The Specac Quest™: How the ATR accessory works

The Specac Quest™ accessory lets the user record accurate IR spectra without timely sample preparation.

TRADITIONALLY, recording IR spectra of samples involved making KBr discs or dissolving the sample in solvents.

However, this is an inefficient and time consuming practice, especially for multiple recordings.

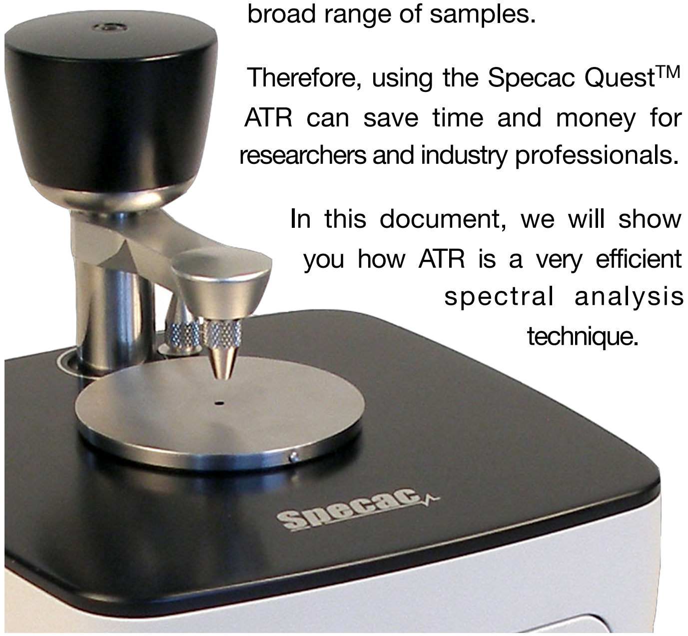
Furthermore, solvents present hazards and can be expensive, while making KBr discs requires user training. Diluting samples in this way helps to prevent the intense absorption bands saturating the detector.

Attenuated Total Reflectance (ATR) eliminates these issues because little or no sample preparation is needed.

ATR is an effective FTIR sampling technique to produce qualitative or quantitative spectra of a broad range of samples.

Therefore, using the Specac Quest™ ATR can save time and money for researchers and industry professionals.

In this document, we will show you how ATR is a very efficient spectral analysis technique.



So how does it work?

Figure 1 demonstrates the principle behind the single bounce ATR, where IR light from a spectrometer is internally reflected in the ATR crystal and interacts with the sample at the crystal interface.

Partial penetration of the IR light by an evanescent wave allows an absorption spectrum to be recorded.

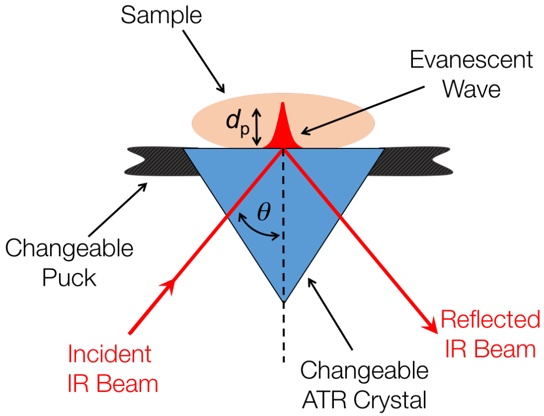


Figure 1: Graphical representation of a single bounce ATR.

The absorbance and depth of penetration (d_p) of the evanescent wave depend on the following factors:

- the refractive index of the crystal, n_1
- the refractive index of the sample, n_2
- the angle of incidence, θ
- the wavelength of the light, λ

Table 1 shows the physical properties of different ATR crystals and the value of d_p when $n_2 = 1.5$, 0 - 45° and λ = 10 µm. The typical value of d_p ranges from 2–4 µm depending on the crystal.

$$d_p = \frac{\lambda}{2\pi\sqrt{(n_1^2 \sin^2\theta - n_2^2)}}$$

For example, a Ge crystal provides a shorter sample penetration depth than a ZnSe crystal, which is ideal for strongly absorbing samples.

However, its effective IR absorption range is limited, whereas a range of 4000–400cm^{-1} can be achieved by using the extended mono-crystalline type IIIa diamond crystal.

The Specac Quest™ ATR allows the user to change the crystal to suit the experimental requirements by swapping the top-plate puck.

Crystal	n_1	d_p / µm	IR range / cm^{-1}	Uses	Part no.
ZnSe	2.4	2.01	7800 – 500	General	GS10812
Diamond	2.4	2.01	7800 – 400	Harder & chemically resistant	GS10810
Extended Diamond	2.4	2.01	10000 – 40	Shorter freq.	GS10811
Ge	4.0	0.66	5500 – 480	Highly conc. samples	GS10813

Table 1: Physical properties of the ATR crystals.

Figure 2 shows that ZnSe and Diamond have similar refractive indices and hence have similar penetration depths, while Ge is significantly different, so provides a lower penetration depth.

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> As the IR light frequency decreases, so does the crystal's refractive index.

Intensity Corrections

At higher frequencies, the relative absorbance of a sample is lower in the spectrum recorded using the ATR.

This is because the penetration depth of IR light into the sample depends on the refractive index of the crystal, which changes as a function of frequency.

