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DETERMINATION OF CHRYSOTILE IN AIRBORNE ASBESTOS BY AN INFRA-RED SPECTROMETRIC TECHNIQUE

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Abstract—A new method of determining chrysotile in airborne asbestos using infra red absorption at 2·72 μm is described. The technique is rapid, simple and allows detection of chrysotile down to about 20 μg in the sample, provided that the serpentine minerals of related structure (e.g. Kaolinite) are absent.

INTRODUCTION

INCREASING attention is now being paid to the health hazards arising from the inhalation of airborne asbestos (H.M. FACTORY INSPECTORATE, 1968), and in consequence of this, improved methods for the determination of chrysotile and other asbestos minerals in airborne dusts are of wide interest. Recently GOODHEAD and MARTINDALE (1968) have reported an X-ray diffraction technique which possesses a number of advantages, but which needs about 30 mg of sample. In this note we describe an i.r. spectrometric method used in this Laboratory for the determination of chrysotile. The technique is rapid, simple and allows detection of chrysotile down to about 20 μg in the sample. The method suffers from the disadvantage of being non-specific within the serpentine class of minerals. In the absence of other serpentine minerals e.g. kaolinite, as was usually the case, the method is specific for chrysotile asbestos, the form of asbestos most used commercially.

EXPERIMENTAL TECHNIQUE

Chrysotile in common with other serpentine minerals possesses a sharp i.r. absorption band at 2·72 μm which is associated with the stretching vibration of the lattice hydroxyl groups present in the layer silicate structure. The band is fairly well separated from the broad absorption arising from the free or weakly adsorbed water molecules present in most solid samples (see FIG. 1). The analogous bands in the amphibole asbestos minerals (amosite, crocidolite, etc.) are considerably weaker and are not suitable as the basis for an analytical technique.

In this work potassium bromide discs containing known amounts of chrysotile in the range 0·04–0·65 mg were prepared by the standard procedure (see, for example, MARTIN, 1966). Values of extinction $\log_{10} I_0/I$, where I_0 and I are the incident and emergent intensities of radiation respectively, measured at the peak of the 2·72 μm band, were determined from spectra recorded on a Grubb–Parsons "Spectromaster" i.r. spectrometer, and were found to be proportional to the mass of chrysotile present in the disc (FIG. 2). The sensitivity of the method is such that at the lowest levels of chrysotile content (< 100 μg) difficulties arose in preparing standard discs containing such small amounts of material; however, the calibration graph is sufficiently linear to allow a reasonable extrapolation, and indicates a lower limit of detection of $\sim 20\mu$g.

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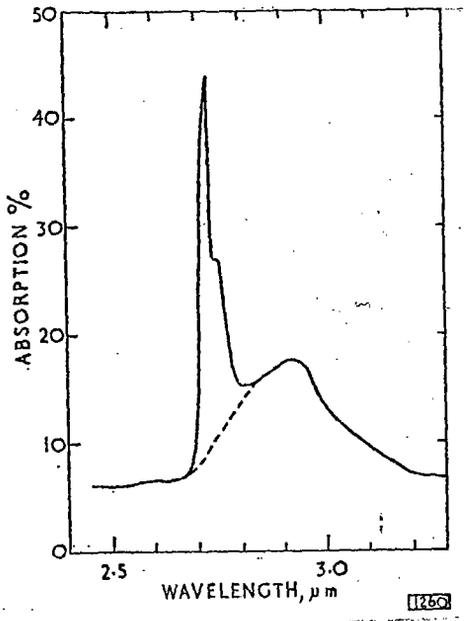

FIG. 1. Infra-red spectrum of chrysotile between 2·5–3·2 μm.

