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Application News

Fourier Transform Infrared Spectrophotometer IRXross

High-Speed Mapping Measurement of Micro Sample Using IRXross™/AIM-9000

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User Benefits

- High-speed measurement is possible by using the IRXross FTIR and AIM-9000 infrared microscope.
- High-speed mapping measurement is also possible by using this system in combination with optional mapping software.
- Measurement throughput is greatly enhanced by shortening the measurement time.

■ Introduction

The IRXross Fourier transform infrared spectrophotometer (FTIR) released recently by Shimadzu Corporation offers the highest class sensitivity and resolution and can support high-speed scanning at a maximum of 20 spectra/second. Shimadzu original software IR Pilot™ for navigation of analysis work is also provided as a standard feature, realizing excellent operability together with outstanding performance.

Among these features, this article introduces an example of a micro sample mapping analysis, focusing on high-speed measurement using the IRXross in combination with an AIM-9000 infrared microscope.

Fig. 1 shows the appearance of the instruments. The IRXross uses the same stable moving mirror system as the IRTracer™-100 to realize high-speed measurement. In particular, measurement time can be substantially shortened by using this instrument in combination with the AIM-9000 infrared microscope equipped with a high-response speed MCT detector *.

* The MCT detector is a semiconductor type detector using mercury (Hg), cadmium (Cd), and tellurium (Te).

Fig. 1 Appearance of IRXross and AIM-9000

■ Sample Material

The sample used in this experiment was prepared by filtering microscopic contaminants contained in a solution with a polytetrafluoroethylene (PTFE) filter paper. If contaminant is small and is also white or transparent, determining the position of the contaminant on the filter paper can be extremely time-consuming. In such cases, area mapping measurement of a certain region that includes the position of the contaminant is effective.

Fig. 2 shows a visual image of the particle of contaminant measured in this experiment. The approximate position of the contaminant can be understood, but due to the irregular surfaces of the filter paper and contaminant, the visible light image is poorly focused at all positions and the boundary between the contaminant and the filter paper is unclear. Therefore, high-speed mapping measurement was carried out in a range of 250 µm (vertical) × 250 µm (horizontal) which included the position of the contaminant.

Fig. 2 Visual Observation Image of Contaminant on Filter Paper

■ Measurement Conditions

Fig. 3 shows the measurement range of 250 µm × 250 µm. Apertures of 25 µm × 25 µm were set at measurement position steps of 25 µm at each measurement position, making it possible to measure the selected range with no gaps. The yellow square indicates the background position. Table 1 shows the measurement conditions.

Since the PTFE filter paper does not display infrared absorption except in the vicinity of 1150 cm^{-1} to 1250 cm^{-1}, the transmission method was selected as the measurement method.

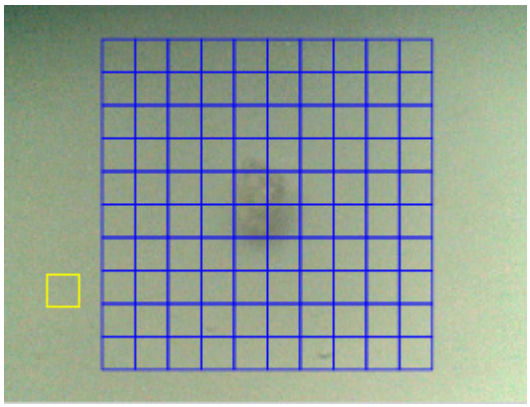

Fig. 3 Visual Observation Image After Setting Measurement Range

Table 1 Measurement Conditions

Instruments	: IRXross Fourier transform infrared spectrophotometer (KBr window) AIM-9000 infrared microscope
Resolution	: 16 cm^{-1}
Accumulation	: 1 time
Apodization function	: SqrTriangle
Detector	: MCT

■ High-Speed Mapping of Contaminant on Filter Paper

High-speed mapping (accumulation: 1) was carried out for 100 set measurement points, and a chemical image (see Fig. 4) of the infrared spectra obtained from all measurement points was created by using the peak areas in the entire region from 4000 cm^{-1} to 700 cm^{-1}. The measurement time for the total region was about 60 seconds. Since the location shown in red had a broad peak area, it can be understood that this is the position of the contaminant. Although the chemical image was prepared by using the peak area in this experiment, it is also possible to select "Intensity of specified position," "Intensity ratio of specified position," "Peak height," "Peak height ratio," "Peak area ratio," "Degree of similarity" as the calculation formula for the displayed results.

