

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

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(First received 1 June 1970 and in final form 10 August 1970)

Abstract—A new method of determining chrysotile in airborne asbestos using infra red absorption at $2.72 \mu\text{m}$ is described. The technique is rapid, simple and allows detection of chrysotile down to about $20 \mu\text{g}$ 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 GOODHARD 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 $20 \mu\text{g}$ 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 $2.72 \mu\text{m}$ 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 $2.72 \mu\text{m}$ 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 ($< 100 \mu\text{g}$) 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 $\sim 20 \mu\text{g}$.

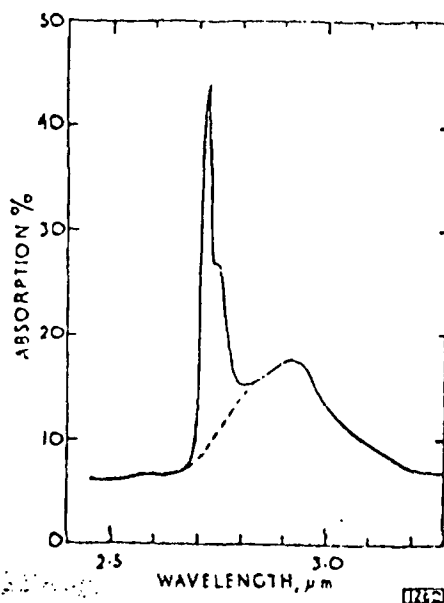
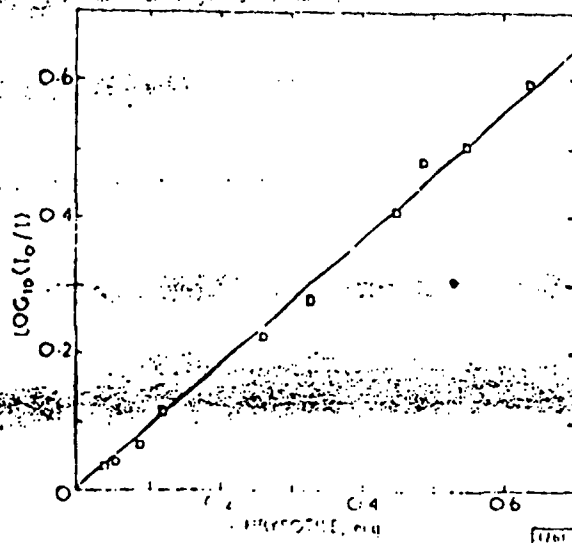
FIG. 1. Infra-red spectrum of chrysotile between 2.5-3.2 μm .

FIG. 2. Calibration graph for chrysotile determination.

All samples to be analysed filters. These were ashed at 450°C and any carbonaceous residue removed. The transmission of the final disc after decomposition of chrysotile is graph was considered necessary: preferable to construct the curve in the same way. The residue in the filter and the amount of chrysotile present was paid of the data set out in Figure 1. A lamina of KBr is pressed at 1000 psi. Since the material was concentrated, a considerable increase in sensitivity was obtained by comparing and calibrating such curves. Nevertheless, careful use of a satisfactory measurement to

This method has been used in this Laboratory.

A certain amount of field work has been done with the standard volumetric instrument in the United Kingdom, with Whatman No. 1 paper. Filter for long periods. Short-term, high efficiency, has been used in a car park with a foundation of asbestos waste.

Where long period sampling the limit of detection of the technique was below a value of 0.05 $\mu\text{g m}^{-2}$ papers by examination under a microscope in length.

During short-term sampling found in measurable quantities about 1 $\mu\text{g m}^{-2}$.

Amounts of chrysotile in the samples. The method combines a low limit of detection; other serpentine minerals have similar absorption bands at 2.7 μm . Determination based on the infra-red spectra of dust samples collected from asbestos was not expected) were from kaolinite or other mineral used critically; its value is likely to be continuously monitored, when it

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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

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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.

REFERENCES

- GOODHEAD K. and MARTINDALE R. W. (1969) The determination of amosite and chrysotile in airborne dusts by an X-ray diffraction method. *Analyst* 94, 985-988.
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SOURCE TEST

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Department of Civil Engineer

(First received 8 Decem

Abstract—The source test cascade is a method of determining the distribution of particles in stacks and ducts. It is inserted inside the duct or stack to measure the distribution of particles emitted from a K cell. A fluidized bed sewage sludge incinerator was used as a source.

Nomenclature

- C = Cunningham correction factor
 d = particle diameter
 d_{50} = particle diameter collected with 50% efficiency
 D = diameter of jet (hole in impactor)
 M = molecular weight of gas
 N = number of jets per impactor stage
 P = gas pressure
 Q = gas volumetric flow rate through impactor
 Re_j = Reynolds number of jet
 T = gas temperature
 V_j = velocity of gas in jet

Greek symbols

- λ = mean free path of gas molecules
 μ = gas viscosity
 ν = 3.14159
 ρ_p = density of particle
 δ = inertial impaction parameter
 δ_{50} = inertial impaction parameter at 50% efficiency

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THE SIZE distributions of aerosol particles are of interest to:

- (1) design new particulate air pollution control equipment
- (2) evaluate the performance of existing equipment
- (3) characterize the aerosol emission sources

Unfortunately a satisfactory technique for measuring emissions from emission sources has not been available. The size measuring systems which include isokinetic sampling, losses and vapor condensation, are expensive and difficult to use. They can be inserted inside a duct or stack to measure the distribution of particles. By operating the impactor cascade with a minimum of wall losses,

