INFRARED SPECTROMETER ACCESSORIES

Quantitative Analysis of Powdered Solids

Introduction

Infrared quantitative analyses are an everyday requirement in the analytical laboratory. While liquid solutions are commonly analyzed, mixtures of powdered components also exist and often require quantitative analysis without dissolution into a solvent.

This paper outlines the development of quantitative analysis methods for two powdered mixtures.

The traditional infrared analysis method for powdered samples is the collection of a KBr pellet spectrum of an aliquot of the powdered sample. However, preparation of KBr pellets requires skill, and precise weighing of every component for each sample. The Golden Gate™ singlereflection diamond ATR provides a simple and effective alternative, suitable for the infrared analysis of powders.

The powdered sample is simply placed onto the ATR crystal and the sample spectrum is collected. The sapphire anvil ensures that up to 100 lbs load is evenly applied to the sample lying on the diamond micro-ATR crystal during sample analysis. The sample is then cleaned from the crystal surface and the accessory is ready to collect additional spectra. ATR analysis is less complicated than using KBr pellets, is fast and only requires a very small amount of the sample.

Specac's Golden Gate ATR Accessory.

To develop the quantitative analysis method, a number of standard powder mixtures of known concentration are prepared and spectra are collected from aliquots of these standards. Specified analyte absorption bands are identified and the peak heights, or areas are calculated for the various standards.

The resulting quantitative data is examined by a least squares analysis based on **Beer's Law**, **A = abc**. Where, **A** = the absorbance value of an analyte band; **a** = the absorptivity coefficient of the analyte band - a constant; **b** = the pathlength - generally considered a constant; and **c** = analyte concentration.

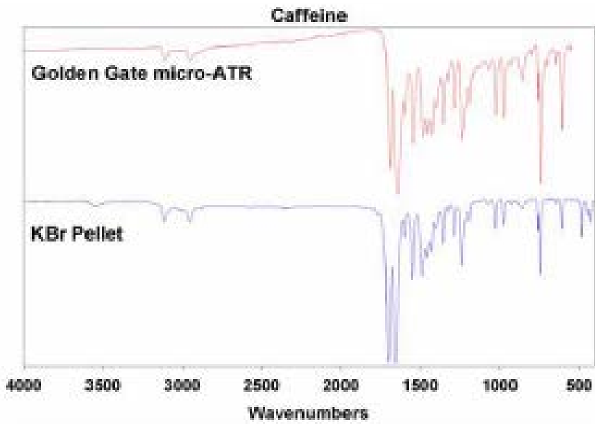

Figure 1

ATR spectra cannot be used with a quantitative method previously developed using transmission spectra due to differences in the relative peak intensity of the absorption bands - this is a result of the internal reflection mechanism of ATR accessories. – as seen in the example, Figure 1, a plot of the transmission and ATR spectra of caffeine.

However, a least squares relationship can be drawn for ATR spectra of quantitative samples.

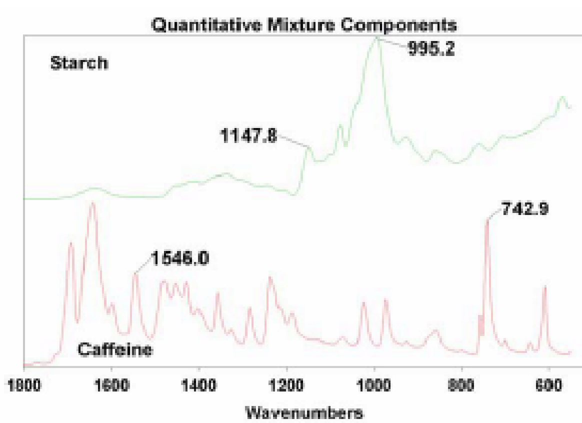

Figure 2

Experiment and Results

Spectra were collected using a Golden Gate™ Diamond ATR accessory equipped with ZnSe lenses and a single-reflection diamond ATR element. Without additional sample preparation sample aliquots of 5-10 milligrams were placed onto the ATR element and spectra collected.

For correlation with the ATR data, aliquots drawn from the standard powders were mixed with KBr powder, pressed into pellets and analyzed to provide transmission spectra. FT-IR spectra were collected at 4 cm^{-1} resolution to obtain the single-beam background, micro-ATR spectra and KBr pellet transmission spectra.

Figures 2 and 3 compare the fingerprint regions of the Golden Gate™ ATR spectra for the components used in the study; caffeine and starch in the first mixture, Ibuprofen and starch in the second. The analyte bands selected for the analysis of each component are labeled.

