SHIMADZU
Excellence in Science

Fourier Transform Infrared Spectrophotometer IRSpirit™-X

Application News

Analysis of Minute Objects Using the SurveyIR Sample Compartment Type Infrared Microscopy System

Shoko Iwasaki

User Benefits

- The infrared microscope SurveyIR can be installed in the IRSpirit-X sample compartment to analyze minute objects in a compact space.
- It can easily measure minute objects of about 100 µm and store images of the measuring points.
- Because the standard detector of the FTIR instrument is used, measurement is possible even in environments where liquid nitrogen cannot be used.

■ Introduction

The IRSpirit™-X Series features the highest signal to noise ratio and maximum resolution in its class. The highly compact FTIR has a footprint smaller than an A3 sheet of paper, with dimensions of just 390 (W) × 250 (D) × 210 (H) mm. The sample compartment of the IRSpirit-X series is easily accessible whether installed in "landscape" or "portrait" orientation, so even a narrow space on a lab bench can accommodate it. It also has a compact main body and a large sample compartment that is designed to accommodate transmission accessories, such as a KBr pellet holder and demountable cells and existing Shimadzu and third-party accessories, such as single reflectance ATR attachments and diffuse reflectance attachments. The IRSpirit-X series includes TX and ZX models equipped with a DLATGS and LX models equipped with a $LiTaO_3$ as infrared detectors. The DLATGS detector is a high-performance model featuring a temperature control function that minimizes the influence of ambient temperature changes on measurement results.

This article introduces examples of analyses of minute objects using the sample compartment type infrared microscopy system SurveyIR and the IRSpirit-TX, the most sensitive of the IRSpirit series.

■ SurveyIR, Sample Compartment Type Infrared Microspectroscopy System

The SurveyIR is a simple microspectroscopy system that can be installed in the sample compartment of the FTIR instrument to measure minute objects. Infrared spectra are detected using the FTIR standard detector, and minute areas can be measured using the SurveyIR if the sample is about 100 µm in size.

The SurveyIR is capable of transmission, reflectance, and ATR measurement (diamonds or Ge crystals), and an aperture can be selected from six sizes (2000, 250, 200, 160, 100, and 60 µm). Even in cases where minute objects for measurement are dispersed in multiple points, it enables accurate centering of each point without moving the objects since the X, Y stage can be moved manually.

Fig. 1 shows a system that combines Shimadzu's FTIR spectrophotometer the IRSpirit-X series with the SurveyIR.

Fig. 1 Sample Compartment Type Infrared Microscopy System (IRSpirit™-TX/SurveyIR)

■ Analysis of Colorless Minute Objects (Transmission Method)

Colorless minute objects were sampled on a diamond cell and thinly compressed for transmission measurements. Fig. 2 shows a minute object after compression. Since the sample stage of the SurveyIR can accommodate a diamond cell like general infrared microscopes can, the process from pretreatment to measurement can be done smoothly. The minute object in Fig. 2 is about is about 200 × 100 µm, so the aperture size was set to φ160 µm. Other measurement conditions are shown in Table 1.

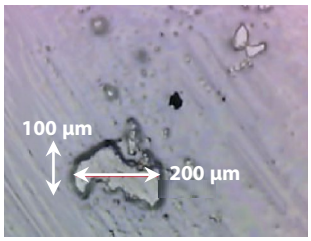


Fig. 2 Image of Minute Object after Compression

Table 1 Measurement Conditions

Instrument:	IRSpirit-TX, SurveyIR
Resolution:	8 cm^{-1}
Number of Scans:	128
Apodization Function:	SqrTriangle
Detector:	DLATGS

Transmission measurements were carried out on minute objects compressed on a diamond cell. Fig. 3 shows the infrared spectrum and search results obtained. The Shimadzu Standard Library showed that the minute object was a mixture of oil and sugar.

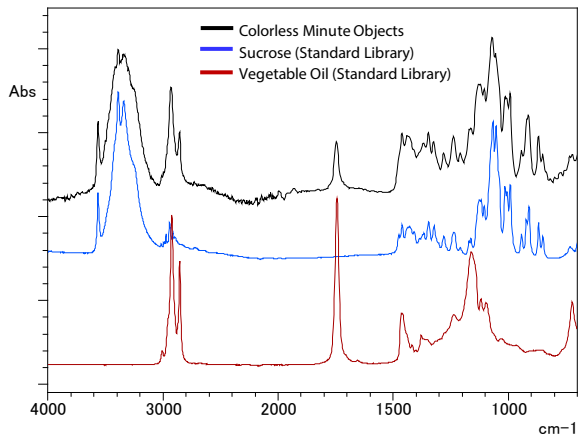


Fig. 3 Infrared Spectra and Search Results for Colorless Minute Objects

■ Analysis of Contaminant Fibers (Transmission Method)

Contaminant fibers were found and measured using the transmission method. The fibers were compressed in a diamond cell in the same way as the colorless minute objects mentioned above. Fig. 4 shows the fibers after compression. The short side was about 100 µm, while the long side was fibrous and long. Therefore, the aperture size was set to 160 µm. The other measurement conditions were the same as those in Table 1.

