Development of a method for the determination of low contents of asbestos fibres in bulk material

Thomas Schneider, Laurie S. T. Davies, Garry Burdett, Jan Tempelman, Salvatore Pulledda, Ole Jørgensen, Duncan Buchanan and Luigi Paolotti

Asbestos is a category 1 carcinogen under the EU classification, but in the absence of a method to quantify asbestos in a matrix at the 0.1% level, there has been a delay in implementing relevant directives to asbestos. An analytical scheme for identification and quantification of asbestos using polarised light microscopy (PLM) and phase contrast optical microscopy (PCM) has now been developed. When used on artificial mixtures by an experienced laboratory, it achieved the required target performance, at 0.1% asbestos concentration by mass in a bulk sample, to obtain a result, which with 90% probability, is correct within a factor of two. The method of identification by PLM and quantification by PCM has been assessed by interlaboratory comparisons. The method begins with an initial identification using PLM, and depending on asbestos type and matrix a combination of preparation procedures are used to produce the analytical filter. A gentle comminution method was used which reduces the risk of overmilling. The asbestos mass percentage on the filter is quantified using PCM in combination with a PLM attachment for identification of possible non-asbestos fibres. The final method is supported by efficient methods for fibre identification for size determination and calculation of total fibre volume. A statistical analysis of mass concentration estimates was made and the effect of preferred orientation of fibres on the analytical filter was quantified.

Keywords: Asbestos mass determination; optical microscopy; bulk material; comminution
continuous range of morphological forms only some of which may be asbestos. The temperature, pressure, and concentration of mineral forming medium may vary from place to place within the same rock. Therefore, serpentine may occur as chrysotile, antigorite or lizardite. The amphiboles may form acicular crystals (Fig. 1), fibres (Fig. 2) or prismatic crystals which by cleaving may generate elongated cleavage fragments (Fig. 3). These fragments are part of a crystal, while asbestos fibres are aggregates of numerous fibrils. Ultimately, it may not be possible either to define the exact amount of asbestos by mass or by number in a material, as there is no strict dividing line between the formation of acicular crystals and asbestos.

The method is needed for classifying hazard, i.e., the potential of a material to release fibres. A standard challenge was developed to maximise the amount of fibres originally present in clumps or bound in the matrix then be released in the sample and thus be detected as fibres, while minimising the risk of destroying fibres by overmilling. It was not the purpose of the challenge to relate directly to exposure risk from airborne fibres, which is handled under EC Directive 83/477/EEC. However, for such fibres the shape criteria is: width < 3 μm, length to width ratio (aspect ratio, AR) > 3 : 1, length > 5 μm. Since asbestos fibres in a bulk sample can only disintegrate into fibres with smaller width or into shorter fibres during use, the implications for the present method is that all fibre widths, but only fibres longer than 5 μm, will need to be evaluated in the bulk material. The aspect ratio AR < 3 is also used as an exclusion criteria in the method.

Further problems related to shape arise from the use of PCM for quantification. The analyte is observed as discrete entities and their volume has to be estimated from the projected image. Thus efficient counting and sizing methods and a mechanism for extrapolation from the two to the three dimensional shape had to be developed.

**Experimental**

**Overview**

The method begins with an initial identification using PLM, and depending on asbestos type and matrix a combination of preparation procedures are used to produce the analytical filter. The asbestos mass percentage on this filter is quantified using PCM in combination with a PLM attachment for identification of possible non-asbestos fibres. Although measurement of extinction angle, birefringence, pleochroism, texture, and elongation would have allowed classification of fibres, it was decided to use only a standard PCM with polariser and analyser and to rely on observations that a non-specialist microscopist could reliably and reproducibly record. The analytical scheme allows additional analysis by scanning (SEM) or transmission electron microscopy (TEM) for difficult samples. The flow diagram, Fig. 4, illustrates the sample preparation scheme. The exact choice of individual steps must be made by an experienced person, who can write the detailed procedure to be applied in the laboratory for each specific type of sample. The individual steps are described below.

