ENERGY & CHEMICAL ANALYSIS

ADVANTAGES OF THE AGILENT CARY 630 DIAMOND ATR – SEE WHAT YOU'VE BEEN MISSING!

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Author
Rob Wills
Agilent Technologies

There's no doubt that the advent of single-bounce, diamond crystal Attenuated Total Reflectance (ATR) accessories has totally revolutionised modern FTIR analysis. Traditional, time consuming, transmission based sample preparation methods such as KBr disk and Nujol mull have now been all but forgotten in favour of using a diamond ATR which requires no sample preparation, and which can be used to analyse a wide range of sample types including solids, liquids, powders, pastes and gels, across the complete pH range from strong acid to strong base.

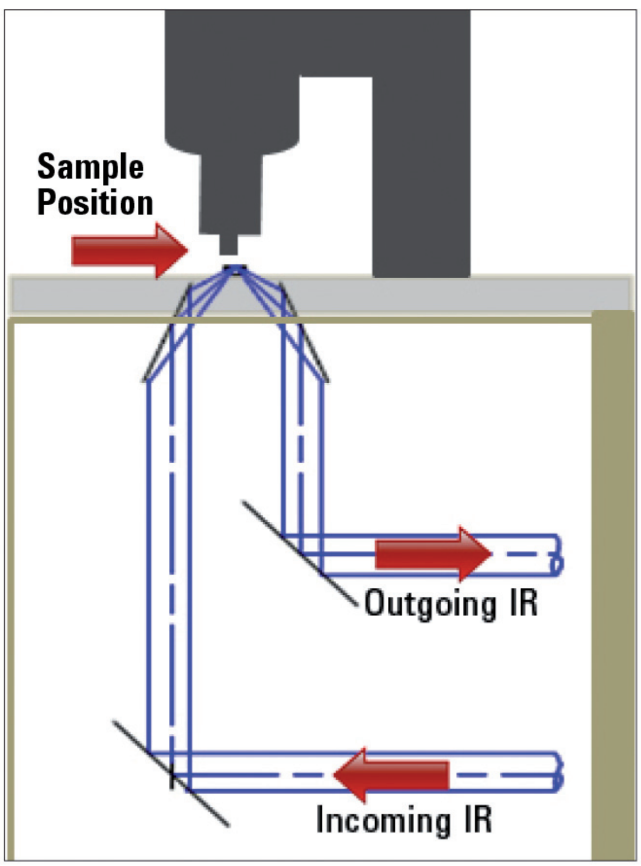
However, not all ATR accessories work in the same way and, in this article, we will briefly discuss these differences and look at the advantages of the Cary 630 diamond ATR.

How ATR Works and Comparison of ATR Types

The principle of operation of a single-bounce diamond ATR is relatively simple to understand. The diamond crystal is typically a small (circa 2mm diameter), high purity Type IIa diamond fixed into a stainless steel plate which acts as a sampling stage. The sample is placed on top of the diamond and it is important to ensure that the sample is in intimate contact with the sampling crystal, so for the analysis of solid samples a pressure clamp is used to press the sample down firmly onto the crystal. The IR beam is directed towards the diamond, usually at an angle of 45°, and is largely reflected back from the internal surface, such that it exits the crystal and is detected by the spectrometer. However, some of this IR energy escapes from the surface of the crystal in the form of an evanescent wave and may be absorbed by the sample. Ultimately what we end with is a spectrum that looks very similar to the transmission spectrum of the sample.

It's important to note that this ATR phenomenon only works if the refractive index (RI) of the sample is lower than that of the diamond (diamond RI = 2.4). For samples with higher refractive indices a Germanium crystal must be used (RI = 4.0).

Generally speaking, diamond ATR accessories fall into two distinct groups depending on their optical characteristics, and these groups can be described under the headings of either "monolithic" design or "composite" design.



