Advantages of the Agilent Cary 610 FTIR microscope

Technical Overview

Advantage statement

With the most flexible area of analysis, unrivalled sensitivity, versatility of experiment types and ease-of-use, the Agilent Cary 610 FTIR microscope reduces measurement time and maximizes productivity without compromising results.

Introduction

The Agilent Cary 610 FTIR Microscope is an infinity-corrected optical system, providing superior sensitivity at high spectral and spatial resolution, fast analyses and maximum productivity. It delivers a selection of measurement modes and provides the complete solution for automated infrared mapping experiments. With many advantages over in-bench FTIR spectroscopic analysis of small (hundreds to ~10 µm) samples, the Agilent Cary 610 FTIR is an essential tool for numerous applications such as polymers and material sciences, pharmaceuticals and biotechnology, electronics and electronic materials, forensics, homeland security and academic research. The Agilent Cary 610 FTIR excels in the four most important characteristics of an FTIR microscope system:

- Area of analysis
- Sensitivity and analytical performance
- Versatility and upgradeability of the system
- Simplicity of sample analysis - “Ease of use”

Area of analysis

Agilent's Cary 610 FTIR Microscope is a diffraction-limited spectroscopic instrument that allows for the investigation of sample features and chemical heterogeneity from sample areas as small as 10 µm. The desired sampling size is attained by adjusting the size of the microscope's field stop aperture prior to spectral acquisition. This effectively focuses the infrared radiation onto the sampling area of interest and eliminates spectral interference from the surrounding areas. Agilent's 'View-Thru' aperture is controllable from the microscope and the PC, which allows for accurate and convenient positioning of the blades. The aperture also enables users to see the entire sample while collecting data from only the area of interest as shown in Figure 1a. The design allows for independent adjustments of the rotational position to any angle as well as for independent adjustments of its length and width, allowing users to match the aperture size and orientation to their samples as shown in Figure 1b.

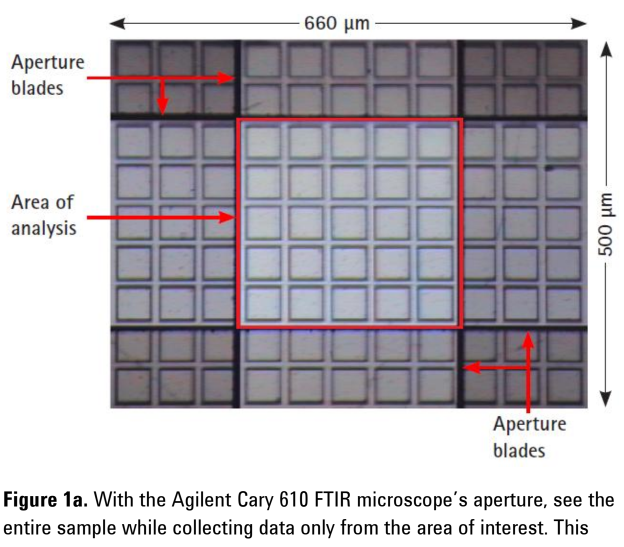

Figure 1a. With the Agilent Cary 610 FTIR microscope's aperture, see the entire sample while collecting data only from the area of interest. This makes positioning easier, ultimately making analyses faster

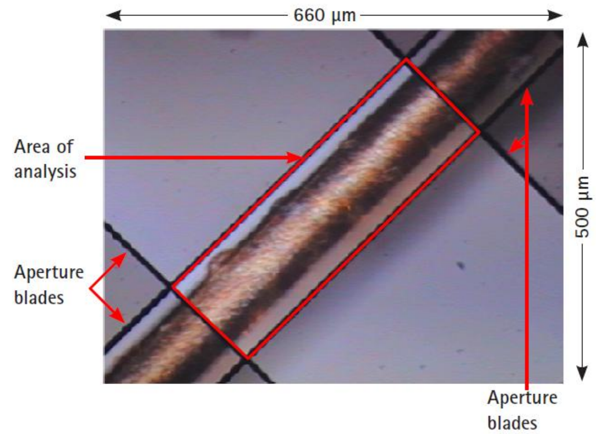

Figure 1b. The rotating aperture and fully adjustable aperture blades in the Agilent Cary 610 FTIR microscope allow you to customize the precise area of analysis to suit your sample, in this instance a human hair. This has application in the forensics market and can be extended to similar types of samples

Sensitivity and analytical performance

One of the most important parameters in selecting and assessing the performance of an FTIR spectrometer and microscope is the sensitivity [as measured by the signal-to-noise ratio (S/N)] provided by the system. An FTIR microscope's spectral performance is dependent on the infrared energy throughput and the optical configuration of the spectrometer to which it is interfaced. Agilent's Cary 600 FTIR spectrometers provide unrivalled sensitivity and analytical performance under real-world measurement conditions.[1,2]

The all-reflective and infinity-corrected optical design of the Cary 610 FTIR microscope has also focused on maximizing performance in both transmission and reflection modes. The open beam S/N ratio of an FTIR system is considered to be a measure of its sensitivity and is often viewed as the primary specification for determining whether or not an instrument will meet the requirements of the intended application. For FTIR microscopy, this S/N ratio value is obtained by sequentially measuring two open beam (single beam) spectra using an MCT detector, then ratioing one spectrum to the other to obtain a baseline at 100% transmission.

