

Application News

No. A587

Spectrophotometric Analysis

Analysis of a Filter Paper Surface Contaminant with the GladiATR™ Vision Observation-Type ATR

ATR (attenuated total reflection) is a powerful type of reflectance method that enables measurement of minute or thick samples which are difficult to measure using the transmission method. The ATR method acquires an absorbance spectrum of a sample surface by measuring the total reflection of light from the sample surface. Characteristics of this method include simplicity compared to other surface analysis methods, absorbance intensity that depends on wavelength, and the ability to adjust the penetration depth of light into the sample by changing the incident angle and crystal refractive index.

Shimadzu offers an extensive variety of ATR accessories, including those that enable observation and others equipped with a pressure sensor. This article introduces an example of analyzing a contaminant using one of these ATR accessories, the GladiATR Vision (Fig. 1) manufactured by PIKE Technologies.

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Fig. 1 GladiATR Vision

■ GladiATR Vision Features

The GladiATR is an ATR accessory that features high throughput, a high clamping pressure, a wide measurement wavenumber range, and optional crystal plates that support heating to high temperatures.

In addition to the above, the GladiATR Vision also includes a function for observing minute samples. The diamond crystal has a monolithic structure that is difficult to damage by pressure.

Observation on the GladiATR Vision is performed through the diamond crystal. Locating the correct measurement position is easy since the position for measurement can be observed in real-time on the LCD screen. Observation is also possible through the diamond crystal for thick opaque samples.

The GladiATR Vision, as illustrated in Fig. 2, employs an innovative optical design that enables infrared measurement and visual observation to be performed simultaneously. The measurement position on the sample can be observed at 110 times magnification. The ability to optimally place and analyze samples at the center of the diamond crystal allows measurement at sizes down to 50 μm.

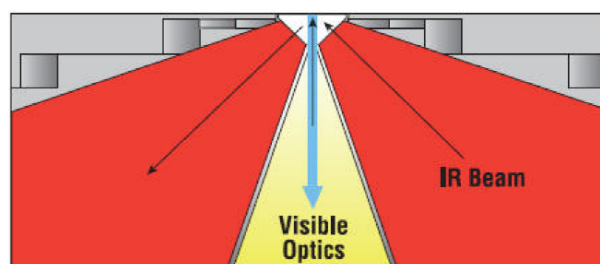


Fig. 2 Optical Method Employed by the GladiATR Vision

■ Analysis of a Filter Paper Surface Contaminant

The ATR method involves irradiating an infrared beam onto the sample from below, as illustrated in Fig. 3. This means that the actual measurement position may be incorrect if the position of the target contaminant cannot be determined from the other side such as on a piece of paper or resin.

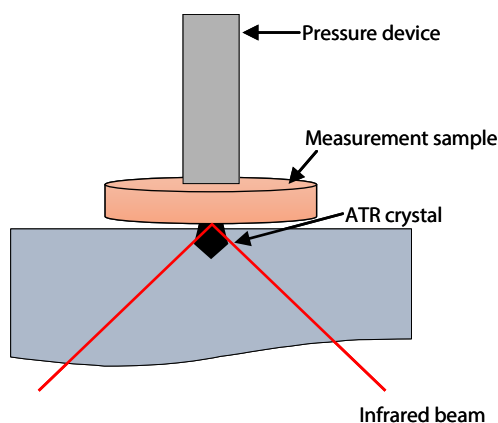
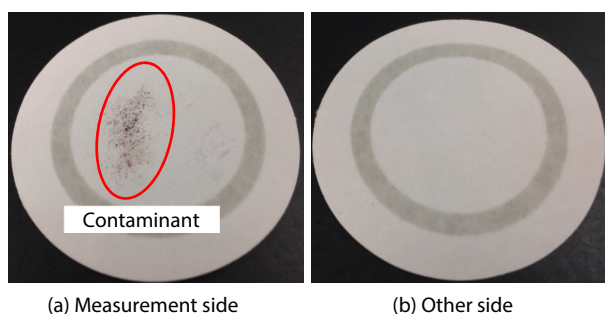


Fig. 3 ATR Method Principle

Fig. 4 (a) and (b) show the sample for measurement. Since the location of the contaminant cannot be confirmed from the other side, the observation-type ATR GladiATR Vision was used to perform measurement.



