MDHS

Methods for the Determination of Hazardous Substances

Health and Safety Laboratory

HSE Health & Safety Executive

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Fibres in air

Guidance on the discrimination between fibre types in samples of airborne dust on filters using microscopy

November 1998

INTRODUCTION

1 At present there are two approved methods for determining airborne fibre concentration; one for asbestos (MDHS 39/4[1]) and one for man-made mineral fibres (MMMF) (MDHS 59[2]). Both methods are based on collecting particulates by drawing air through a membrane filter which is analysed at 500x light microscopy. As the analysis relies on manual fibre counting of a relatively small number of fibres, the method is recognised as one of the least precise analytical techniques used in the occupational environment. The fibre count is subject to a number of systematic and random errors as well as individual counter bias. This bias is particularly important if an attempt is being made to discriminate between fibre types and may have a large influence on the accuracy of the results. This MDHS gives a strategy and method for fibre discrimination based on the optical, crystallographic and chemical properties of the fibres. It should be emphasised that discrimination using a light microscope is, in general, not easily carried out, and depends on the skills of the analyst and the techniques available.

2 For regulatory purposes in the UK, the Control of Asbestos at Work Regulations (CAWR)[3] define asbestos as any of the following fibrous materials (or any mixture containing them): chrysotile, amosite, crocidolite, fibrous anthophyllite, fibrous actinolite and fibrous tremolite; the diseases asbestosis, lung cancer and mesothelioma have been associated with inhalation of asbestos. MMMF is the term used for various inorganic materials which have been made into fibres: they have names such as 'rock wool', 'slag wool' and 'refractory (or ceramic) MMMF' (sometimes referred to as MMVF), and are subject to a maximum exposure limit (MEL) under the Control of Substances Hazardous to Health (COSHH) Regulations, COSHH ACOPs and EH40 (revised annually).[4] In some situations it is important to distinguish between regulated and non-regulated fibre types when fibre evaluations are carried out. This may be applicable to situations described in MDHS39/4[1] as background sampling, leak (enclosure check) sampling for assessment of respirator performance and reassurance. *It is not recommended that this be generally applied to clearance testing, since the determination of results above the clearance indicator demonstrates that the enclosure is not clean, even if some of the dust is non-asbestos.* However, in exceptional circumstances it can be applied to clearance testing. For example in asbestos removal operation, after cleaning or where non-asbestos dust is suspected of being drawn into the enclosure from operations outside and increasing the 'fibre' concentration (eg gypsum particles from plaster board, or MMMF fibres from glass fibre insulation). This discrimination method must not be used for the assessment of compliance to the control limits for asbestos in MDHS 39/4[1] or to the maximum exposure limit for MMMF in MDHS 59.[2]

Note: *The European Reference Method and the Approved Methods described in MDHS 39/4[1] and MDHS 59[2] could be superseded in 1999 by a World Health Organisation method which may be adopted by the European Union.*[5]

3 To assess compliance with regulations, airborne fibre concentrations are measured by sampling a known volume of air through a membrane filter and counting the numbers of fibres (>5µm in length, <3µm in width and with an aspect ratio >3:1) in a number of graticule areas using 500x phase contrast light microscopy (PCM). However, the use of PCM alone does not give sufficient information to positively discriminate between respirable fibre types. *No single technique is capable of identifying all fibres: different techniques must be used for different fibres.* Specific methods are available for most fibre types: for example, analytical transmission electron microscopy (TEM) has the potential to identify all airborne asbestos fibres and ultra-violet (UV) fluorescence microscopy can identify para-aramid fibres. Considerable care must be taken when discriminating between fibre types.

GENERAL METHOD

Principle

4 This method provides guidance on various techniques which may be used to discriminate between fibre types.

It should only be used after the routine evaluation by PCM has been completed. The recommended techniques covered by this MDHS are:

- Polarised Light Microscopy (PLM);
- UV Fluorescence Microscopy;
- Scanning Electron Microscopy (SEM), with Energy Dispersive X-ray Analysis (EDXA);
- Transmission Electron Microscopy (TEM), with EDXA and Selected Area Electron Diffraction (SAED).

The last two of these techniques rely either on duplicate samples having been taken or on half of the original filter being available. (Re-sampling might be adopted but the airborne fibres, both type and concentrations, may be different to the initial sample.)

Scope

5 Following evaluation of fibre concentration by PCM, this method employs various microscope techniques to examine crystallographic and optical properties, and element compositions, which may permit discrimination between certain fibre types. Discriminating decisions should be taken with caution and should not be based on the observation of a single characteristic (such as morphology or UV fluorescence) unless additional information on the environment from which the sample was taken is available. The extent to which this method can be implemented will depend on the equipment available and upon the training of, and care taken by, the microscopist.

Strategy

6 Various strategies for fibre discrimination may be considered depending on the purpose of the analysis and the degree of identification required: Table 1 lists the microscopical methods which are the most useful, and notes their applicability to various fibre types. Discrimination can be applied in two ways, either 'POSITIVELY' when a fibre is IDENTIFIED and then INCLUDED in the count, or 'NEGATIVELY' when a fibre can be shown to differ from the type being evaluated and therefore is EXCLUDED from the count. *Thus it is essential first to define the purpose of the fibre count, and second to decide which strategy is appropriate***.**

Examples of strategies are:

- positive identification of asbestos fibres present by TEM-EDXA;
- positive identification of para-Aramid fibres by UV fluorescence;
- exclusion of MMMF from a PCM/PLM fibre count;
- exclusion of para-Aramid from a PCM/UV fibre count.

Each technique (PCM/PLM, SEM-EDXA or TEM-EDX-SAED) has limitations and may give different answers. Therefore, it is necessary to understand the techniques and how they can be used. Table 1 provides a guide for the main classes of fibres encountered. Even when populations of fibres exhibit certain characteristics, these may not be shown by all individual fibres: thus, unless further information is available (as noted in paragraph 5 above) *at least two characteristic properties of each fibre should be examined to permit discrimination.*

7 The analyst should choose the most appropriate technique for the strategy selected (see Table 1). However, if the types of fibre are not known, a decision hierarchy may be adopted (see Appendix 1). The results and their implications should be evaluated after each analysis.

Table 1: Guidance in selection of the appropriate method

This table helps the analyst to select the appropriate method and strategies for different fibre types based on the capabilities and limitations of the methods.

	Methods			
	PCM/PLM	PCM/UV fluorescence	SEM-EDXA	TEM-EDXA-SAED
Primary fibre analysed	**Strategies**			
Asbestos	Exclude other fibres >1µm diameter (limited use if some types of other mineral fibres are present)	Not recommended	Include asbestos fibres>0.2µm diameter	Include all fibres
MMMF	Include MMMF fibres > 1µm diameter which are isotropic	Not recommended	Include MMMF fibres >0.2µm diameter, can be useful if source of fibre is known	Can be useful for fibres from known source
Other mineral fibres	Exclude other fibres >1µm diameter, eg rutile needles (cannot always be used to discriminate between different types of mineral fibres)	Not recommended	Include fibres >0.2µm diameter, if source is known: can be used to discriminate between some types of mineral fibre	Include all fibres; can identify or discriminate between fibre types
Synthetic organic fibres	Not recommended, except for a known source which may have a characteristic property	Inlcude certain types of organic fibre from a known source which fluoresce at specific wavelengths	Exclude inorganic fibres >0.2µm diameter	Exclude other inorganic fibres of all widths which give an EDX spectrum

Reference material

8 It is essential that the laboratory has a range of reference materials appropriate to the fibre types being discriminated. Bulk samples should be collected, where possible, as a reference material, preferably at the same site as the air sample. Laboratories can prepare their own sets of reference samples containing fibres of interest.

