Determination of airborne fibre number concentrations

A recommended method,
by phase-contrast optical microscopy
(membrane filter method)

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Preface

The inhalation of airborne fibres in the workplace can cause a variety of occupational respiratory diseases, which contribute appreciably to morbidity and mortality among workers in both developing and developed countries. Monitoring airborne fibre concentrations is an important tool for occupational health professionals for assessing exposure and establishing the need to control it, evaluating the efficiency of control systems, and characterizing exposure in epidemiological studies. However, a proliferation of methods has hitherto hindered the comparability of results, as well as the possibility of having worldwide proficiency testing to ensure the reliability of obtained data.

A methodological framework that allows for meaningful comparisons between results obtained by different researchers and laboratories is of immense benefit for all aspects of occupational hygiene, but particularly for exposure assessment and environmental monitoring. In addition to proficiency testing, quality assurance schemes, comparisons and exchanges of data and international collaborative studies depend on the use of compatible methodologies. Furthermore, to ensure that preventive control systems in the workplace are adequate and effective, the reliability and comparability of monitoring and exposure data are essential. Occupational health surveillance and the ability to establish correlations between epidemiological and environmental indicators also depend on the ability to compare data from diverse sources.

A project aiming to establish a unified methodology for the evaluation of airborne fibres in the work environment was therefore carried out by the World Health Organization (WHO). A draft text of the present method specification was initially prepared by Dr N. P. Crawford, Institute for Occupational Medicine, Edinburgh, Scotland. During the course of four meetings of an international working group of experts (see Annex 3 for a list of participants at the final meeting),

current evaluation methods were compared and their differences identified and analysed, with a view to understanding the effect of these differences on the results of counting airborne fibres. Consensus was reached by the working group on a recommended method for the determination of airborne fibre number concentrations by phase-contrast optical microscopy. Dr Crawford's role as rapporteur at the final meeting and his work in revising the draft manuscript are gratefully acknowledged.

Collaboration from the International Labour Office (ILO), the European Commission (EC), the International Organization for Standardization (ISO), the European Committee for Standardization (CEN), the International Fibre Safety Group (IFSG), as well as national agencies, particularly the Health and Safety Executive (HSE), United Kingdom, and the National Institute for Occupational Safety and Health (NIOSH), USA, has been fundamental to this project and is also gratefully acknowledged. Special thanks are offered for the financial support provided by the EC and the IFSG.

Particular acknowledgement should also be made of the valuable personal contribution of several members of the working group, particularly Dr N. G. West (HSE), Dr P. Baron (NIOSH), and Mr S. Houston (IFSG), as well as Mrs B. Goelzer, Occupational Health, WHO.

In its first phase, this project has focused on methodology, so that authoritative scientific knowledge can be utilized to ensure accurate and precise measurements of airborne fibre number concentrations. In its second phase, the project will consider worldwide efforts for proficiency testing, quality assurance, and technical cooperation, including training and education.

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Chief, Occupational Health
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Outline of the method specification

Principle of the method

A sample is collected by drawing a known volume of air through a membrane filter by means of a sampling pump. The filter is rendered transparent ("cleared") and mounted on a microscope slide. Fibres on a measured area of the filter are counted visually using phase-contrast optical microscopy (PCOM), and the number concentration of fibres in the volume of air is calculated.

Sampling

Filter:	Membrane of mixed esters of cellulose or cellulose nitrate, 0.8–1.2μm pore size, 25mm diameter.
Filter holder:	Fitted with an electrically conducting cowl.
Transport:	In closed holders.
Flow rate:	0.5–16 litres·min^{-1}. Adjust to give 100–650 fibres·mm^{-2}.
Blanks:	Sampling media, 4% of filters. Field, ≥2% of samples. Laboratory, optional.

Sample preparation

Acetone–triacetin for fibres with a refractive index >1.51; stable for ≥1 year.

Acetone/etch/water for fibres with a refractive index ≤1.51; unstable.

Sample evaluation

Technique:	Phase-contrast optical microscopy.
Microscope:	Positive phase contrast, ×40 objective, ×400–600 magnification.

	Walton–Beckett graticule, type G-22 (100 ± 2 μm diameter).
	HSE/NPL Mark II test slide.
	Stage micrometers (1 mm long, 2-μm divisions).
Calibration:	To meet visibility requirements of the test slide.
Analyte:	Fibres (visual count).
Counting rules:	Select counting fields at random, subject to defined criteria.
	A countable fibre is >5 μm long, <3 μm wide and with a length:width ratio >3:1, subject to defined rules when it overlaps the graticule perimeter and when it touches other fibres or particles.
Lower limit of measurement:	10 fibres per 100 graticule fields.
Bias and reproducibility:	See sections 4 and 5.

1. Introduction

Many countries have established personal exposure limits for airborne fibres in workplace atmospheres in terms of fibre number concentrations, i.e. the number of fibres in one millilitre of air. The method typically used to determine these concentrations for comparison with exposure limits is the so-called membrane filter method. However, experience has shown that this method does not always produce comparable results when used by different laboratories and different analysts. In fact, its precision is among the poorest of any occupational hygiene assessment method. Differences in results can arise through variations in sampling method, sample preparation, optical microscopy, the calculation of results and other factors, but particularly as a result of subjective effects associated with the visual counting of fibres. Such differences have both systematic and random components. The application of standard procedures and the establishment of a reproducible routine is the only way of controlling most of the sources of error inherent in the membrane filter method, which despite its limitations is the only method suitable for widespread international use in developed and developing countries.

Various specifications for the membrane filter method have been published by international, regional and national organizations. Differences in detail between the specifications are found when they are applied both to different fibre types and to the same fibre type (e.g. EEC, 1983; WHO, 1985; ISO, 1993; NIOSH, 1994). There are now fewer differences in the specifications than in the past, but such differences as still exist can have important systematic effects on results. The magnitude of this systematic variation depends on the sampling method, filter type and process employed. Further harmonization is needed to eliminate method specification as a source of variation and, with proper training and quality control, to ensure comparability between results produced by different microscopists and laboratories.

The method recommended by WHO, as set out in the main text of

this publication, relates to measurement of the number concentration of airborne fibres of all types for the purpose of assessing personal exposure in the occupational environment. Modifications are needed for application to static monitoring; these are described in Annex 1. These specifications have benefited from a review of the relevant literature previously prepared (Crawford, 1992).

Sampling strategies are not covered by this publication, but a training manual on this method, as well as sampling strategies specific to fibres, is envisaged. Sampling strategies have also been well covered in the specialist literature (e.g. NIOSH, 1977; ACGIH, 1991; AIHA, 1991; BOHS, 1993; NIOSH, 1994; ACGIH, 1995; CEN, 1995).

Reliable results depend on participation in a suitable quality assurance programme. The general requirements for the technical competence of testing laboratories published by the International Organization for Standardization (ISO, 1990) should therefore be followed. Microscopists should participate in intralaboratory counting checks, and laboratories should participate in a proficiency testing scheme.

It is hoped that this publication will motivate a review of the various specifications currently in use and that the method presented here will eventually be used by all countries, irrespective of the fibre type being assessed.

2. Scope of application

The method set out in the following pages is applicable to the assessment of concentrations of airborne fibres in workplace atmospheres—most commonly personal exposures—for all natural and synthetic fibres, including the asbestos varieties, other naturally occurring mineral fibres and man-made mineral fibres. The method can be used in sampling or monitoring carried out for the purposes of:

— comparison with occupational exposure limits
— epidemiology
— assessing the effectiveness of control measures and monitoring the effects of process modifications.

The method is appropriate for the above applications when information is required about the number concentration of airborne fibres. Occupational exposure limits for some fibres may also be expressed in gravimetric units ($mg \cdot m^{-3}$); in these cases, mass concentrations are measured by other methods referred to in *Safety in the use of mineral and synthetic fibres* (ILO, 1990). Unlike the membrane filter method, these gravimetric methods are not specific for fibres, since particles and non-countable fibres are included in the mass.

The method presented here measures the number concentration of airborne fibres, defined as objects with a length >5 µm, a width <3 µm and a length:width ratio (aspect ratio) >3:1, using a phase-contrast optical microscope.

Setting an upper width limit means that, for some fibre types, some wide fibres will not be counted. All fibres satisfying the dimensional criteria and counting rules defined in section 3.2.3 of this method should be counted.

Many fibres are too small to be visible by optical microscopy. The minimum visible width depends on the resolving power of the optical system, the difference in refractive index between the fibre and the surrounding medium, and the visual acuity of the microscopist. With

a good, correctly adjusted microscope which conforms to the specification of this method, the limit of visibility is in principle about 0.13–0.15 μm. However, in practice, the smallest visible fibres will be about 0.2–0.25 μm wide. Since some fibres fall below the limit of visibility, the PCOM fibre count represents only a certain proportion of the total number of fibres present (the exact proportion varies and depends on factors such as the sample type and the analyst). Thus the count represents only an index of the numerical concentration of fibres and is not an absolute measure of the number of fibres present.

Use of this method has other limitations when applied to samples containing "platy" (flat) or acicular (needle-shaped) particles and consequently should not be implemented without a full understanding of the workplace atmosphere (ISO, 1993). The method does not permit the determination of the chemical composition or crystallographic structure of fibres and therefore cannot be used on its own to distinguish between different fibre types. However, supplementary information on fibre type or size may be obtained by using other methods when necessary, such as polarized light microscopy, scanning electron microscopy and transmission electron microscopy. Annex 2 provides guidance on the scope and application of these methods. Such methods may be particularly appropriate when different fibre types are present in the same workplace and when airborne dust is a mixture of fibres and other types of particle. Supplementary methods are also useful for epidemiological studies, where a more detailed characterization of the properties of airborne contaminants is usually required.

3. Specifications of parameters

For the method parameters presented below, the recommended specification is generally given in italics following the section heading. Supplementary information is given in normal text. This supplementary material may include one or more other specifications that may be used, provided that they are demonstrated by the user to produce equivalent results to those obtained by the recommended specification.

3.1 Sampling

3.1.1 Filter

The membrane filter should be of mixed esters of cellulose or cellulose nitrate and should have a pore size of 0.8–1.2 μm and be 25 mm in diameter.

Lower fibre counts have been observed when filters of larger pore size have been used. The range of pore sizes above has suitable filtration and pressure-drop characteristics; it also allows scope for the reasonable variations in flow rate that may be required to obtain optimal fibre densities. Filters with pore size <0.8 μm offer an increased resistance to flow that may cause problems for some pumps.

Measured fibre concentrations have been found to be comparable for filters of diameter 13 mm, 25 mm and 37 mm when exposed to the same face velocity during sampling. However, 25-mm filters have advantages over 37-mm filters in that the smaller size of the sampling head is more amenable to personal sampling, and the whole filter can be mounted if required; 25-mm filter holders are now in widespread use internationally. There have been reports of unevenness of deposit on both 25-mm and 37-mm filters. Notwithstanding this problem, there remains scope for variations in filter diameter to be used to obtain optimal fibre densities; in particular, 13-mm filters may become more useful in future as fibre concentrations are progressively reduced in most workplaces.

