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ASBESTOS AND OTHER FIBRE LEVELS IN BUILDINGS

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Abstract—A Nuclepore filter/SEM method was used to determine the airborne concentration of asbestos and other fibres in office and plant buildings. The monitoring/SEM/XRMA conditions were chosen to match the collection efficiency, fibre detectability and convenience of the membrane filter/optical microscope method. Additionally, the SEM/XRMA technique provides identification of counted fibres and a detection limit of 1 fibre l.⁻¹ under the routine conditions of occupational hygiene sampling.

Application of the method to the collection of 193 air samples in the occupied areas of 22 asbestos-containing office and plant buildings gave the following asbestos counts: 22 fibre l.⁻¹ (1 sample); 3 fibre l.⁻¹ (3 samples); 2 fibre l.⁻¹ (5 samples); 1 fibre l.⁻¹ (22 samples); 0 fibre l.⁻¹ (162 samples).

Organic fibre levels ranged from 63-0 fibre l.⁻¹ (median = 4 fibre l.⁻¹).

Mineral (non-asbestos) fibre levels ranged from 13 to 0 fibre l.⁻¹ (median = 1 fibre l.⁻¹).

INTRODUCTION

THE RECOGNITION of the carcinogenic potential of respirable asbestos dust under occupational and para-occupational exposure has led to concern regarding the cancer hazard from asbestos in the environment. In Australia, this concern has focused on the question of airborne asbestos exposure in buildings containing asbestos fire-proofing, thermal insulation, etc.

The determination of asbestos in environmental samples is generally recognised as requiring the application of electron microscopy (BECKETT, 1973; MIDDLETON, 1982). The membrane filter/optical microscope [MF/OM] method (NH & MRC, 1976; WALTON, 1982) used for the evaluation of asbestos at occupational levels is unsatisfactory for this task because (i) the method gives non-zero and variable fibre counts for the blank collection filter (NH & MRC, 1976; RAJHANS and SULLIVAN, 1981) and (ii) the method cannot identify the counted fibres.

Point (i) limits the detection limit of the method for a 4-h sample to 10-20 fibres l.⁻¹. Point (ii) is important because, at environmental levels, the majority of respirable fibres found are generally not asbestos.

The Nuclepore polycarbonate filter/scanning electron microscope method [NPF/SEM] can match the collection efficiency, fibre-width detectability, and convenience of the MF/OM method but, additionally, provides a significant improvement in fibre number detection limit and a means of individual fibre identification (BECKETT, 1973; SPURNY *et al.*, 1970; MIDDLETON, 1982). It was therefore selected for use in the present study of airborne concentrations of asbestos and other

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fibres in office and plant buildings. The monitoring/SEM/XRMA conditions were designed to match the fibre size (width) detection limit of the MF/OM method that is commonly used for the measurement of asbestos fibres in occupational situations.

THE NPF SEM METHOD USED

1. Sampling

Head. Open face, 25 mm dia. (Gelman catalogue No. 1107). Brass cowl, length = 55 mm, internal diameter = 32 mm.

Filter. Nuclepore polycarbonate, 0.8 μm pore-size, 25 mm dia. (Nuclepore stock No. 110609). No filter pre-treatment was made. Samples were collected on the shiny side of the filter. Collection area = 370 mm².

Technique. Fixed-point samples were collected. The sampling head was located at chest height, with the collection filter facing downwards. The samples were collected under conditions of dynamic ambient air-flow during office hours.

2. Analysis

Instrumentation. Cambridge Stereoscan 180 scanning electron microscope with Link 290 energy-dispersive X-ray microanalysis unit.

Preparation. A sector was cut from the Nuclepore collection filter by scalpel, directly mounted on a 12.5 mm carbon stub by use of a natural organic liquid adhesive, coated with a 10 nm conducting layer of carbon using a liquid nitrogen trapped evaporative coating unit, and then counted and analysed. The specimen, as prepared, was capable of practical inspection to a resolution of 20 nm.

SEM conditions. Accelerating voltage = 30 kV
Beam diameter at specimen = 12.5 nm
Signal mode = secondary electrons
Stage = greatest practical working distance of 35 mm
Specimen tilt = 25°
Nominal magnification = 1200 ×
Average magnification at tilt = $1250 \pm 50 \times$
Field area at tilt = $0.0073 \pm 0.0004 \text{ mm}^2$

Counting. Quasi-rectangular fields counted along radial traverses.