The peak-to-peak (p-p) value in a region of the spectrum (typically between 2200–2100 cm^{-1}) is measured as the noise, and divided into 100 (for 100% T) as the signal. Reflection measurements are made with the use of a gold-coated mirror.

Signal-to-noise ratio is a common specification reported by FTIR instrument manufacturers. Table 1 shows typical long term open beam S/N (p-p) values between 2200–2100 cm^{-1} for the Cary 610 FTIR microscope equipped with a narrow-band 250 µm MCT detector using a 100 µm aperture when paired to a Cary 600 FTIR series spectrometer.

Table 1. Typical signal-to-noise (peak-to-peak) [S/N (p-p)] values between 2200-2100 cm^{-1} for Agilent's Cary 610 FTIR microscope when paired with Agilent Cary 600 FTIR series spectrometers. This level of sensitivity is unrivalled—so you don't miss a thing. These values highlight the superior performance of the microscope

Spectrometer and Microscope	Typical long term transmission S/N ratio (p-p)	Typical long term reflection S/N ratio (p-p)
Cary 640 FTIR + Cary 610 FTIR	7000	4000
Cary 660 FTIR + Cary 610 FTIR	9500	5500
Cary 670 FTIR + Cary 610 FTIR	12500	7700
Cary 680 FTIR + Cary 610 FTIR	12500	7700

The spectral acquisition parameters are:

- Detector: MCT
- Source: MIR (normal mode)
- Long term: 120 s
- Spectral resolution: 4 cm^{-1}
- Source aperture: 2 cm^{-1}

It should be noted that by convention, FTIR instrument manufacturers report the S/N values of a microscope MCT detector while using a 100 µm aperture (even on a 250 µm detector). As such, the 250 µm detectors are significantly under-filled when the performance tests are conducted, which lowers the detector's achievable S/N values. If the aperture size was matched to the detector format (that is, a 250 µm aperture with a 250 µm MCT detector, or a 100 µm aperture with a 100 µm MCT detector), then the values would evidently increase.

Another factor that can impact on the sensitivity of a system when investigating very small samples is the size of the detector element that is mounted on the microscope. The Cary 610 FTIR's detector elements are available in both 250 × 250 µm and 100 × 100 µm formats, and the microscope can be configured with one or two single-element detectors. Selection between the two formats is typically based on a user's current and potential applications. The 250 µm detector is the most popular selection as it allows for the most flexibility in sampling size for single-element FTIR microscopes, is well-suited to a multi-user environment, and it provides excellent spectral performance. The 100 µm format is the detector size of choice for dedicated applications that involve the investigation of samples that are ≤100 µm, as it provides superior sensitivity (highest signal-to-noise ratio) for such samples in a shorter period of time than the 250 µm format requires because it has proportionally less contribution from noise, as illustrated in Figure 2.

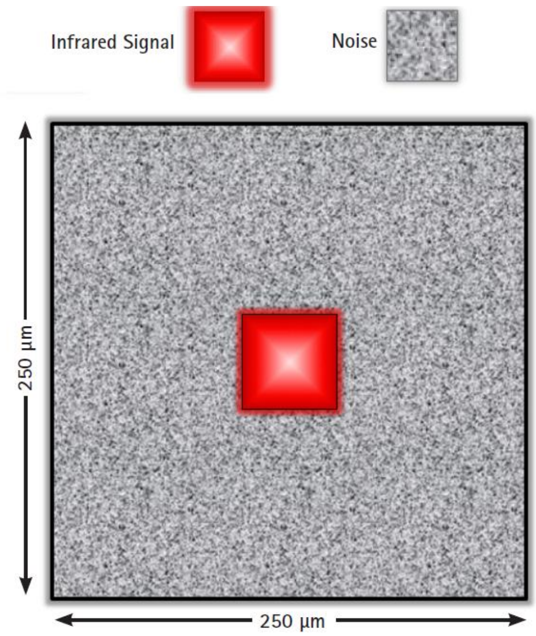

Figure 2a. 250 x 250 µm square MCT detector element

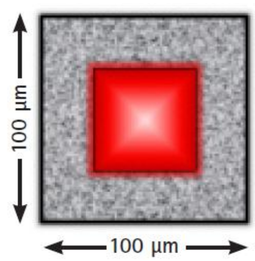

Figure 2b. 100 x 100 µm square MCT detector element

In practice, the Cary 610 FTIR's spectroscopic performance will reduce the overall time of analysis as well as provide the ability to detect extremely low concentrations of challenging samples that could otherwise be missed when using conventional FTIR analysis.

Versatility and accessories

The third important characteristic that must be considered when selecting an FTIR microscope is the overall versatility of the system. With the cost of current analytical instruments, flexibility and upgradeability translate into cost-savings over the life of the equipment. The Agilent Cary 610 FTIR is built on a foundation to optimize optical performance and provide maximum versatility. Infinity-corrected optical systems are designed to have the focal point at infinity for higher precision (sharper focus) in both visible and infrared analyses by collimating the two beams within the microscope. All of Agilent's Cary 600 FTIR microscopes are infinity-corrected to ensure maximal versatility.