Either gridded or ungridded filters may be used. Printed grids aid

both focusing the microscope on the plane of the fibres and position identification. Moreover, any distortion of the filter grid lines indicates a disturbance of fibre distribution associated with errors in the filter mounting procedure.

The quality of each batch of filters should be checked before use (see section 3.1.8).

The diameter of the exposed area of the filter must be known. If components of the filter holder that are likely to affect this (e.g. O-ring) are changed, the effective diameter should be remeasured. An acceptable method involves collecting a sample from a thick cloud of dark-coloured dust and mounting the filter on a microscope slide in the normal way. The diameter of the dark deposit may be measured with vernier callipers, but the slide is usually placed on a microscope stage and the filter observed at low magnification while a diameter of the dark area is traversed by moving the stage. The distance moved is obtained from the stage vernier scale. Two diameters on each filter should be measured, and three filters in separate holders should be checked in this way. If different types of filter holder are in use, the process should be repeated for each type. Provided that the three filter diameters differ by no more than 1 mm, an arithmetic mean is taken and used as the effective filter diameter. If differences in diameter are found to be greater than 1 mm, the sampling and filter-mounting techniques should be investigated; the gross appearance of the filter may reveal problems with the filter holder or the mounting procedure (e.g. a leak or an uneven deposit). The effective filter diameter should not be less than 20 mm.

The appearance of needle-like objects has been reported on membrane filter samples that have been wetted (and have retained some moisture) before mounting. The objects, which appear about 4 days after clearing and mounting with acetone–triacetin, satisfy the definition of a countable fibre in section 3.2.3. Filters should therefore be dry before they are cleared and mounted.

Electrostatic repulsion of fibres by the filter may occur and is more likely under conditions of low humidity. In such cases, the filter may be pretreated with a suitable surfactant to eliminate electrostatic charge. The surfactant should be certified as particle-free. One suitable procedure is to immerse the filter in a 0.1% solution of benzethonium chloride and dry it overnight on a sheet of blotting paper to prevent localized globules of detergent from blocking up part of the filter (Mark, 1974).

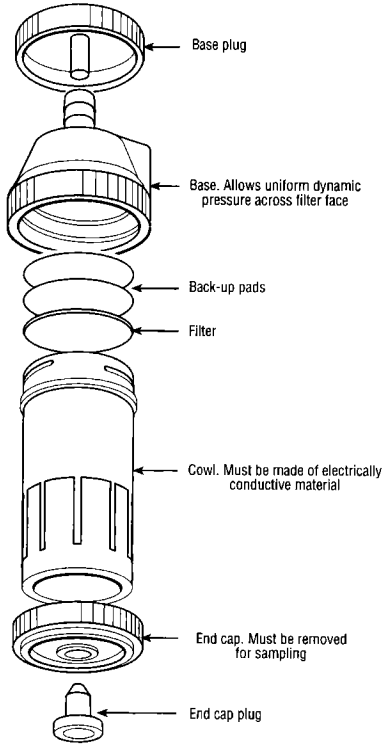


Fig. 1 Exploded view of a personal sampling head

3.1.2 Filter holder

An open-faced filter holder fitted with an electrically conducting cowl should normally be used.

An example of a filter holder suitable for personal sampling is illustrated in Figure 1.

The cowl helps to protect the filter from accidental contamination and unintentional physical damage while permitting uniform fibre deposit. The cowl length is normally 1.5–3.0 times the effective filter diameter. Shorter cowls may be used if they can be shown to yield the same results as longer ones. Care should be taken to ensure that the cowl is well seated in the cassette so that leakage is minimal.

Shrink-tape may be employed to help seal the cowl to the filter holder; it keeps joints clean and prevents contaminants from getting onto the filter when the filter holder is disassembled.

A conducting cowl should be used to minimize the risk of fibre loss due to electrostatic effects. Such effects are most likely to occur under conditions of very low humidity and with highly charged fibrous aerosols. Fields of view towards the centre of the filter should be selected for counting (avoiding the outer 4-mm region of the filter circumference), since fibre loss is most likely to occur near the edge of the filter where static charge accumulates (see section 3.2.3). It is important to ensure that filter loadings fall within the optimal range for fibre density specified in this method (see section 3.1.5), in order to minimize counting bias.

In some situations, fibres may be deposited on the cowl because of electrostatic effects or accidental overloading of the filter. Occasionally cowls are rinsed in order to collect and count these fibres. It is possible that rinsing fibres from the cowl and depositing them on the filter may introduce counting biases. Moreover, unused plastic cowls can be significantly contaminated with "background" fibres (i.e. shreds of plastic) that may be released on rinsing. Therefore, any fibres rinsed from the cowl should be disregarded when determining airborne fibre number concentration. Where it is foreseen that fibre deposition on the cowl will be appreciable, an open-head filter holder without a cowl may be used.

If O-rings are used, they should be made of polytetrafluoroethylene. Airtight flexible tubing is required to connect the filter holder to the pump.

In correct use, the cowl will point downwards. For personal sampling, the filter holder should be fixed to the upper lapel or shoulder of the worker's clothing, as close to the mouth and nose as practicable but in any case no more than 300 mm away. Wherever possible, the same sampler position should be adopted for each worker. In some circumstances, a higher concentration may be expected on one side of a worker than the other; the sampler should be then positioned on the side expected to give the higher result.

In the future, a size-selective sampler, replacing the cowled sampler, may be adopted as a more specific and accurate way to sample fibres; this would obviate the need to determine fibre width and would reduce the background disturbance caused by the presence of large particles.

3.1.3 Storage and transport

Fixatives should not be used. Filters should be transported in closed holders which should be opened only immediately before use and sealed immediately afterwards.

Experience has shown that fixing fibres to the filter surface with cytological or other fixatives is unnecessary and should not be done.

Transport of filters in the filter heads is preferred. If this is impracticable, clean, degreased tins with close-fitting lids, or similar containers, may be used to transport the filters. Adhesive tape can be used to secure the clean, unexposed edge of the filter to the tin (if used), and the tape can subsequently be cut from the filter with a surgical scalpel. The filter holders or tins should be packed into a rigid container with sufficient soft packing material to prevent both crushing and vibration of the filters. Containers should be unambiguously labelled, and caution is necessary to ensure that filters cannot be accidentally reused. The filters themselves should not be marked for this purpose because of the risk of damaging them.

Care should be taken to minimize electrostatic effects in handling filters. Boxes fitted with expanded polystyrene should not be used.

To minimize contamination, the filter holders and cowls must be clean before use, and the filters should be packed, unpacked and analysed in an area as free from fibre contamination as practicable. Care must be taken to handle the filter at all times with only good quality tweezers and only by the edge. The entrance to the cowl should be closed with a protective cap or bung before and during transport.

3.1.4 Sampling pump

A portable, battery-operated pump is normally used for personal sampling. The capacity of the battery should be sufficient to operate continuously over the chosen sampling time. The flow, with the filter connected to the pump, should be free from pulsation.

The pump should give a smooth flow which should be known to be within ± 10% of the required flow rate for flow rates ≤2 litres · min^{-1}, and to within ± 5% for flow rates >2 litres · min^{-1}. It should be capable

of maintaining this flow rate through the filter during the period of sampling. This acceptable variation includes any change of flow rate caused by changes in the pump orientation. The pump should be light and portable, and a belt may be required to hold it if it is too large to fit in a worker's pocket. The battery should be powerful enough to operate the pump within the specified flow limits for the duration of measurement. Although pumps are usually equipped with pulsation dampers, an external damper may have to be installed between the pump and the filter.

It may be necessary to warm up the pump, depending on the pump type and the environmental conditions. To do this, the pump should be attached to a loaded filter holder using flexible tubing and allowed to run at the chosen flow rate for 15 minutes so that the flow rate can become steady (experience may show warm-up to be unnecessary with some types of stabilized-flow pumps). The filter should then be discarded and a new one fitted for collecting the sample (or a separate filter holder, loaded with a filter, may be dedicated for the warm-up of several pumps). The flow should be readjusted after warm-up to the chosen flow rate using a calibrated airflow meter. One satisfactory procedure is to connect the calibrated meter to the entrance of the cowl by a bung and tube. When the flow-setting is finished, the pump should be switched off and a protective cap fitted to the cowl entrance. The pump should not be operated without a filter, to avoid any damage to it by dust particles.

The sampling system may be checked for leaks by activating the pump with the filter-holder assembly closed and a flow-measurement device in-line. Any measurable flow indicates a leak that must be eliminated before the sampling operation is started.

3.1.5 Optimal filter fibre loadings

Where contamination from non-fibrous particles is low, the target range for fibre density for optimal accuracy and precision should be about 100–650 fibres·mm^{-2}. The upper density limit of the range may be extended to 1000 fibres·mm^{-2} if few interfering particles are present but may need to be reduced where many non-fibrous particles or agglomerates are present. In special circumstances the lower density limit may also be reduced.

Biases in the estimation of fibre concentration can arise from subjective differences in visually counting the fibres. Generally, but not invariably, fibre counts are underestimates at high densities and overestimates at low densities. Also, at lower densities, the variability of counts increases. Both subjective bias and density-dependent variability are minimized with the fibre density range specified above for "clean" samples. Counts in this density range can be achieved by a suitable choice of flow rate, sampling time and filter diameter.

In situations where non-fibrous particles or agglomerates are present, it may be necessary to reduce the maximum fibre density target to minimize obscuring of fibres by other particles. When fibre loadings are less than about 100 fibres·mm^{-2}, results are subject to greater error and may be biased. Such results may still be used in certain circumstances (e.g. when an indication of concentration with low precision is acceptable), provided that they are above the lowest measurable fibre density (see section 4.3).

3.1.6 Flow rate

The flow rate used in sampling should be in the range 0.5–2.0 litres·min^{-1} when comparisons are to be made with fibre-concentration limit values defined for 4-hour or 8-hour reference periods. Flow rate should be adjusted, wherever possible, to give fibre densities in the optimal range for accuracy and precision.

For comparisons with limit values defined with reference to short periods (e.g. 10 minutes) the flow rate may be increased to 16 litres·min^{-1}.

Over a wide range of flow rates, sampling efficiency is largely independent of flow rate for the fibres of interest. This means that flow rates can be varied, provided that they give the same filter loading or give loadings within the optimal range for fibre density specified in this method.

The lower flow rate of the defined range should be such as to minimize loss of precision, and the upper rate should be within the efficient working capacity of the sampling pumps operating under normal conditions. The limiting flow rates specified above take account of these factors under most conditions.

Operationally, it will be most convenient to use a single flow rate

for the majority of workplaces in a factory; the flow rate should be changed only for those workplaces where experience or historical data indicate that a different flow rate (or a change in filter diameter) is needed to give acceptable filter loadings.