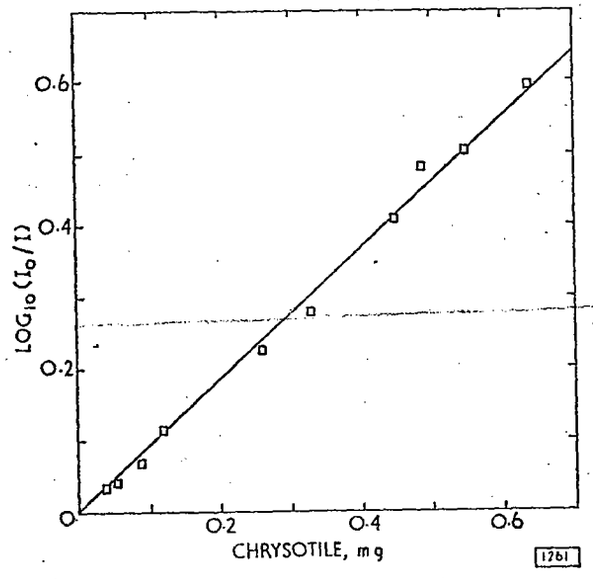

FIG. 2. Calibration graph for chrysotile determination.

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All samples to be analysed were received as deposits on 4·7 cm dia. "Millipore" filters. These were ashed at 450°C for ~ 1 hr, during which process the material of the filter and any carbonaceous matter present in the sample (which may reduce the transmission of the final disc) were oxidized or volatilized. Under these conditions decomposition of chrysotile is barely detectable, and no correction to the calibration graph was considered necessary. In the most careful work, however, it would be preferable to construct the calibration graph from standard samples heated in the same way. The residue in the crucible was incorporated in a potassium bromide disc and the amount of chrysotile present determined from the infra-red spectrum with the aid of the data set out in FIG. 2. The use of a microdisc, where a small rectangular lamina of KBr is pressed at the centre of a cardboard former, was also examined. Since the material was concentrated into an area roughly the same size as the beam, a considerable increase in sensitivity resulted; unfortunately difficulties arose in preparing and calibrating such discs, and they were not adopted in routine analyses. Nevertheless, careful use of a microdisc offers the possibility of lowering the limit of satisfactory measurement to ~10 μg.

APPLICATION

This method has been used successfully for short-term sampling at workshop sites in this Laboratory.

A certain amount of field work has also been carried out using, as far as possible, the standard volumetric instrument employed in the National Survey of Air Pollution in the United Kingdom, with the substitution of Millipore paper for the normal Whatman No. 1 paper. Filter papers were changed every 2 or 3 days when operating for long periods. Short-term, high-volume sampling was also carried out at two sites—a car park with a foundation of chrysotile waste and a refuse tip used for dumping asbestos waste.

Where long period sampling was carried out, in no case was chrysotile found above the limit of detection of the technique. This meant that the airborne concentration was below a value of 0·05 μg m^{-3}. A few chrysotile fibres were found on the Millipore papers by examination under an electron microscope; the majority were around 0·3 μm in length.

During short-term sampling at the sites mentioned above, chrysotile was only found in measurable quantities on two occasions and in both instances the value was about 1 μg m^{-3}.

CONCLUSION

Amounts of chrysotile in the range 20–700 μg may be determined by this procedure. The method combines a low limit of detection with speed and simplicity, but is non-specific; other serpentine minerals of related structure (e.g. kaolinite) show very similar absorption bands at 2·7 μm and their presence can invalidate any chrysotile determination based on the infra-red absorption in this region. However, in this work, spectra of dust samples collected from 12 industrial and other environments (where asbestos was not expected) were examined, and it was found that absorption at 2·7 μm from kaolinite or other mineral matter was rare. Nevertheless, this technique must be used critically; its value is likely to be greatest in environments which are to be continuously monitored, when the extent of likely interference can be established by a

preliminary investigation (using perhaps X-ray diffraction techniques). If such interference is found to be negligible, subsequent routine measurement may be conducted with comparative ease by the technique described here; periodic tests for the presence of interfering material will, however, still be desirable. In all circumstances, however, the method gives a valuable upper limit to the amount of chrysotile present in a sample.

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d = particle diameter
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D = diameter of jet (hole in
M = molecular weight of gas
N = number of jets per impa
P = gas pressure
Q = gas volumetric flow rate
Re_j = Reynolds number of jet
T = gas temperature
V_j = velocity of gas in jet

Greek symbols

λ = mean free path of gas m
μ = gas viscosity
π = 3.14159
ρ_p = density of particle
ψ = inertial impaction param
ψ_{50} = inertial impaction param

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