INITIAL PCM FIBRE COUNT

Sample preparation

9 The membrane filter must be prepared using the relevant procedure (MDHS 39/4[1] or MDHS 59[2]). If additional analysis (for example, TEM) is anticipated, samples and blank filters should be cut in half with a scalpel using a rolling action with the filter carefully held at the edge. Half of the filter then can be mounted, and the other half can be kept for the subsequent investigation.

Fibre counting

10 Equipment and fibre counting procedures must accord with those specified in MDHS 39/4[1] or MDHS 59.[2] *There should be no discrimination at this stage.*

Results

11 The results of the initial PCM fibre count will determine if there is a need for further analysis. For example, if reassurance sampling is being carried out and the result falls below the clearance indicator level, then no further action may be deemed necessary. In any case, *PCM should not be used alone to discriminate between asbestos and non-asbestos fibres to produce a fibre measurement.* The strategy used in the discrimination must be stated in the final report.

DISCRIMINATION BY LIGHT MICROSCOPY

PCM/PLM fibre discrimination

12 The discrimination between fibre types is only undertaken after the initial analysis of the sample. In order to discriminate between fibres in the field of view, it is necessary to change from PCM to PLM: the additional accessories required are given in Appendix 2. An example of a completed count sheet is given at Appendix 6.

Fibre discrimination

13 The procedure of a normal PCM count is followed until a countable fibre is found. PLM is used to examine the characteristic properties of the fibre, eg relief, shape, birefringence and pleochroism, as discussed in MDHS 77.[6] Examination of the properties will require the following sequence:

- the phase condenser annulus and the light filters are removed;
- crossed polarising filters are introduced;
- the rotating stage locking mechanism is released;
- the first order red (or other compensator) is introduced if required;
- appropriate observations are made and recorded;
- when observations on the field are complete, the microscope is returned to phase contrast mode.

It is important that the fibres which generate a PCM count are those which undergo the additional PLM examination. Therefore, care should be taken when the stage is rotated, or when other changes (for example, introduction of a high resolution objective) are made, that the same fibres are analysed. Analysts should be aware that the discrimination between fibres <1µm diameter may not be possible using 40x, 0.65NA light microscope objectives. In these circumstances, discrimination may be possible only by using additional equipment with higher resolution objectives.

The characteristics which can be observed by the respective techniques are summarised in Table 2 and detailed in Appendix 4.

Table 2: Properties, and the techniques by which fibres may be observed

Techniques and identification mode

	PCM	PLM			UV	SEM	TEM
Property		Plane polarised light	Crossed polars	First order red (or other compen-sator)	Fluores-cence	EDXA	EDXA-SAED
Morphology	✔	✔	✔	✔	✔	✔	✔
Relief	✔	✔	✔				✔
Colour		✔					
Pleochroism		✔	✔				
Birefringence			✔	✔			
Extinction angle			✔				
Sign of elongation				✔			
Fluorescence					✔		
Elemental composition						✔	✔
Crystal structure							✔

PCM/UV fluorescence microscopy

14 Many organic fibres, both naturally occurring and synthetic, fluoresce when exposed to UV light of suitable wavelength: aromatic structures such as para-Aramids (for example Kevlar and Twaron) fluoresce strongly, and this property is used in their analysis[7,8] (see also Appendix 4). Also, many synthetic organic fibres have added optical brighteners which are highly fluorescent. Untreated asbestos does not fluoresce. Fibres of one composition will fluoresce in a particular wavelength range; this will be manifest as a particular 'colour', and will be a strong indicator of fibre identity. As described in paragraph 8, a range of standards can be prepared for comparison. The general analytical procedure will be similar to that described in paragraph 13, changing between PCM and UV fluorescence as appropriate. However, additional care must be taken to ensure that the counted fibres are those which undergo further examination. If the sample is analysed on a separate microscope to the PCM count, it should be used to recount the sample and not reduce the original PCM count. The characteristics of a satisfactory UV fluorescence microscope are similar to those given in

Appendix 2 for PCM, with the important exception that special optics are needed for fluorescence work.

DISCRIMINATION BETWEEN FIBRE TYPES BY SEM

Strategy

15 For SEM-EDXA analysis each fibre is identified and the result of this reported independently of the PCM count. This section describes the treatment of mixed cellulose ester filters, upon which fibres are collected in the first instance for PCM, and from which portions are available for analysis by alternative techniques to distinguish the fibre types. The aim is to provide a SEM analysis of the fibre type on the filter. In general SEM-EDXA should be able to identify fibres which are visible by PCM. As an alternative, a fibre count on a sample collected on a polycarbonate or pvc filter may be used if the sample was collected simultaneously with the cellulose ester filter. An ISO method,[9] based on a German method[10] using previously gold-coated polycarbonate filters, is currently in preparation.

Sample preparation

16 The exact sample preparation procedure used will depend on the type of filter and on other factors. Either a segment is cut from the original filter before it is cleared and mounted for PCM (see paragraph 9) or a separate sample is used that was collected at the same time as that used for the PCM count. In the case of a mixed cellulose ester filter (as used for PCM) a section is cut from the filter, cleared as normal on a suitable substrate, etched in a low temperature oxygen plasma incinerator, mounted on a suitable SEM sample stub using carbon dag, and coated with carbon in an appropriate carbon evaporation vacuum coating unit (which should be capable of a vacuum of $<10^{-4}$ torr). The etching process removes part of the surface of the collapsed filter and exposes fibres that might otherwise be embedded within the filter. Examples of filters from batches to be used for SEM fibre counting should be checked by SEM before use to ensure the absence of fibre contamination. Organic volatile components of conducting metal paints or glues have been found to interfere with the deposition of the carbon coat unless the adhesives are properly cured. Carbon coating of the samples is preferred because although gold coating may produce a clearer SEM image, the presence of gold may impede EDX analysis of the fibres.

SEM

17 The SEM operating conditions should be adjusted to produce a visible image, when scanning at 2000x or greater magnification, of a 0.2µm diameter chrysotile fibre (0.5mm on the screen) and with the lens current's accelerating voltage, spot size, working distance, apertures etc, set in the format for routine fibre counting and analysis. A suitable reference sample should be maintained for these parameters to be tested. The SEM should be fitted with a suitable EDXA system for elemental analysis of fibres. In general, the following operating conditions are recommended:

- the sample surface should be perpendicular to the electron beam when seeking, counting and measuring fibres (EDXA will be possible only if a high 'take-off angle detector' is fitted);
- the magnification used for searching the filter should be 2000x or greater;
- the accelerating voltage used should be between 15 and 20 kilovolts (kV);
- the working distance, beam diameter, alignment and astigmatism should be adjusted for optimum visibility of fine fibres with a secondary electron image while at the same time ensuring that resolution of fine fibres is satisfactory and that X-ray yields are sufficient for identification;
- the EDXA detector and spectrometer should be capable of detecting X-ray energies at least within the range 980 to 10 000 electron volts (eV) to detect elements from sodium (1050 eV) to iron (6400 eV), should have a channel width of between 10, 20 or 40 eV per channel, and should have a peak resolution (peak width as half peak height) of at least 160 eV (for manganese Kα).