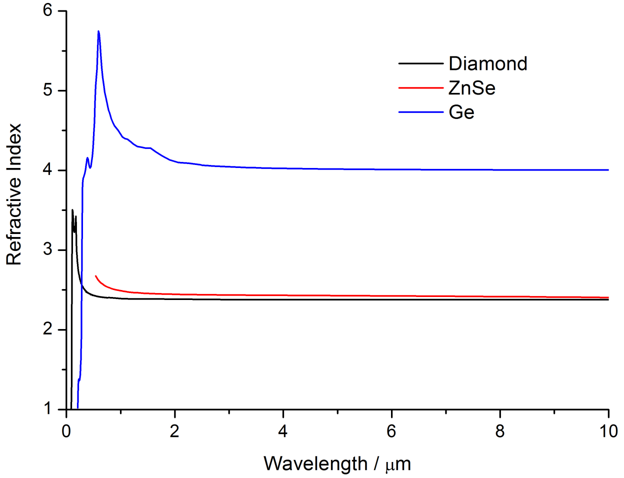

Figure 2: The refractive index of Ge, ZnSe and Diamond.

Figure 3 shows in more detail how the refractive index of ZnSe changes with wavelength at standard room temperature and pressure.

Consequently, the depth of penetration changes with the refractive index and hence the amount of light absorbed by the sample. Most FTIR spectrometers come with a correction package.

Figure 4 shows the transmission spectra of DuPont Krytox Gpl205 lubricant recorded using a traditional transmission method and the Quest™ ATR.

Using a transmission measurement technique, there is a strong absorption by the bands around the fingerprint region (1500–500 cm^{-1}) as well as some saturation at 1250 cm^{-1}. But the peaks are more resolved using the ATR because of its short *effective pathlength*.

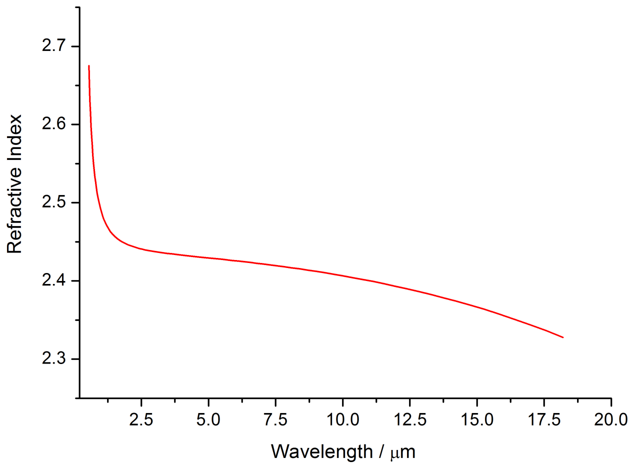

Figure 3: The refractive index of ZnSe.

Conclusions

The Quest™ ATR is a single bounce ATR that provides a quicker alternative for acquiring IR spectra for a range of sample types, such as gels, pastes, liquids and powders.

As the IR light frequency decreases, so does the crystal's refractive index, which results in an increase in sample absorbance. Interchangeable pucks with 4 different crystals allow the user to control the IR light penetration depth and *effective pathlength*, as well as the spectral range.

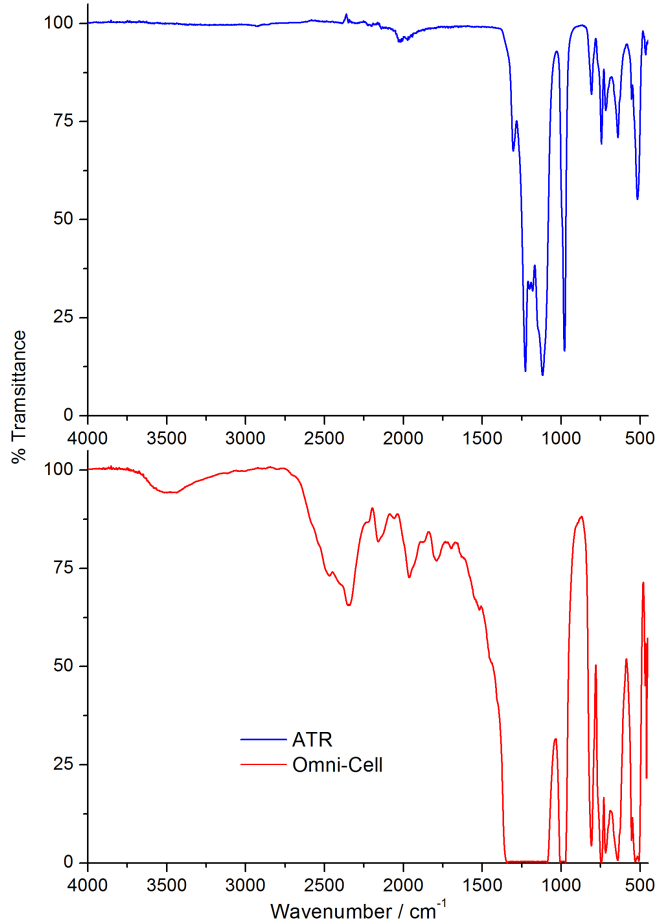

Figure 4: Transmission spectra of Gpl205 lubricant.

References

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