In a monolithic type ATR the IR beam is directed to the crystal using only mirrors. The advantage of this design is that you can make full use of the spectral range of the instrument, but the downside is that efficiently focussing a relatively large diameter IR beam onto a small diameter diamond crystal using only mirrors is quite a challenge, and inevitably much of the IR energy is wasted, so monolithic ATR's can suffer from relatively low energy throughput, resulting in poor signal-to-noise and therefore needing long data collection times.

By comparison, a composite ATR exhibits the opposite phenomenon. In a composite design, the IR beam is directed onto the diamond using a focussing lens which is much more energy efficient and results in roughly a doubling of the energy throughput compared to the monolithic design. However, the disadvantage of the composite design is that the focussing lens does limit the measurable spectral range – for example, when a ZnSe focussing optic is employed the lower spectral range is limited to around 600cm^{-1}, and many compounds have valuable spectral information below this lower limit.

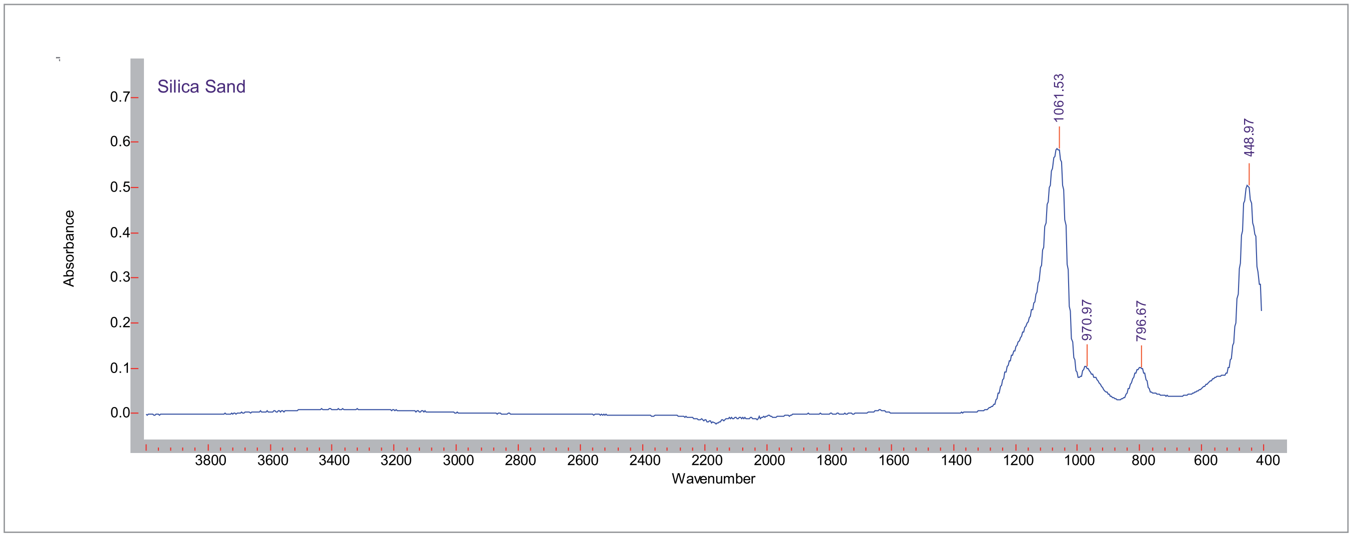
Clearly, the ability to collect data over the widest possible spectral range has benefits, especially when performing library searches to identify materials – the more spectral bands you can match, the greater will be the confidence that the library match returned from the search is correct.

Cary 630 Diamond ATR – The Best of Both Worlds!