A calibrated working flow-meter is necessary when the pump is first set up and when the flow is checked subsequently. This would normally be a portable flow-meter of the variable-area, or supported-float, type (i.e. "rotameter"), previously calibrated against a master flow-meter. The master flow-meter should preferably be a flow-meter whose accuracy is traceable to national standards, used with careful attention to the conditions of the calibration certificate. A "bubble" flow-meter may also be used (Figure 2). This is a device in which the pump being tested draws a soap film through a calibrated tube. The passage of the film is timed between two marks whose separation defines a known volume. A 1-litre burette can serve as a suitable tube. The volume between the marks can be checked by filling the burette with distilled water, allowing temperatures to stabilize, drawing off the volume and weighing the water, making allowance for the dependence of volume on temperature. A suitable bubble solution can be made by mixing one part of concentrated washing-up liquid (detergent), two parts of glycerol, and four parts of water. The burette must be thoroughly wetted with the solution, and several attempts at drawing the film through the tube may be necessary before the tube is wet enough for this to be achieved consistently. (For this calibration to be

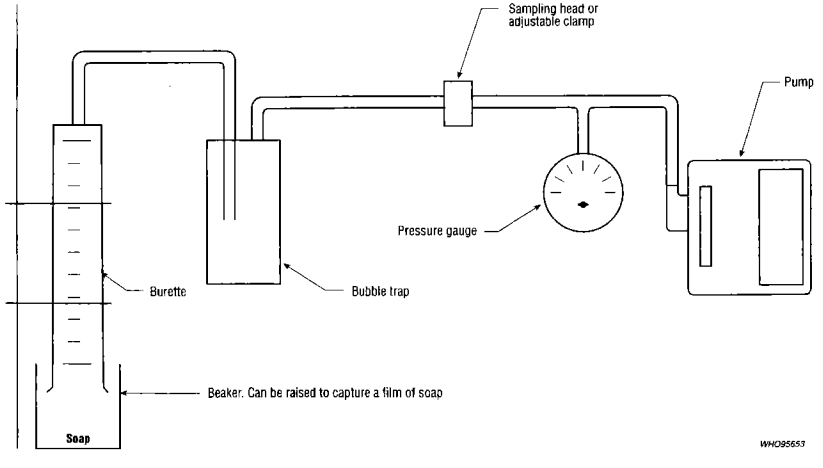


Fig. 2 Calibration of flow using a bubble flow-meter

traceable also requires checking the timing devices against a traceable timepiece and the use of certified weights.)

The working flow-meter must be sufficiently sensitive to permit the flow to be read within ± 10% for flow rates ≤2 litres · min^{-1} and within ± 5% for rates >2 litres · min^{-1}. If it meets this requirement, a flow-meter incorporated into the pump can be used, but it must be calibrated with a filter holder and filter in-line, and read in the vertical position if it is of the rotameter type. If there are any leaks between the sampling head and the flow-meter, the reading will be inaccurate.

The flow rate should be checked, as a minimum, before and after sampling. If the measured difference in the readings is greater than 10%, the sample should be rejected.

The master flow-meter should be used only to calibrate the working flow-meters, and for no other purpose. The frequency of the recalibration of the master and working flow-meters will depend on the amount and type of use to which the flow-meters are subjected. Generally, calibration should be at least annual and monthly for the master and working flow-meters, respectively, unless documentary evidence to justify longer intervals can be provided.

The following additional points about the calibration procedure should be emphasized:

- Working flow-meters should normally be calibrated in series with the master flow-meter.
- In order to minimize errors due to pressure-drop between the two meters, the tube connecting the flow-meters should not be of a smaller diameter than the connection ports of the flow-meters, and its length should be kept to a minimum. All connections should be airtight.
- No restrictions or valves should be fitted between the two flow-meters.
- Where variable-area flow-meters are chosen for both the master and working flow-meters, the scale length of the master flow-meter should be equal to or greater than the scale length of the working flow-meter.
- Calibration should cover the expected range of flow rate for each flow-meter used.

Working flow-meters are used in widely varying environmental conditions. All air sampling measurements are concerned with volu-

metric flow rate (i.e. flow rate measured and expressed at the prevailing temperature and pressure) and not mass flow rate (i.e. flow rate corrected to standard temperature and pressure). Recalibration or correction of flow rate is therefore essential if the pump is operated under conditions substantially different from those of calibration (e.g. differences in altitude). If possible, calibration should be carried out at the sampling site. If this is not possible, a correction may be needed if the pump is affected by temperature and pressure changes. The actual flow rate will be given by:

$$Q_a = Q_c \sqrt{\frac{P_c T_a}{P_a T_c}}$$

where Q_a = actual flow rate
Q_c = calibrated flow rate (the rotameter value)
P_c = air pressure at site of calibration
P_a = air pressure at sampling site
T_a = air temperature at sampling site
T_c = air temperature at site of calibration

Flow rate should be corrected if differences in ambient temperature or pressure between calibration and sampling sites are greater than 5%.

At the start of the sampling period, the protective cap must be removed from the filter holder, the pump started and the time noted. The flow rate should be checked periodically (e.g. hourly) during sampling, using a calibrated flow-meter, and readjusted to the chosen rate. Operating experience may show this to be unnecessary with some types of pump, but it is still advisable to confirm that the equipment is functioning satisfactorily. At the end of the sampling period, the time should be noted, the flow rate checked, the pump switched off and the protective cap replaced on the filter holder.

3.1.7 Single sample duration

The sampling duration for each sample should take account of the fibre loading considerations detailed in section 3.1.5.

The following formula can be used to determine the sampling duration for each sample:

$$t = \frac{A}{a} \times \frac{L}{c_c} \times \frac{1}{r}$$

where t = single sample duration (min)
A = effective filter area (mm^2)
a = graticule area (mm^2)
L = required fibre loading (fibres/graticule area)
c_c = average fibre concentration (fibres $\cdot$ ml^{-1}) expected during the single sample duration
r = flow rate (ml $\cdot$ min^{-1})

Where information about past measurements is available, this should be used to estimate the single sample duration required; otherwise a limit value is a useful starting point. Where an exposure limit value is specified for a short time period in national legislation, that period can be used as the duration of a single sample. Obtaining optimal fibre loadings requires consideration of the interaction between sampling time, volume, flow rate and likely fibre concentration. Sampling time should be measured accurately (to within ± 2.5%), preferably using timepieces that are traceable to national standards.

3.1.8 Blanks

The maximum acceptable fibre count for blank filters is 5 fibres per 100 graticule areas.

Blank filters are used to check for contamination of filters as supplied and during handling, storage and transport at all stages (in the field and in the laboratory).

There are three types of blanks:

(1) those that are extracted from each box of 25 filters in the laboratory and mounted and counted before sampling to check that the batch of filters is satisfactory ("sampling media" blanks);
(2) those that are taken to the sampling area and subjected to the same treatment as normal samples but without having the caps of the filter holders removed, having air drawn through them or having them attached to the worker; they are then mounted and counted ("field" blanks);
(3) those that are extracted from satisfactory filter batches (see (1), above) and mounted and counted to check for laboratory contamination ("laboratory" blanks).

For sampling media blanks, the usual procedure is to select four blank filters from each batch of 100 (i.e. one filter from each small box of 25). The proportion of field blanks should normally be about 2% of the total number of samples, unless there are reasons to believe that more field blanks may be needed. A laboratory blank may be evaluated along with each batch of routine samples, or afterwards if contamination due to laboratory sources is suspected.

Median fibre densities for blank filters included in counting comparisons range from 0.3 to 6.7 fibres·mm^{-2}. The latter value corresponds to about 5 fibres per 100 graticule areas, the maximum acceptable count for a blank filter in this method.

Wherever possible, the identity of blank filters should be unknown to the microscopist until all counts have been completed. If elevated counts are obtained, potential internal causes should be investigated first (e.g. microscopist error, contamination of coverslip). If it is concluded that the problem lies with the filter, the whole batch of 100 should be rejected. In the event of contamination, the measurement should be regarded as only a rough estimate of the airborne concentration.

Counts on field blanks should normally be subtracted from sample counts, but if counts on field blanks are high (i.e. above the maximum acceptable limit) the reasons should be investigated.

3.2 Evaluation

3.2.1 Sample preparation

The acetone–triacetin method should be used to clear and mount the filter for fibres with refractive indices greater than 1.51. For inorganic fibres with lower refractive indices, the acetone/etch/water method should be used.

The entire filter, or a portion of it, may be mounted. It may be necessary to retain a portion of the filter unmounted, to follow up if evaluation of the mounted portion indicates that further analysis is needed (e.g. fibre identification). If it is necessary to subdivide the filter, cutting should be done with a scalpel by "rolling" the blade from toe to heel across the filter. Scissors should not be used. The filter should be cut along diameters into wedge-shaped segments that are at

least one-quarter of the filter in area. For some organic fibres, cutting filters may result in significant fibre loss.

Systematic variations in counting levels of as much as 20–30% are associated with different methods of mounting the filter. For a given fibre, the use of a single procedure eliminates such differences. A method for clearing the filter based on the use of acetone vapour is widely used internationally but must be applied with care: any serious distortion of the filter will invalidate the sample. However, for some organic fibres, this method needs to be validated by appropriate controls because the fibres may dissolve to some extent in acetone.

The principle of this filter-clearing method is that the filter is exposed to acetone vapour, which condenses on the filter, collapsing its pores and making it transparent. The filter is then fixed to a glass slide, where it appears as a transparent and, ideally, uniform plastic film with any fibres on the upper surface. A liquid must be added to provide optimal contrast. If the refractive index (RI) of the fibres exceeds 1.51, triacetin (glycerol triacetate) is satisfactory; for inorganic fibres with RI $\leq$1.51, the filter surface must be etched to expose the fibres, and water is used as the contrast liquid. Mounted filters for which the acetone–triacetin method is used deteriorate very slowly, if at all; the acetone/etch/water method is not permanent. It is important to check reagents and materials used for mounting for possible contamination. Practice in mounting clean filters is recommended in order to gain proficiency before a real sample is used. Sampling filters that have been exposed to high humidity should be dried gently before exposure to acetone, to improve their clarity.

Caution: Acetone vapour is highly flammable and slightly toxic. It should never be used in the vicinity of an open flame.

The acetone vapour may be generated by any one of three methods: the "hot block" method (preferred), the "boiler" method and the "reflux condenser" method. The latter two methods must be used in a fume cupboard.

