

Applications of a New Single Reflection ATR Accessory

Attenuated total reflection (ATR) is a method of obtaining a sample spectrum by contacting the sample to the prism and conducting the measurement. As even resin and rubber samples need not be thinly stretched or pulverized, it is one of the most frequently used infrared spectroscopy measurement methods. This Application News introduces the single reflection (infrared light is reflected once) ATR measurement accessory.

In the past, the technique that was almost always

adopted to increase sample absorption involved the use of a flat prism, and performing multiple reflections (bounces). However, with the development of more sensitive FTIR instruments, sufficient absorption can now be obtained with a single reflection. Therefore, measurement of small samples has become possible. Moreover, since the direct contact area is small, sufficient contact is possible, even with curved samples such as curved plastic.

■ Outline of ATR Sampling Accessory

Fig.1 shows a drawing of the "MIRacle" Single Reflection ATR accessory. The micrometer-equipped pressure clamp can apply finely-adjusted pressure from above onto the sample to obtain the appropriate contact with the approximately 1.5 mm-diameter circular prism (crystal).

Moreover, as the prism (crystal) is mounted in metallic housing to form a single unit, replacement is easy when using crystals consisting of different materials, nearly eliminating the need for optical adjustment.

■ ATR Spectra of Polymers

The ATR method is often used for measurement of polymers and rubber. We conducted measurements of a nylon pellet and polyurethane. It is evident that sufficient spectral intensity is obtained using the single reflection method.

Here, we used zinc selenide (ZnSe) as the prism. In the past, "KRS-5" was often used, but currently, ZnSe has been receiving attention as an alternative material. Compared to KRS-5, the measurement range with ZnSe is narrower at the lower wavenumbers; however, its hard, tough polycrystalline state imparts excellent mechanical strength. As they have the same refractive index, measurement of ZnSe and KRS-5 provide the identical spectrum.

The refractive index, hardness and usage wavenumber range are shown below for ZnSe, KRS-5 and diamond, respectively.

Table 1 Physical Properties of Various ATR Prisms

	Refractive Index	Hardness (200g Knoop No.)	Wavenumber Range (cm ⁻¹)
ZnSe	2.4	250	~550
KRS-5	2.4	40	~250
Diamond	2.4	Hardest	~12.5

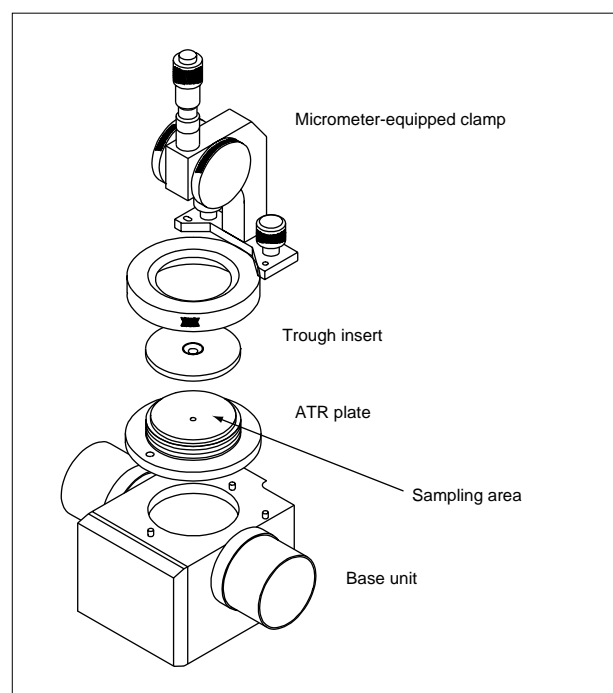


Fig.1 Overview of Single Reflection ATR MIRacle

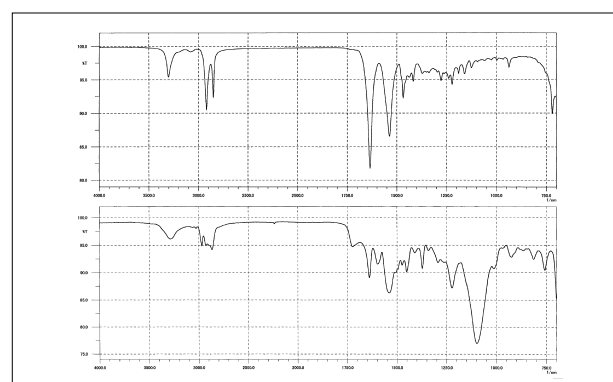


Fig.2 ATR Spectrum of Nylon 11 Tip (upper) and Polyurethane (bottom)

■ ATR Measurement of Rubber

Resins and rubbers are easily measured by ATR. When measuring rubber, the selection of the type of prism is important, and depends on whether or not the rubber contains carbon black. As the ATR method utilizes the phenomenon of total reflectance, the prism that is used must have a higher refractive index than the sample. Carbon is mixed in with the rubber used in automobile tires, etc. to increase the strength of the rubber; however, the refractive index of the sample increases due to the presence of carbon. Therefore, when measuring black rubber, it is necessary to use germanium (Ge) for its high refractive index ($n=4$). Here we show the results using both Ge and ZnSe as prisms.