The SEM operating conditions may need to be modified on occasions in order to obtain suitable X-ray count rates for identification (for example, by changing magnification and increasing the spot size); if so, the conditions should be returned to those used for searching before the search is resumed. High beam intensities will distort cellulose ester filters and conditions will need to be adjusted as a compromise.

Calibration of the SEM and EDXA system

18 The SEM should be calibrated against a standard grating covering the magnification range (2500x-10 000x) used for measuring and sizing fibres. The grating should be traceable to a recognised standard. Normally the EDXA will require calibration using one or more known elements in a reference sample to position the peaks at the correct energy.

Counting and sizing fibres

19 Before any systematic search is started, it is important that a low magnification scan of the filter surface is conducted - to ensure that the dust deposit is uniform, that it is not too dense for fibre counting and that the surface has not been damaged. Searching is best conducted by examining fields of view separated by short distances from each other along a long axis of the available filter area. Counting must be inside the effective filter area and at least 1 mm from any cut edges. A field of view near one edge is chosen and processed, and then this is changed to a new area by adjustment of one axis of the SEM stage control; this process is continued by traversing away from the edge across to the other edge. It is important that the approach is systematic and that step scanning is performed in one direction only so that fields of view are separate and the same traverses are not repeated. The fibre counting rules in MDHS 39/4 are used with the full screen area as the graticule. Measurement of fibre sizes can be made directly from the SEM screen at an appropriate magnification (adjusted differently for separate length and diameter measurements if required), or by an electronic image measuring device. A predetermined

number of fields of view at a set magnification is searched systematically to scan a fixed total area of the filter. The exact number of fields of view, or the area specified, depends on the desired detection limit of the test. Also, it is normal to specify a maximum number of fibres to be counted and analysed, and a minimum number of fields of view to be searched. The maximum fibre number sets a limit on the likely cost of the test in time and expense. For discrimination of the fibre types present approximately the same number of fibres should be counted as in the PCM count.

Fibre analysis

20 Any counted fibre of length >5µm is analysed by EDXA. Magnification should be increased so that the static beam can be placed and maintained accurately on the fibre of interest, before turning to the 'spot' or 'analysis' mode (which stops the scan and allows the electron beam to be positioned directly on the fibre). X-rays generated from within the fibre are collected over a set period of time. It is important that sufficient X-ray energy is counted so that peak to background ratios are suitable for X-ray identification, and so that peak height comparison can be made with standard spectra from known samples: emission rates >1000 counts per second (cps) are suggested to give a total count of 60 000. The EDX spectrum is used as a 'fingerprint' for fibre identification, and the relative integrated intensities of peaks seen should be recorded. Care must be taken that a given X-ray spectrum is derived from the fibre of interest only; if possible, choose a part of the fibre where there are no contaminating surface particles or adjacent particles which may contribute additional elements to the spectrum (since this may otherwise preclude identification of a fibre as asbestos). It is accepted that the substrate filter (and the gold coat if used; see paragraph 19) will make some contribution to the spectrum and to the background, but this is unavoidable. However, possible contribution to the EDX spectrum from aluminium stubs must be monitored. It is unlikely that fibres having diameters <0.1µm will generate enough X-rays above background to give an identifiable spectrum.

Fibre classification

21 Fibres are classified on the basis of the comparison of their EDX spectrum with spectra collected from reference standards analysed using the same SEM operating conditions. The same types of strategies can be applied as for discrimination by light microscopy. SEM-EDXA is limited in its ability to discriminate by the quality of the EDX spectrum generated. The morphology of the fibres is also important and must be considered at all times in combination with the EDX spectrum.

A fibre is counted as asbestos if:

- the EDX spectrum is effectively the same as that of a reference asbestos type;
- the EDX spectrum has the same elements as a reference asbestos type but the elemental X-ray proportions differ by up to ±30%;
- the EDX spectrum contains elements typical of a reference type but also contains elements from a known contaminant dust;
- the fibre diameter is too small to produce an EDX spectrum (0.2 µm or less).

A fibre is counted as a non-asbestos fibre if:

- the fibre is >0.2 µm diameter and contains no major metal elements or silicon;
- the EDX spectrum contains some or all of the elements found in one of the reference types but the proportions are very different, eg high Ca, very low Si, very low Mg;
- the EDX spectrum contains high proportions of elements that are not found in any of the reference asbestos types and there are no signs of obvious contamination of the fibre, eg very high Fe, very low Si.

Quantitative analysis of EDX spectra and comparison with known variability in reference materials may permit more confident identification of asbestos minerals but it must be emphasised that, for SEM analysis, chemical composition and simple fibre morphology alone are not sufficient for positive identification, unless there is a known source.

Therefore for SEM discrimination between asbestos and other fibres, from background or environmental samples only non-asbestos fibres should be used to reduce the count recorded. Characteristics of fibres are given in Appendix 5.

DISCRIMINATION BETWEEN FIBRE TYPES USING TEM

22 TEM has the ability to discriminate between mineral fibres of all sizes, but is not recommended for organic fibres or MMMFs.

Sample preparation

23 Samples should be prepared by the 'direct transfer method' which is described in the International Standards Organisation Method ISO 10312.[11] This method covers cellulose ester filters, cellulose nitrate filters and polycarbonate filters.

Counting and sizing fibres

24 ISO 10312[11] gives a comprehensive method for the assessment of airborne asbestos fibre concentrations. However, Appendix D of that method should be followed for discrimination of >5µm long fibres when the results are to be compared with PCM counts. The main components of the analysis are described below.

- TEM specimen grids are examined at both low and high magnifications to check that they are suitable for analysis before conducting a quantitative structure count on randomly selected grid openings. In the TEM analysis, selective area electron diffraction (SAED) may be used to examine the crystal structure of a fibre, and its elemental composition is determined by EDXA. For

various reasons, it may not be possible or necessary to identify every fibre unequivocally, and fibres are classified according to the techniques which have been used to identify them. A simple code is used to record the manner in which each fibre was classified as it was counted. The fibre classification is based on successive inspection of the morphology, the SAED pattern and/or qualitative and quantitative EDXA. Unequivocal confirmation of the identity of a chrysotile fibre is made from a measured ED pattern, and of an amphibole fibre from quantitative EDXA and measured zone axis ED. Discrimination can be based on less onerous analyses.

- In addition to single fibres, airborne dust samples often may contain complex aggregates of fibres with or without other particles, some of which are composites of asbestos fibres with other materials. Individual fibres and these more complex aggregates are described as 'asbestos structures'. Coding systems are used to record the type of asbestos structure and to provide the optimum description of each of them. The two codes remove from the microscopist the requirement to interpret the structure counting, and permit this evaluation to be made later without the need to re-examine the TEM specimens.

