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Application News

No. A557

Spectrophotometric Analysis

Introduction to the QATR™ 10 Single-Reflection ATR Accessory with a Diamond Crystal

The attenuated total reflectance (ATR) spectroscopy is widely used for various purposes such as chemical identification tests and contaminant analysis. This article introduces example analyses of resin, powder, and liquid samples using the QATR 10 single-reflection integration-type ATR accessory with a diamond crystal.

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Features of the QATR 10

The QATR 10 is a single-reflection ATR accessory with a diamond crystal and is designed especially for the IRTracer-100 and the IRAffinity-1 series. Fig. 1 shows the QATR 10 mounted in the IRTracer™-100. It is designed to fit perfectly in the sample compartment and also features smooth mounting and dismounting from the sample compartment since no screws are necessary. In addition, the QATR 10 is equipped with a chip so that it will be recognized by the FTIR upon initialization when mounted in the sample compartment. Automatic loading of specified parameters can also be set to take place simultaneously, enabling inexperienced operators to perform analyses using the appropriate conditions.

The crystal of the QATR 10 is a Type IIIa monolithic diamond (wide-band type) with a contact area diameter of 1.8 mm. The incident angle is 45 degrees and a wavenumber range from 4,000 to 400 cm^{-1} can be measured with good sensitivity. As options, there are also crystal materials of high-throughput diamond, zinc selenide (ZnSe), and germanium (Ge), which the user can easily exchange. The front cover is provided with a hole for passing through a purge tube to allow measurements while purging with nitrogen gas or dry air.

Fig. 1 QATR 10 Mounted in IRTracer-100

Example Analysis of a Resin Fragment

Resin and rubber are often measured by ATR spectroscopy. In this analysis, we measured the spectra of acrylonitrile butadiene styrene (ABS) and polyvinyl chloride (PVC) pellets using a diamond crystal. Measurement was done using the conditions listed in Table 1 with the pellet pressed on the crystal using the flat anvil as shown in Fig. 2. The measurement result of the ABS pellet is shown in Fig. 3. A clear peak of C≡N stretching vibrations is detected at about 2,237 cm^{-1}. Fig. 4 shows the measurement result of the PVC pellet with a peak of C-Cl stretching vibration at about 611 cm^{-1}. The peaks at about 1,420 cm^{-1} and 870 cm^{-1} (marked with a ★) are likely to be peaks originating from the calcium carbonate that is used in PVC as a filler.

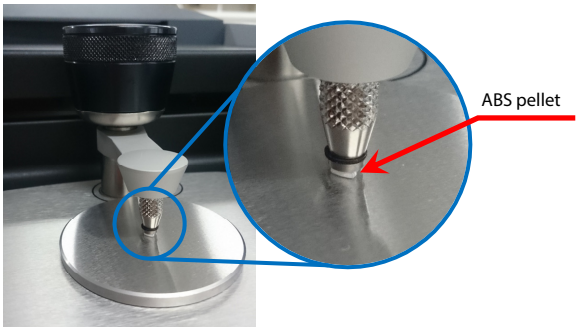

Fig. 2 Pellet Set on Instrument

Table 1 Measurement Conditions

Instruments	: IRTracer-100, QATR 10 (Diamond crystal)
Resolution	: 4 cm^{-1}
Accumulation	: 20
Apodization	: Happ-Genzel
Detector	: DLATGS

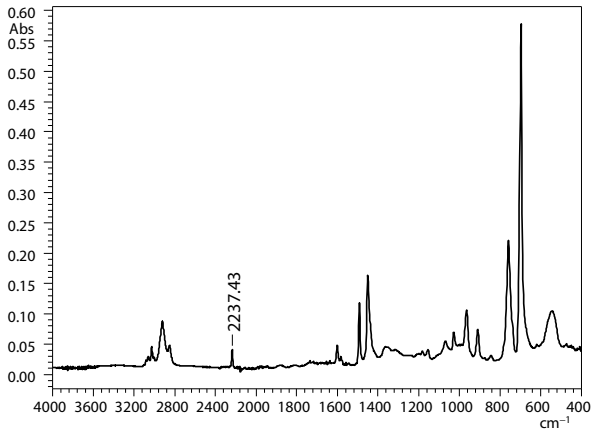

Fig. 3 ATR Spectrum of the ABS Pellet

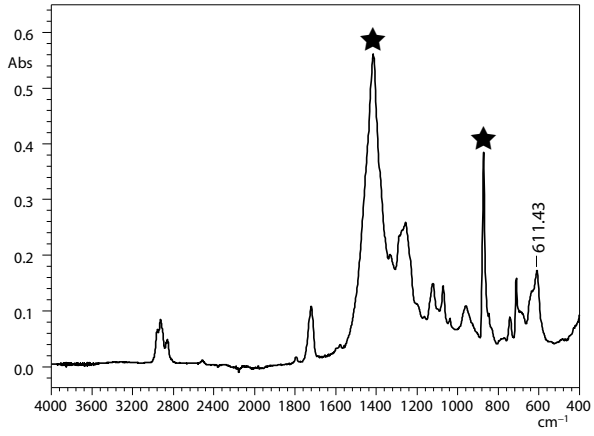

Fig. 4 ATR Spectrum of the PVC Pellet

Example Analysis of a Powder Sample

Samples in powder form are often analyzed using the KBr pellet method or the diffuse reflection spectroscopy. However, by using the ATR spectroscopy, samples can be measured easily as is without any need for pretreatment such as dilution. Since the contact between powders and the ATR crystal changes depending on how the sample is pressed against the crystal, spectra are measured while monitoring the peak intensity.

