)  $2 \frac{1}{2}$  Fibres: 5 Ends in the counting field

b)  $1 \frac{1}{2}$  Fibres

c) 3 Fibres: The two adjoining fibres can be discriminated easily. Convexity is ignored for width determination

d) 2 Fibres: The visible section of the fibre is taken into account

e)  $4 \frac{1}{2}$  Fibres: Agglomerate consisting of 3 fibres; splits are ignored. The ends of the fibre located at the right edge are considered to be out of field.

f) 0 Fibres: The fibrous particles are too short or too thick

**Fig. 5.** Examples for the application of the fibre counting rules (the length of the edge of the image is equivalent to  $38 \mu\text{m}$ ).