SHIMADZU APPLICATION NEWS

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• SPECTROPHOTOMETRIC ANALYSIS

No.A264

Analysis of Contaminants on Paper by ATR Microspectroscopy

Analysis of microscopic foreign substances is regarded very important for quality control; the infrared microscope when applied to that purpose enables measurement easily and in a short time, being applied widely in different fields. Among the applications of the infrared microscope, the ATR microscopy doesn't require any sample pretreatment such as scratching off substance from the surface or thickness adjustment of the sample, and is free from such influence as interference infringes which are often observed in transmittance measurement or influence from reflection and absorption due to rough surface of foreign substances or the substrate observed in reflectance measurement, giving full play to the analysis of foreign substances deposited on the surface of the sample.

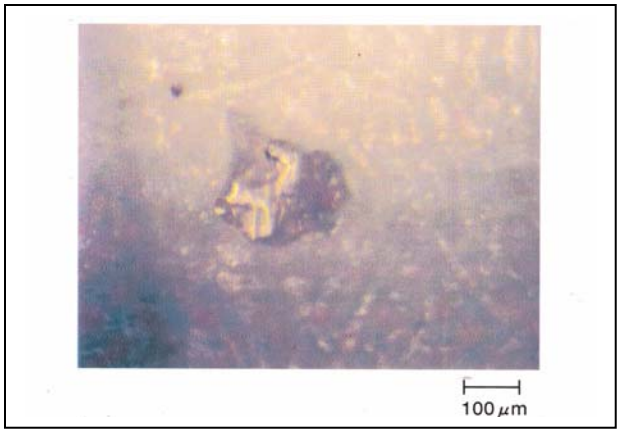
When performing the ATR microscopy, the sample is put on the stage of the microscope, and as the stage is raised, the foreign substance is brought into close contact with the prism. The condition of the contact is what greatly affects the quality of the data to be obtained. Consequently, in general, a somewhat hard foreign substance having a flat and soft substrate surface is easy to measure, but in the case of soft substrate or undulating surface, the measurement becomes difficult. In this article, examples of measurements of foreign substances on paper or filter, which belong to the category of difficult measurements, are introduced.

Measurement of Contaminants on Paper

ATR microspectroscopy is different from the transmission method and reflectance method in that when the sample is subjected to the measurement by the ATR microspectroscopy, a physical pressure is applied to the foreign substance which is the object of measurement. Here, if the foreign substance is softer than the foundation, the foreign substance comes into close contact with the prism, but if the foundation is soft, it will be imbedded in the foundation and in some cases, the prism comes into close contact even with the foundation. Spectrum obtained under this condition may include absorption by the foundation, which may induce true character of the foreign substance to be misjudged. Therefore, in order to measure contaminants on paper, it is necessary to measure the foundation first, and check if there is any influence of it on the result of the measurement of the contaminant. Shown in Figure 1 is a microgram of a contaminant imbedded in paper, and in Figure 2, an ATR spectrum of paper and a contaminant imbedded in paper. In this measurement, as the contaminant was sufficiently large and was easy to come into contact with the prism, a favorable spectrum free from the influence of paper was obtained. This spectrum shows an absorption of a second class amid substance, and the contaminant is assumed to be part of skin.

Table 1 Analytical Conditions

Resolution : 8cm^{-1}
Accumulation : 60
Detector : MCT Detector
Prism : Ge



Flg.1 Micrograph of a Contaminant Imbedded in Paper

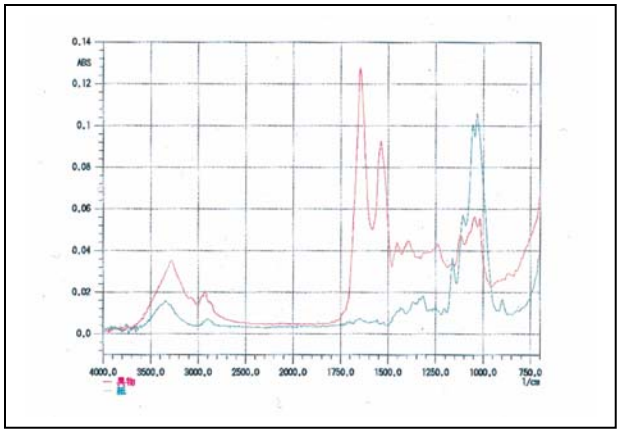


Fig.2 ATR Spectrum of Paper (green) - and a Contaminant in Paper (red)

Measurement of a Fiber on Filter

By the ATR microspectroscopy, measurement of part which comes into close contact with the prism is performed. Consequently, minimum measurement area (region of measurement) differs depending on the form of the prism and the sample to be measured. In the above measurement for which an ATR Cassegrain prism (slide-on type germanium prism) was used, it is known that in the case of measuring a plastic with flat surface, the minimum measurement area is approximately 20 - 30 ,U m. Consequently, when using a smaller substance or part than that, it is conceivable that absorption of other things than the target substance may be picked up, too.

