

Application News

No. A563

Spectrophotometric Analysis

Analysis of Minute Objects Using a Sample Compartment Type Infrared Microscopy System

The sample compartment type infrared microscopy system is a combination of an FTIR instrument and the microspectroscopy accessory SurveyIR™ is a product manufactured by Czteck, LLC., which is designed to fit in the FTIR's sample compartment. The sample compartment type infrared microscopy system is capable of measurements on minute objects down to about 100 μm. Infrared spectra are detected using the FTIR's standard detector. SurveyIR is capable of transmission, reflectance, and ATR measurement (diamond or Ge prism), and an aperture can be selected from six sizes (2000, 250, 200, 160, 100, 60 μm). Therefore, measurement of minute areas is possible using SurveyIR if the sample is about 100 μm in size. In cases where the sample size is a few dozen μm, analysis using an infrared microscope equipped with an MCT detector is effective.

Fig. 1 shows an example system which combines Shimadzu's FTIR spectrophotometer IRSpirit™ (referred to as IRSpirit hereafter) with the SurveyIR.

IRSpirit features the highest signal to noise ratio and maximum resolution in its class. A highly compact FTIR, the instrument has a footprint smaller than an A3 sheet of paper with dimensions being a mere 390 (W) × 250 (D) × 210 (H) mm. In addition, the sample compartment of IRSpirit is easily accessible whether installed in "landscape" or "portrait" orientation, so that even a narrow opening on a lab bench can accommodate it. IRSpirit is also designed to accommodate transmission accessories such as a KBr pellet holder and demountable cells as well as existing Shimadzu and third party accessories such as single reflectance ATR attachments and diffuse reflectance attachments.

There are two models to IRSpirit which differ by the infrared detector that is used. One is the IRSpirit-T which is equipped with a DLATGS detector and the other is the IRSpirit-L which is equipped with a LiTaO₃ detector. The DLATGS detector is a high performance model featuring a temperature control function which can minimize the influence of ambient temperature changes on measurement results.

This article introduces example analyses of minute objects using a sample compartment type infrared microscopy system employing IRSpirit-T.

R. Fuji



Fig. 1 Sample Compartment Type Infrared Microscopy System (IRSpirit-T and SurveyIR)

■ Analysis of Fiber Using the ATR Method

The fiber shown in Fig.2 with a diameter of about 80 μm was measured using the ATR method. Visible light observation of the area for measurement is possible through the diamond crystal, allowing for accurate centering and measurement while observing the sample. Table 1 lists the measurement conditions.

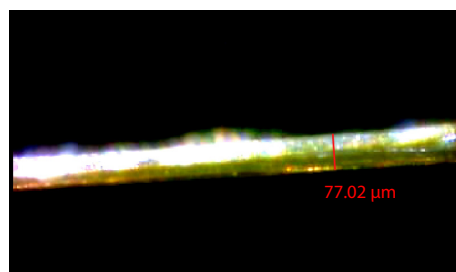


Fig. 2 Fiber

Table 1 Measurement Conditions

Instrument	: IRSpirit-T (KRS-5 window) SurveyIR
Resolution	: 8 cm ⁻¹
Accumulation Times	: 128
Apodization Function	: SqrTriangle
Aperture Size	: 100 μm
Detector	: DLATGS
ATR Prism	: Diamond

Fig. 3 shows the search result from the Shimadzu Standard Library based on the obtained infrared spectrum. The results indicate that the fiber is a material that is similar to polyester.

Use of SurveyIR facilitated the specification of the measurement position thanks to the clarity of the sample observation image. In addition, a favorable infrared spectrum was obtained because unlike the conventional single reflectance ATR attachment, an aperture size appropriate for the sample size can be selected.

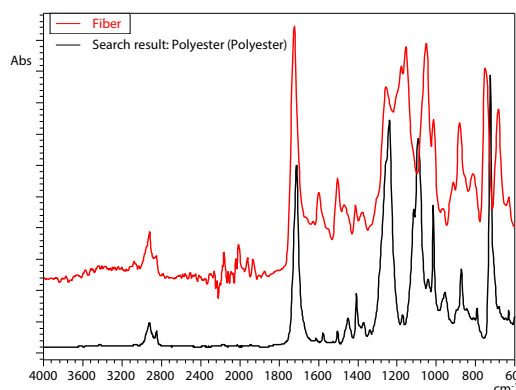


Fig. 3 Infrared Spectrum Obtained from Fiber and Search Result

■ Analysis of a White Minute Object Using the ATR Method

The white minute object shown in Fig. 4 with a length of about 130 μm was measured using the ATR method. Even in cases where minute objects for measurement are dispersed in multiple points, SurveyIR enables accurate centering of each point since the X, Y stage can be moved freely by manual operation. Also, samples with an uneven surface can be focused on fairly easily using the fine adjustment function of the Z axis.

