

INTERNAL REFLECTION SPECTROSCOPY APPLIED TO AIR POLLUTION

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Abstract--Five internationally standardized samples of types of asbestos were identified by multiple internal reflectance spectroscopy in the infra-red region between 650 cm^{-1} and 4000 cm^{-1} . Previously, identification of asbestos types has required personal visual recognition through light microscopy, electron microscopy, or electron microprobe techniques.

The type of samples used in this investigation were undiluted, uncontaminated masses of large sized asbestos fibres less than $200\text{ }\mu\text{m}$ in length, similar to refined production material. The ability to identify this material in bulk, readily, can be expected to be useful when relating atmospheric asbestos fibre samples to specific sources and types.

The lower limits of resolution as to mass, fiber length, and dilution with other air pollutants will next be determined, along with methods of atmospheric samples preparation, such as a slow ashing method.

1. INTRODUCTION

SURFACE spoiling, including soiling, of materials by particulate and gaseous air pollutants is a recognized undesirable effect encountered almost daily in our environment. One type of particulate pollutant, asbestos, seems to have more than just a surface soiling effect. Asbestos fibers are generally characterized and identified by visual or electron microscopy methods requiring skilled visual observers.

A different class of instrumental technique that may be useful in identifying particulate pollutants and pollutant effects is known variously as multiple internal reflection spectroscopy and attenuated reflectance spectroscopy. Pollutants may be chemically identified using this technique by determining the infra-red absorption characteristics of the collected pollutant or its effects on some materials in the 4000 cm^{-1} – 650 cm^{-1} region of the spectrum.

2. EXPERIMENTAL

Internal reflection spectroscopy was used to characterize 5 standard reference samples of asbestos obtained from the International Union Against Cancer as described by TIMBRELL, GILSON and WEBSTER (1969). These sample consisted of Amosite, Anthophyllite, Crocidolite, Chrysotile "A", and Chrysotile "B". These samples were assembled and distributed by the Pneumoconiosis Research Unit of the International Union Against Cancer primarily for biological tests of the effects of asbestos in animal experiments. The asbestos samples are fibrous in nature and approximately $200\text{ }\mu\text{m}$ in length. The Amphibole samples (Amosite, Anthophyllite and Crocidolite) fibers are coated. The 5 standard reference samples of asbestos were all Grade IV on the Canadian scale or the nearest equivalent in order to have materials of relatively short fiber that together now account for 98 per cent of all commercially used asbestos.

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The asbestos samples were deposited onto both sides of a KRS-5 reflector plate approximately $50 \times 20 \times 2$ mm with a 45 degree angle face that yielded approximately 25 reflections. Approximately one third to one half of the KRS-5 reflector plate surface area was covered with the asbestos sample supported in a Wilks MIR-2 steel holder. Pressure was applied to achieve efficient intimate contact of the asbestos with the reflector plate.

Internal reflectance measurements were made by means of the Wilks Scientific Model 9 Single Beam Internal reflectance attachment, including a Wilks Model 2 sample holder, on a Perkins Elmer Model 137B. Infra-red Spectrophotometer with sodium chloride optics. A Wilks MIR-11 beam attenuator was used to attenuate the reference beam. The optical efficiency of the Wilks Model 9 attachment with no sample present is about 50 per cent after reflection losses.

Fine adjustments of the optical alignment were made without the reflectance unit to produce a recorder response of 98 per cent transmission at 2000 cm^{-1} . The multiple internal reflection unit was inserted and produced a recorder response of 37 percent transmission at 2000 cm^{-1} . The reference beam was then attenuated using a Wilks Model 11 beam attenuator such that the recorder pen response was 75 per cent transmission at 2000 cm^{-1} with no sample present.

The infra-red internal reflectance spectra obtained for the asbestos samples are shown in Figs. 1-5.

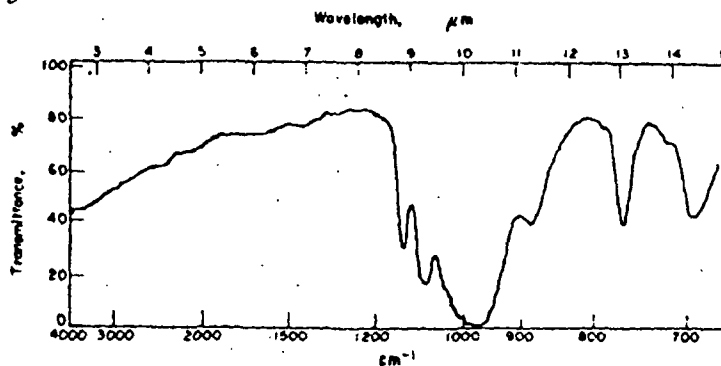


FIG. 1. Infra-red internal reflectance spectrum of Amosite.

3. DISCUSSION

No attempt was made to note band assignments since this part of the study has been directed only to detect instrumentally measured differences between the various UICC standard reference asbestos samples. BELLAMY (1964) has noted the region of transmission absorption area between 1100 cm^{-1} and 900 cm^{-1} for silicate compounds. LAUNER (1952) also shows a transmission spectrum of anthophyllite that was obtained from thin films of powder asbestos (fiber length of less than $2 \mu\text{m}$) deposited on a sodium chloride plate. Launer indicates that the amphibols have a band near 769 cm^{-1} which is characteristic of the double-chain silicates.