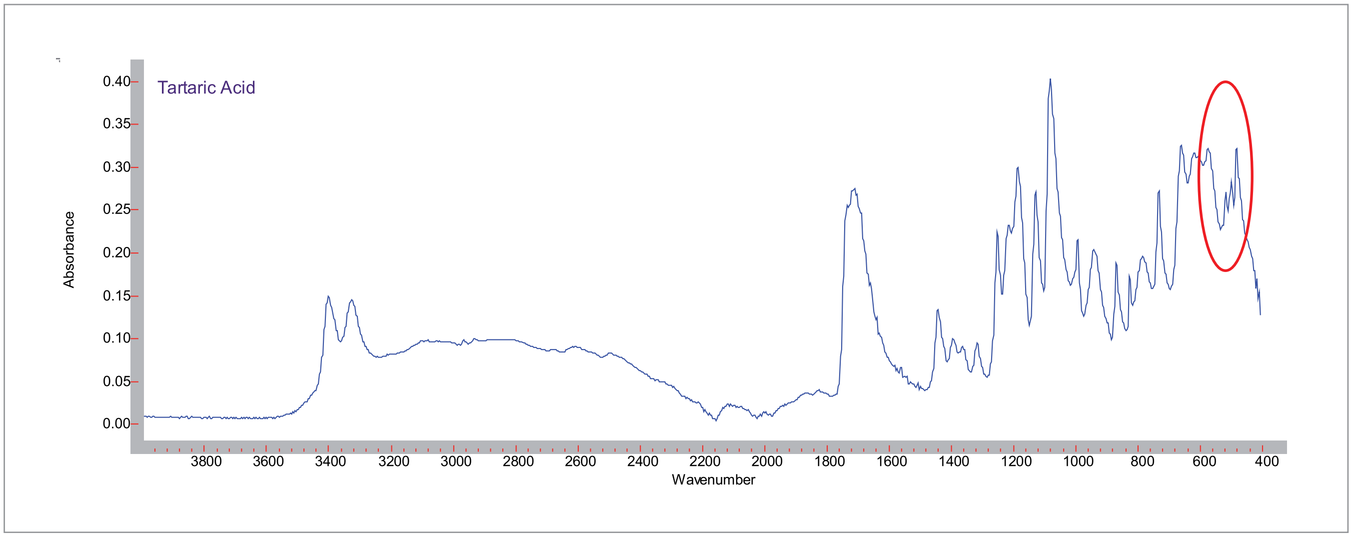
The Cary 630 diamond ATR is a monolithic design offering full spectral range, but the very short IR beam path from the interferometer exit to the ATR crystal allows the IR beam energy to be very efficiently transferred with minimal internal energy loss, such that the energy throughput is comparable to the highest performing composite type ATR. In other words, the Cary 630 ATR gives the best of both Worlds!

Example Data

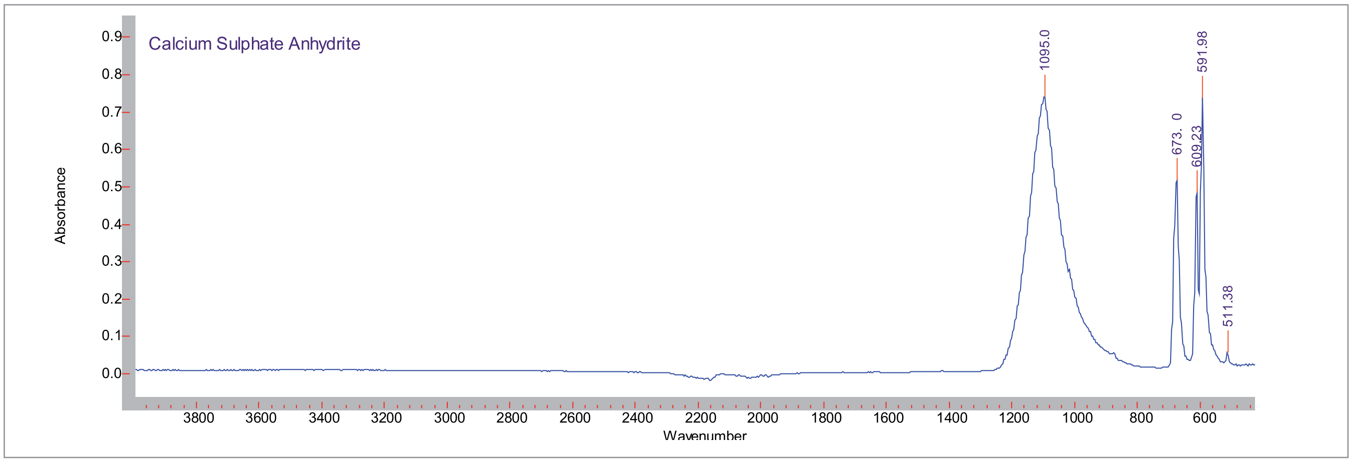
Here follows a selection of spectra measured on a variety of compounds using the Agilent Cary 630 FTIR equipped with diamond ATR interface. All of these spectra were collected using only 32 co-averaged scans at 4cm^{-1} spectral resolution (approx. 15 second measurement time).