Fig. 5 shows the obtained spectrum and the search result. Measurement conditions are the same as those listed in Table 1. The results indicate that the white minute object is calcium carbonate.



Fig. 4 White Minute Object

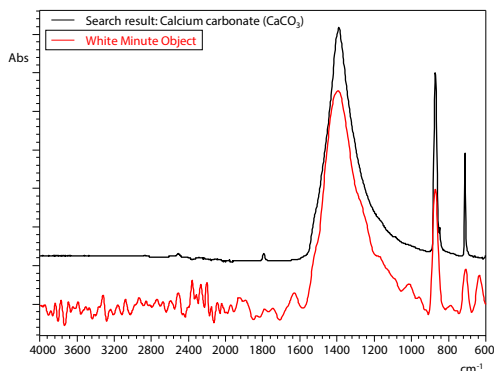


Fig. 5 Infrared Spectrum Obtained from White Minute Object and Search Result

■ Analysis of Transparent Film Using the Transmission Mode

The fairly large-sized transparent film shown in Fig. 6 was compressed in a diamond cell and measured using the transmission mode. Since the sample stage of SurveyIR can accommodate a diamond cell likewise general infrared microscopes, the process from pretreatment to measurement can be done smoothly. Fig. 7 shows the cell set on the sample stage.

LabSolutions™ IR is a control software for Shimadzu FTIR instruments that allows measurement of spectra while monitoring. Since both an image and the infrared spectra can be observed while adjusting the stage position, it is possible to find a measurement position that can yield the optimum peak intensity.

This measurement was done by setting the aperture size to 160 μm . Other conditions are the same as those listed in Table 1. Fig. 8 shows the obtained spectrum and the search result which indicate that the film material is polyethylene.

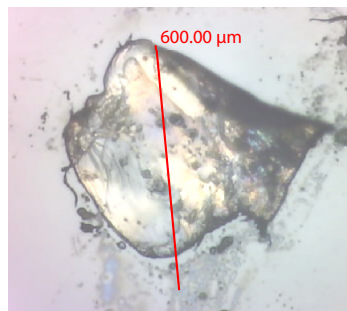


Fig. 6 Transparent Film

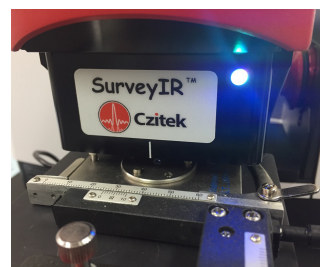


Fig. 7 Sample Stage with a Diamond Cell Set in Place

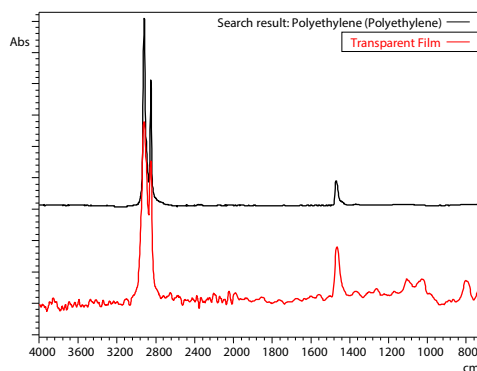


Fig. 8 Infrared Spectrum Obtained from Transparent Film and Search Result

■ Conclusion

Analysis of minute objects about 100 μm in size was done with good sensitivity using a sample compartment type infrared microscopy system. The system also has an outstanding observation function which allowed smooth acquisition of clear images using the fine adjustment function of the Z axis. The function also enabled measurement of sample dimensions. Switching between transmittance, reflectance, and ATR measurement is easy on the SurveyIR, making it possible to select the measurement method that is most appropriate for each sample. SurveyIR also features six aperture sizes which enable measurement according to the size of the sample.

In cases where the sample size is a few dozen μm , use of an infrared microscope equipped with an MCT detector is recommended.

SurveyIR is a product purchased from CziTek, LLC.

IRSpirit and LabSolutions are trademarks of Shimadzu Corporation. SurveyIR is a trademark of CziTek, LLC.