(a) Measurement side (b) Other side

Fig. 4 Optical Method Employed by the GladiATR Vision

Table 1 lists the measurement conditions, Fig. 5 shows the observation image, and Fig. 6 shows the measurement results. The filter paper sample was placed directly onto the GladiATR Vision and the contaminant on the surface of the filter paper was measured. Since influence from the filter paper component was expected, the other side without the contaminant was also measured for comparison.

Table 1 Measurement Conditions

Instrument	: IRTracer™-100, GladiATR Vision
Resolution	: 4 cm ⁻¹
Accumulation	: 40
Apodization function	: Sqr-Triangle
Detector	: DLATGS

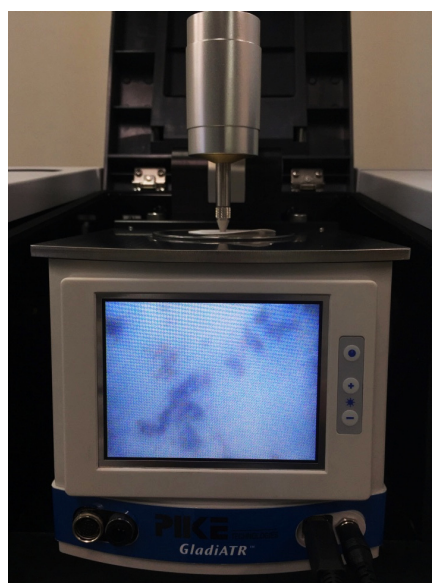


Fig. 5 Observation Image of Contaminant

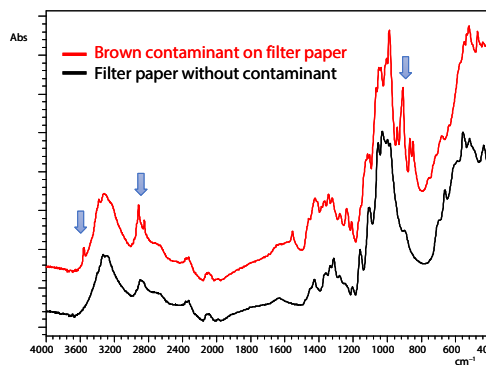


Fig. 6 Measurement Results

In Fig. 6, a difference in peaks in the vicinities of 3600 cm⁻¹, 2800 cm⁻¹, and 900 cm⁻¹ can be observed (blue arrows in the figure). By performing analysis using the library provided as standard on the FTIR, the contaminant appears to be sucrose, as shown in Fig. 7 (a) and (b). (The filter paper component is cellulose.)

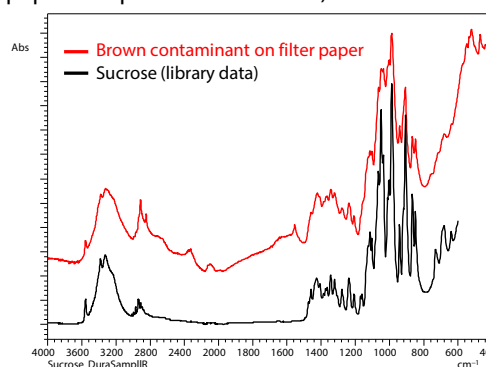


Fig. 7 (a) Analysis Result of Contaminant

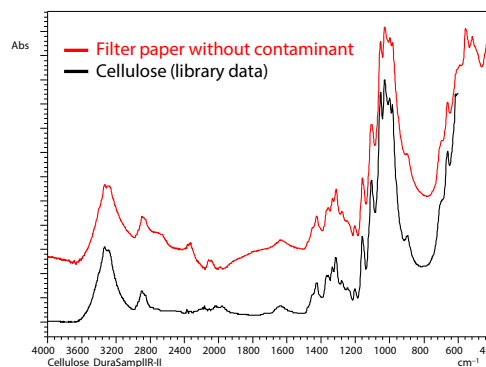


Fig. 7 (b) Analysis Result of Filter Paper

■ Conclusion

An observation-type ATR accessory is helpful for samples on which the position of a contaminant cannot be determined from the other side. By using the GladiATR Vision, fine adjustment of the sample position is made easy by checking the image on an LCD screen. The GladiATR Vision can be attached to the Shimadzu IRTracer-100 and IRAffinity™-1S.

IRTracer is a trademark of Shimadzu Corporation
GladiATR is a trademark of PIKE Technologies.

First Edition: Dec. 2018



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