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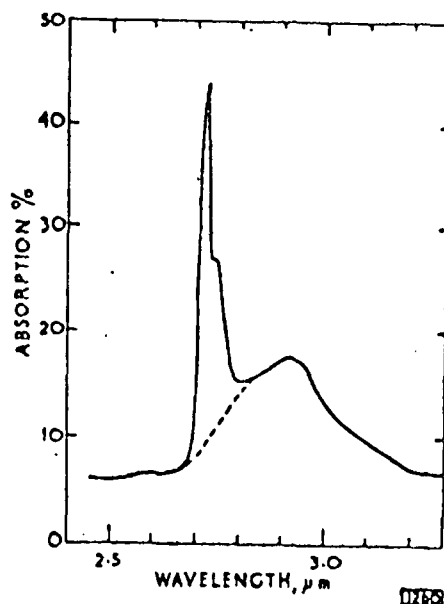
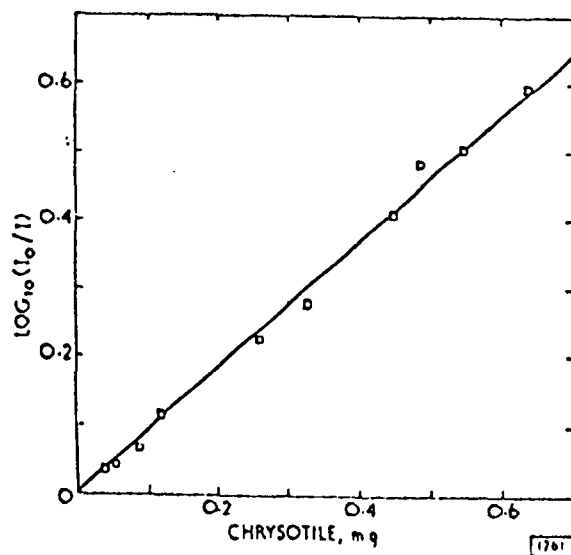
FIG. 1. Infra-red spectrum of chrysotile between 2.5-3.2 μm .

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Where long period samples are taken, the limit of detection of the method was below a value of 0.05 μg per square metre of paper by examination under a microscope.

During short-term sampling, the limit of detection found in measurable quantities was about 1 $\mu\text{g m}^{-2}$.

Amounts of chrysotile in dust samples. The method combines a highly specific method for the determination of chrysotile. The determination based on the spectra of dust samples of asbestos was not expected, from kaolinite or other minerals. The method is used critically; its value is continuously monitored, and

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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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SOURCE TEST

MICHAEL J. PILAT,

Department of Civil Engineering

(First received 8 Dec 1969)

Abstract—The source test cascade distribution of particles in stacks and inserted inside the duct or stack problems. The new source test cascade distribution of particles emitted from a fluidized bed sewage sludge incinerator cell.

Nomenclature

- C = Cunningham correction factor
 d = particle diameter
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Greek symbols

- λ = mean free path of gas molecule
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 π = 3.14159
 ρ_p = density of particle
 ψ = inertial impaction parameter
 ψ_{50} = inertial impaction parameter

THE SIZE distributions of aerosols are:

- (1) design new particulate air
- (2) evaluate the performance of
- (3) characterize the aerosol

Unfortunately a satisfactory size measuring system includes losses and vapor condensation cost and ability to determine the amount of material that can be inserted inside a duct or stack. By operating the impactor at a minimum of 100 ft/min, the efficiency is achieved with a minimum of 100 ft/min.

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DISCUSSIONS

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

WE AGREE with the authors' conclusion that the technique would be valuable mostly in monitoring air for asbestos where possible interferences would be negligible. Also we agree that the detectability of the method makes it applicable primarily to point source emission areas. Ambient air concentrations of asbestos generally range from 0.001 to $1 \mu\text{g m}^{-3}$.

We feel it would be useful to comment on possible use of the silicate absorption in the 9–10 μm , wavelength region which may be much stronger than the OH absorption at 2.72 μm and thus may provide for better measurements at low concentrations of chrysotile.

It would be useful if the known or possible interferences were listed. Finally we feel that with a longer collection period and with a separation of interfering materials, this technique might have a wider application and possibly be applicable to the low concentrations found in ambient air.

Also useful could be comments of the extent to which the method differentiates chrysotile from its thermally degraded form, Forsterite, and from other silica particulates found in air such as quartz, talc, and mica.

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AUTHOR'S REPLY

WE SHOULD like to make the following reply to the points which Mr. HEFFELINGER has raised:

We consider the silicate absorption at 9–10 μm to be unsuitable for the purposes of this analysis because

- (i) The absorption occurs in the infra-red spectra of most dust samples and is hence very unspecific
- (ii) the band is broad and ill-defined
- and (iii) is frequently overlapped by other broad and ill-defined absorptions.

If the use of the method becomes widespread a list of interfering substances would be useful, but it would be preferable to wait until further experience has revealed the commonest sources of interference. In the meantime, workers may find it useful to remember that interfering bands are given by minerals which are related to chrysotile (e.g. those of the serpentine family).

We believe, but have not checked experimentally, that the ashing technique employed here would free forsterite and quartz from adsorbed water molecules and since these minerals possess no structural OH groups, chrysotile is readily distinguishable from them. However adsorbed, i.e. lattice, hydroxyl groups are not easily eliminated in amorphous silicas and do give rise to absorption bands at $\sim 2.7 \mu\text{m}$.

Talc and mica are likely to interfere and their absence should be confirmed before regarding the method as being suitable. Finally, the length of the collecting period is limited by the amount of opaque material which can be tolerated in the final potassium bromide disc.

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* J. A. GADSDEN, J. PARKER and W. L. SMITH, *Atmospheric Environment* 4, 667–670 (1970).