In the hot block method, which is illustrated in Figure 3 (p. 18), just enough acetone to clear one filter is injected into a block with an integral heater. The acetone is vaporized and emerges as a vapour jet from an orifice, below which the filter is placed. Commercial versions of the hot block should be used in accordance with the manufacturer's instructions. The filter is placed centrally on a clean microscope slide, sample-side up, with the grid lines parallel to the slide edges. About 0.25 ml of acetone is injected into the block, so that the vapour emerges

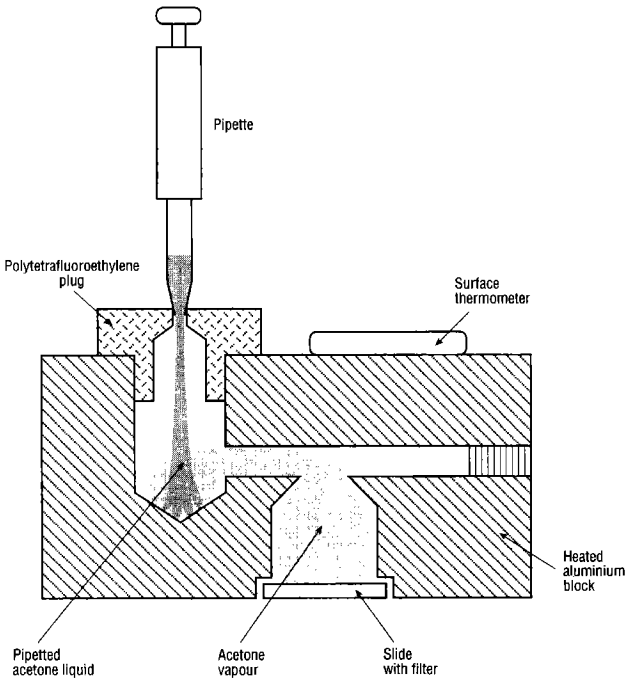


Fig. 3 The hot block method

in a stream over the filter. The filter clears. The small amount of acetone used minimizes the risk of fire, although sources of ignition should be kept away, and the acetone bottle should be kept closed when not in use.

In the boiler method, illustrated in Figure 4 (p. 19), the acetone is vaporized in a tall, narrow, flat-bottomed vessel with a cooling coil near the top. The lid should have a stiff wire cradle attached to it, to hold a microscope slide securely. The coil carries cold water, which confines most of the acetone vapour to the lowest part of the vessel. The water line should include an indicator that shows clearly when water is flowing. A source of heat is required that cannot ignite acetone vapour, such as a recirculating oil-bath. Possible sources of ignition, including electrical switches liable to sparking, should be avoided. The water vapour produced by a hot water-bath, an alternative source of heat, may lead to poor clarity of the cleared filters. Use of a fume cupboard is necessary with the boiler, and a surrounding reservoir to contain any spilled acetone is advisable.

The acetone boiler creates a region of vapour confined by the cool-

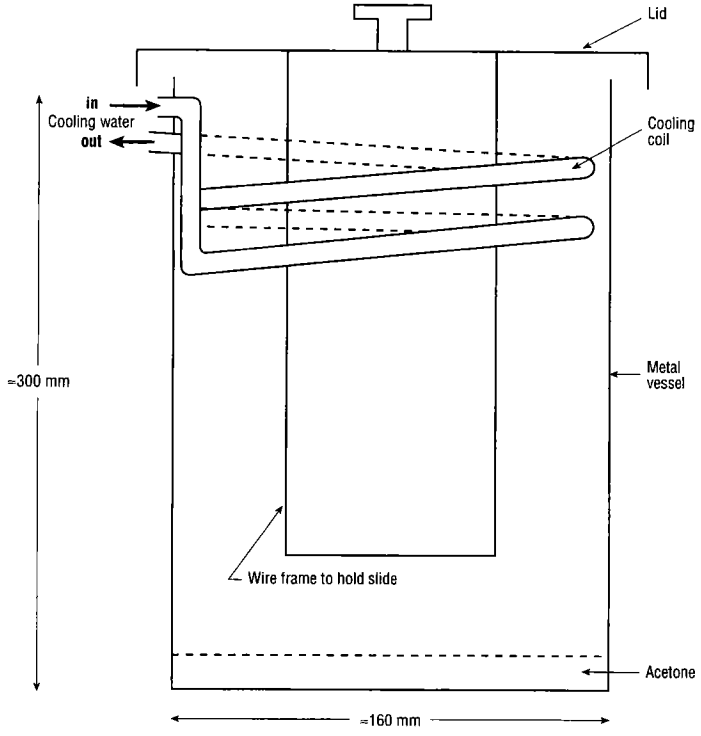


Fig. 4 The boiler method

ing coil, which reduces the risk of fire. The quantity of acetone used should be kept to a minimum, it should not be left in the apparatus when it is not in use, and it should not be left boiling when clearing is not actually in progress. The lid should be kept in place whenever possible. Sources of ignition should be kept well away from the equipment, and a "No Smoking" rule should be enforced.

The cooling water in the coil is turned on, a volume of about 30 ml acetone is put into the vessel, the lid is replaced and the vessel heated. When the acetone boils, the lid is removed, a microscope slide is placed in the cradle with the membrane filter placed centrally on the slide, sample-side facing up and the grid lines parallel to the slide edges. The lid is replaced, lowering the slide into the vapour in the vessel; the slide should clear in a few seconds, and it is then removed.

In the reflux condenser method, shown in Figure 5 (p. 20), the acetone liquid is contained in a three-necked flask fitted with a reflux condenser on one neck outlet. One of the other two neck outlets is

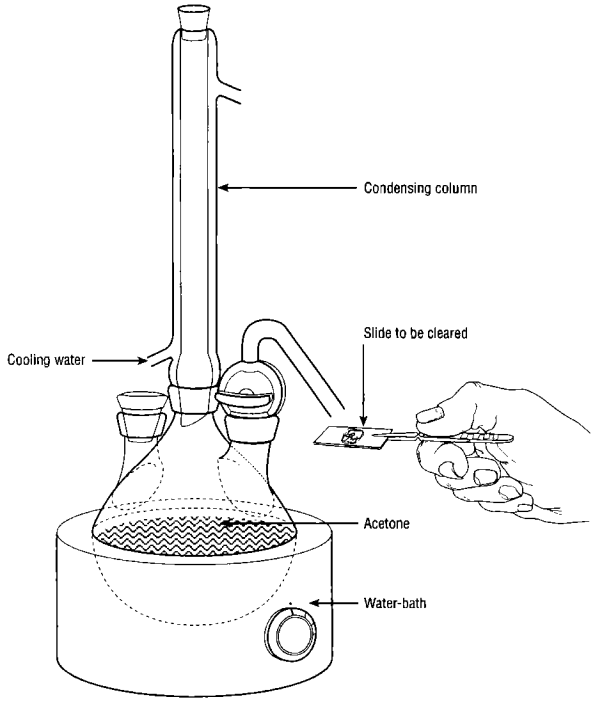


Fig. 5 The reflux condenser method

plugged, and the remaining outlet is fitted with a two-way tap to release the acetone vapour. The acetone is boiled until a steady stream of acetone vapour emerges from the outlet. The filter is placed sample-side up on a clean microscope slide, which is then held with clean forceps directly in the stream, approximately 15–25 mm from the outlet, for 3–5 seconds; the filter is moved slowly across the outlet to ensure even coverage until the filter is transparent. Too little vapour will fail to render the filter transparent; too much vapour (and especially drops of liquid acetone) will destroy the filter by dissolving or shrinking it beyond usability. The slide should not be prewarmed, as the acetone vapour must condense on the slide for correct clearing of the filter.

In each method, a few minutes are allowed for the acetone to evaporate from the filter, and a micropipette is then used to place a drop (about 10 µl) of triacetin on the filter or coverslip, enough to cover the whole filter when the coverslip is in place without excessive overflow around the edges. The coverslip is gently lowered onto the filter at an

angle, so that all the air is expelled, but it should not be pressed onto the filter. The filter becomes less granular in appearance within a short time, and counting may begin as soon as the sample is clear. In some cases, if clearing is slow, the slide may need to be left for 24 hours or heated for 15 minutes at about 50 °C before counting. The mounted slide will usually keep for a year or more without noticeable deterioration, although some small-scale fibre movement can occur; it is desirable to store the slides flat. The long-term keeping qualities can be improved by painting around the edges of the coverslip with a microscopical embedding agent.

If the fibre RI is ≤1.51 (as is true of most inorganic fibres), the following method should be used. The acetone clearing procedure collapses the filter to about 15% of its original thickness, forming a clear plastic film that has minimal distortion and does not deteriorate. The fibres are embedded in the surface but may be invisible under phase-contrast illumination if the refractive index of the fibre is close to that of the filter. The fibres must therefore be exposed by etching away the surface of the filter and immersing them in a liquid of markedly different RI.

To perform the etching, the slide and filter are placed in a plasma asher to remove the surface of the filter. This leaves the fibres exposed but still attached to the filter. An oxygen flow rate of $8\,cm^3 \cdot min^{-1}$ for about 7 minutes, with a forward and reflected radio frequency power of 100 and 2 W respectively is recommended by Le Guen et al. (1980). Information is available elsewhere on the calibration of other types of plasma asher (Burdett, 1988).

A drop or two of distilled water is placed on the etched filter, and a coverslip is placed on top; again, care should be taken not to trap air bubbles. The amount of water should be sufficient to fill the space between the filter and coverslip without excessive overflow. Water has an RI of 1.33 and provides a good contrast, even with fibres of low RI. It is necessary to mount blank filters as controls to ensure that the water is free of fibrous or bacterial contamination; depending upon the findings, it may be necessary to filter the water.

Clean conditions should be maintained at all times. Dirty slide preparation will result in sample contamination and erroneous results. Slides, coverslips, scalpel and forceps should be cleaned with lens tissue or industrial paper tissue and should be checked to be free from contamination and placed on a clean surface, e.g. a lens tissue sheet, before samples are mounted.

3.2.2 Optical requirements

Microscope

The microscope should be set up so that the ridges in block 5 of an HSE/NPL[1] Mark II test slide are visible; those in block 6 may be partially visible, but those in block 7 should not be visible.

Other characteristics of satisfactory microscopes are:

- A Köhler or Köhler-type light source, to provide even illumination of the sample.
- A substage assembly incorporating an Abbe or achromatic phase-contrast condenser in a centring focusing mount, and a phase-contrast ring with a centring adjustment independent of the condenser-centring mechanism.
- A built-in mechanical stage, with slide clamps and x–y displacement.
- A low-power objective, e.g. ×10 magnification, which is useful for locating the stage micrometer and test-slide grids and for carrying out a preliminary check on the evenness of dust deposit on the filter.
- A high-power positive phase-contrast objective, normally with a magnification of ×40, which is the objective used for counting. The numerical aperture of the objective, which determines the resolving power of the microscope, should be between 0.65 and 0.75, and preferably between 0.65 and 0.70. The phase-ring absorption should be between 65% and 85%, and preferably between 65% and 75%.
- Binocular eyepieces, preferably of the wide-field type, yielding a total magnification of ×400–600 but preferably ×500 (corresponding to a ×40 objective and a ×12.5 eyepiece). At least one of the eyepieces must be of the focusing type and permit the insertion of an eyepiece graticule.
- A Walton–Beckett eyepiece graticule, type G-22, matched to the user's microscope.
- Various accessories, including:
  - — a centring telescope or Bertrand lens, to ensure correct alignment of the phase rings in the condenser and objective;
  - — a green filter, which makes viewing easier since microscope optics are designed for the wavelength of green light;

[1] HSE/NPL = Health and Safety Executive, National Physical Laboratory, UK.