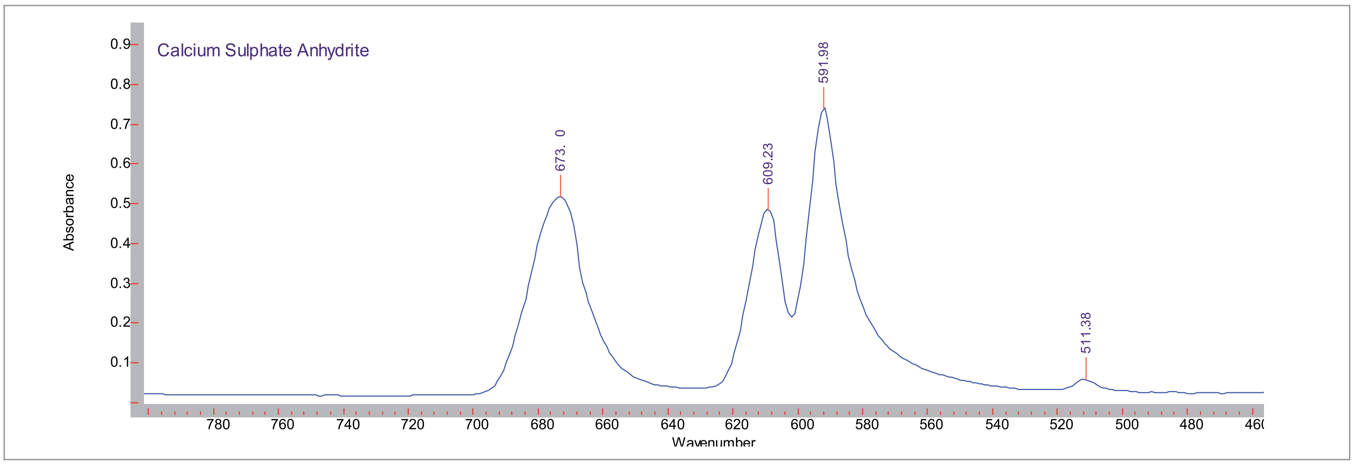
Spectrum of silica sand – the peak at 448.97cm^{-1} is clearly visible.



Tartaric acid spectrum shows peaks at 573.39, 516.05, 499.99 and 482.00cm^{-1} (circled).



Spectrum of calcium sulphate anhydrite – this compound exhibits a cluster of peaks below 700cm^{-1}.



A magnified view of this cluster is shown in the spectrum. The peak at 591.98cm^{-1} is clearly visible, as is the tiny peak at 511.38cm^{-1} which is testament to the excellent energy throughput of the Cary 630 diamond ATR, which gives the benefit of fantastic signal-to-noise.

Summary

The above data clearly shows that the Agilent Cary 630 equipped with diamond ATR is capable of measurements below 600cm^{-1}, and that this low wavenumber spectral region can exhibit significant absorbance bands.

The data also shows the excellent signal-to-noise ratio of the Cary 630, verified by its ability to detect even very weak signals using only a short measurement time.

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