- Several levels of analysis are specified, the higher levels providing a more rigorous approach to the fibre identification. The procedure allows a minimum required fibre identification criterion to be defined on the basis of previous knowledge, or lack thereof, about the particular sample. It also allows the counts to be interpreted using different counting rules, eg the European Reference Method given in MDHS 39/4.

- Attempts are made to achieve this minimum criterion for each fibre, and the degree of success is then recorded. The lengths and widths of all classified structures and fibres are recorded. The number of asbestos structures found on a known area of the microscope sample, together with the equivalent amount of air filtered through this area, is used to calculate the concentration of asbestos structures in the air.

To discriminate against asbestos it is necessary only to demonstrate adequately that a fibre is not asbestos. This can be achieved at the level of morphology with one other observation which is not consistent with the identification of asbestos. Chemical composition determined by EDXA is probably the quickest and easiest method to combine with morphology for this purpose. The absence of an ED pattern is not a good discriminator because many genuine asbestos fibres and non-crystalline glassy materials in a sample may fail to give a recognisable pattern for various reasons. *The only good discrimination in ED is a pattern which is not consistent with one of the asbestos minerals.*

CALCULATION

25 The initial PCM analysis should be calculated and reported as set out in MDHS 39/4[1] or MDHS 59.[2] When discrimination is applied during a second PCM/PLM the strategy used and the observations made must be recorded on an appropriate count sheet. An example worksheet for this purpose is given in Appendix 6. Where a discrimination count is made, it should replace the original count and must not be used to ratio the original count.

Calculation of light microscopy results

The airborne concentration is given by the formula:

$$C_{PCM} = 1000\ N\ D^2 / V\ n_1\ d^2 \text{ fibres per millilitre (f/ml)}$$

where:

- N is the number of fibres counted;
- n_1 is the number of graticule areas examined;
- D (mm) is the diameter of the exposed filter area;
- d (µm) is the diameter of the Walton-Beckett graticule;
- V (litres) is the volume of air sampled.

Results reported for SEM or TEM analyses should be reported to PCM equivalent fibres on the basis that fibres >5µm long and >0.2µm diameter should be visible with a light microscope.

Calculation of SEM results

The airborne concentration is given by the formula:

$$C_{SEM} = 1000\ N\pi\ r^2 / V\ n_2 a \text{ fibres per millilitre (f/ml)}$$

where:

- N is the number of fibres counted;
- n_2 is the number of screen areas examined;
- r (mm) is the radius of the exposed filter area;
- a (mm^2) is the calibrated screen area;
- V (litres) is the volume of air sampled.

Calculation of TEM results

The airborne concentration is given by the formula:

$$C_{TEM} = 1000\ N\pi\ r^2 / V\ n_3 g \text{ fibres per millilitre (f/ml)}$$

where:

- N is the number of fibres counted;
- n_3 is the number of grid openings examined;
- r (mm) is the radius of the exposed filter area;
- g (mm^2) is the average area of a grid opening;
- V (litres) is the volume of air sampled.

Accuracy and precision of optical methods

26 The precision of the fibre counting strategy (constrained as it is by the Poisson distribution) outlined in this method is not expected to be better than that discussed in MDHS 39/4.[1] Precision is influenced by two factors: (i) mistaken identity of asbestos and non-asbestos, and (ii) errors due to the Poisson distribution for the number of fibres counted (see tables and graph in MDHS 39/4[1]). Considering the second factor, when the numbers of non-asbestos fibres exceed the numbers of asbestos fibres, the latter will have the greater error associated with the count and therefore would not affect

the final result significantly. However, when non-asbestos fibres represent only a small proportion of the total fibres and accordingly have a large imprecision associated with their count, it is vital that identification of a reasonable number of them is made before subtraction. *Therefore it is recommended that at least TEN non-asbestos fibres are counted before subtraction from a total.* This will give confidence that a substantial number of non-asbestos fibres has been observed and that it is reasonable to subtract them from the count.

Bias may arise which must be controlled by training, internal quality control and external quality assurance. Analysts should be aware of the large imprecision associated with evaluations in which (i) relatively few fibres are counted and (ii) very dense deposits are present. However, a systematic bias which might have arisen due to non-target fibres being counted should decrease.

Limit of quantification

27 In so far as this method is concerned, the lower concentration limit (as given in MDHS 39/4[1]) is unaffected as long as twenty fibres are counted. Theoretically, the limit of detection would remain at 0.010 f/ml as long as 480 litres of air are sampled onto an effective filter area of about 385 mm^2 of which the equivalent of about 1.5 mm^2 is evaluated.

REPORTING OF RESULTS

28 When the results are reported, the original PCM result must be given together with the discriminated count. For asbestos and MMMF, the SEM and TEM results are for guidance only; see note in paragraph 2.

Results should be reported along with the upper and lower 95% confidence limits.

ACKNOWLEDGEMENT

29 Preparation of this document was overseen by Working Group Two of the Committee of Fibre Measurement. At the time of writing, CFM/WG2 consisted of Dr G Burdett, Mr B Tylee (both of Health and Safety Laboratory), together with Mr John Addison (John Addison Consultancy), Mr L Davies (Institute of Occupational Medicine), Miss J Prentice (McCrone Scientific Ltd), Mr W Sanderson (Casella London Ltd) and Mr T Shenton-Taylor (United Kingdom Accreditation Service).

ADVICE

Advice on this method and the equipment used may be obtained from the Health and Safety Laboratory, Broad Lane, Sheffield S3 7HQ (telephone 0114 2892000). The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would, therefore, be welcomed and should be sent to the above address.