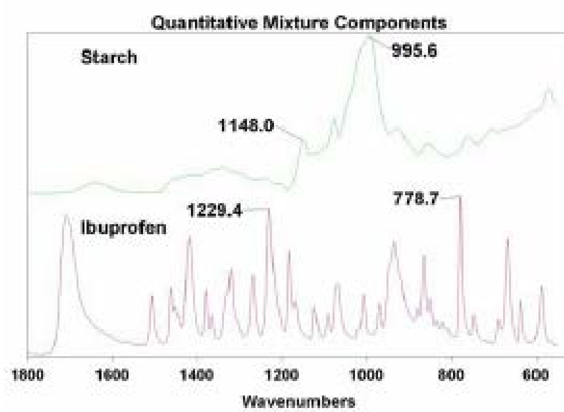

Figure 3

Three aliquots were analyzed for each powder standard using the ATR and KBr pellet methods. Two analyte bands were selected for each component in each mixture and peak ratios calculated based on the ratio of the peak heights of the active ingredient vs. the starch. An example of the repeatability of the Golden Gate™ ATR measurement is demonstrated in Figure 4, containing three spectra of a 25%concentration of ibuprofen in starch.

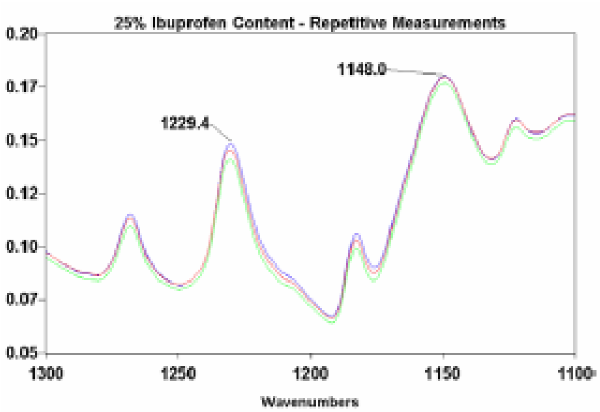

Figure 4

Figure 5 is a least squares plot of the ATR data, following the 743 and 995 cm-1 bands for caffeine and starch, respectively. Figure 6 is the least squares plot of the KBr pellet data using the same analyte bands. Table 1 contains the R and R2 values for the least squares analysis of the data from the two mixtures using the different analyte bands for each component.

These results demonstrate that the ATR method can provide repeatable quantitative results that are comparable to KBr pellet data.

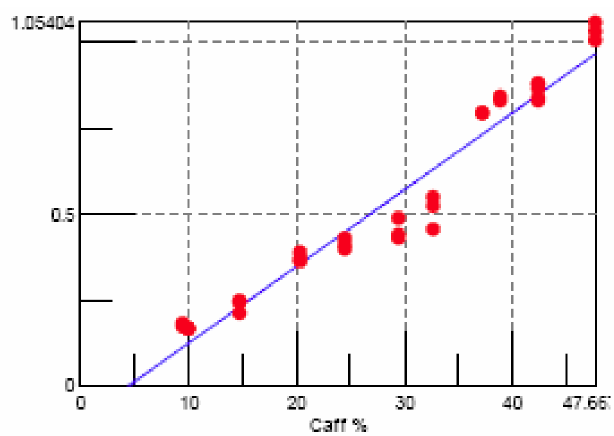

Figure 5

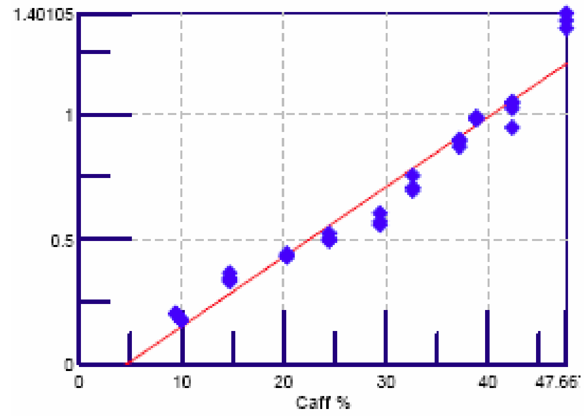

Figure 6

Table 1: Least Squares Residuals Data

Caffeine/Starch Mixture	R	R^2
ATR - 743/995 cm^{-1}	0.9738	0.9484
ATR - 1546/1148 cm^{-1}	0.9589	0.9196
KBr - 743/995 cm^{-1}	0.9764	0.9533
KBr - 1546/1148 cm^{-1}	0.9616	0.9246
Ibuprofen/Starch Mixture		
ATR - 1230/995 cm^{-1}	0.9474	0.8976
ATR - 779/1148 cm^{-1}	0.9535	0.9091
KBr - 1230/995 cm^{-1}	0.9731	0.9469
KBr - 779/1148 cm^{-1}	0.9741	0.9489

Conclusion

The results show that the Golden Gate Diamond ATR can be used to produce quantitative data – the ability to clamp samples at very high loads ensures consistent results, while the use of a type IIA natural diamond allows for extreme durability of the accessory.

The Golden Gate Diamond ATR is a simple and easy to use accessory providing a non-destructive technique to collect data from a minimal amount of sample, providing quantitative results similar to KBr pellet spectra in a faster less complicated fashion.

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