Performance of Slide-On Attenuated Total Reflectance Accessories: FT-IR Microscopy

APPLICATION BRIEF

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Introduction

Attenuated Total Reflectance (ATR) is widely used to simplify sample preparation. This technique relies on total internal reflection. The sample is brought into direct contact with a crystal (internal reflection element, IRE) that has a refractive index that is much higher than the sample. When the angle of incidence of the light from the IRE to the sample exceeds the crystal angle, total internal reflection takes place. When this total internal reflection takes place, the light penetrates slightly into the sample producing an absorbance-like spectrum. This phenomenon has been used since the early 1960's to obtain spectra of difficult samples such as rubbers, fibers, minerals etc.

With a microscope, this technique is used to analyze small samples and eliminate microtoming. Since the ATR technique requires contact with the sample, it is crucial that the design of the IRE must ensure that it neither pierces fragile samples nor does it exert so much pressure that the surface of the sample to be examined is destroyed.

Experimental Details

To demonstrate that the ATR technique is truly reproducible and that the crystals do not destroy the sample, multiple spectra were collected from a coated aspirin sample. The spectra were collected using a Varian UMA 600 microscope interfaced to an Excalibur FTS-3000 spectrometer. The system was controlled using Resolutions Pro™, the Varian software designed for research applications.

The experiments were conducted using the Varian slide-on ATR crystal. This crystal mounts on the Cassegrain objective and slide out of the way for sample viewing or transmission analysis and can be slid into position for ATR analysis. This design eliminates the needs for either two 15x objectives or for the constant swapping of microscope objectives. A germanium crystal was used. The geometry of the crystal is shown in Figure 1. This geometry is designed to provide good optical contact with the sample to be analysed without damaging the surface of the sample. The samples examined were commercially available coated aspirin tablets. These samples are delicate and the coating can easily be broken.

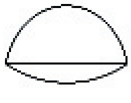


Figure 1a

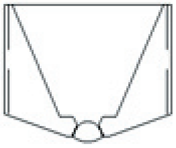


Figure 1b

Figure 1a shows the shape of the germanium ATR IRE. The larger radius surface is the one brought into contact with the sample. **Figure 1b** shows a view of the germanium IRE in this holder. This holder then fits into the slide on ATR.

Results and Discussion

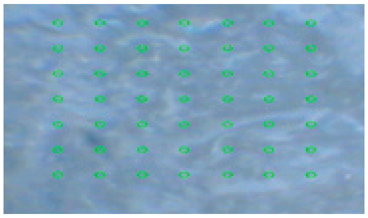


Figure 2.
Visual picture of the surface of a coated aspirin tablet

At each indicated point in Figure 2, a spectrum was collected using software-controlled ATR mapping. Figure 3 shows a series of spectra collected by: touching the crystal to the sample, collecting the spectrum, raising the crystal to make sure that no material is adhered, lowering it and collecting again. No special caution was taken to ensure that the contact was equal. As the data indicates, the spectra obtained are very reproducible.

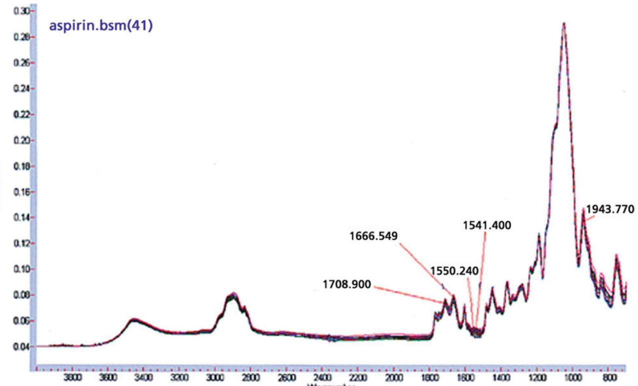


Figure 3. Series of sequential spectra collected by touching the ATR element to the aspirin coating

Results and Discussion (continued)

The spectra are shown in an overlaid mode on an absorbance scale of 0 to 0.3. There is no evidence of saturation in the absorbance and no indication of the underlying aspirin, the spectrum of which is shown in Figure 4.

Figure 4.
Spectrum of aspirin for comparison

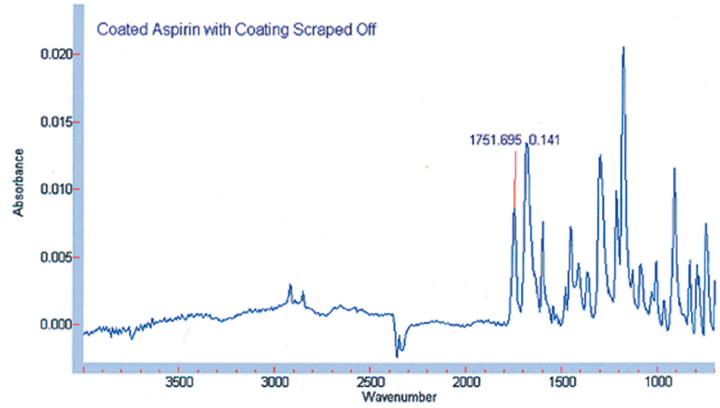


Figure 5 shows a contour plot of the data collected on the sample. The contours represent the intensity of the coating peak at 1708 cm^{-1}.

In a second experiment, we examined a sample after we had scraped off a small amount of the coating to expose the underlying aspirin. In this case, a map was also done. When a contour plot of the intensity of a peak of interest at 1708 cm^{-1} was generated, the hole in the coating is clearly indicated by the increased intensity of the peak at 1708 cm^{-1}. This is shown by the red contours on the plot in Figure 6.

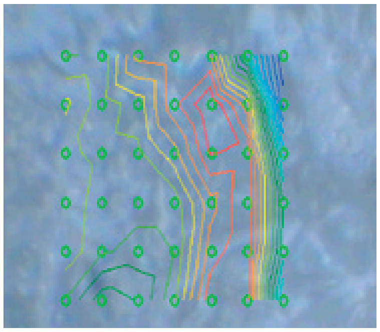
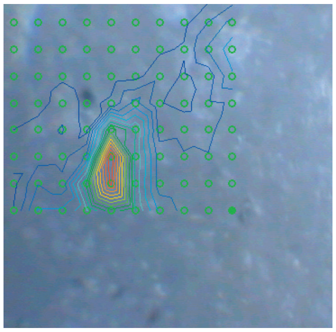


Figure 5.
Contour plot of the coating distribution of the surface of the aspirin

Figure 6.
Contour of hole in the coating



Conclusion

Varian's slide-on ATR delivers superior performance on delicate materials. It can be used to obtain multiple reproducible spectra of a sample and can also be used to map chemical features. Depending on the chemical compatabilities and crystal hardnesses requried, other crystal materials are available such as ZnSe, Si, KRS-5 and diamond.

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