We measured acryl rubber containing carbon. With the high-refractive index Ge, the baseline is high and the absorption peak is clearly observed, but with the low-refractive index ZnSe and diamond, the total reflectance condition is not attained, so that the peak is distorted and the baseline is low due to the carbon absorption. (Fig.4)

A good spectrum is obtained with germanium. (Fig.3)

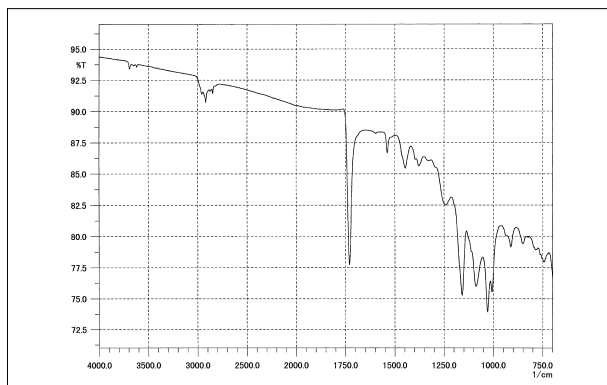


Fig.3 ATR Spectrum of Rubber with Ge prism

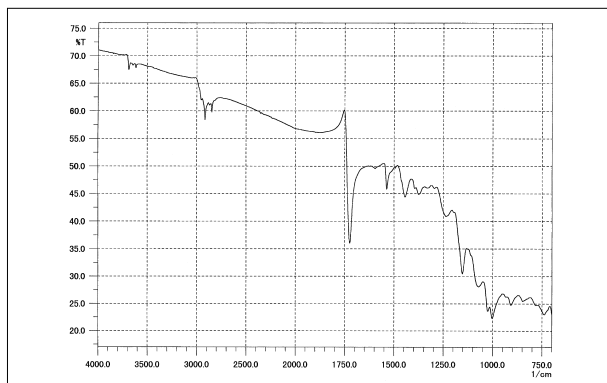


Fig.4 ATR Spectrum of Rubber with ZnSe prism

■ Measurement of Powder

Powder measurement is most often conducted by the KBr pellet method and diffuse reflectance method; however, these measurement methods have previously required mixing with KBr. Accordingly, this has involved time-consuming pretreatment.

With the ATR method, powder measurement can be conducted easily by merely establishing contact between the powder sample and the prism. Moreover, as this also eliminates the need to mix in KBr, there are no spectral changes due to substitution.

The Japan Pharmacopeia also prescribes ATR as a measurement method. Fig.5 and Fig.6 show ATR spectra of galactose and lactose samples, respectively.

In the single reflection ATR method introduced here, since measurement is easily conducted by bringing the sample in close contact with the prism, measurement time is shortened and pretreatment is greatly simplified.

In addition, although a specific measurement example is not presented here, measurement of liquid samples is also easy with ATR.

Since this single reflection ATR attachment "MIRacle" can operate using both ZnSe and Ge prisms, the appropriate crystal can be selected and mounted according to the sample, enabling measurement of a wide variety of samples.

The ATR method is expected to undergo further development as an analytical technique, and to be used in a wider range of application fields.

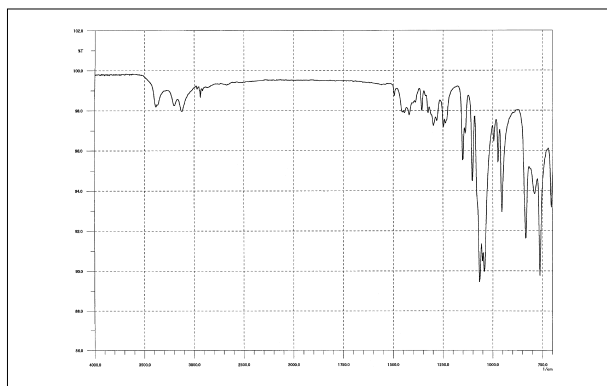


Fig.5 ATR Spectrum of Galactose

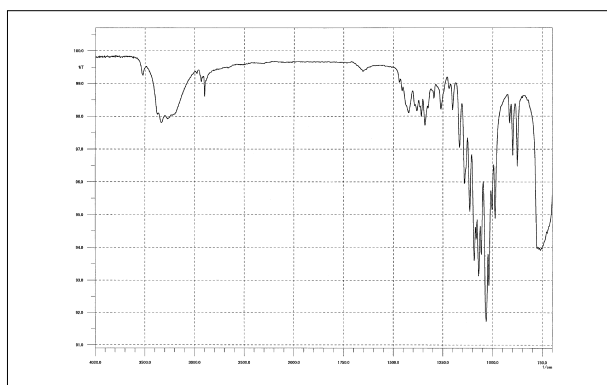


Fig.6 ATR Spectrum of Lactose

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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