— a stage micrometer;
— an HSE/NPL Mark II phase-contrast test slide;
— high eye-point eyepieces with flexible or removable eye caps for microscopists who wear spectacles;
— glass coverslips of a thickness compatible with the design of the objective (e.g. no. $1^1/_2$, normally 0.16–0.19 mm thick) and about 25 mm in diameter or width;
— glass microscope slides of approximately 76 × 25 mm and of thickness 0.8–1.0 mm.

Differences in the smallest widths of fibres detectable by different phase-contrast microscopes may contribute to interlaboratory differences in counts, since typical fibre diameter distributions extend well below the detection limit of phase-contrast microscopes. It is therefore essential to maintain a uniform level of detection at the limit of visibility, and thus the quality of the microscope, including its adjustment and maintenance, is critically important.

Satisfactory counting performance can be achieved by laboratories using a variety of microscopes with specifications consistent with the characteristics described above. It is advisable for all components of a microscope to be from the same manufacturer. The microscope should be regularly calibrated and maintained; the calibration and maintenance arrangements should be documented.

Eyepiece graticule

The type G-22 Walton–Beckett graticule is the only graticule specifically designed for counting fibres of the dimensions specified in the method. This graticule (see Figure 6) is circular, with a diameter of 100 μm in the object plane, and is divided into quadrants by lines scaled in 3-μm and 5-μm divisions; there are also shapes of critical length, width and aspect-ratio values shown around the periphery.

The purpose of the eyepiece graticule is to define the area of the field of view within which fibres are counted and to provide reference images and scales of known dimensions which are used to size fibres. Different shapes and sizes of graticule can affect counting results. The graticule must be made for the microscope with which it is used, taking into account the true magnification. When the graticule is

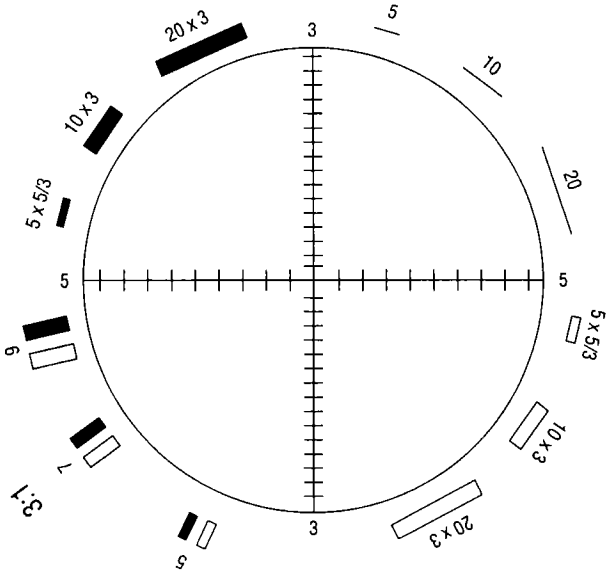


Fig. 6 Walton–Beckett graticule, type G-22

ordered it is therefore necessary to specify the microscope type, the outer diameter of the glass disc of the eyepiece graticule, and also the distance in millimetres on the graticule that corresponds to 100 µm in the object plane of the microscope. This distance can be measured with any other eyepiece graticule as follows:

- The microscope should be set up as if for counting with the available graticule in place.
- A calibrated stage micrometer should be used to measure the apparent length L, in µm, of the scale on the graticule.
- The graticule should then be removed from the eyepiece and the true length Y, in mm, of the graticule scale measured; the true length X, in mm, that corresponds to an apparent length of 100 µm should be calculated as follows:

$$X = 100\,Y/L$$

The value X is the length on the graticule corresponding to 100 µm in the object plane that should be given when ordering. Y should be measured to within ± 2%. If the microscope has a vernier gauge on its stage, and if no better method is available, Y can be measured by placing the available graticule on the stage, observing it at low

magnification, and seeing how far (in millimetres) the stage must be moved to cover the length Y on the graticule.

When received, the Walton–Beckett graticule should be inserted in the microscope for which it was ordered. Its apparent diameter in the object plane should be checked with a stage micrometer and should be between 98 and 102 μm.

This diameter should be checked with the microscope set up for use, and with the correct tube length if it is adjustable. With some microscopes, adjustment of the interocular distance changes the magnification; the microscope should be checked for this effect. If the effect is apparent, the graticule diameter should be measured at the same interocular separation as used during counting. The measured diameter should be used in calculations.

Stage micrometer

The stage micrometer is used to measure the diameter of the Walton–Beckett graticule. Wherever possible, the stage micrometer should be calibrated to national or international standards.

A laboratory should preferably have at least two stage micrometers:

- *One that is an internal reference standard and is traceable.* Such a reference standard should be used for internal calibration of the other stage micrometers and for no other purpose. Traceability is obtained by using the services of either an organization holding national accreditation for the calibration concerned or the organization holding the national standard. The fibre-measurement laboratory should obtain from the calibration organization evidence of the calibration in the form of an official certificate. The traceable stage micrometer need be recalibrated only if its condition or accuracy appears to have changed.
- *One (or more) that is used to measure the diameter of the graticule.* This working stage micrometer must be calibrated when new and at least annually thereafter (or more frequently as required), utilizing the traceable stage micrometer. This is done by measuring the diameter of the graticule using each stage micrometer and comparing the results.

As mentioned above, depending on the microscope, the tube length and interocular distance can affect magnification; these should be set correctly by the microscopist before measuring the graticule. The object-plane diameter of the graticule should be checked daily when in use, before each session of fibre counting. Each analyst should separately measure the diameter of the field of view, with the microscope correctly adjusted for his or her vision. The stage micrometer for this should have a scale preferably 1 mm long and with 2-μm divisions, since the graticule has to be measured to 100 ± 2 μm. The result should be recorded and the measured diameter used in the calculation of fibre densities or concentrations.

Test slide

The HSE/NPL Mark II test slide (see Figure 7, p. 27) is the only commercially available detection-limit test slide.

A test slide checks or standardizes the visual detection limit of phase-contrast microscopes. An important factor contributing to inter-laboratory differences in results is differences in the smallest widths of fibres that can be detected. This is particularly true when microscopes are not set up correctly before evaluation of samples.

The HSE/NPL Mark II test slide consists of a conventional glass microscope slide bearing parallel ridges of resin of decreasing widths (not depicted in Figure 7) in seven blocks, on which is mounted a coverslip with a second resin layer of slightly lower RI. The first resin layer is a replica of a master slide; the second layer produces an optical phase shift representative of that seen when fibre-bearing filters are mounted. A microscope (and observer) can be judged by the finest resin ridges that can be seen. For the microscope/observer combination to be satisfactory, the ridges in block 5 should be visible; those in block 6 may be partially visible, but those in block 7 should not be visible.

The microscope should be set up according to the manufacturer's instructions, and its performance should be checked with the test slide at the beginning of each session of fibre counting. The fine focus and condenser focus may need readjustment before a sample is counted.

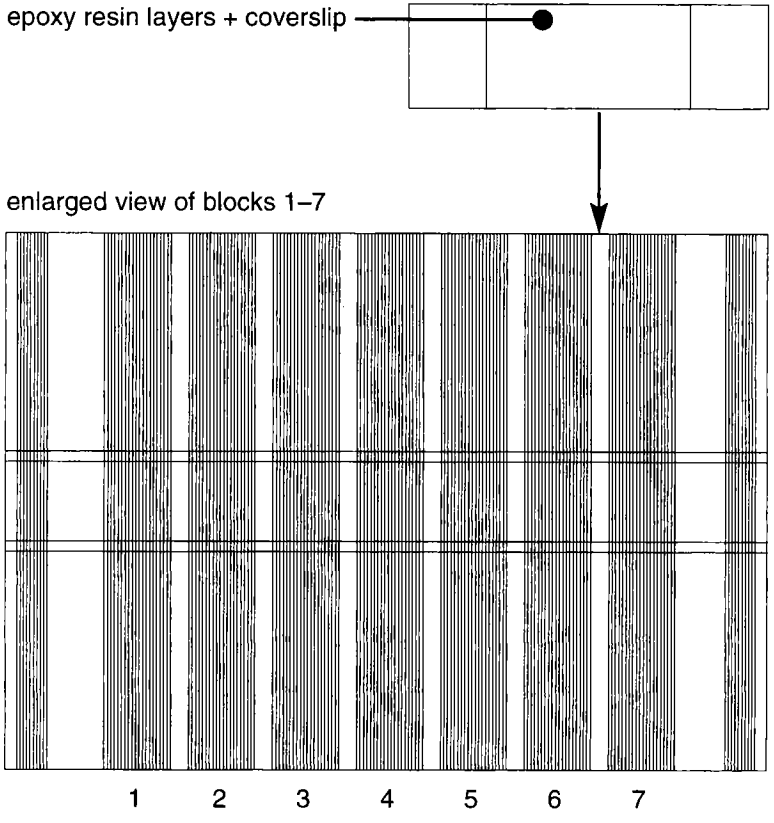


Fig. 7 Schematic representation of HSE/NPL Mark II test slide

3.2.3 Counting and sizing fibres

Low-power scanning

The entire filter area should be scanned with a low magnification (e.g. ×10) objective to check for uniformity of fibre deposit.

A slide with a mounted filter is set up on the microscope stage, and the fibres on the top surface of the filter are brought into focus. Fibres should be evenly deposited over the filter surface (except in the margin normally covered by the filter-holder rim; this should be free of dust and fibres). If the fields observed during low-power scanning show marked differences in loading, or gross aggregation of fibres or dust, the filter should be rejected.

Graticule field selection

Counting fields should be chosen at random throughout the exposed area of the filter to be evaluated. Fields lying within 4 mm of the filter edge (or 2 mm of a cutting line) should be rejected. Fields should also be rejected if (a) a filter grid line obstructs all or part of the field of view, (b) more than one-eighth of the graticule field area is occupied by an agglomerate of fibres and/or particles, by discrete particles or by air bubbles, or (c) the microscopist judges that fibres are so obscured that they cannot be counted reliably.

A random selection procedure is more representative of the whole filter than others, e.g. selecting fields along a diameter of the filter. It is a good idea to divide (notionally) the filter into equal areas (e.g. quadrants), within which approximately equal numbers of random fields are examined. The fine focus must be adjusted for each new field and may need to be changed from fibre to fibre; the microscopist should focus continually on each fibre counted. Counting should take place towards the centre of the filter, where losses due to electrostatic effects are negligible, avoiding the outer 4-mm region of the filter circumference. If condition (a), (b) or (c) occurs, the microscopist should ignore the field and move on to the next one. If the number of rejected fields exceeds 10% of the number accepted, or if the microscopist judges the sample to be uncountable or biased, this should be reported.

