IR Spectra of Grease: Pearl™ vs Quest™ ATR

Which of Specac's latest IR accessories is more suitable for testing grease samples?

GREASES AND LUBRICANTS are important to help moving parts and machinery operate smoothly. Over time, they can degrade and pick up contaminants. Therefore, we need reliable ways to inspect them and literally keep things running smoothly.

Attenuated Total Reflectance (ATR) is an established technique used in the IR spectral analysis of many solid and liquid samples, such as organic polymers[1] and solvated proteins[2].

Minimal sample preparation is required but the sample has to make good physical contact with the ATR crystal to obtain an IR spectrum.

This is easy for liquids, but solids need to be pressed onto the crystal.

This application note will show that **The Pearl™** liquid transmission accessory is perfect for measuring IR spectra of **'sticky' and relatively immobile** liquid samples like greases and lubricants as easily, quickly and reliably as a comparable ATR technique, such as with the Quest™.

Equipment and Methods

A Quest™ ATR fitted with an **extended range diamond crystal puck** was used to collect ATR spectra. It had an effective **pathlength of 4.5 µm**.

The Pearl™ accessory was used to record IR transmission spectra via its Oyster Cell with **wedged ZnSe windows** and a **50 µm pathlength** (P/N GS31221).

DuPont Krytox GPl205 and Castrol LM greases were tested; < 1 ml of sample without dilution.

The spectra were recorded with a **1 cm^{-1} in an FTIR spectrometer**. Their absorbance traces are shown in Figures 1 and 2 respectively.

- For the Quest™, the samples were spread over **a diamond crystal**.
- For the Pearl™, the greases were spread on the bottom **ZnSe window** of the Oyster Cell.

The sample introduction, spectral data acquisition and subsequent clean-up were all quick and easy due to the design of both the Quest™ and the Pearl™.

Results and Discussions

Figure 1 shows the IR absorption spectrum from 3600 – 600 cm^{-1} of Castrol LM grease recorded with both the Quest™ ATR and the Pearl™.

The two techniques capture the same peaks but with different resolution.

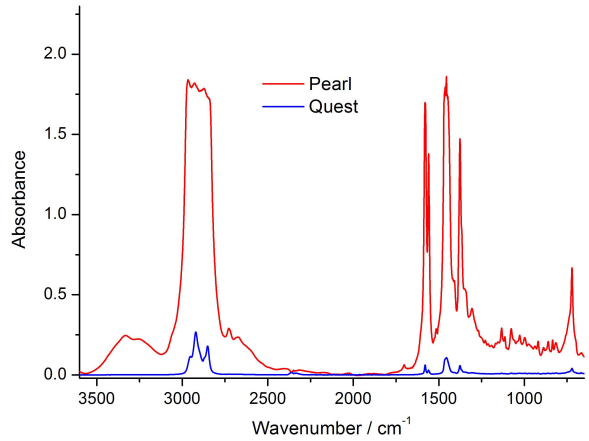

Figure 1: Castrol LM grease spectrum, Pearl™ vs Quest™

The clearest difference is the stronger absorption of light at **2900 cm^{-1}** in the Pearl™.

Another key observation is that the signal intensity in the Pearl™ is 10 times stronger than that recorded in the ATR, reflecting the **~10 times longer pathlength**.

Figure 2 shows a similar comparison for the Krytox lubricant.

In both figures, the weaker absorption bands, such as those that lie within the 500 – 1500 cm^{-1} fingerprint region, appear more intense when measured using the Pearl™ accessory.

A **Ge puck** can be used instead of diamond in the Quest™ ATR to give a lesser depth of IR beam penetration. This would decrease the effective pathlength in the ATR measurement and the sample absorbance.

@Specac
Specac Ltd

> The Pearl™ offers greater spectral resolution.

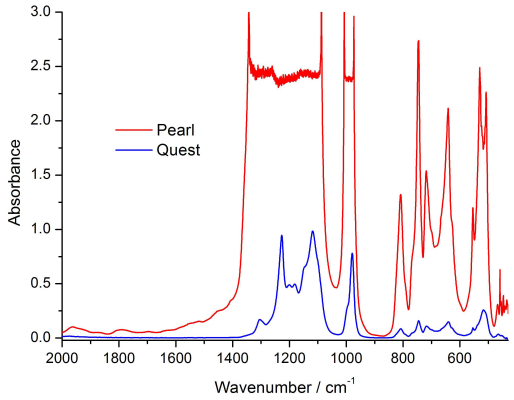

Figure 2: DuPont Krytox spectrum, Pearl™ vs Quest™

Conclusion

IR absorption spectra for fairly immobile greases were recorded using the Quest™ ATR and the Pearl™ liquid transmission accessory.

The spectral features recorded in the Quest™ were quite weak because the ATR technique provides much shorter pre-defined *effective pathlengths* that are **dependent on a sample contact** effect with the ATR crystal and the incident angles being used.

Conversely, the spectra were amplified using the Pearl™, which offers a **longer pathlength**.

This means that vibrational modes at larger wavenumber, such as -OH or -NH molecular stretching vibrations, can be more readily identified by this transmission technique rather than the ATR alternative.

Overall the Pearl™ offers greater spectral resolution, even for samples which weakly absorb in the IR.

There is also no need for a complicated spectral intensity correction with the Pearl™. Furthermore, the **pathlength can be easily controlled** and determined in the Pearl™, unlike in the Quest™.

The Pearl™ and Quest™ are available in various colours.

Nevertheless, ATR is a very **reliable and fast** method and perfect when only a small amount of sample is present and absorbs strongly in the IR.

Email **sales@specac.com** or visit **www.specac.com**

References

[1] Blanc, F., Blanc, Chong, S.Y., McDonald, T.O., Adams, D.J., Pawsey, S., Caporini, M.A., Cooper, A.I., Dynamic Nuclear Polarization NMR Spectroscopy Allows High-Throughput Characterization of Microporous Organic Polymers. *J. Am. Chem. Soc.* **135**, 14, 15290 – 15293 (2013).

[2] Boulet-Audet, M., Byrne, B., Kazarian, S.G., High-Throughput Thermal Stability Analysis of a Monoclonal Antibody by Attenuated Total Reflection FT-IR Spectroscopic Imaging. *Anal. Chem.* **86**, 9786 (2014).

Pearl™ Wedged Lower Windows

	CaF_2	ZnSe
25 µm	GS31326	GS31226
50 µm	GS31321	GS31221
100 µm	GS31322	GS31222
200 µm	GS31323	GS31223
500 µm	GS31324	GS31224
1000 µm	GS31325	GS31225

Did you know parallel windows are also available?

Quest™ ATR Models

	Diamond	ZnSe	Ge
Standard	GS10800	GS10802	GS10803
Extended Range	GS10801	N/A	N/A

Quest™ ATR Accessories

Volatiles Cover	Part Number
Purge Bellows	GS10707
Steel Flat Anvil	GS10820
Steel Pellet Anvil	GS10821