For development of this method a basic stock of 12 artificial materials were produced. They comprised: (1) New York talc undiluted, 20–40% anthophyllite; (2) New York talc, (1) dilution 1 : 10; (3) New York talc, (2) dilution 1 : 10; (4) acid washed dolomite, contains tremolite asbestos; (5) 1% amosite in soil, spiked; (6) fibrous wollastonite, no asbestos; (7) vermiculite with tremolite asbestos, spiked, 0.05%; (8) amosite present as asbestos cement fragments in demolition waste, spiked, 0.25%; (9) chrysotile in olivine sand, spiked, 0.01%; (10) crocidolite in sepiolite, spiked, 0.1%; (11) amosite in talc, spiked, 0.15%; and (12) dolomite.

**Initial identification of fibres using polarised light microscopy (PLM).**

The method begins with a preliminary evaluation by stereo microscopy. Based upon the observation of physical appearance, the fibres are mounted in the most suitable refractive index liquid. PLM is used to identify fibres using morphology, pleochroism, birefringence, extinction, and sign of elongation. The refractive index is assessed by observing relief, Becke line,
or dispersion staining colours. The method is described in detail in Burdett. 3

Optical properties alone may not be sufficient to distinguish between tremolite and actinolite from some sources (because these minerals are members of a ‘solid solution series’ for which there is continuously varying composition giving a continuous range of refractive index) or between tremolite and anthophyllite (because they have similar birefringence and ranges of refractive index). Should such distinctions be necessary, additional methods (such as electron microscopy) may be used.

**Fibre counting and sizing using PCM–PLM**

Four types of microscopical methods are commonly used for quantification of asbestos in bulk samples:

1. Comparison with reference slides, see for example method 9002 in NIOSH. 4 This method is semi-quantitative at best, and is not suitable for concentrations below 1% m/m.
2. Determination of fibre number per unit weight of material. The result will depend critically on the preparation step prior to microscopy.
3. Projected area percentage determined by point counting. 4 This method is inherently biased. 5 The result will also depend on the preparation step prior to microscopy.
4. Volume determination based on measured fibre length and projected width.

Because of the limitations of the methods 1–3, quantification was based on volume determination.

When counting fibres using PCM, rules are needed to decide if a fibre not completely contained in the field of view has to be included and with which statistical weight. The following rules and weights for a fibre overlapping the field of view were

![Diagram](image-url)  
**Fig. 4** Preparation of sample for PLM–PCM quantitative analysis.
Comminution

Comminution is a key step and represents the major part of the standard challenge. A range of milling methods were initially considered. The final selection for the study were the following three methods:

1. The McCrone Micronizing Mill. It uses a wet technique, is relatively small, and is compact. The comminution process is standardised.

2. A steel percussion mortar, which is used by mineralogists for comminuting minerals without damaging crystal structure.

3. Mortar and pestle. The main reason for its inclusion was that this technique is widely used for preparation of powders.

Milling of acid washed dolomite and of talc in a McCrone mill showed that with increasing milling time the response for tremolite as measured by X-ray diffractometry, fibre length, and more important diameter, continued to decrease, whereas the total number of fibres per unit weight of material increased (Table 1). After 45 min milling of the acid-washed dolomite no fibres were visible in PLM.

The risk of overmilling has also been demonstrated by others. A steel percussion mortar was constructed from a commercial steel mortar with pestle and cylinder. A ‘pile driver’ ensured a reproducible stroke on the pestle. Calcite, tremolite and quartz were comminuted, and the resulting size distribution determined by sieving. An almost linear relationship was found between the potential energy of the pile driver and the median of the diameter distribution.

The percussive mortar was used to comminute steel mortar with pestle and cylinder. A ‘pile driver’ ensured a reproducible stroke on the pestle. Calcite, tremolite and quartz were comminuted, and the resulting size distribution determined by sieving. An almost linear relationship was found between the potential energy of the pile driver and the median of the diameter distribution.