Fibre selection criteria. Length > 5 μm , Width < 3 μm ,
Aspect ratio > 3:1

The practical limit on the size of a fibre that was consistently seen under the routine sampling and counting conditions was 0.3 μm fibre width at 5 μm fibre length. Fibres of width = 0.2 μm were not uncommon, while fibres of width = 0.1 μm were occasionally found. Such fibres were counted when found.

X-ray microanalysis. Each fibre found was excited by a static electron beam and the X-rays emitted from the fibre were sampled with an energy-dispersive X-ray detector of 149 eV resolution, under conditions of take-off angle and proximity to give a count rate of 500 counts s^{-1} for a 3 μm dia. amosite fibre. Sufficient counting time was allowed to give a total count of 15,000 counts over a range of 0–10 keV, thus providing good peak-to-background ratios for all elements of interest. The X-rays emitted from the fibre thereby provide a measure of the major and minor chemical elements ($z > 10$) in the individual fibre and so allow its identity to be determined (CHAMPNESS *et al.*, 1976).

3. Factors considered in developing the method

Nuclepore polycarbonate filter (NPF). The use of SEM strongly favoured the use of the Nuclepore polycarbonate filter for dust sampling. The NPF is made of solid, flat plastic (about 10 μm thick) through which nominal pore-size holes have been etched by collimated atomic particle bombardment and chemical dissolution. The flat surface makes the NPF an excellent choice for SEM work (BECKETT, 1973; SPURNY *et al.*, 1979).

The choice of pore-size for the NPF was determined by the detectability requirement for fibre width. The fibre-width detectability of the MF/OM method is 0.3 μm (for 5 μm fibre length). This limit allowed an 0.8 μm pore-size filter to be used. SPURNY *et al.*, (1979) have shown that for face velocities of 85 $mm s^{-1}$ and above the collection efficiency of the 0.8 μm NPF for 0.3 μm amosite fibres is 80% or higher.

Specimen preparation and mounting. To reduce the possibility of ambiguous EDXRMA results (and hence false identification of fibres), it was important that stub, collection filter, adhesive, and coating be limited to materials composed of light elements (e.g. $z < 10$) whose characteristic X-rays are not detected by EDXRMA. It is this concern which contributes to the reason for avoiding coating the collection filter with gold. Gold, when excited with 30 kV electrons, will produce X-rays of the following energies (in keV): $M\alpha = 2.123$, $M\beta = 2.205$, $L\alpha = 9.711$, $L\beta = 11.5$, $L\gamma = 13.4$. Three problems arise because of the presence of such background X-rays:

- (i) some element peaks from fibres are obscured by overlapping gold peaks (e.g. overlap of Au $L\alpha$, β with S $K\alpha$),
- (ii) additional preferential excitation of some elements in the fibre will occur (e.g. Fe $K\alpha$ by Au L lines; Si $K\alpha$ by Au M lines),
- (iii) the excitation of elements in a nearby or attached fibre or particle may occur and be interpreted as emanating from the target fibre.

SEM magnification. The fibre-width detectability also determined the SEM magnification that needed to be used and, hence, the SEM field size. A nominal magnification of 1200 $\times$ was found to be adequate to ensure routine detectability of all fibres 0.3 μm wide at 5 μm length.

Airborne fibre concentration detection limit.

$$\text{Airborne fibre } l^{-1} = \frac{AN}{anrt} \quad (1)$$

where A = collection area of filter, mm^2
 a = area of SEM counting field, mm^2
 N = number of fibres found
 n = number of fields counted
 r = sampled air flow rate, l. min^{-1}
 t = sampling time, min.

Equation (1) shows that, if a typical occupational hygiene air sampling rate and time (2 l. min^{-1} , 4 h) is used and if 1 l. is chosen as the volume of sampled air actually examined under the SEM at a magnification of $1200\times$, then about 100 SEM fields must be counted. This number of fields can be counted in about 1 h.