REFERENCES

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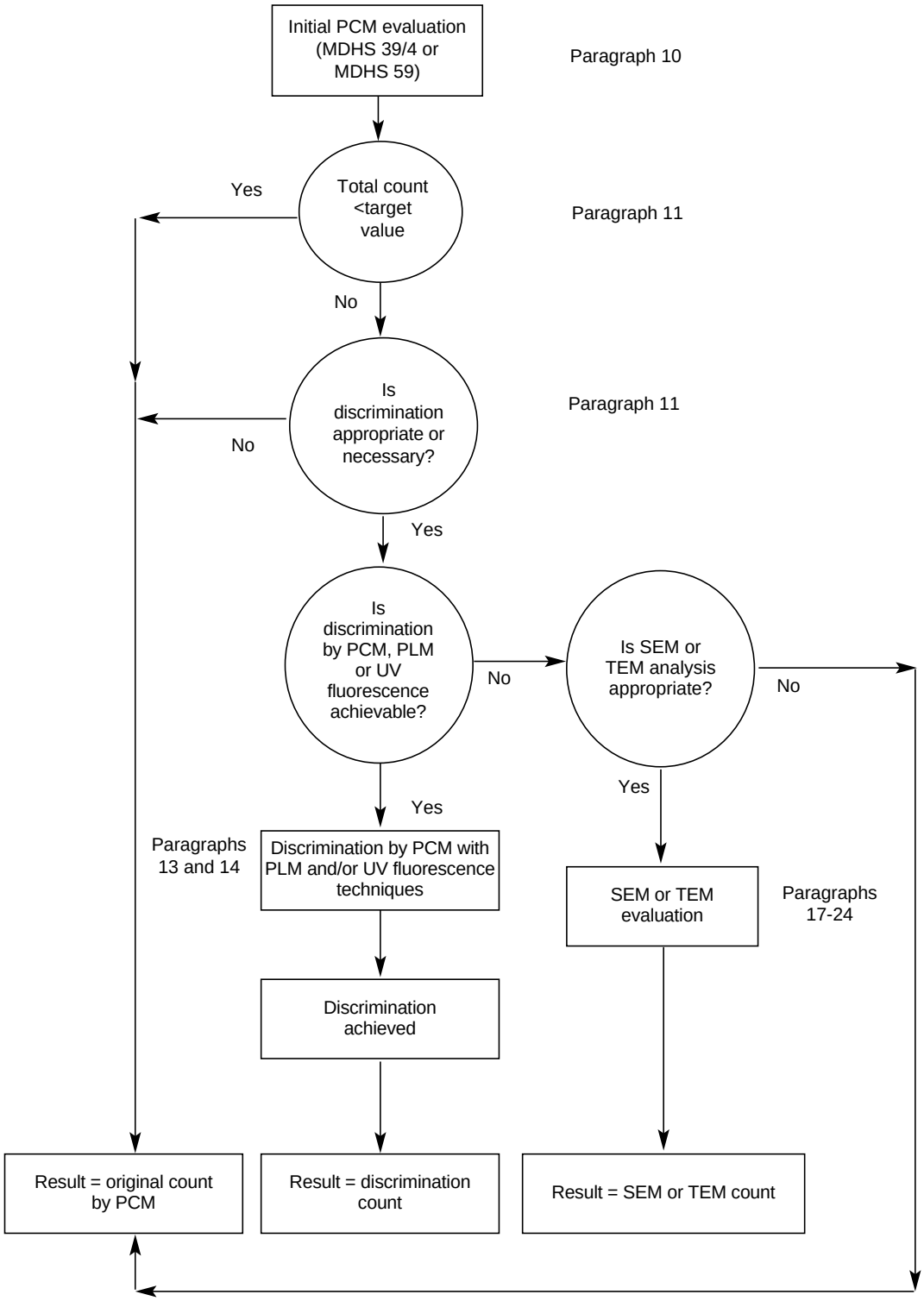
APPENDIX 1: Hierarchy of techniques and decision making



APPENDIX 2: Additional equipment required

ADDITIONAL EQUIPMENT FOR PLM

In addition to the equipment specified in MDHS 39/4,[1] the following are required to permit observations under PLM conditions:

- a polariser;
- a removable analyser;
- a removable first order red or other suitable compensator;
- a removable phase annulus in the condenser;
- a level rotating and independently centrable stage (or a level rotating stage and a centrable objective);
- individual components of the microscope should be from the same manufacturer and must be optically compatible.

Also, the following components may be useful:

- a high resolution objective (eg 100x oil immersion, 1.25 NA). Note that the NA of the condenser must be greater than that of the objective's);
- immersion oil.

Additional equipment for UV fluorescence microscopy

Most UV fluorescence microscopes are specially designed, but many can be fitted with equipment and accessories (as above) which will enable PCM and PLM to be conducted. Conversely, many microscopes configured for PCM and/or PLM can be fitted with accessories which will permit UV fluorescence work to be performed. Any modification may affect the tube factor and the graticule must be checked.

APPENDIX 3: Observations which can be made by PCM

The following properties can be observed by PCM and may aid identification:

- size;
- shape and morphology;
- aspect ratio;
- relief and refractive index (RI) relative to the filter and other particles.

They are discussed in relation to particular fibre types observed with positive phase contrast.

Amphibole asbestos

These properties apply to all amphibole asbestos fibres unless otherwise stated. Generally they are straight fibres; often their sides are parallel, but they may occur as bundles with split and splayed ends. They have high relief because their RIs are considerably greater than that of the mounted filter: fine fibres appear black against a grey background, while thicker fibres have white centres and black outlines (blue for crocidolite), all with surrounding bright phase halos. Their diameters seldom are >1µm in static samples taken for background, leak, clearance or reassurance investigations; however, personal samples taken may contain fibres with diameters >1µm. Many fibres have aspect ratios >10:1. These properties should be compared with those for MMMF, gypsum, plant and insect hairs, slivers from platy minerals (such as talc, exfoliated vermiculite and mica) and fibres released during incineration of natural organic matter (see below). Positive identification of crocidolite is possible when fibre diameters are large enough to show the characteristic pleochroic blue colour. Crocidolite fibres often show more curvature than other amphibole asbestos.

Chrysotile asbestos

Chrysotile fibres often are curved; their sides can be parallel, but they also occur as bundles with split and splayed ends. They have lower relief than amphiboles because their RIs are relatively low (although still higher than the cleared filter RI): fine fibres appear dark grey to black against a grey background, while thicker fibres have white centres and black outlines, all with surrounding bright phase halos. Their diameters seldom are >1µm in static samples taken for background, leak, clearance or reassurance purposes; however, personal samples taken in asbestos working environments may contain fibres of diameters >1µm. Many fibres have aspect ratios >10:1.

MMMF (including extruded fibres, wools, and ceramic fibres)

Generally these fibres are straight or curved with parallel sides (although some wools contain irregular shapes). There is no splitting of the fibre ends; comparatively thick fibres may show conchoidal fracture. Generally they have relatively large diameters; extruded fibres have mean diameters >3µm, and most others have diameters >1µm. Glass wools and extruded glass fibres show low relief because their RIs are close to that of the cleared filter; however, fibres having relatively large diameters show white centres and black outlines with phase halos. Mineral wools have comparatively high RIs and therefore show high relief. Ceramic fibres and alkaline earth silicate fibres have RIs which overlap those for the glass and mineral wools. Many fibres have aspect ratios >10:1. Insulation fibres often are bonded with resin and are distinguished readily by the resulting 'beads' along their lengths.

Diatoms

Diatoms are the silica skeletal remains of small aquatic organisms. Accumulations of these in geological deposits are mined as 'diatomaceous earths' and are often used in hard set thermal insulation on pipes and boilers. RIs of diatoms are lower than that of the mounted sample filter; hence they have low relief and appear white against the grey background. They may occur as acicular fragments resembling straight fibres, and in other shapes such as discs and fragments of discs. They sometimes have distinctive small holes throughout their bodies. The fragments do not split or splay at their ends. Their fibre diameters are often greater than 1µm. Usually they have aspect ratios <10:1.

Gypsum

Gypsum may appear as straight 'fibres' with parallel sides. The ends of larger particles may show the distinctive trapezoid shape of a single crystal or the characteristic 'V' shape or 'arrowhead' of a twin. These crystals have low relief because their RIs (1.52-1.53) are less than asbestos (although still higher than the mounted filter). Also they are lath shaped and tend to lie on their largest faces: thinness of the laths contributes to low relief; usually they appear grey-black against the background. The visible diameter is often >1µm. The aspect ratio is usually <10:1 and is not as variable as for asbestos. When gypsum is present there will be many particles of similar appearance which will be too short to be included in the fibre count.