The key problem in using manual grinding by mortar and pestle is to standardise the pressure and movement of the pestle. This problem was resolved by using a large mortar with a heavy pestle and by moving the pestle in a circular movement at 100 rev min⁻¹, without pressing the pestle. The degree of grinding can be changed by changing the grinding time. Calcite was ground to below 100 µm in 0.25 min, tremolite in 1 min, and quartz in 10 min. This mortar was finally chosen because the test showed that most types of asbestos are more easily comminuted by ceramic mortar than by percussion mortar. An exception is chrysotile which can neither be easily comminuted by ceramic mortar nor by percussion mortar. Furthermore a mortar is readily available and inexpensive. It is easy to clean.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Grinding time/min</th>
<th>Length/µm *</th>
<th>Diameter/µm *</th>
<th>Counts s⁻¹ for 400 µg †</th>
<th>Number ×10⁶/kg</th>
<th>RSD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tremolite in acid-washed dolomite</td>
<td>0</td>
<td>22 (2.2)</td>
<td>1.6 (2.6)</td>
<td>18.7 (9.2)</td>
<td>3.82</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>14.2 (2.4)</td>
<td>1.2 (2.4)</td>
<td>46.6 (6.9)</td>
<td>15.6</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>11 (2.2)</td>
<td>1.05 (2.2)</td>
<td>39 (3.4)</td>
<td>14.5</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>9.3 (2.0)</td>
<td>0.75 (2.2)</td>
<td>31.7 (7.0)</td>
<td>32.5</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>8.0 (1.9)</td>
<td>0.66 (2.0)</td>
<td>24.3 (1.7)</td>
<td>49.4</td>
<td>5</td>
</tr>
<tr>
<td>Tremolite in talc</td>
<td>0</td>
<td>11 (2.1)</td>
<td>4.3 (2.1)</td>
<td>144 (12.5)</td>
<td>10.6</td>
<td>8.1</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>9.2 (1.8)</td>
<td>3.7 (1.8)</td>
<td>130 (11.8)</td>
<td>14.1</td>
<td>7.6</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>8.4 (1.8)</td>
<td>2.8 (1.7)</td>
<td>96.2 (4.8)</td>
<td>26.3</td>
<td>6.6</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>6.4 (1.8)</td>
<td>2.3 (1.8)</td>
<td>69.9 (2.4)</td>
<td>37.0</td>
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<tr>
<td></td>
<td>45</td>
<td>6.0 (1.7)</td>
<td>2.2 (1.7)</td>
<td>4.3 (5.8)</td>
<td>34.6</td>
<td>4.5</td>
</tr>
</tbody>
</table>

* Geometric mean (geometric standard deviation in parentheses). † X-ray diffractometric response in counts s⁻¹, arithmetic mean (standard deviation in parentheses).
or more specific; cleaning by grinding a portion of quartz sand followed by washing in water eliminates the risk of cross contamination with fibres. In order to prevent over-comminution the grinding is carried out by repeated grinding for 1 min and sieving (106 μm sieve) to remove the generated fine fraction from the material in the mortar. It is not possible to sieve chrysotile, as the wire screen will trap the fibres. Communion of chrysotile to below 100 μm remains a challenge and no suitable method of reducing chrysotile to below 100 μm could be found. However, because the concept of a standard challenge is essential it was decided also to grind chrysotile, disregarding that this may be difficult to a degree depending on the chrysotile concentration and the matrix. The grinding time was set at 5 min, being an expert judgement.