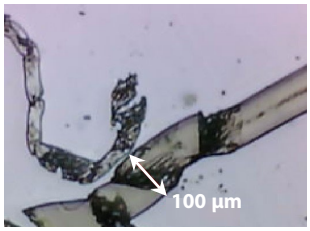
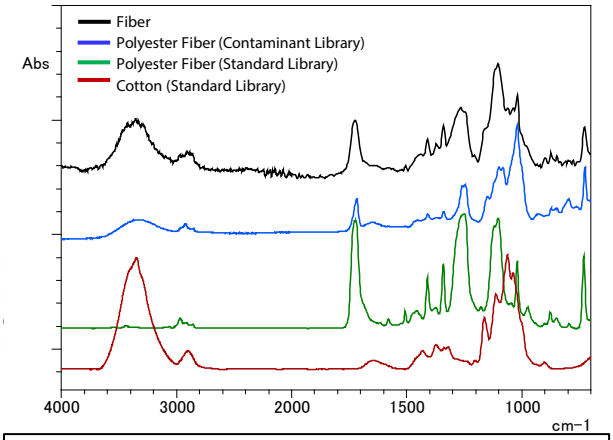


Fig. 4 Image of Fibers

Fig. 5 shows the spectrum and the search results obtained using the Standard Library and the original Contaminant Library (optional). Polyester and cotton were hit separately from the Standard Library, while the polyester fibers mixed with polyester and cellulose were hit from the Contaminant Library.



Infrared Spectrum Information for Polyester Fiber
Materials; Polyester Fiber, Cellulose Major Elements; Below 1 % Color; Purple
Shape; Fiber Hardness; Soft Metallic Luster; No Technique; ATR (Diamond)

Fig. 5 Results of Search of Infrared Spectrum and Detailed Information on Library Data for Hit (Using Contaminant Library)

While many single components are registered in the Standard Library, it should be noted that a close investigation of each individual component is necessary when foreign matter consists of multiple components, and a level of skill in spectrum analysis is required. However, unlike commercially available libraries that record only data for single components, the Contaminant Library, an original library created by Shimadzu, contains data on foreign substances that were actually captured (data provided by water supply utilities and food product companies) and mixtures, such as packing. Therefore, it provides dramatically improved search accuracy and also includes various types of supplementary information, such as the major elements and the color, shape, hardness, and metallic luster of contaminants.

■ Analysis of Colorless Minute Objects (ATR Method)

Fig. 6 shows a colorless minute object that is large, approximately 600 × 400 µm in size, and because it is black, despite being colorless , it was expected to be very thick. For this reason, it was difficult to adjust the thickness to make it suitable for the transmission method simply by compressed it on a diamond cell. Therefore, analysis was performed using the ATR method (diamond crystal) without any pretreatment.

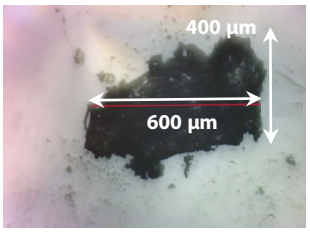


Fig. 6 Image of Colorless Minute Objects

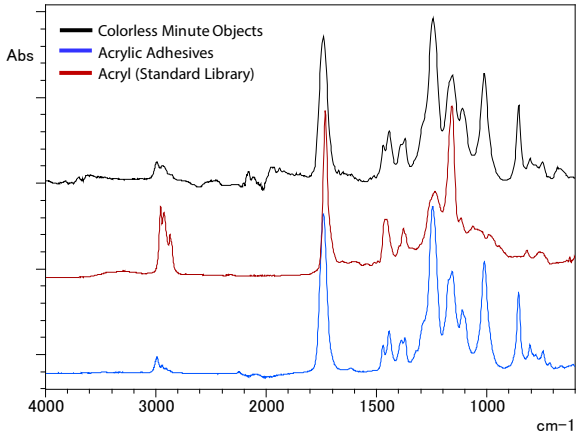


Fig. 7 Infrared Spectra and Search Results for Colorless Minute Objects

In the ATR method, the contact area between the ATR prism and the sample is important. So by setting an aperture larger than the contact area, it is possible to obtain a good spectrum because more light can be used. This time, the maximum aperture size was set to 2,000 µm for ATR measurement. (The size of the SurveyIR diamond ATR is φ3 mm, and it is slightly rounded, so it is a point contact for hard samples.) Fig. 7 shows the obtained infrared spectrum and the search results. The measurement conditions were the same as those listed in Table 1. The colorless minute object was found to be acrylic adhesive. If the object to be measured is a black rubber-like sample containing a large amount of carbon, the diamond prism may cause spectral distortion. So in that case, using the Ge crystal ATR is recommended.

■ Conclusion

Analysis of minute objects was done with good sensitivity using a sample compartment type infrared microspectroscopy system. In addition, by utilizing the original Contaminant Library for searching infrared spectra, it was possible to accurately analyze mixtures.

The SurveyIR also has an outstanding observation function that allows smooth acquisition of clear images using the fine adjustment function of the Z axis. This function also enables measurement of sample dimensions. Switching between transmittance, reflectance, and ATR measurement is easy with the SurveyIR, making it possible to select the measurement method that is most appropriate for each sample. Two types of crystals, diamond, and germanium (Ge), are provided in the ATR method. In the case of diamond, the sample can be attached to the crystal while confirming the sample through the crystal. In addition, the optional ATR proximity warning system is available to prevent crystal damage.

If the size of the object to be measured is several micrometers to several tens of micrometers, use the infrared microscope AIMsight™ or the Infrared/Raman Microscope AIRsight™ equipped with a T2SL detector cooled with liquid nitrogen.

01-00654-EN First Edition: Dec. 2023

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/