Slivers from talc, exfoliated vermiculite, mica and other platy minerals

These can appear as straight 'fibres' with parallel sides which often show steps. Split or splayed ends which are characteristic of asbestos are not seen generally. For some micas the RIs are considerably greater than that of the filter, but for talc and vermiculite the lowest RIs are comparable to those of chrysotile; generally, fine 'fibres' are black against the background, while thicker 'fibres' have white centres and black outlines with bright phase halos. There is a wide range of apparent diameters, some being 'sub-micron'. If slivers of these minerals are present (interfering with the fibre count), plates of the materials will be present as well. The aspect ratios usually are less than those for asbestos, but may be >10:1.

Aramids and other macerated synthetic organic materials

Often the fibres are curved with sides appearing parallel. Also, they can appear as bundles with splayed ends. Aramid fibrils often have a characteristic 'C' shape caused by relaxation when they become detached from the larger manufactured fibres;[7] they show high relief because of the high RI along their lengths, and frequently show white centres and black outlines with phase halos although they are relatively fine (<1µm). A particular problem is Aramid 'pulp': this can appear as fine fibres, more highly twisted than chrysotile, and with more splayed ends. The RIs of polypropylene (PP) are close to that of the mounted filter and the finest such fibres will not be visible: diameters usually are >1µm and many are too large to be included in the count; PP fibres have high aspect ratios, seldom <20:1. Polyethylene (PE) has refractive indices of similar magnitude to those of chrysotile and hence fibres of similar size show similar relief. Its morphology is similar to that of polypropylene but it has high birefringence: otherwise the properties of macerated fibres are similar to polypropylene.

Incinerated natural organic materials (such as burnt stubble)

These can occur as straight single fibres, often with parallel sides, and without split or splayed ends. Generally they are opaque and have high relief. Their diameters seldom are >1µm, although they may be larger than this if originating from close to the source of ignition. They have lower aspect ratios than asbestos. Usually there will be particles of similar appearance present which will be too short to include in the fibre count. If samples are collected close to the source of ignition, there will be other opaque or blackened particles present (some of which will still show plant morphology). Fine fume may be present.

Dead skin swarf

This material can appear as 'curved fibres' for which the sides are seldom parallel. These 'fibres' often have thickened sections at one end (a residue from the original cell shape) and seldom show the split ends characteristic of chrysotile. They have low relief because their RIs are close to that of the mounted filter; generally they appear as dark grey to black against the background, and usually are too thin to show white centres or phase halos. Their diameters usually are >1µm, and their aspect ratios usually are <10:1. When dead skin swarf is present, other dead skin cells with more characteristic morphologies normally are present on the mounted samples. Dead skin cells can be found in all human environments.

Paper swarf

These fibres often are curved; the sides seldom are parallel, and they often have thickened sections at one end (from the original cell shape); they never show split ends, characteristic of asbestos, although the thicker fibres may appear to have jagged ends characteristic of mechanically pulped wood fibre. They have low relief (with RIs of similar magnitude to chrysotile) and they generally appear dark grey to black against the background. Usually they are too thin to show white centres or phase halos. Normally their diameters are >1µm and their aspect ratios are <10:1. When paper swarf is present, usually other paper fibres with more characteristic morphologies are present on the filters.

Plant and insect hairs

These can appear as curved or straight fibres. Some, particularly insect hairs, can have smooth sides but seed hairs can show regular protrusions including barbs. They frequently taper towards one end, often part of the cellular structure can be seen, and occasionally the thicker end of the fibre may retain part of its 'root'. Insect hairs can have high relief. Plant hairs have low relief because their RIs are closer to that of the mounted filter. Usually diameters are >1µm and aspect ratios can be around 20:1. On some mounted samples where hairs are present, other signs of life (including pollens, leaf trichomes, moth scales and fragments of moth scales, which also can appear as fibres) may be seen.

TABLE 3: Summary of properties observed by PCM

	Morphology	Diameter	Aspect ratio	Relief
Amphibole	straight; often parallel sides or splayed ends	seldom >1µm	often >10:1	high
Chrysotile	curved; often parallel sides or splayed ends	seldom >1µm	often >10:1	moderate
MMMF	straight or curved	often >1µm	often >10:1	(depends on type)
Diatoms	acicular or circular; holes in structure	often >1µm	usually <10:1	RI < filter
Gypsum	often straight with parallel sides	often >1µm	usually <10:1	low
Talc, exfoliated vermiculite, mica, etc	often straight	wide range (sometimes sub-micron)	~10:1	moderate for talc and vermiculite; high for mica
Incinerated organics	straight (often with parallel sides)	seldom >1µm	~10:1	high
Dead skin swarf	curved; often thick sections at one end	usually >1µm	usually <10:1	moderate (similar to chrysotile)
Paper swarf	curved; often thick sections at one end	usually >1µm	usually <10:1	moderate (similar to chrysotile)
Plant and insect hairs	curved or straight tapered ends	usually >1µm	up to 20:1	plant hairs moderate (similar to chrysotile) insect high
Aramids and macerated organic material	Aramids curly; polypropylene straight	usually >1µm	Aramids ~ 20:1; polypropylene >20:1	Aramids high; polyethylene medium; polypropylene low

APPENDIX 4: PLM examination of mounted acetone/triacetin filters

For the purposes of fibre discrimination in fibre counting, the direct observation of at least two properties should be made before any fibre is eliminated from the count. The absence of one expected optical effect cannot be used to imply the absence of a particular fibre type. The ability to use this technique for discrimination depends on an understanding of relief, pleochroism, birefringence, extinction and extinction angle, and sign of elongation. (Further information, particularly regarding the problems in evaluations of very small diameter fibres on mounted slides, is given in Appendix 3.)

Properties of fibres seen by this technique

The following is a discussion of the properties of asbestos and non-asbestos fibres which can be observed using optimum PLM conditions, *that is, using oil immersion to maximise resolution*. Some of these effects may be visible using a 40x objective of NA 0.65, but high magnification objectives (particularly 1.25 NA oil immersion types) will give better resolution and hence better discrimination between fibres.

Pleochroism

The asbestos varieties chrysotile, amosite, fibrous tremolite and fibrous anthophyllite are virtually colourless in plane polarised light. Crocidolite has a natural strong absorption which gives a dark blue colour when parallel to the polariser and pale blue-grey when parallel to the analyser. Fibrous actinolite often has a pale green colour when parallel to the polariser. However, it is difficult to observe these properties on airborne fibres on mounted filters in plane polarised light; a more sensitive test (especially for crocidolite) is to align the fibre at 45° between crossed polars and to rotate the polariser or analyser 5° in either direction from the extinction position: the fibre will show different colours in the '-5°' and '+5°' analyser positions. Fibres which show such a change in colour are pleochroic.

Birefringence

Asbestos

Birefringence of the fibres is the numerical difference between their highest and lowest RIs. It can be assessed with the mounted filter between crossed polars and the fibre aligned at 45° to the plane of vibration of the polariser when maximum interference colours are seen. Interference colours are also dependent on the thickness of the fibre; for asbestos fibres only first order colours are seen for those <3µm diameter. Amosite, fibrous tremolite and fibrous anthophyllite have moderate birefringence, crocidolite and chrysotile have low birefringence.

