ALB/MF
To : Mr. S.F. Heron (4)

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The Detection and Estimation of Asbestos as a Contaminant

Introduction

Due to the widescale usage of asbestos and the association of the minerals with lung diseases it is becoming necessary to develop methods for detecting the presence of minute amounts of asbestos minerals in the presence of large amounts of other materials.

Asbestos is a name given to several silicate minerals and serves only to define a fibrous form of the mineral. Due to the confusion which exists concerning the precise nature of the health hazard it is considered that methods must be developed which will allow the specific identities of the various minerals to be determined and not to rely solely upon the detection of a fibrous shape. Due to the crystalline nature of the minerals, and in general the mineral names can be considered to be crystallographic definitions of the mineral, it is possible in principle to use the characteristic x-ray and electron diffraction patterns to determine the precise identity of a mineral. Further if a specimen can be presented in a suitable form so that an x-ray diffraction pattern can be obtained it is possible by measuring the intensities of the diffraction lines to obtain quantitative estimates of the relative proportions of all the crystalline components.

The problem of collecting and preparing specimens for analysis is greatly simplified when the contaminant is present in large quantities compared to other materials, but becomes extremely difficult when the levels of contamination are low compared with other pollutants such as would be found in a normal urban atmosphere.

The purpose of the work described here was to investigate methods of collecting samples and to obtain a rough estimate of the amounts of asbestos contaminants. The analysis techniques are limited for the present to qualitative x-ray powder patterns and "semi quantitative" electron diffraction microscopy.

1. The urban atmosphere.

Sampling.

Samples were collected by filtration of volumes of air with "Sartorius" membrane filters having a nominal pore size of 0.1u. Air was drawn through a 25 mm filter by means of a rotary oil filled vacuum pump and metred through a water filled gas meter attached to the pump exhaust.

A sampling point was chosen situated on the S.E. corner of a building about 300 yds N.E. of the main T.B.A. factory buildings. The sampling head was positioned about 30 ft. above ground level and was fixed in a position approximately nine inches from the wall. The filter head was held with the filtering face downwards and protected by an inverted filter funnel. No account was taken of weather conditions or wind directions though the normal prevailing wind is S.W. and the samples were collected over a period in winter.

Using the system described above, it was possible to obtain sampling rates of 1 litre per minute until the filter became blocked after about 10,000 litre had been passed. Using pumps other than a vacuum pump, blockage of the filter occured after passing of one or two hundred litres, particularly if the air was wet.

Figure 1 is an electron micrograph of a typical grid area obtained from a specimen after passage of 1000 litre of air. A typical particle counting result was:-

The deposits from 1000 litre deposited onto 21 mm filter

2.83 mm diameter taken from 21 mm diameter

Hence reduction of 115 x

1 - 4 "fibres" observed/grid (entire

i.e. 4 x 115 "fibres"/1000 litre

= 460 "fibres"/cu metre.

The fibres observed were not all definable as chrysotile or any other asbestos form as not all the fibres gave a usable diffraction pattern, possibly due to screening by organic material, or the result of decomposition in the atmosphere besides which the fibres may not have derived from asbestos. Considerable care has been taken to remove the possibility of decomposition of the specimen in the electron microscope by the effect of the electron beam.

Many fibrous shapes were observed but only those which appeared to resemble asbestos were counted. The size of the fibres observed was of the order of 1 u long and a fibril or two fibrils in width.

Thus:

1u x 300A° containing a mass of approximately 2×10^{-15}g

for 460 "fibres" = 10^{-12}g/cu metre.

2. Beer.

Samples of beer were examined by centrifuging any suspended material onto carbon films supported on the normal electron microscope grids. Specimens were examined without any ashing treatment to remove organic material, despite the disadvantages caused by this form of specimen it was considered preferable due to the uncertainty of the nature of the specimen and its behaviour when heated. There are also problems of handling the minute amounts of residue and the unpredictable levels of contamination which could occur when a specimen is left exposed to the atmosphere of a laboratory normally handling asbestos minerals.

The method which was finally adopted was as follows:-
After shaking the container at least two separate 15 ml samples were centrifuged for one hour in tubes which had previously been shown to be free from contamination by centrifuging deionised water and examining the residue. The centrifuged beer was decanted from the residue which was then washed by resuspending and centrifuging successively with acetone and deionised water. The washed residue was finally resuspended in a few drops of acetone and transferred to a cleaned tapered centrifuge tube at the bottom of which was placed an uncontaminated carbon coated electron microscope grid, carbon side uppermost. Further centrifuging deposited the residue onto the grid which was then examined in the electron microscope. With beers that contained large amounts of sediment, instead of centrifuging all the residue onto grids, the residue was resuspended into 1 ml of water and 1 drop (1/10 ml) of the solution was allowed to dry onto the carbon coated grid.

At all times considerable care was taken to exclude contamination and several blanck reference specimens were examined.

As with the atmospheric specimens, counts were attempted for

3. Brake lining dusts.

Attempts to make x-ray diffraction observations with dusts produced under normal and experimental wear conditions have demonstrated that similar problems of detection exist for these materials as for the beer and atmospheric dust samples. Examination in the electron microscope again proved to be the most sensitive detection technique, though as with the other exercises accurate quantitation was difficult if not impossible.

A method for extracting the chrysotile component of a dust has been used and found to be satisfactory though further work will be necessary to make the technique usable as an accurate quantitative method.

Method.

An amount of dust was added to a few mls of deionised water and suspended in a clean tube in an ultra sound field for about 15 mins. After removal from the bath the suspended dust was allowed to settle for a few mins. e.g. until the water was cloudy rather than black. It was assumed that any chrysotile would be dispersed by the ultra sound, large agglomerates of non asbestos material would be broken down, releasing any fibres enclosed by the agglomerates. The chrysotile would be more stable in suspension than most of the brake lining dust thus allowing a large part of the chrysotile to be separated upon standing.

It has been found that by using this method x-ray diffraction lines could be identified from the dried suspension when nothing was apparent from the original material. Electron microscope observations are generally improved as a large part of the dust has been removed allowing clear observations of the chrysotile. It is important to note though that the effect of the ultra sound will be to totally alter the original particle size distribution so the method can only be applied to exercises for mass determination.

Calibration of fibre density on electron micrographs.

In all the exercises attempted it has only been possible to obtain very approximate estimates of amounts of asbestos seen on electron microscope specimens and in general the method is little better than a detection technique. In order to rectify this inadequacy known amounts of chrysotile have been prepared on membrane filters this allowing a series of pictures and counts to be obtained which can be related to an order of magnitude of mass in micrograms.

Method

A known weight of finely divided, high surface area chrysotile was suspended using ultra sound in 1 litre of deionised water. 100 ml of the suspension was made up to 1000 ml, thus giving a dilution of X10. This operation was repeated several times until the required small mass of chrysotile was obtained in a 100 ml portion. After filtration of the diluted portion through a 25 mm. an electron microscope specimen was produced using the method described in 1. Thus by producing a series of specimens from differing diluted aliquots it was possible to obtain electron micrographs which could be related to a known mass of chrysotile on a 25 mm filter. Also, by knowing the area of filter taken to make the specimen mass figures could be determined for extremely small amounts.

By carrying out a similar exercise with specimens to which carbon black has been added, it is hoped to be able to demonstrate the reliability of the methods adopted for preparing specimens for observation from samples collected from the atmosphere.

Though by no means is this method ideal for quantitative estimations it is hoped it will be capable of giving meaningful figures until a more satisfactory approach can be taken.