We measured talc using the conditions listed in Table 1. Talc is a powdered, selected, natural hydrated magnesium silicate that is used in a wide variety of fields for varying purposes such as a reinforcing filler for plastics and rubber, a mold release agent, a pharmaceutical excipient, and a cosmetics ingredient. Fig. 5 shows the measurement result of talc. The red line is the measured ATR spectrum and the black line is the spectrum after applying advanced ATR correction under the condition that the refractive index of talc is 1.54.[*1] By principle, when compared with transmission spectra, ATR spectra show small shifts along the vertical and horizontal axes. Particularly, peak shifts toward lower wavenumbers may inhibit qualitative analysis. ATR spectra with shifts in either axis direction can be made to closely match their transmission counterparts by applying advanced ATR correction.[*2] For details on advanced ATR correction, please refer to Application News No. A476. The corrected spectrum exhibits peaks at about 3,674 cm^{-1}, 1,014 cm^{-1}, and 669 cm^{-1}, as described in the chemical identification test specified by the Japanese Pharmacopoeia. As such, we were able to obtain a favorable spectrum with no saturated peaks for talc, which is a sample with a strong absorption, simply by measuring the sample as is.

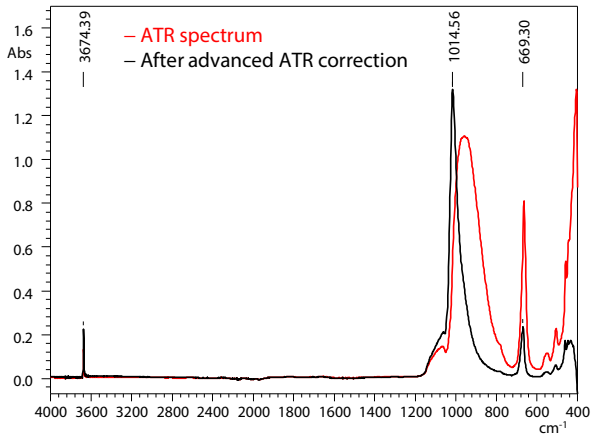

Fig. 5 ATR Spectrum and Spectrum after Advanced ATR Correction of Talc

Example Analysis of a Liquid Sample

The ATR spectroscopy is also used for measuring liquid samples. Since liquids achieve a good contact with the ATR crystal, measurement is possible by merely pipetting the sample onto the crystal. Cleaning after measurement is also easy. In addition, measurement is not influenced by the viscosity of the liquid so samples in paste form can be measured in the same way. However, since the crystal plate is made of stainless steel (SUS), measurement of corrosive liquids is not possible. If measuring corrosive liquids, please use a disposable IR card. Details of IR cards are described in Application News No. A448.

The QATR 10 is provided with a volatiles cover to enable the measurement of volatile liquids. Measurement is done by pipetting the sample onto the crystal and placing the volatiles cover over the sample. Fig. 6 shows a measurement using the volatiles cover. Ethanol was measured three times consecutively using the conditions listed in Table 1. Fig. 7 shows the obtained spectra superimposed on one another. We can see that the spectra were obtained with no changes in peak intensity, indicating that vaporization of the sample did not occur.

Fig. 6 Measurement Using the Volatiles Cover

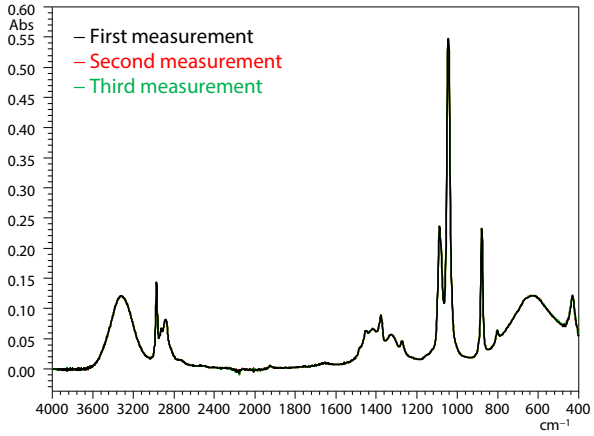

Fig. 7 ATR Spectra of Ethanol (n = 3)

Conclusion

This article introduced example measurements of resin, powder, and liquid samples using the QATR 10 single-reflection integration-type ATR accessory. The accessory enables the measurement of a wide variety of samples regardless of their form or properties. We expect the QATR 10 to be a highly effective tool in acceptance inspections for materials and contaminant analyses.

References
*1 Dictionary of Chemistry (Simplified Edition), Representative Editor Shoji Shida, Morikita Publishing
*2 Shimadzu Application News No. A476

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