MMMF

Glass, mineral wool and ceramic fibre are isotropic, but strain and size effects occasionally produce low order interference colours in some areas of some fibres.

Other minerals

The birefringence of gypsum is low and needles collected from an airborne cloud seldom show observable interference colours. Another commonly encountered mineral fibre is calcium carbonate which has extreme birefringence. It may be readily distinguished from other fibres because when aligned perpendicular to the polar it has high relief while it disappears when aligned parallel to the polar as its RI is almost the same as the filter. Rutile has very high birefringence and even 1µm diameter fibres have bright interference colours (1st order white). Also platy materials on edge show higher interference colours than do the equivalent diameter asbestos fibres. Slivers of talc, mica and vermiculite etc have no birefringence when the sliver seen is part of the flat face of the plate. However, when the sliver is 'on edge' the birefringence is high and, depending on the thickness, interference colours above first order white may sometimes be seen.

Animal and plant fibres

Some plant materials, especially if thick, display distinctly

higher interference than chrysotile of apparently similar dimensions. Dead skin cells will lie on their largest face, have very low birefringence and may not show visible interference colours between crossed polarisers. However, increased thickness of the rolled edges can enhance interference, and it is these edges which generally resemble 'fibres'; crystalline matter adheres to the surface frequently. Paper fibres (swarf) have moderate birefringence, the colours depending on thickness and tightness of the cellulose spiral (which vary with location in the cell); thus, the swarf can show virtually no interference colour for very thin fibres and higher interference colours for thicker fibres (especially if the flattened cell is 'on-edge' or if the swarf originates from near the cell tip).

Para-aramid fibrils

Para-aramid fibres have extreme (very high) birefringence and fibrils of respirable sizes (abraded from large fibres) show bright high order white interference colours between crossed polars.

Angle of extinction

The asbestos varieties chrysotile, amosite, crocidolite and fibrous anthophyllite show straight ('parallel') extinction when the fibres are parallel to the vibration direction of either the polariser or the analyser; fibrous actinolite and fibrous tremolite show parallel extinction or nearly so (<5° from parallel). Cleavage fragments from non-fibrous serpentine or amphibole minerals often show oblique extinction. *Fibres showing oblique extinction* >5°*from parallel are not asbestos.* When 'on edge', the common minerals mica, talc and vermiculite show parallel extinction; when lying flat there is no visible interference. Gypsum and wollastonite have variable angles of extinction depending on orientation. Inclined plates and slivers differ in their angle of extinction depending on their orientation.

Sign of elongation

Crocidolite fibres have a negative ('length fast') sign of elongation. All other asbestos fibres have a positive ('length slow') sign of elongation (see note below). This property can be determined for a fibre under observation only if the interference can be seen. The first order red plate should only be used with caution for fibres which show interference colours above first order white when alternative compensators such as a quarter wave plate can and should be used. Fibres with extreme birefringence such as aramids can be confirmed as such by using the first order red plate when they still appear white in the position of maximum interference. Thus, together with the curly 'C-shaped' morphology, this provides a means of distinction from other fibre types.[7] (Another characteristic of p-aramid is its white fluorescence under broad band UV radiation: this permits discrimination from other fibre types while counting;[6,7] see also paragraph 14.)

Note: It has been reported that shortly after sampling onto filter material, crocidolite fibres can initially appear length slow: the reason for this is unclear.

Table 4: Summary of properties observed by PLM

	Pleochroism	Birefringence	Extinction	Sign of elongation
Amphibole	crocidolite and actinolite are pleochroic	crocidolite low; others moderate	usually parallel; (actinolite and tremolite sometimes up to 5°); and complete	crocidolite usually length fast (negative)
Chrysotile	not pleochroic	low	parallel/ undulose	length slow (positive)
MMMF	not pleochroic	not birefringent (unless strained)	if showing strain complete, parallel	none, except where showing strain it can be either length fast or slow
Gypsum	-	low	complete, angle varies with orientation	+ve
Calcium carbonate	-	extreme	complete, parallel	-ve
Talc, exfoliated vermiculite, mica, etc	pleochroic only in some high refractive index micas	depends on orientation: ranges from high down to zero	parallel 'on edge'; total when flat	+ve
Wollastonite	-	low	varies with orientation	varies with orientation
Dead skin swarf	-	weak when flat; rolled edges may give some birefringence	attached particles interfere	
Paper swarf	-	none when thin; low to moderate when thick	parallel, can be incomplete	+ve
Plant fibres	-	some thick show moderate	parallel	+ve
Aramids and macerated organic material	-	Aramids extreme (bright white); PE moderate PP low	Aramids-undulose	+ve

APPENDIX 5: Observations which can be made by SEM

A combination of morphology and chemistry can be used to discriminate chrysotile from amphibole asbestos and to an extent between each of the regulated amphibole asbestos types. The typical relative intensity obtained from an EDXA detector fitted with a beryllium window are described. Improved detection of light elements will be obtained with a thin window or windowless detector, which will change the relative peak intensities given below.

Chrysotile

Normally chrysotile in aerosols is found as fine fibres (curly or straight) with diameters <1µm. The fibres often display splitting at the ends and often occur in bundles. When examined at high magnification their fibrillar nature may be seen. The EDXA spectrum shows peaks for magnesium and silicon in the approximate height ratio 3:4/Mg:Si; also a very small iron peak may be seen.

Amphibole asbestos

Generally the amphibole asbestos minerals are parallel sided, needle-like or lath-shaped, fibres with aspect ratios >10:1 (sometimes up to 100:1). Fibres with widths >1µm usually show splitting at the ends, occur in bundles and show gentle curving (without sharp angular bends). Usually in airborne dust samples fibres are <1µm in diameter and appear straight with parallel sides.

- *Amosite* is characterised chemically by the presence of a strong iron and silicon peak with a peak height ratio around 4:5/Fe:Si; a small magnesium peak may be found: the presence of a small/trace manganese peak is indicative of amosite as opposed to crocidolite. Amosite fibres usually have rectangular ends.

- *Crocidolite* is characterised chemically by the presence of strong iron and silicon peaks in the EDXA spectrum in the approximate height ratio 4:5/Fe:Si, and by a small sodium peak and a slightly larger magnesium peak. The sodium content is 5-6%, but at the low energy range of NaKα X-rays, X-ray yields are low, absorption is high and detector efficiency is low, so that the sodium peak may not be detected for fine fibres. The use of gold coating makes detection of sodium even more unlikely. Hence the EDXA spectra of crocidolite and amosite may appear similar.

- *Anthophyllite* contains silicon, magnesium and iron in varying proportions, although the iron content usually is the lowest of these. Frequently anthophyllite is found in association with talc. Talc 'fibres' may have compositions similar to that of anthophyllite; the main distinction between them is by morphological observations: anthophyllite has a straight needle-like or lath-like form, often with stepped ends, while talc has a platy or ribbon-like form showing kinks, steps and angular folds as a result of inelastic deformation.

- *Tremolite* is characterised chemically by the presence of strong peaks for silicon, magnesium and calcium in the EDXA spectrum (possibly also with a small iron peak).

- *Actinolite and ferro-actinolite* give strong calcium, iron, magnesium and silicon peaks. It is necessary to determine the proportions of iron in addition to the other peaks for these species. Proportions of iron and magnesium are continuously variable in the mineral solid-solution series tremolite/actinolite/ferro-actinolite.