Fibre dispersion

The final residue is dispersed in water for preparation of the analytical filter. Dispersion is assisted by ultrasonic treatment and use of a dispersing agent. The effect of using acetic acid as a dispersion agent on the fibre size, fibre mass and number concentration (measured by PCM) was determined on acid washed dolomite containing tremolite asbestos. It was found that dispersion in water adjusted to pH 3.0–4.0 with acetic acid did not change the size distribution. This was consistent with the results that within experimental uncertainties, no change in concentration by mass or by number could be detected. However, since use of acetic acid facilitates dispersion of chrysotile, and since its use is recommended by ISO, use of water with adjustment of the pH between 3–4 using acetic acid was adopted. Sonication for 30 min and 1 h gave a statistically significant lower mass percentage (two-sample t-test, \( p = 0.02 \)) and higher fibre number concentration (\( p < 0.001 \)) of tremolite asbestos. On the other hand, sonication reduced by a factor of 2 the RSD both for mass and for number concentrations. Use of a high energy ultrasonic probe will actually break down the fibres, but an ultrasonic bath used for a short time will only disperse fibres. A sonication time of 10 min was finally chosen. After sonication, the suspension is filtered onto a 0.8 μm membrane filter and cleared using the acetone–triacetin method.

Determination of fibre volume

When using microscopy only two dimensions; the fibre length \((L)\) and the projected fibre width \((B)\) is determined for each fibre, \(i\). The volume \(W_i\) of the single fibres has to be estimated as

\[
W_i = L_i (B_i)^2
\]

Obviously the true cross section area will usually not be square and fibres may exhibit preferred orientation on the filter. Thus some kind of correction factor is needed. Suppose that the true volume \(W_{\text{true}}\) is known. Then the shape correction factor, \(k\), is defined as

\[
\sum w_i^{\text{true}} = k \sum L_i B_i^2
\]

The correction factor applies to the total volume of a fibre population, not to the volume of individual fibres. The exact value of \(k\) was left open until the entire method had been developed and recovery studies were made. The overall best fit of PCM quantification of samples with known asbestos concentration was obtained for \(k = 0.48\).

Determination of asbestos mass percentage

Filter samples are prepared in replicate from each dispersion. For both filters 50 fibres are counted or 100 fields per filter, whichever comes first. At least 20 fields are counted. Each fibre is observed under crossed polarisers. The stage is rotated 180° and it is observed if any part of the fibre is illuminated (undulating or partial extinction). If this occurs, the microscope stage is rotated through 360°. An asbestos fibre will extinguish at four positions each 90° apart. If extinction occurs to within ±5° of the vibration orientation of the polariser or analyser, the fibre is classified as asbestos. All other fibres are classified as non-asbestos. These rules do not apply for thin fibres, which should always be classified as asbestos. The lower limit of width is approximately 1 μm, but may be larger, depending on the birefringence. The experience of the microscopist is essential. Pure samples of the regulated fibre types should be analysed at regular intervals to maintain familiarity with the performance of the classification procedures.

Chrysotile may be distinguished from amosite, crocidolite, tremolite asbestos, anthophyllite asbestos, and actinolite asbestos by its yarn-like morphology. Amphibole asbestos fibres are straight but long fibres can bend in curves with a large radius of curvature.

For each individual fibre, \(i\), \(W_i (\mu m^3)\) is calculated as

\[
W_i = L_{i,\text{f}} B_i^2
\]

where \(L_{i,\text{f}}\) is length of fibre within counting field and \(B_i\) is width of fibre \(i\), both measured in μm. The total fibre volume in the investigated number of fields is calculated as:

\[
\hat{W}_A = k_A \sum_{i=1}^{n} W_i
\]

\[
\hat{W}_C = k_C \sum_{i=1}^{n} W_i
\]

A refers to all individual fibres classified as amphibole and C to all individual fibres classified as chrysotile. The volume is corrected for influence of cross section shape by the factor \(k\), where \(k_A = 0.48\) and \(k_C = \pi/4\).

It was found that \(W_i\) were not log-normally distributed. Thus maximum-likelihood estimators or minimum variance estimators assuming log normality should not be used.

Rules for determining effective volume of fibre bundles and clusters were not specifically developed. Fibre densities were compiled from the literature. The fibre density may be lower than the non-fibrous mineral, which is given in the tables.