Laboratory working conditions

Care should be taken to ensure that the working practices and the working environment in the laboratory do not adversely influence the accuracy of counts.

Laboratory working conditions may influence the counts produced by different microscopists. Different practices of recording data may also cause disagreement between microscopists, because of differences in the rate of eye fatigue. Field-by-field recording of data involves refocusing the eyes after counting each field, whereas continuous registering with an electric or mechanical counter involves only a single

period of continuous concentration. Where possible, the environment should be vibration-free and such that the microscopist can sit in a relaxed and comfortable manner. To minimize eye fatigue, any peripheral view beyond the microscope should preferably be an unobstructed distant view in unchanging subdued light; alternatively, a matt background-shield can be used. Counting should never be carried out in bright sunlight, as this can reduce the contrast between fibres and background.

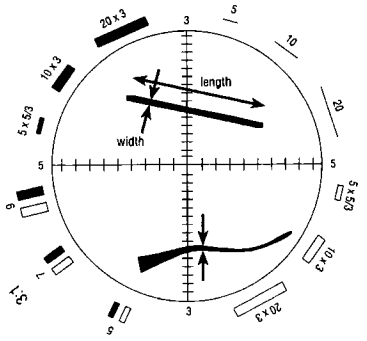
The amount of fibre counting undertaken by analysts in specified periods should be limited, since eye fatigue can adversely affect the quality of counts. A maximum daily working time of about 6 hours is recommended, with microscopists taking 10–20 minute breaks after about 1 hour, although the length of breaks will depend on the microscopist, the samples and laboratory conditions. Eye, upper back and neck exercises should be performed during these periods. The number of samples evaluated in a day will differ from microscopist to microscopist; typically, it takes 10–25 minutes to evaluate a sample.

Counting rules

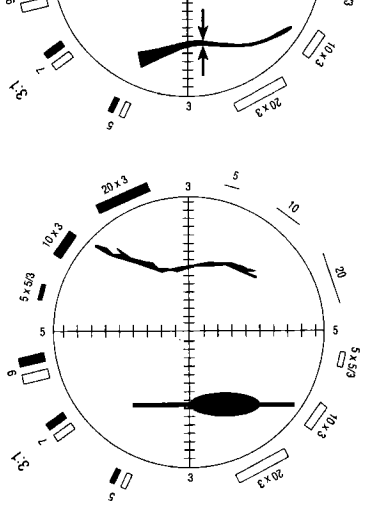
The counting rules described below should be followed.

Airborne fibres collected on membrane filters appear in a wide variety of forms, from single fibres to complex configurations and agglomerates. When presented with the latter, a microscopist can experience difficulty in defining and counting the fibres. In addition, different counting rules can produce differences in results. It is therefore essential when counting fibres (with the ×40 objective) that a single set of rules be used. The rules described below are adapted from those agreed upon for man-made mineral fibres by a WHO Technical Committee (WHO, 1985); examples of the application of these rules are illustrated in Figures 8–11.

- A countable fibre is a particle longer than 5 µm, with a width less than 3 µm, and with a length:width ratio greater than 3:1. A countable fibre with both ends in the graticule field is counted as 1 fibre: a countable fibre with only one end in the field counts as half a fibre. A fibre completely crossing the graticule, with neither end in the field, is not counted.

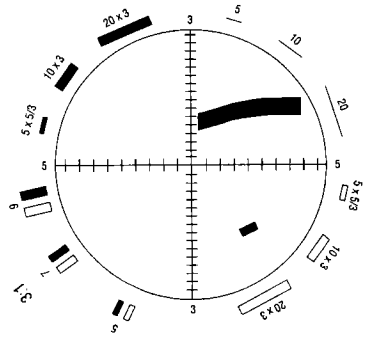


1 fibre; meets length, width and aspect ratio criteria



1 fibre; the width being measured at the 'average' point

1 fibre



1 fibre; ignore the particle or 'bulb' of resin when estimating the width

0 fibres; width too large

0 fibres; aspect ratio is less than 3:1

WHO95658

Fig. 8 Examples of application of fibre-counting rules: single fibres

- If the width of a fibre varies along its length, a representative average width should be considered. Bulges in the fibre, such as are sometimes caused by resin, should be ignored. In case of doubt, the width should be taken to be <3 µm. Fibres that are attached (or apparently attached) to non-fibrous particles should be assessed as if the par-

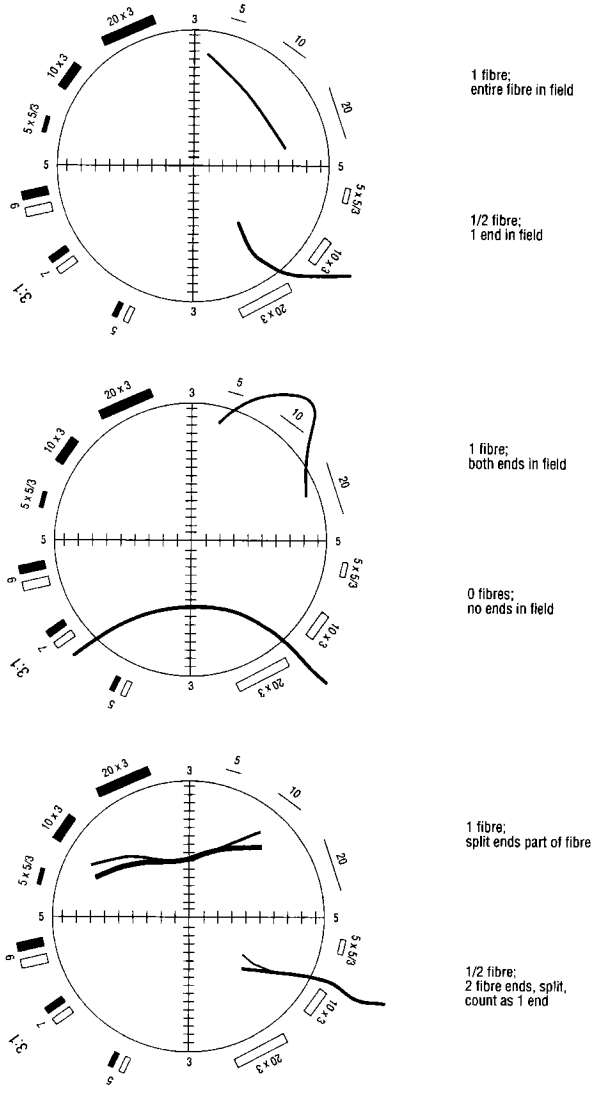


Fig. 9 Examples of application of fibre-counting rules: ends-in rule and split fibres

ticle did not exist; however, only the length of the visible part of the fibre is considered, not the part obscured by the particle (except where a fibre passing through a particle can be seen to be continuous).

- A split fibre is defined as an agglomerate of fibres that, at one or

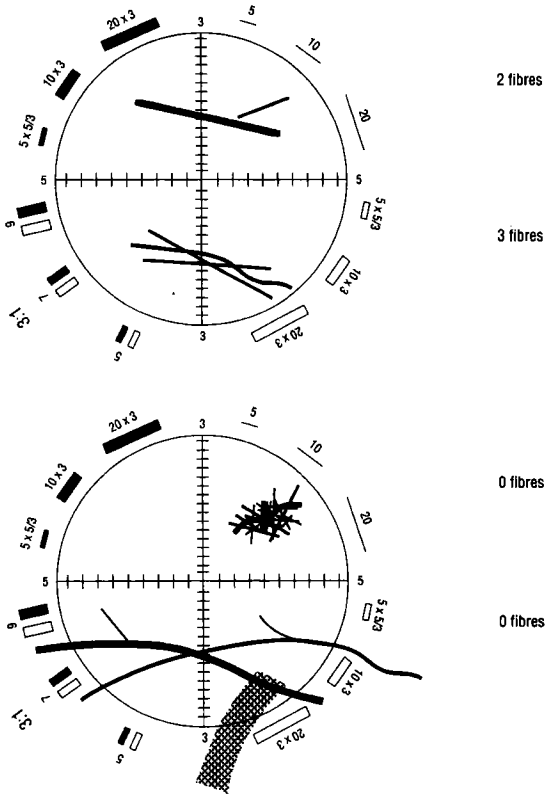


Fig. 10 Examples of application of fibre-counting rules: grouped fibres

more points on its length, appears to be solid and undivided but elsewhere appears to divide into separate strands. Split fibres should be treated as single fibres. The width of a split fibre is measured across the undivided part, not the split part. When several fibres occur together in a group, the fibres should be assessed as separate fibres if they can easily be distinguished. Where they form a clump in which they cannot easily be distinguished, the clump should be ignored unless overall it satisfies the dimensions of a countable fibre, in which case it is counted as 1 fibre.

- The field selection and rejection rules described in section 3.2.3 should be applied.
- At least 100 fibres must be counted or 100 graticule areas inspected. However, fibres must be counted in at least 20 graticule areas, even if these contain more than 100 fibres.

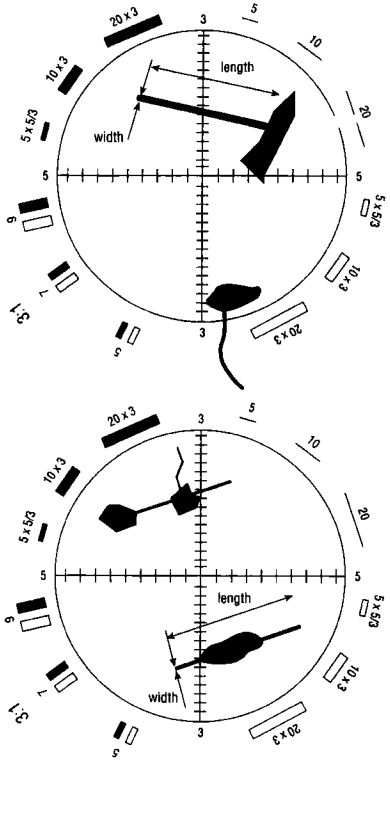


Fig. 11 Examples of application of fibre-counting rules: fibres in contact with particles

3.2.4 Calculation of fibre concentration

(a) For each single sample, the airborne fibre concentration is given by:

$$c = \frac{A \cdot N}{a \cdot n \cdot r \cdot t}$$

where c = concentration (fibres · ml^{-1})
A = effective filter area (mm^2)
N = total number of fibres counted
a = graticule counting area (mm^2)
n = number of graticule areas examined
r = flow rate of air through the filter (ml · min^{-1})
t = single sample duration (min)

Alternatively, this can be expressed as:

$$c = 10^3 \cdot \frac{D^2 \cdot N}{d^2 \cdot n \cdot v}$$

where c = concentration (fibres·ml^{-1})
D = diameter of the exposed filter area (mm)
N = total number of fibres counted
d = diameter of the graticule (μm)
n = number of graticule areas examined
v = volume of air sampled (litres)

(b) When several samples of different durations are taken, the time-weighted average concentration (c_{twa}) is given by:

$$c_{twa} = \frac{\Sigma c_i \cdot t_i}{\Sigma t_i} = \frac{c_1 \cdot t_1 + c_2 \cdot t_2 + \cdots + c_n \cdot t_n}{t_1 + t_2 + \cdots + t_n}$$

where c_{twa} = time-weighted average concentration (fibres·ml^{-1})
c_i = single sample concentration (fibres·ml^{-1})
t_i = single sample duration (min)
Σt_i = sum of the sample durations (min)
$\Sigma c_i \cdot t_i$ = sum of the products of c_i and t_i
n = total number of samples

If the sample durations are equal, average concentration (c_a) can be obtained from the following simplified equation:

$$c_a = \frac{\Sigma c_i}{n} = \frac{c_1 + c_2 + \cdots + c_n}{n}$$

where c_a = average concentration (fibres·ml^{-1})
c_i = single concentration value (fibres·ml^{-1})
Σc_i = sum of the sample concentrations (fibres·ml^{-1})
n = total number of samples

(c) The average concentration for an actual working period (shift) may be obtained in different ways. If the sample duration corresponds to the duration of a shift, the fibre concentration calculated from (a) above yields the shift-average concentration (c_{sa}). If the duration of a single sample is less than the duration of the shift, and the sample is assumed to be representative of the entire

shift, the shift-average concentration is equal to the single sample concentration. If several consecutive samples are taken, and the total duration of the samples is equal to the duration of the shift, the shift-average concentration is calculated according to one of the equations in (b) above, depending on whether the sample durations are equal or not. If several samples are taken randomly during a shift, and the total duration of the samples is less than the duration of the shift, the shift-average concentration can again be calculated as in (b) above, provided that the samples can be assumed to be representative of the entire shift.

