

## Application News

# No. A552

### Spectrophotometric Analysis

## An Introduction to the QATR™-S Single-Reflection ATR Accessory with a Diamond Crystal - Example Analysis of a Food Contaminant -

Shimadzu's newly introduced IRSpirit™ is a highly compact FTIR featuring the highest signal to noise ratio and maximum resolution in its class (Fig.1). 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 the 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 reflection integration-type ATR attachments and diffuse reflection attachments.

The IRSpirit also features the dedicated QATR-S single-reflection ATR accessory with a diamond crystal which perfectly fits in the sample compartment of the IRSpirit. This article introduces the advantages of the QATR-S together with actual measurement results.

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Fig. 1 Appearance of IRSpirit, a Compact FTIR

### ■ QATR-S

The QATR-S is a single-reflection ATR accessory with a diamond crystal and is designed especially for the IRSpirit. Fig.2 (a) shows a view of the IRSpirit with the QATR-S mounted in the sample compartment from above and (b) shows a view of the same instrument from the front. Designed to be flush with the sample compartment on all sides, the QATR-S creates a wide top-sampling surface that can easily accommodate large samples for measurement without having to cut them.

The QATR-S also features smooth mounting and dismounting from the sample compartment since no screws are necessary. In addition, the accessory is equipped with a chip so that it will be recognized by the IRSpirit 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.

Furthermore, for pushing the sample against the ATR crystal, the QATR-S employs a swing clamp mechanism which incorporates a torque limiter so that damage to the ATR crystal can be prevented.

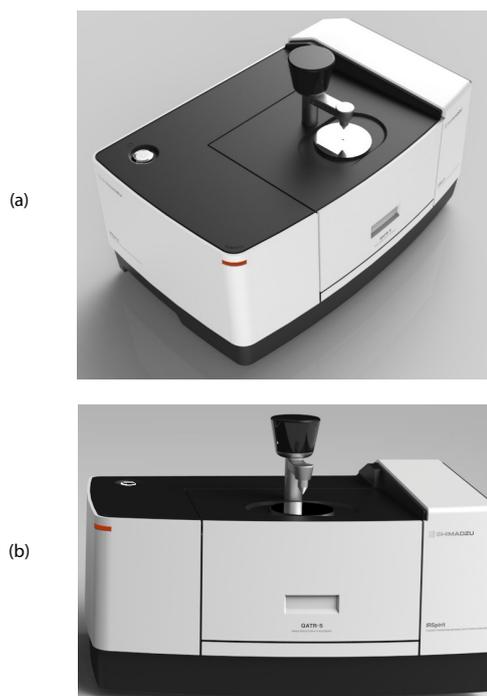


Fig. 2 IRSpirit with QATR-S Mounted  
(a) View from Above, (b) Front View

The crystal of the QATR-S 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  $\text{cm}^{-1}$  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. Fig.3 shows an ATR crystal being removed to be exchanged.



Fig. 3 Removal of ATR Crystal

## Contaminant Analysis Using QATR-S

A common complaint regarding food products is the inclusion of bone particles. When measuring a bulk sample such as bone using the transmission method with the KBr pellet method, pretreatment including pulverizing the sample becomes necessary. However, there are many samples which are difficult to pulverize. On the other hand, measurement using the ATR method requires no pretreatment of the sample and is therefore widely used to measure contaminants.

In this analysis, we measured the bone particle sample shown in Fig. 4 as an example of a bulk sample. Fig. 5 shows the sample set on the instrument with the QATR-S and being measured using the ATR method. Table 1 lists the measurement conditions that were used and Fig. 6 shows the measurement results. In using the ATR method, it is essential to achieve a good contact between the sample for measurement and the ATR crystal. The QATR-S is designed to cover this issue with a clamping mechanism that presses the sample against the ATR crystal from above for a firm contact and, as described earlier, the clamping mechanism incorporates a torque limiter so that an excess force is not applied to the ATR crystal.



Fig. 4 Photograph of Bone Particle Sample

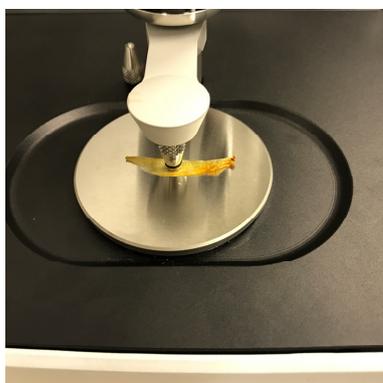


Fig. 5 Measurement of the Bone Particle Sample Using the ATR Method

Table 1 Measurement Conditions

Instrument	: IRSpirit-T (KBr window), QATR-S (wide-band diamond crystal)
Resolution	: 4 cm <sup>-1</sup>
Accumulation Times	: 20
Apodization Function	: Happ-Genzel
Detector	: DLATGS

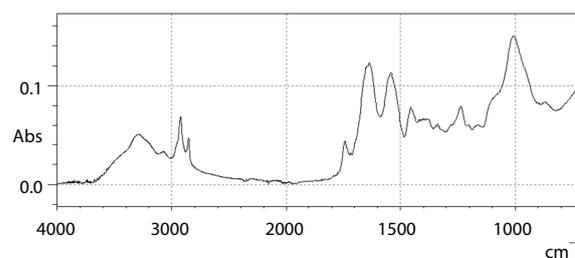


Fig. 6 Measurement Results of Bone Particle

The IRSpirit comes with a library containing approx. 2,000 spectra by standard specifications. By combining the optional contaminant library for searches, more detailed analysis results can be obtained with respect to the measurement results of contaminants. Fig. 7 shows the spectra search result using the contaminant library. The top match was the library spectrum of bone particle.

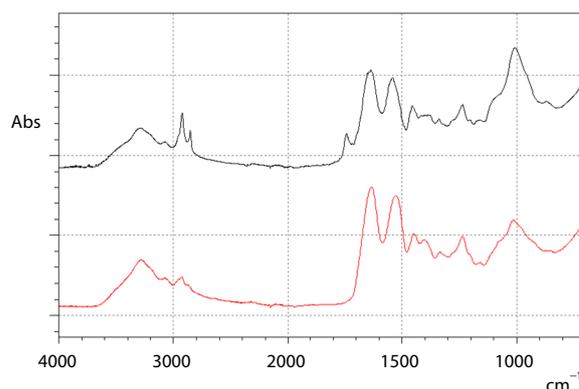


Fig. 7 Spectrum Search Result for Bone Particle (Using the Contaminant Library)  
Top: Measured Spectra of Bone Particle,  
Bottom: Library Spectrum of Bone Particle

In addition, the comment field of the spectrum search result shows the following library information which can be used for comparison with the actual measurement sample.

Name: Bone particle\_white  
Materials: Bone particle (Calcium phosphate, Protein)  
Major elements: Ca, P, S    Color: White  
Shape: Stick    Hardness: Hard  
Metallic luster: No    Technique: ATR (Diamond)

## Conclusion

This article introduced an example measurement of a contaminant using the QATR-S single-reflection integration-type ATR accessory with a diamond crystal. The ATR method can be used for measuring a wide variety of samples whether they be in solid, powder or liquid form. As such, the QATR-S is highly recommended for use in contaminant analyses and acceptance inspections for materials.

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