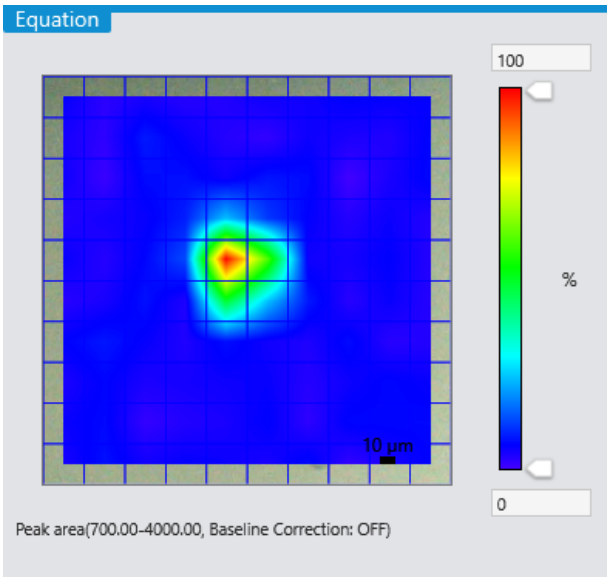

Fig. 4 Chemical Image of Contaminant on Filter Paper and Its Range

■ Identification of Contaminant

Since it was possible to determine the position of the contaminant accurately as described above, a high-quality infrared spectrum was measured by increasing the number of scans (accumulation number). Table 2 and Fig. 5 show the measurement conditions and the acquired infrared spectrum, respectively.

Table 2 Measurement Conditions

Instruments	:	IRXross Fourier transform infrared spectrophotometer (KBr window) AIM-9000 infrared microscope
Resolution	:	16 cm^{-1}
Accumulation	:	40 times
Apodization function	:	SqrTriangle
Detector	:	MCT

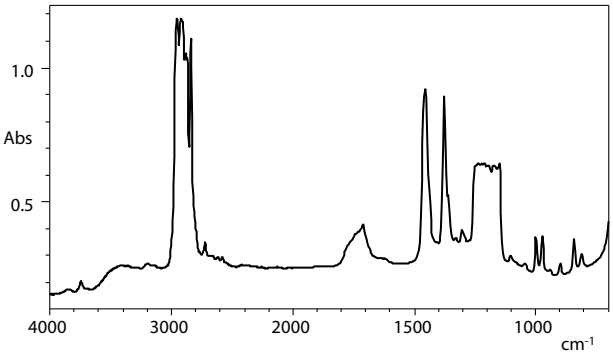

Fig. 5 Infrared Spectrum of Contaminant on Filter Paper

Fig. 6 shows the result of a library search of the acquired infrared spectrum.

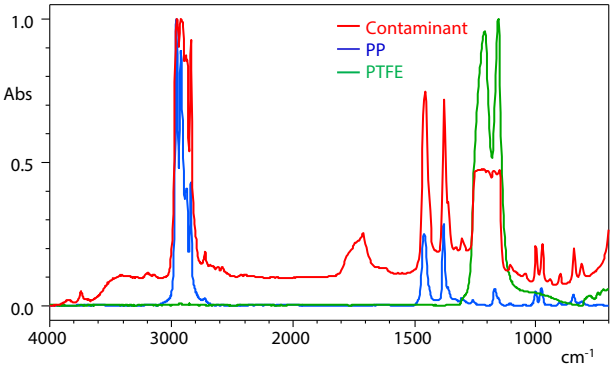

Fig. 6 Result of Search of Contaminant on Filter Paper

As a result of this search, the contaminant was identified as polypropylene (PP). In the comparison of the spectra of the contaminant and PP, it may be noted that the peak shapes are not in agreement in the range from around 1280 cm^{-1} to 1100 cm^{-1}. However, this is attributed to absorption of the PTFE of the filter paper (see the green line in Fig. 6).

■ Conclusion

High-speed mapping measurement of a micro sample was carried out using the IRXross FTIR and the AIM-9000 infrared microscope. Although the actual measurement time will vary depending on the aperture size and measurement position step to be set, and the number of scans, measurement throughput was greatly improved by using the IRXross, which is equipped with an interferometer that supports high-speed movement, making it possible to acquire data for 100 points in approximately 60 seconds.

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