The significance of the fibre count number, N , can be directly assessed from a knowledge of the volume of air sampled that was actually examined in the SEM and from the known confidence intervals for the Poisson mean (HALL and SELINGER, 1985; CROW and GARDNER, 1959). For a single sample taken under the given conditions, with 0 asbestos fibres counted, the 80% confidence interval for the fibre count collected on the filter is 0–1.82 fibre l.^{-1} . For two samplings and a 0 asbestos fibre total count, the upper confidence limit falls below 1 fibre l.^{-1} .

Thus, the combination of *circa* 100 SEM fields with routine hygiene sampling conditions gives a practical detection limit for duplicate samples of 1 fibre l.^{-1} of air (i.e. 0.001 fibre ml.^{-1} of air). This is an improvement of at least 10 times on the detection limit obtainable by the MF/OM method.

COMPARISON WITH THE RTM2 METHOD

The direct use of the Asbestos International Association's RTM2 method (1984) for this work was investigated. It was not found to be the optimum method for the task at hand. The RTM2 method is also a NPF and SEM-based method. However, it uses a gold-coated Nuclepore filter for dust collection and ashes the collected sample before SEM/EDXRMA analysis. RTM2 has been developed for application to the task of determining mineral fibres in samples of high organic-fibre content (e.g. air samples in an asbestos-cement factory that is in the process of change to an organic-fibre-cement factory). The gold-coating protects the collection filter while ashing eliminates organic fibres in the sample. For the task of determining the respirable asbestos fibre content in office and plant buildings, however, the RTM2 method has the following disadvantages in relation to the original Beckett method as developed and reported here:

- (i) The ashing step eliminates organic fibres in the sample and, in so doing, negates the use of the NPF/SEM method as a supplementary technique to the MF/OM method for samples of low and/or mixed fibre content.
- (ii) The gold-coating on the collection filter forms a highly emissive background in the secondary electron mode used in SEM counting. This significantly reduces the contrast between the collected fibre and the background, thus necessitating the use of higher magnifications. The magnification of $2000\times$ needed in the RTM2 method, triples the SEM counting time relative to this method.
- (iii) The gold-coating produces gold X-ray lines that interfere with the EDXRMA, as discussed above.

- (iv) Specialized pre-treatment of the collection filter is required by the SEM laboratory before air sampling can be undertaken.
- (v) Specialized ashing of the collection filter is required after sample collection and before scanning electron microscopy.

RESULTS

The NPF/SEM Method has been applied to monitoring the air in twenty-two office and plant buildings. The majority of these buildings contained asbestos sources (three were monitored after the removal of asbestos sources, two had no known asbestos source) and these sources had been installed at the time of construction of the buildings. All sources had been *in situ* for at least 10 yr.

The details of the asbestos type and source within each building and of the results found in the monitorings are given in Table 1. The monitoring results refer to samples taken in the occupied parts of each building and generally in those areas most likely to be contaminated with airborne asbestos from the nominated sources. A summary of the monitoring results follows:

Buildings: Total = 22 (Office = 19, Plant = 3).

Asbestos present: Chrysotile = 9, Amosite = 9, Crocidolite = 2.

Asbestos removed = 3, No asbestos = 2.

Total number of air samples collected = 193.

Asbestos fibre concentrations found:

1 sample	22 fibre l. ⁻¹
3 samples	3 fibre l. ⁻¹
5 samples	2 fibre l. ⁻¹
22 samples	1 fibre l. ⁻¹
162 samples	0 fibre l. ⁻¹

The 22 fibre l.⁻¹ result was obtained in a room containing a man-hole access to a roof space in which loose floc amosite insulation was spread on top of the ceilings.

Organic fibre concentrations found:

1 sample	63 fibre l. ⁻¹
1 sample	59 fibre l. ⁻¹
1 sample	25 fibre l. ⁻¹
13 samples	10-20 fibre l. ⁻¹
58 samples	5-9 fibre l. ⁻¹
119 samples	0-4 fibre l. ⁻¹

Mineral (non-asbestos) fibre concentrations found:

3 samples	10-13 fibre l. ⁻¹
10 samples	5-9 fibre l. ⁻¹
36 samples	2-4 fibre l. ⁻¹
144 samples	0-1 fibre l. ⁻¹

