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The Automation of Microscopic Analyses: Ease of Use

APPLICATION BRIEF

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Introduction

Typically, infrared analysis is carried out in a single analysis mode. Most analysts have learned to prepare one sample while another is being run in an instrument. This speeds up the analysis but it does not give the analyst a block of time to accomplish another task. Chromatographic instrumentation has used auto samplers for a number of years. For FT-IR, auto samplers have been introduced in the past. Most were, at best, slightly successful. Effort has not been expended on this type of auto sampler because most FT-IR spectroscopists only analyze between 5 and 10 samples per day.

The workloads in most analytical laboratories have changed dramatically over the past few years. Companies have cut back on staff and analysts are now expected to do more than one job. Chemists who operate FT-IRs usually have responsibility for many more instruments than their counterparts a few years ago. This has made the ability to analyze many samples, unattended, a necessity in many labs.

The most complicated and time consuming operation in an FT-IR analysis is sample preparation. Attenuated total reflection (ATR), diffuse reflection and specular reflection all simplify the sample preparation. An infrared microscope can also simplify this sample preparation.

The next step in the automation of infrared analysis would be to automate microscopic analysis. Varian has taken this approach.

Experimental Details

All spectra were collected using Varian's Excalibur with UMA 600 microscope. This microscope offers an automatic stage that can move in precise steps and auto-focus the sample in both the visible and infrared spectrum. To illustrate the auto sampling mode, a multi-layer laminate was used (Figure 1).

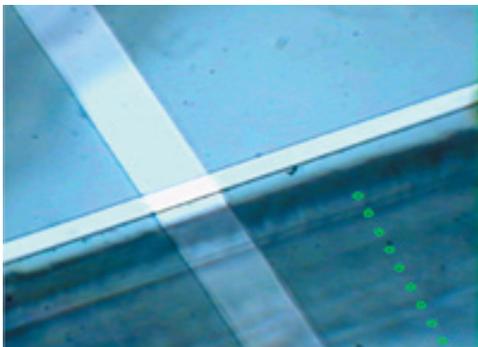


Figure 1. Multi layer laminate with nine points defined. These nine points represent positions on the sample where data will be collected.

The sample was placed in the home position on the microscope and then a series of points to collect were defined. All spectra were collected at 8 cm^{-1} resolution and 64 scans at a scan speed of 20 kHz. This translates to a data collect time of less than 3 minutes for the data set in (Figure 2).

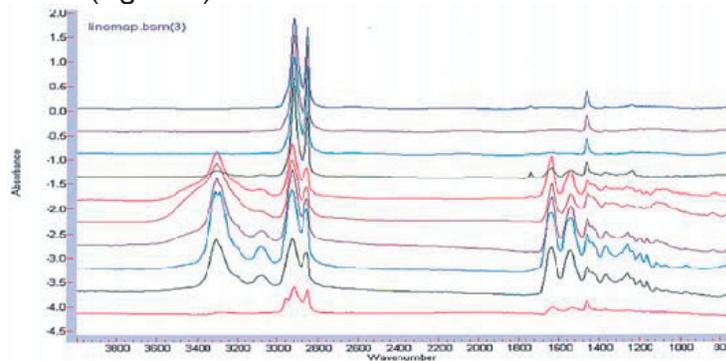


Figure 2 shows the spectra collected from the sample described above. Each individual spectrum exhibits excellent signal to noise as well as differentiation of each of the layers.

Results and Discussion

In order to optimize the analysis of multiple samples, it is desirable to define points on a sample or a sampling substrate and repeatedly analyze different samples using the same set of spatial sampling points. This can easily be accomplished on the UMA 600 using a feature called point templates. Figure 3 shows the dialog associated with these templates.

To use the template, samples are loaded into an IR substrate and positions of the samples of interest are defined. When the samples are changed, the new sample holder is placed in the home position and the point template is recalled. This means that multiple microscopic samples can be analyzed in a single experiment. This allows for an entire series of unattended analyses. Figure 4 illustrates this.

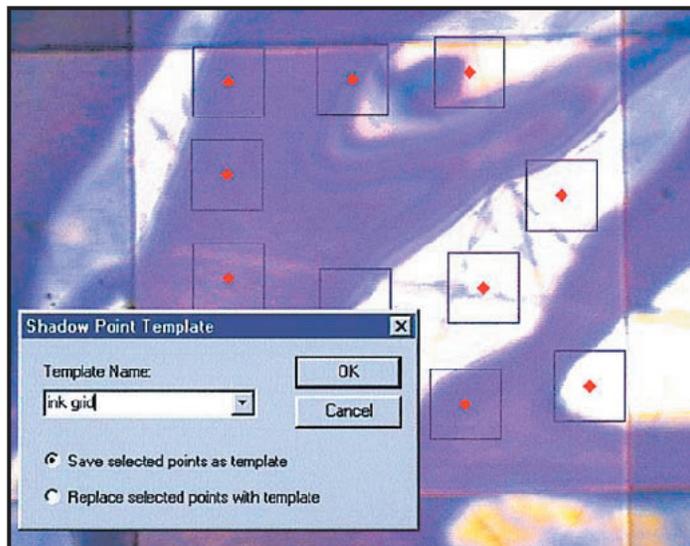


Figure 3. This illustrates the point templates mode. Selected points can be saved into files which can be recalled later to position all points on a new sample.

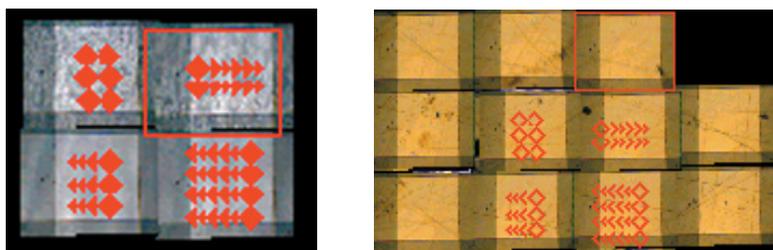


Figure 4. This illustrates the use of a point template. In the left hand figure, the series of desired points were defined on one sample. A new sample was placed on the stage in the home position and the point template recalled. Data collection at all points would then take place unattended.

Conclusion

The auto-sampling mode on the Varian UMA 600 allows unattended analysis of multiple samples. It increases the analyst's productivity and provides time to conduct more analyses.



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