There is a strong similarity between the absorption spectrum obtained for anthophyllite by direct transmission measurements by Launer and that which was obtained

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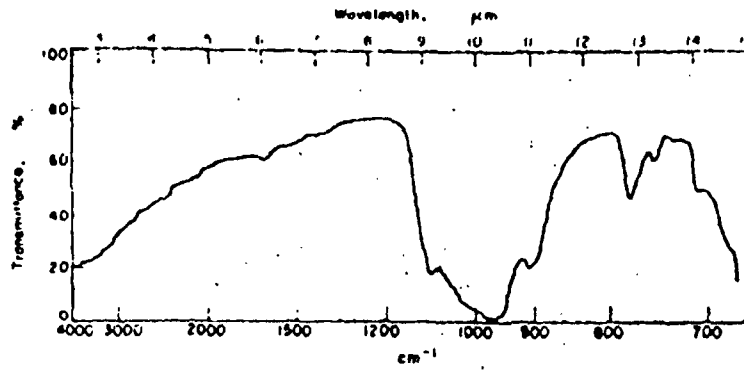


FIG. 2. Infra-red internal reflectance spectrum of Anthophyllite.

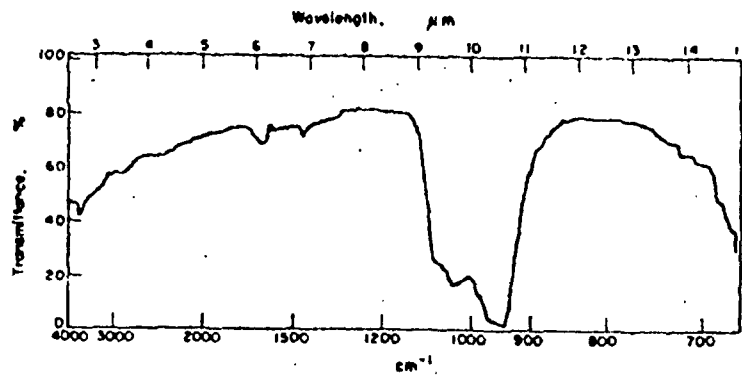


FIG. 3. Infra-red internal reflectance spectrum of Chrysotile "A".

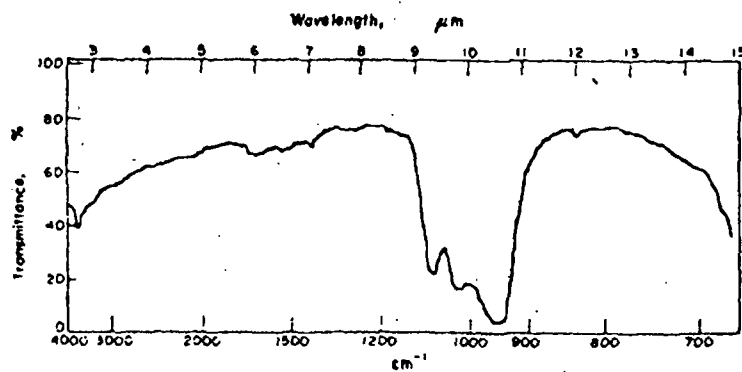


FIG. 4. Infra-red internal reflectance spectrum of Chrysotile "B".

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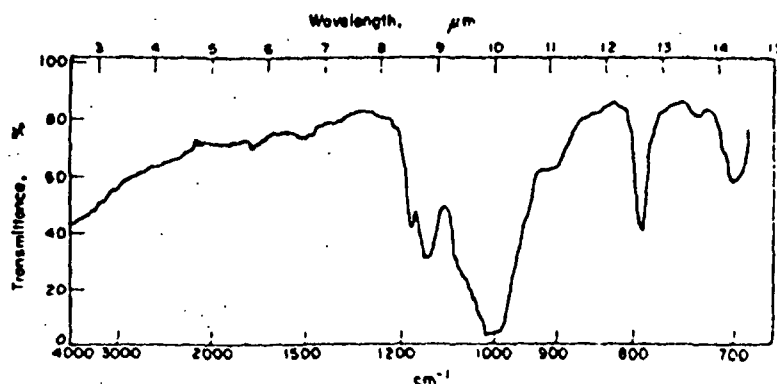


FIG. 5. Infra-red internal reflectance spectrum of Crocidolite.

in this investigation using internal reflectance spectroscopy on the UICC standard reference sample.

There appears to be a wavelength shift to the longer wavelength which may be expected as one changes from transmission to internal reflectance.

The transmission absorption peak at 1639 cm^{-1} has been associated by CORLENTZ (1906) in his early research on both water of crystallization and liquid water.

When the sources of air pollutants in a community's atmosphere are being sought, or when an undesirable effect of air pollutants is being investigated, it is important to be able to determine the probable sources and types of the pollutants. Various types of asbestos have been implicated in varying degrees in human diseases.

Heretofore, it was necessary to rely upon a highly skilled microscopist, visual or electronic, to distinguish between the major types of asbestos that are used in production at any one time.

This investigation attempted to use an instrumental technique that might lend itself to a totally numerical, and, therefore, computerized, identification system for a larger number of samples with less manpower and lesser manpower skills.

The continuing investigation is extending the technique to the characterization of air pollution soiled and otherwise spoiled surfaces and to extend the technique of asbestos identification to smaller quantities above and in the presence of other pollutants. Sample preparation techniques such as slow ashing will be investigated, together with spectrum signal peak amplification and masking.

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