But, if the target substrate is protruding from the surface of the foundation and is easy to come into close contact with the prism, even in case it is smaller than the minimum area mentioned, it may be measured without being affected by the foundation. Shown in Figure 3 is a micrograph of a fiber imbedded in film. It is longer than 100 μm but is no more than 10μm in width. Shown in Figure 4 is spectra of a fiber and a filter. It is comprehended that the spectrum of the filter, which is identified to be that of polyethylene telephthalate, is free from the influence of the foundation.

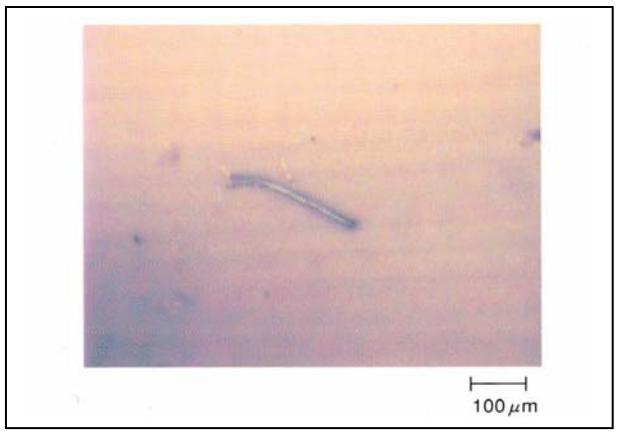


Fig.3 Micrograph of a Fiber Imbedded in Filter

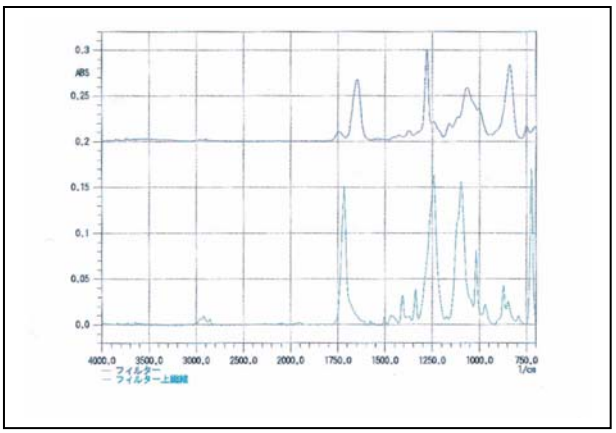


Fig.4 ATR Spectra of a Filter and a Fiber Imbedded in Filter

Measurement of a Stain

It is difficult to measure a substance permeated into paper without being influenced by the paper. Consequently, it is necessary to eliminate this influence by the calculation of difference spectrum; in the case the peameated substance is a liquid or a soft material in the form of powder, it may be attached on the prism when it comes into contact with it. In such a case, by measuring again the same substance with the sample separated from the prism, spectrum of only the stain can be obtained. Furthermore, by letting the stain come into contact again with a gold mirror, high- sensitivity measurement is possible. Shown in Figure 5 are an **ATR** spectrum of a plain paper (upper) and a spectrum of stained part (lower), and in Figure 6, an **ATR** spectrum of a stain on the prism (lower) and a spectrum which again came into contact with a gold-evaporated mirror (upper).

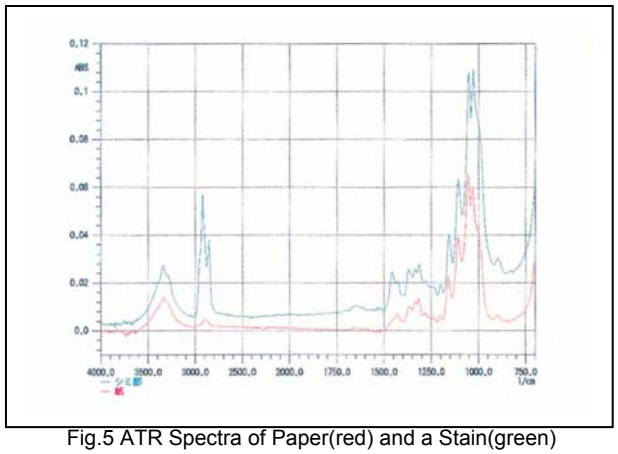


Fig.5 ATR Spectra of Paper(red) and a Stain(green)

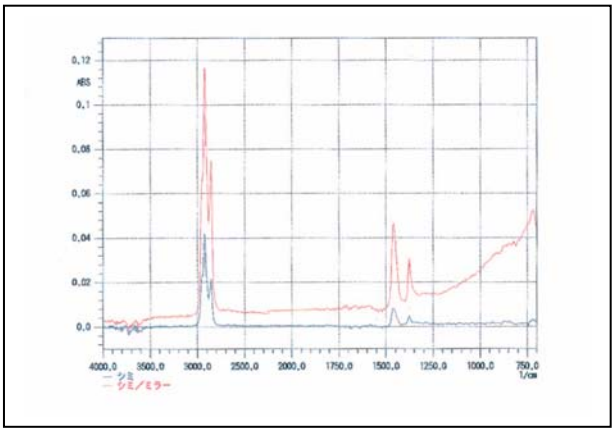


Fig.6 ATR Spectra of a Stain Imbedded in Prism