Table 2 Results from residual maximum likelihood (REML) analysis of milling time. Mass percentage estimates were calculated using a cross-sectional correction factor of \(k = 0.48\). The predicted mean is that calculated from the model.
of densities. Thus for chrysotile a density of 2.5 g cm\(^{-3}\) is adopted. A common density of 3.0 g cm\(^{-3}\) is applied for all amphiboles, as the PCM–PLM method does not always distinguish between the different amphibole asbestos.

The mass (g) of fibres on the filter is calculated as:

\[
M_{\text{A}} = \frac{10^{-12} \hat{W}_p A}{NG}
\]

\[
M_{\text{S,L}} = \frac{10^{-12} \hat{W}_p \rho_c A}{NG}
\]

where \(\rho_A = 3.0\) (g cm\(^{-3}\)); \(\rho_c = 2.5\) (g cm\(^{-3}\)); \(N = \) number of fields analysed; \(G = \) graticule area (mm\(^2\)); and \(A = \) dust covered area of filter (mm\(^2\)).

The asbestos mass percentage is calculated as:

\[
P = \frac{100F}{M_A} [M_{\text{A}} + M_{\text{S,L}}]
\]

\[
M_A = \frac{M_A Q_p}{Q_{\text{tot}}}
\]

where \(M_A = \) mass of powder (g) dispersed in total volume \(Q_{\text{tot}}\) (cm\(^3\)); \(Q_p = \) volume of liquid (cm\(^3\)) from dispersion filtered onto filter surface; \(A = \) mass of sample on filter (g); and \(F = \) mass fraction remaining after pre-concentration.

**Discrimination by aspect ratio**

In regulatory methods, an aspect ratio, AR > 3 : 1 has been widely used to discriminate between a particle and a fibre. However, crushing non-asbestos forms of anthophyllite, tremolite and hornblende produces fragments with AR > 3 : 1. In order to differentiate between asbestos and non-asbestos fibres it has been argued that a higher minimum aspect ratio (i.e. 10 : 1 to 20 : 1) should be used. Aspect ratio distributions of particles from six different samples including amosite in soil and demolition waste, tremolite in vermiculite, crocidolite in serpilolite, chrysotile in olivine sand and New York talc have been measured. It was found that an aspect ratio of about 8:1 is the best compromise to exclude most cleavage fragments without excluding more than about 10% of the asbestos fibres. Unfortunately, the crude sizing bins used for the PCM sizing do not allow this precision and the 5 \(\mu\)m length bins and 0.5 \(\mu\)m width bins make a 10 : 1 aspect ratio the pragmatic choice. Also the 10 : 1 aspect ratio is shown on the graticule.

Aspect ratio discrimination is used as follows. If a non-asbestos fibre population is indicated (the majority of fibres having AR < 10) an estimate of the non-asbestos component can be made by removing all fibres with AR < 10. This allows an estimate of the remaining possible asbestos fibres to be made. It is important that this additional discriminant analysis is reported as an observation and the initial result without applying the discrimination must be reported.

**Results**

**PLM identification**

The PLM method was tested on 57 samples taken from the stock of 12 materials. Very good agreement between the PLM result and the true asbestos component for all 57 samples analysed was obtained. Only for undiluted and diluted New York talc, and digested dolomite, were there any differences. However, for all of these samples the differences were for samples containing tremolite or anthophyllite. For all other samples the type and proportions of asbestos found by PLM were consistent with the known components of the original mixtures. For a sub-sample of 22 samples, analysis was repeated. All of the original results were reproduced.

**Aashing**

On ‘difficult-to ash’ samples containing bitumen, paint and epoxy the relative standard deviation (RSD) of the ash content was 1% or less (at 50% ash). For bitumen, with an ash content of 2.1%, RSD was 5%. On samples of dolomite containing tremolite, the RSD of the residue was 8%.