TABLE 1. AIRBORNE RESPIRABLE ASBESTOS AND OTHER FIBRES IN OFFICE AND PLANT BUILDINGS

Building number	Asbestos source type and location	Number of samples	Asbestos fibre l. ⁻¹	Organic fibre l. ⁻¹ (Number of samples)	Mineral fibre l. ⁻¹
1	Amosite, fire doors, timber faced; unsealed at top and bottom	3	1 (1) 0 (2)	0.4 (3)	0.1 (3)
2	Chrysotile; asbestos-cement board surrounding re-heaters in air conditioning ductworks	8	1 (1) 0 (7)	5.6 (3) 1.4 (5)	2 (3) 0.1 (5)
3	Chrysotile; asbestos-cement board on walls, painted	8	0 (8)	5.6 (1) 1.4 (7)	2 (1) 0.1 (7)
4	Crocidolite; fire-proofing on steel structural members	8	0 (8)	5.8 (3) 1.4 (5)	2.3 (2) 0.1 (6)
5	Amosite; loose floc in roof space above ceiling	59	22 (1) 3 (2) 2 (4) 1 (9) 0 (43)	10.16 (6) 5.9 (14) 0.4 (39)	5.6 (1) 2.4 (9) 0.1 (49)
6	Amosite; spray insulation, under-roof on top floor	4	3 (1) 1 (3)	5.9 (2) 3.4 (2)	0 (4)
7	Chrysotile; spray insulation residual on plenum wall after replacement of steel roof	8	1 (1) 0 (7)	5 (2) 2.4 (6)	4 (1) 0.1 (7)
8	Amosite; fire-proofing on steel members on top floor	4	1 (3) 0 (1)	2.4 (4)	0.1 (4)
9	Chrysotile and amosite; asbestos-cement wall board and compressed board, unpainted	3	0 (3)	5 (1) 3 (2)	0.1 (3)
10	Amosite; plant insulation, after removal	2	0 (2)	5 (1) 2 (1)	0.1 (2)
11	No asbestos source identified in building	8	0 (8)	1.2 (8)	7 (1) 3 (1) 0.1 (6)
12	No asbestos source identified in building	4	0 (4)	2.4 (4)	0.1 (4)
13	Amosite; steam-pipe insulation enclosed by metal or cloth binding	8	0 (8)	5.6 (3) 1.4 (5)	10 (1) 2.4 (2) 0.1 (5)
14	Amosite; fire-proofing on steel members on top floor	4	2 (1) 1 (1) 0 (2)	25 (1) 5.8 (3)	0.1 (4)
15	Amosite and crocidolite; fire-proofing on steel members of building	4	0 (4)	5 (1) 1.4 (3)	2 (1) 0.1 (3)
16	Amosite; fire-proofing on steel members of building and roof	4	0 (4)	5.7 (3) 4 (1)	2.3 (2) 0 (2)
17	Chrysotile; minor component in under-roof spray insulation	10	0 (10)	10 (1) 5.6 (4) 0.4 (5)	2 (1) 0.1 (9)
18	Chrysotile; minor component in spray-on ceiling finish; before removal	5	0 (5)	5 (1) 0.4 (4)	2 (2) 0.1 (3)
19	As in 18; after removal	2	1 (1) 0 (1)	3.4 (2)	10 (1) 8 (1)
20	Chrysotile; minor component in spray insulation on concrete slabs	5	0 (5)	10.14 (1) 7.8 (2)	6 (2) 2.4 (3)

(continued)

TABLE 1. AIRBORNE RESPIRABLE ASBESTOS AND OTHER FIBRES IN OFFICE AND PLANT BUILDINGS (continued)

Mineral fibre l. ⁻¹	Building number	Asbestos source type and location	Number of samples	Asbestos fibre l. ⁻¹	Organic fibre l. ⁻¹ (Number of samples)	Mineral fibre l. ⁻¹
0.1 (3)	21	Amosite; fire-proofing on steel members of building	5	1 (1) 0 (4)	5 (2) 2.4 (3)	2.3 (3) 0.1 (2)
2 (3)	22	Amosite; plant insulation, after removal	27	1 (1) 0 (26)	59.63 (2) 10.20 (3)	13 (1) 5.8 (5)
0.1 (5)					5.9 (12) 1.4 (10)	2.4 (5) 0.1 (16)

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