(d) To compare shift-average concentration with an exposure limit value that is specified for a defined reference period (usually 8 hours), if the actual shift is either longer than, or shorter than, the defined reference period, the shift-average concentration must be multiplied by a certain factor (f). This adjustment yields a hypothetical concentration to which a worker whose shift duration was equal to the defined reference period would have to be exposed in order to have an equivalent level of exposure. This hypothetical concentration is termed equivalent exposure concentration (c_{eq}) and is calculated as follows:

$$c_{eq} = f \cdot c_{sa}$$

where $$f = \frac{\left(\text{duration of actual working period (shift) in hours}\right)}{\left(\text{duration of defined reference period in hours}\right)}$$

Example 1

Defined reference period = 8 h
Actual working period (shift) = 12 h
Shift-average concentration (c_{sa}) = 1.2 fibres · ml^{-1}
f = 12/8 = 1.5
c_{eq} = 1.5 · 1.2 = 1.8 fibres · ml^{-1}

Example 2

Defined reference period = 8 h
Actual working period (shift) = 5 h
Shift-average concentration (c_{sa}) = 1.2 fibres · ml^{-1}
f = 5/8 = 0.625
c_{eq} = 0.625 · 1.2 = 0.75 fibres · ml^{-1}

4. Accuracy, precision and lower limit of measurement

4.1 Accuracy

Since it is not possible to know the "true" fibre concentration of a given dust cloud, the absolute accuracy of the described method cannot be assessed. However, something is known about the bias associated with sample evaluation.

Microscopists generally undercount dense and overcount sparse dust samples. When fibres are sampled in atmospheres relatively free from interfering particles, the sample density range for optimal accuracy is between 100 and 1000 fibres·mm^{-2}; for densities above this, results can be underestimates while for lower densities, results can be overestimates. No corrections should be made, however. Results outside the optimal range should simply be reported as having reduced accuracy.

In environments where there is more than one type of fibre, the presence of different fibre or particle types may interfere with the accuracy of results. Chance superimposition of non-fibrous particles may cause fibres not to be counted fully, by a proportion that depends on the mean size and concentration of the interfering particles. In practice, the effects of chance superimposition on counting results are small compared with effects due to differences in individual microscopists and will not be important for the counting rules described in this method.

The counting procedure that is used can result in differences in the counts produced by different microscopists within and, more particularly, between laboratories. Such differences should be minimized by proper training in the recommended procedure, internal quality control and proficiency testing (see section 5).

4.2 Precision

Factors contributing to poor precision are the relatively small portion of the filter surface that is examined and the variable distribution of fibres on the surface (statistical variation), the existence of different method specifications (systematic variation), and the possibility for differences in counts made by different microscopists (subjective variation). Systematic and subjective variation can be reduced by harmonizing the methods used, training personnel, ensuring that laboratories participate in proficiency testing, and performing internal quality control testing (see section 5). Even when such factors are addressed, statistical variation is an unavoidable source of error. In this method, statistical variation depends on the total number of fibres counted and on the uniformity of fibre distribution on the filter.

Fibre distribution may reasonably be supposed to be approximated by the Poisson distribution. Theoretically, the process of counting randomly distributed (Poisson) fibres yields a coefficient of variation (CV) equal to $(\sqrt{N})^{-1} \times 100$, where N is the number of fibres counted. The CV is thus 10% for 100 fibres and 32% for 10 fibres. Table 1 gives theoretical CVs for different numbers of fibres counted, taking into account only statistical variation.

In practice, however, the actual CV will be greater because of the additional component of variation associated with subjective differences within and between microscopists. Table 2 (p. 38) gives typical CVs *within* a laboratory where a satisfactory quality control scheme operates and N fibres are counted.

It can be seen from Table 2 that counting more than 100 fibres gives only a small improvement in precision. Also, methods lose precision as

Table 1. Coefficients of variation for a Poisson fibre distribution

N	CV (%) (from the Poisson distribution)
5	45
7	38
10	32
20	22
50	14
80	11
100	10
200	07

Table 2. Typical coefficients of variation within a laboratory

		90% confidence limits for the mean of repeated determinations	
N	**Typical CV (%)**	**Lower**	**Upper**
5	49	2.0	11.0
7	43	3.2	14.0
10	37	5.1	18.5
20	30	11.7	33.2
50	25	33	76
80	23	53	118
100	22	68	149
200	21	139	291

fewer fibres are counted; this loss of precision accelerates as counts drop below about 10 fibres.

Interlaboratory CVs can be twice as great as intralaboratory CVs, or even larger if quality control is poor. Mean interlaboratory CVs for more than 100 fibres counted range up to 45% for laboratories performing satisfactorily in proficiency testing.

The counting rules specified in this method were formulated to minimize subjective decisions and optimize precision and are therefore expected to have a beneficial impact on intra- and interlaboratory precision.

4.3 Lower limit of measurement

Errors become large when the number of fibres counted is small. It can be seen from Table 2 that a true mean count of 10 fibres per 100 graticule areas will give a count of 5 fibres or fewer per 100 areas on about 5% of occasions. This is the maximum acceptable count for a blank filter; therefore it is reasonable that 10 fibres per 100 graticule areas should be regarded as the lowest measurable fibre level above "background" contamination. This level corresponds to a filter fibre density of about 13 fibres·mm^{-2}; such a measurement is approximate, and has 90% confidence limits of about 6.5 and 23.5 fibres·mm^{-2}. The actual lowest measurable air concentration depends on the sample volume and effective area of the filter. For example, this level (13 fibres·mm^{-2}) corresponds to 0.02 fibres·ml^{-1} for a 240-litre sample and an effective filter area of 380 mm^2.

This analysis takes account of only intralaboratory precision. Bias and interlaboratory differences can further degrade the reliability of low density (and low concentration) results and raise the effective lower limit of measurement.

5. Quality assurance

The results obtained by the membrane filter method are reliable only if a comprehensive quality assurance programme is employed. The provisions of the publication *General requirements for the technical competence of testing laboratories* (ISO, 1990) should be followed. National accreditation schemes based on the requirements of this publication have been established in many countries, and, in some, additional requirements are specified for fibre measurements (e.g. NAMAS, 1989).

It is essential that those engaged in sampling or evaluating airborne fibres not only use a single validated method but also be properly trained in that method and regularly evaluated. Such training and evaluation may require attendance at prescribed in-house or national courses. Authorized personnel may be required to hold, or work under the supervision of a person who holds, a relevant formal qualification; such personnel should also have appropriate experience.

An essential part of quality assurance is the performance of counting checks, because of the large differences in results obtained within and between laboratories using visual counting. Laboratories using the recommended method should participate in a national or international proficiency testing scheme in order to minimize interlaboratory variation. An international proficiency testing scheme is currently envisaged as a supplement to this method specification. Microscopists should also take part in intralaboratory counting checks. Interlaboratory exchange and verification of samples can further complement internal quality control efforts.

Two types of counting check should be performed within a laboratory: counting permanent, well characterized reference samples and making duplicate counts on routine samples. A laboratory should accordingly maintain a set of reference samples that includes a range of fibre and background-material loadings from a variety of routine sample sources. These reference samples should be evaluated periodi-

cally (e.g. four samples per month) by all microscopists, and the counts compared with reference counts made on the same samples. A reference count is usually derived from previous counts and may, for example, be the mean of at least 15 previous visual counts. The results of each microscopist should be judged against defined criteria for satisfactory performance (these criteria should be more stringent than those for proficiency testing schemes). In addition, a defined fraction of a laboratory's routine samples (e.g. 1 in 10) should be re-counted, wherever possible by a different analyst, and the difference between the counts compared with criteria for satisfactory performance. If the results fall outside the acceptable deviation, possible reasons should be investigated and corrective action taken. Systematic records of quality control testing should be kept.

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Related reading

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Annex 1

Static monitoring

Static monitoring is used for assessing the effectiveness of process-control techniques, detecting sources of contamination, determining background fibre concentration, etc. and does not yield a measurement representative of personal exposure. Samples collected at fixed locations—for example, outside asbestos stripping and encapsulating areas, inside decontamination rooms, for clearance monitoring after asbestos stripping and encapsulating and inside buildings or ships that contain asbestos—are called static samples and form the basis of static monitoring. This type of sampling is often conducted where there is a high proportion of fibres other than the one of principal interest, or particles that conform to the definition of a fibre given in section 3.2.3. Such interferences can cause problems in the interpretation of the results obtained by this method, which can be resolved only by obtaining information on fibre composition using other methods (e.g. electron microscopy).

The parameters and methodology specified for personal sampling generally apply to static monitoring. The main differences are indicated in the following discussion.

Sampling

Samples are taken at fixed locations. The sampling head should be mounted on a stand, usually 1–2 metres above floor level, with the cowl facing downwards, allowing free air circulation around the entry. It should be positioned with regard to local sources of dust or clean air. Cross-draughts of more than $1\,m \cdot s^{-1}$ may reduce fibre collection.

Flow rate

The sampling flow rate should be in the range 0.5–16 litres·min^{-1}

Flow rates are usually higher for static sampling than for personal sampling. Over the range specified above, sampling efficiency (for chrysotile) has been found to be independent of flow rate.

Stop-counting rule

One hundred fibres should be counted, or 200 graticule areas inspected, whichever comes first. Fibres should, however, be counted in at least 20 graticule areas.