Neither SEM nor TEM can unambiguously distinguish between fine elongated cleavage fragments of the minerals grunerite, riebeckite, anthophyllite, tremolite or actinolite from their asbestos analogues on a fibre by fibre basis.

Non-asbestos fibres

Many fibres other than asbestos can be found in airborne dust, depending on circumstances. Some of these may be rare in a general sense, but they might be found in particular situations (such as mineral processing). A knowledge of circumstantial factors is important in making a judgement about the identity of an individual fibre. Some fibre types, their possible morphologies and the chemical elements expected in them, are given below.

- *MMMF* usually appear as straight or gently curved cylindrical cross-section fibres, often >2µm in diameter (although some may be much finer). Their chemical compositions are highly variable: some contain silicon and aluminium only (ceramic fibres) while others also contain various alkali elements and elements such as barium, boron, calcium, iron and magnesium. Other refractory fibres contain elements such as zinc and chromium. MMMF may show conchoidal fracture across fibres.

- *Cellulose (wood or paper) and other natural organic fibres* tend to be irregular and curly in shape, but sometimes are ribbon-like or cylindrical (reflecting their biological origins). Being organic they have no distinctive elemental peaks in their EDXA spectra, and would produce very low X-ray count rates for their sizes. Often organic fibres will be distorted by or will suffer damage from the energy of the electron beam during analysis. SEMs with windowless or thin-window detectors can differentiate organic from non-organic fibres by their high carbon and oxygen contents.

- *Gypsum and anhydrite needles* can resemble amphibole asbestos morphologically; they are distinguished easily by their EDXA spectra which contain calcium and sulphur (sometimes with a little aluminium) in contrast to elements in the amphiboles (see above).

- *Calcium silicates (wollastonite), calcium aluminium silicates (tobermorite) and calcium aluminates* are fairly common constituents of building materials and can fracture to give particles which meet the regulatory fibre size criteria, sometimes with aspect ratios in excess of 10:1. Usually they are low aspect ratio particles, and they contain calcium and sometimes aluminium (in the absence of iron and magnesium) to distinguish them from the asbestos minerals.

- *Calcium carbonate* may be found with the correct dimensions for counting, although with low aspect ratios and non-parallel sides terminating in a point. Chemical analysis shows only calcium and carbon and oxygen, on SEMs with windowless or thin window detectors.

APPENDIX 6: Discrimination fibre count sheet for PCM/PLM

The following count sheet is from the analysis of a background count where there are present suspected amosite fibres and MMMF fibres together with other interfering fibres which cannot be positively analysed.

Laboratory sample: 1054/96	Laboratory report
Client sample:	Sheet 1
Analyst: GH	Date 12/05/96
Fibre type being analysed: MMMF	Other known interfering fibres: MMMF

Strategy adopted: Exclusion of MMMF, estimate of amosite fibre concentration

Stopping rules: 200 fields, or 200 fibres, or at least 50 fibres of diameter >1 micron

Graticule diameter: 99 µm

HSE/NPL Mk 2 visibility: Band 5 visible

1. Field number	2. Fibres/ field	3. Fibres to examine	4. Characteristics used for discrimination	5. Fibres subtracted
2	1	1		
5	2	1		
		1	Straight, isotropic	1
7	1			
10	1	1	Straight, isotropic	1
13	1	1		
			... and so on ...	
186	2	1		
		1	Straight, isotropic	1
193	1	1		

analysed fibres (this sheet):	10		*fibres subtracted (this sheet)*	4
analysed fibres (previous sheets):	29		*fibres subtracted (previous sheet)*	12
total analysed fibres so far:	39		*total fibres subtracted*	16

Thus the number of fibres to be used in the calculation of airborne concentration:

= 39 - 16 = 23

- *In another example of a discrimination PCM/PLM count applied to a background sample:* 20 fibres are counted, of which two are identified as being non-asbestos: the calculation must be performed assuming that all 20 fibres are asbestos (as less than 10 non-asbestos have been found).

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1 Acrylonitrile *charcoal tube/gas chromatography (GC)*
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4 Standard atmospheres *permeation tube*
5 On-site validation of methods
6/2 Lead *atomic absorption (AA)*
10/2 Cadmium *AA*
12/2 Chromium *AA*
14 Total inhalable and respirable dust *gravimetric*
15 Carbon disulphide *charcoal tube/GC*
16 Mercury *adsorbent tube (Hydrar) AA*
17 Benzene *charcoal tube/GC*
18 Tetra alkyl lead *continuous monitoring*
19 Formaldehyde *colorimetric (Chromotropic acid)*
20 Styrene *pumped charcoal tube/GC*
21 Glycol ethers *charcoal tube/GC*
22 Benzene *thermal desorption/GC*
23 Glycol ethers *thermal desorption/GC*
24 Vinyl chloride *charcoal tube/GC*
25/2 Organic isocyanates *reagent bubbler/HPLC*
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27 Diffusive sampler evaluation protocol
28 Chlorinated hydrocarbons *charcoal tube/GC*
29/2 Beryllium *AA*
30/2 Cobalt *AA*
31 Styrene *pumped thermal desorption/GC*
32 Phthalate esters *solvent desorption/GC*
33 Adsorbent tube standards
35 HF and fluorides *ion-selective electrode*
36 Toluene *charcoal tube/GC*
37 Quartz in respirable airborne dust *direct infra-red*
38 Quartz in respirable airborne dust *KBr disc technique*
39/4 Asbestos fibres *light microscopy (European reference version)*
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41/2 Arsenic *AA*
42/2 Nickel *AA*
43 Styrene *diffusive/thermal desorption/GC*
44 Styrene *diffusive/solvent desorption/GC*
45 Ethylene dibromide *solvent desorption/GC*
46 Platinum *AA*
47 Rubber fume in air measured as total particulates and cyclohexane soluble material
48 Newspaper print rooms: measurements of total particulates and cyclohexane soluble material in air
49 Aromatic isocyanates *acid hydrolysis/ diazotisation*
50 Benzene *diffusive/thermal desorption/GC*
51/2 Quartz in respirable dusts *X-ray diffraction (direct method)*
52/2 Hexavalent chromium in chromium plating mists *colorimetric (1,5-diphenylcarbazide)*
53 1,3 Butadiene *thermal desorption/GC*
54 Protocol for assessing the performance of a pumped sampler for gases and vapours
55 Acrylonitrile *diffusive/thermal desorption/GC*
56/2 Hydrogen cyanide *ion-selective electrode*
57 Acrylamide *liquid chromatography*
59 Manmade mineral fibres
60 Mixed hydrocarbons
61 Total hexavalent chromium compounds in air *colorimetric*
62 Aromatic carboxylic acid anhydrides
63 Butadiene *diffusive/thermal desorption/GC*
64 Toluene *charcoal diffusive/solvent desorption/GC*
65 Mine road dust: determination of incombustible matter
66 Mixed hydrocarbons (C_5 to C_{10}) in air *diffusive/ thermal desorption/GC*
67 Total (and speciated) chromium in chromium plating mists *colorimetric (1,5-diphenylcarbazide)*
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