**Comminution**

The mortar and pestle method resulted in a reproducible comminution as can be seen from Table 3. Chrysotile containing samples will still contain large particles, as the repeated grinding/sieving procedure cannot be used. For such samples, separation of the ground material into a fine and coarse fraction using, e.g., a modified Andreassen pipette and analysis of both fractions may be the only solution. The coarse fraction should be inspected by PLM at a \(\times 100\) total magnification. If asbestos is present these fibres should be sized and included in the final calculation of asbestos mass percentage.

**Statistical analysis of asbestos number and mass percentage estimates**

During the development work a considerable number of samples were analysed by two counters at one laboratory using quantitative PCM and the standard Walton–Beckett graticule. A statistical analysis of the results was performed to determine accuracy and precision of the estimated mass percentages. A residual maximum likelihood (REML) analysis was used which included effects for counter, milling time, accuracy and precision, sub-samples and microscope slides.

**Accuracy**

Mass percentage estimates were calculated for samples with known asbestos content using maximum-likelihood estimation of fibre volumes. The ratio of observed to nominal asbestos content was calculated and the shape correction factor was adjusted to maximise the number of observations giving ratios in the target range 0.5 to 2. This gave a value \(k = 0.48\), and Fig. 5 illustrates the final result. The number of evaluations falling within the target range were 87, 100, 80, 92 and 83% for samples 5, 7, 8, 10 and 11, respectively. An overall total of 88% of individual results were within the target performance range. The analytical results were obtained using the normal Walton–Beckett graticule and the value may need adjustment when using the modified graticule. Also it will have to be validated against a broader range of amphibole asbestos samples. For chrysotile a circular cross-section is assumed (\(k = \pi/4\)). The value of the correction factor \(k\) is in agreement with the results of Pooley and Clark\(^{21}\) from which it was deduced\(^{22}\) that the
sections for estimating anthophyllite. Results using direct observation of fibre cross sections for estimating $k$ will be published elsewhere.

**Precision**

Due to the discrete nature of the analyse, there will be an inevitable variability in the fibre volume estimate. This variability is controlled by the total number of fibres sized and by the variance of the individual fibre volumes. The actual inevitable variability in the fibre volume estimate. This Due to the discrete nature of the analyte, there will be an equilibrium variability in the fibre volume estimate. This will be published elsewhere. The statistical analysis showed that accuracy and precision of asbestos mass percentage estimates were generally good. Accuracy although ultimately dependent on the most appropriate correction factor, $k$, was close to the method’s target performance criteria. The method was best applied to material containing amphibole asbestos. The method was generally unreliable for materials containing chrysotile.

**PLM intercomparison**

Two round robins, each involving four samples have been completed in which a number of EU laboratories and a selected group of UK laboratories accredited by the United Kingdom Accreditation Service (UKAS) for both the sampling and analysis of bulk asbestos were invited. The laboratories were asked to report whether asbestos was present in the sample and the type of asbestos found. In addition, the laboratories were asked to provide the visually estimated volume percentage of asbestos. No method for this was circulated, but it was seen as an opportunity to measure the bias that would be present if laboratories attempt to estimate asbestos content.

The laboratories were assessed by a scoring system based on analytical errors. Details will be published elsewhere. In summary, none of the laboratories made errors which were analytically unacceptable and which would have serious consequences if committed in reality. Some errors were made which were analytically unacceptable but that might not have significant consequences; such as the failure to detect a proportion of one asbestos component in the presence of an already detected amphibole asbestos, the false positive identification of a very small amount of asbestos in the absence of any other asbestos, or the failure to detect such a component. More errors were made which were analytically almost acceptable and would have no significant consequences; such as the failure to detect a very small proportion of any asbestos in the presence of an already detected amphibole asbestos, or the failure to detect such a component. The result stresses the importance of training and experience of the microscopist and an adequate quality assurance programme. Details will be published elsewhere.