In many static monitoring situations, it is necessary to inspect 200 graticule areas.

If static monitoring is used in making measurements of airborne asbestos for the purposes of asbestos abatement, for example, and therefore comparisons are made with a clearance indicator, it may be unnecessary to evaluate 200 graticule areas or count 100 fibres. For instance, if 30 fibres in 200 fields would indicate a concentration of 0.015 fibres·ml^{-1} (and the clearance indicator is 0.010 fibres·ml^{-1}), it would be possible to report an enclosure as unsatisfactory as soon as a count of 30 fibres is obtained, even if only a few fields have been examined.

The stop-counting rule and minimum total sample volume are usually such that the number of fibres counted in the neighbourhood of typical clearance indicators is below the lower limit of the recommended density range for optimal accuracy and precision, or even below the detection limit of the method. Therefore, concentration estimates can often be only approximate. The detection limit depends on the sample volume and should be reported by the laboratory with its results. For example, the limit of detection, assuming a 480-litre sample, an effective filter area of 380 mm^2 and 200 graticule areas examined, is 0.010 fibres·ml^{-1}. A counting result falling below this limit should not be reported literally, but simply as <0.010 fibres·ml^{-1}.

Annex 2

Characterization of fibres

This publication describes a method for counting fibres that does not differentiate between fibre types. Although knowledge of the type of fibre produced by the bulk material provides a guide to what to look for in airborne samples, the only techniques that permit a scientific determination of the type of fibre present in a sample are based on different forms of microscopy. In choosing an analytical method and a laboratory, several factors need to be considered. The technique should be capable of discriminating between the fibre types in the sample and should be conducted by experienced analysts. The analyst should preferably be a graduate in geology, chemistry, materials science or a similar discipline, with at least 2 years' experience in the appropriate methods of fibre analysis. It is important to assess the laboratory's quality assurance protocol, which should include, wherever possible, participation in an external proficiency testing programme.

The methods listed below were developed for asbestos and other mineral or inorganic vitreous fibres. The cheapest and most readily available technique for fibre characterization is polarized light microscopy (PLM), which can be used to identify many fibre types so long as the fibres are greater than about 1 μm in width. Electron microscopy techniques are more expensive and can be used to provide additional information as needed. Scanning electron microscopy (SEM), in conjunction with energy dispersive X-ray analysis (EDXA), can generally be used to determine the elemental composition of fibres greater than 0.2 μm in width. The most expensive method, analytical transmission electron microscopy (TEM), is generally acknowledged as the most accurate technique for characterizing crystalline inorganic fibres and can be used to provide chemical and structural information for fibres down to about 0.01 μm in width. The costs of TEM analysis are approximately an order of magnitude greater than those of PLM or PCOM; with SEM, costs fall between the two.

Methods applicable to airborne fibres

Polarized light microscopy

For fibres greater than about 1 μm in width, PLM techniques can be used to evaluate the optical properties of individual fibres.

Published methods of fibre characterization by PLM include:

- *Method 9002. Asbestos (bulk): by polarized light microscopy*, 2nd ed. Cincinnati, OH, National Institute for Occupational Safety and Health, 1994 (available from NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, USA).
- *Test method for the determination of asbestos in bulk building materials (EPA/600/R-93/116).* Washington, DC, Environmental Protection Agency, 1993 (available from EPA, 401 M Street SW, Washington, DC 20460, USA).
- *Test method: interim method for the determination of asbestos in bulk insulation samples (EPA/600/M4-82/020).* Washington, DC, Environmental Protection Agency, 1982 (available from EPA, 401 M Street NW, Washington, DC 20460, USA).
- *Asbestos in bulk materials: sampling and identification by polarised light microscopy (PLM).* London, Health and Safety Executive, 1994 (Methods for the Determination of Hazardous Substances, No. 77; available from HSE Books, P.O. Box 1999, Sudbury, Suffolk, CO10 6FS, England).

Scanning electron microscopy

For routine analysis, SEM allows for good visualization of fibre morphology down to widths of about 0.05 μm, depending on the method used. In addition, an energy dispersive X-ray analyser can be used to determine the elemental composition of fibres with widths greater than about 0.2 μm. Sodium and lighter elements cannot generally be observed by SEM/EDXA. Detailed analysis of samples collected on membrane filters is not possible because of the instability of the filter medium; more stable filter media, e.g. polycarbonate filters, should therefore be used if SEM testing is envisaged.

Published methods of fibre characterization by SEM include:

- *Methods of monitoring and evaluating airborne man-made mineral fibres: report on a WHO consultation.* Copenhagen, World Health Organization Regional Office for Europe, 1981 (available from

WHO Regional Office for Europe, Scherfigsvej 8, DK-2100 Copenhagen Ø, Denmark).

- *Method RTM2.* Paris, Asbestos International Association (available from AIA, 10 rue de la Pepinière, 75008 Paris, France).
- *Method for the separate determination of asbestos and other inorganic fibres: raster electron microscopic method (ZHI/120/46).* Sankt Augustin, Germany, Federation of Industrial Injuries Insurance Institutions, 1991 (available from Federation of Industrial Injuries Insurance Institutions, Alte Heerstrasse 111, 53757 Sankt Augustin, Germany).

Transmission electron microscopy

The use of TEM allows characterization of individual fibres as small as 0.01 μm in width. Two powerful qualitative techniques are also available: electron diffraction (ED), which allows the determination of particle crystal structure, and (as with SEM) EDXA, which allows determination of the elemental composition of individual fibres. Combined with ED and EDXA, TEM is particularly useful for identifying inorganic crystalline fibres and generally provides the most definitive identification available. For non-crystalline inorganic materials it is similar to SEM in sensitivity. For qualitative analysis of organic materials, TEM is generally not useful. Sample preparation for TEM is more complex than for SEM.

Published methods of fibre characterization by TEM include:

- *Ambient air determination of asbestos fibres: direct transfer transmission electron microscopy method.* Geneva, International Organization for Standardization, 1991 (available from International Organization for Standardization, 1 rue de Varembé, 1211 Geneva 20, Switzerland).

- *Method 7402. Asbestos: by transmission electron microscopy*, 2nd ed. Cincinnati, OH, National Institute for Occupational Safety and Health, 1994 (available from NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, USA).

- *Interim transmission electron microscopy analytical methods: mandatory and non-mandatory; and mandatory section to determine completion of response actions.* Washington, DC, Environmental Protection Agency, 1987 (Code of Federal Regulations, 40, Part

763, Appendix A to subpart E; available from EPA, 401 M Street SW, Washington, DC 20460, USA).

Methods applicable to bulk materials

X-ray diffraction (XRD)

In XRD analysis, a bulk sample of material is subjected to X-ray bombardment and the angle of the diffracted radiation is measured. This technique allows determination of the crystal structure of mineral compounds.

Published methods of fibre characterization by XRD include:

- *Method 9000. Asbestos (chrysotile): by X-ray diffraction.* Cincinnati, OH, National Institute for Occupational Safety and Health, 1994 (available from NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, USA).
- *Test method for the determination of asbestos in bulk building materials (EPA/600/R-93/116).* Washington, DC, Environmental Protection Agency, 1993 (available from EPA, 401 M Street SW, Washington, DC 20460, USA).

Chemical analysis

Some chemical analyses have been used to indicate the presence of certain elements or functional groups in a bulk sample, but these methods do not distinguish between fibres and other material with the same chemical properties. Commercially available analytical kits are able to detect the presence of magnesium (in chrysotile) and iron (in amphibole asbestos). Other tests may be used for the detection of man-made organic fibres.

A published method of fibre characterization by chemical analysis is:

- *Test for screening asbestos.* Cincinnati, OH, National Institute for Occupational Safety and Health, 1979 (Publication No. 80-110; available from NIOSH, 4676 Columbia Parkway, Cincinnati, OH, 45226, USA).

Infrared (IR) absorption

This technique indicates only the possible presence of certain functional groups in the components analysed and cannot distinguish between fibres and other material with the same chemical properties. The resonant absorption of IR radiation is measured for peaks indicative of various functional groups. The technique may be useful in identifying certain organic fibres.

A published method of fibre characterization by IR absorption is:

- *Method for the determination of chrysotile and amphibole forms of asbestos (ZHI/120/30).* Sankt Augustin, Germany, Federation of Industrial Injuries Insurance Institutions, 1985 (available from Federation of Industrial Injuries Insurance Institutions, Alte Heerstrasse 111, 53757 Sankt Augustin, Germany).

Annex 3

List of participants at the final meeting

Geneva, 31 January–2 February 1994

Dr M. C. Arroyo, National Institute of Occupational Health and Safety, Baracaldo, Spain

Dr P. Baron, National Institute for Occupational Safety and Health, Cincinnati, OH, USA (*Co-rapporteur*)

Mr P. Buchanan, Luxembourg (representing the European Commission)

Mr P. Class, Rueil-Malmaison, France (representing the European Ceramic Fibres Industry Association)

Ms R. Cosca-Sliney, University Institute of Medicine and Occupational Hygiene, Lausanne, Switzerland

Dr N. P. Crawford, Institute of Occupational Medicine, Edinburgh, Scotland (*Rapporteur*)

Dr G. W. Gibbs, Winterburn, Alberta, Canada (representing the International Commission on Occupational Health)

Mrs B. Goelzer, Occupational Health, World Health Organization, Geneva, Switzerland (*Secretary*)

Mr F. I. Grunder, American Industrial Hygiene Association, Fairfax, VA, USA

Mr S. Houston, International Fibre Safety Group, Montreal, Canada

Dr E. Kauffer, National Institute of Research and Safety, Vandoeuvre, France

Professor S. Krantz, National Institute of Occupational Health, Solna, Sweden

Professor Y. Kusaka, Department of Environmental Health, Fukui Medical School, Fukui, Japan

Dr J. LeBel, Asbestos Institute, Sherbrooke, Quebec, Canada

Dr M. Lesage, International Labour Office, Geneva, Switzerland

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Dr A. Marconi, Istituto Superiore di Sanità, Rome, Italy

Dr M. I. Mikheev, Chief, Occupational Health, World Health Organization, Geneva, Switzerland

Dr T. K.-W. Ng, Occupational Health, World Health Organization, Geneva, Switzerland

Mr G. Perrault, Quebec Research Institute for Health and Occupational Safety, Montreal, Canada

Mr A. L. Rickards, Rugby, England (representing Asbestos International Association)

Dr G. Riediger, Sankt Augustin, Germany (representing the European Committee for Standardization)

Dr H. U. Sabir, Occupational Health and Safety Institute, Ankara, Turkey

Mrs M. M. Teixeira Lima, Brasilia, Brazil (representing the National Foundation for Occupational Health (Fundacentro))

Professor F. Valic, Zagreb, Croatia (representing the International Programme on Chemical Safety)

Mr R. A. Versen, Littleton, CO, USA (representing the Thermal Insulation Manufacturers Association)

Dr N. G. West, Health and Safety Executive, Sheffield, England (*Chairman*)