![Fig. 5 Logarithmic ratio of observed to nominal mass percentage asbestos samples](image)

**Table 4** Mean and standard deviations ($s$) of mass percentages calculated as ratios of observed to nominal mass percentage estimates ($k = 0.48$)

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Counter 1</th>
<th>Counter 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>1.33</td>
<td>0.80</td>
</tr>
<tr>
<td>7</td>
<td>0.87</td>
<td>0.22</td>
</tr>
<tr>
<td>8*</td>
<td>0.72</td>
<td>0.37</td>
</tr>
<tr>
<td>9</td>
<td>1.67</td>
<td>1.93</td>
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<tr>
<td>10</td>
<td>1.14</td>
<td>0.42</td>
</tr>
<tr>
<td>11</td>
<td>1.39</td>
<td>0.60</td>
</tr>
</tbody>
</table>

* Zero milling time not included.

**PCM–PLM intercomparison**

An intercomparison in two parts of the PCM–PLM quantification method has been conducted, and details will be given elsewhere. The participants received a prepared slide and a simple bulk sample in the first round and five bulk samples in the second. Results were received from 12 in the first and from 11 in the second round. The results are shown in Fig. 6. One laboratory in particular (number 6) was known to be considerably more experienced than most and had already undertaken a considerable amount of research in developing a similar method for asbestos quantification. One sample contained only chrysotile and most laboratories reported the presence of a varying proportion of amphibole fibres incorrectly applying a correction factor, $k$, for each amphibole fibre reported. The resulting mass percentage estimates for this sample are therefore generally underestimated by an unknown amount. The findings for this first interlaboratory exchange indicate that with care, experience and perhaps considerable practice, it is possible to achieve analytical results approaching the target performance range.
Discussion

Use of optical microscopy limits the lower limit of visibility of fibres. Optical properties such as isotropic–non-isotropic and parallel–non-parallel extinction can only be observed if fibres have a minimum width $B$. For observed fibre width below about 1 $\mu$m no such observations can be made. Thus to minimise risk of false negatives (stating compliance with target concentration when the true concentration exceed the limit), such fibres have to be classified as asbestos. This increases the risk of false positives. However, it is always possible to use electron microscopy for further analysis of samples, e.g., if the result was $> 0.1\%$ $m/m$ and there is reason to believe that a considerable part of the fibre mass is not asbestos. A risk of false negatives cannot be entirely eliminated, e.g., for samples containing extremely thin fibres in large quantities. A person experienced in mineralogy may assess mineral constituents which combined with knowledge of sample origin may give clues about this risk. The method was generally unreliable for materials containing chrysotile.

Microscope samples will always contain particles from coarser materials comminuted during the production process and/or during the standard challenge. This may contaminate the sample with elongated cleavage fragments, which according to the present definition in EU regulation are not asbestos. A considerable part of our research, and of the effort to be spent by laboratories during analysis, stems from the need to separate those fragments from the legally defined asbestos. It is interesting to notice the recent decision to classify the bio-durable man-made ceramic fibres as a category 2 carcinogen. This could support the following hypothesis: provided cleavage fragments if inhaled have the same bio-durability as their asbestiform counterparts of the same size and shape, a difference in health effect is unlikely. If this hypothesis is true, it implies that the analyst at present has to spend large efforts on differentiating between asbestos fibres and fragments from the same parent rock, with no benefit to health protection.

In the interlaboratory comparison one of the most experienced laboratories came very close to the target performance. This is promising if compared with the experience from the Asbestos Fibre Regular Informal Counting Arrangement (AFRICA) scheme. This scheme has shown that: (1) There is a learning curve for microscopists, and it should be expected that full time training over two weeks and supervision during one month’s work with the method is needed to obtain reliable results. (2) Even for the much easier task of only counting fibre number on prepared microscope slides, the AFRICA scheme cannot after years of operation obtain a target performance as the one specified in the present project. Thus the original target performance may have to be relaxed.

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References


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