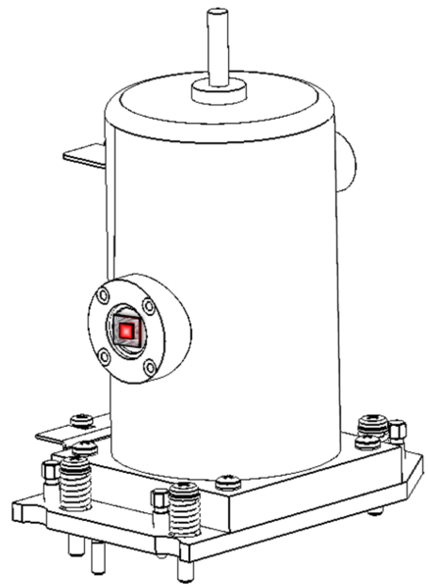

Figure 2c. Element location on an MCT detector

Figure 2. Choose a Agilent Cary 610 FTIR detector element size perfect for your application. Above shows projected images of (Figure 2a) a 250 × 250 µm, and (Figure 2b) a 100 × 100 µm MCT detector element that are both acquiring data from a sample area of 50 µm.
Legend: Red square = infrared radiation; Grey square = non-illuminated detector area (this area is a prime contributor to detector noise). Given an equivalent period of time for spectral collection, detector element B will exhibit better S/N performance than detector element A for this small spatial resolution. Figure 2c shows the location of the detector element on a MCT detector

Advantageously, this enables an extensive range of objectives and accessories to be paired to this optical configuration in order to suit all applications. The Agilent Cary 610 FTIR microscope can include: 4×, 10×, 20×, 40× visible objectives, 15× visible and infrared objectives, large sample microscope objectives, fixed or rotatable visible and IR polarizers, custom-made objectives, as well as grazing angle objectives. The use of a grazing angle objective is ideal for the analysis of ultra thin films (such as monolayers) as it provides increased sensitivity and an improved detection limit over other microscope objectives.

This objective increases the angle of incidence of the infrared radiation to 75 ° nominal, thereby increasing the sampling pathlength and absorbance as illustrated in Figure 3.

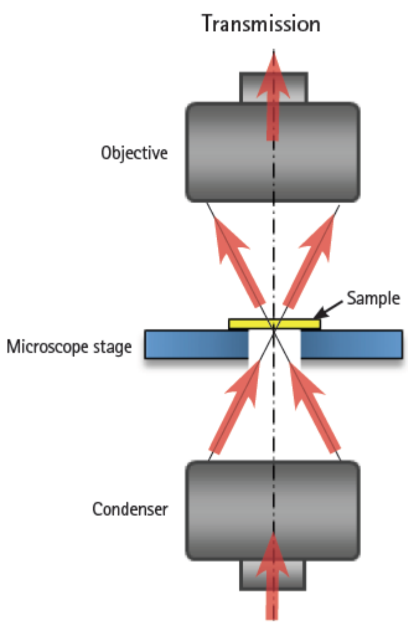

Figure 3a. Transmission analysis

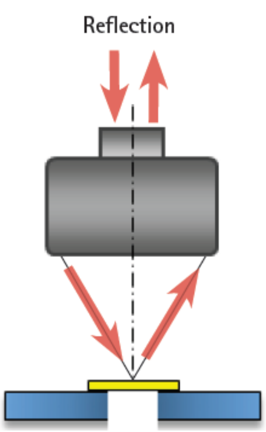

Figure 3b. Reflection analysis

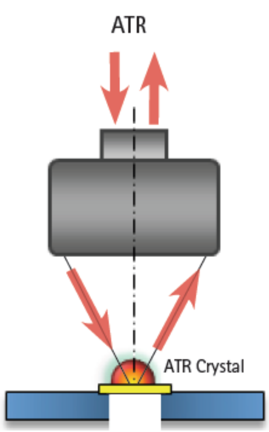

Figure 3c. ATR analysis

The Cary 610 FTIR microscope's versatility results in the ability to acquire high quality infrared spectra in transmission, reflection, and attenuated total reflection modes. This flexibility provides spectroscopists with options for different experimental configurations for the analysis of challenging samples and increases the practicality of FTIR microscopy. In addition, all of Agilent's microscopes have a multi-position turret upon which multiple objectives can be connected simultaneously.

Resolutions Pro software is capable of displaying a live, fully processed infrared spectrum from a Cary 610 FTIR in all modes of spectral acquisition before and during infrared data collection. This provides immediate feedback about the sample's chemistry, and allows users to monitor and modify several parameters in real-time, such as the interferogram signal level, or the single beam spectrum — all prior to spectral data collection. For analysis in micro-ATR mode, this software feature ensures that users have a detailed understanding (in real-time) of when contact is made with a sample, allowing users to optimize the ATR's contact pressure for all sample types.

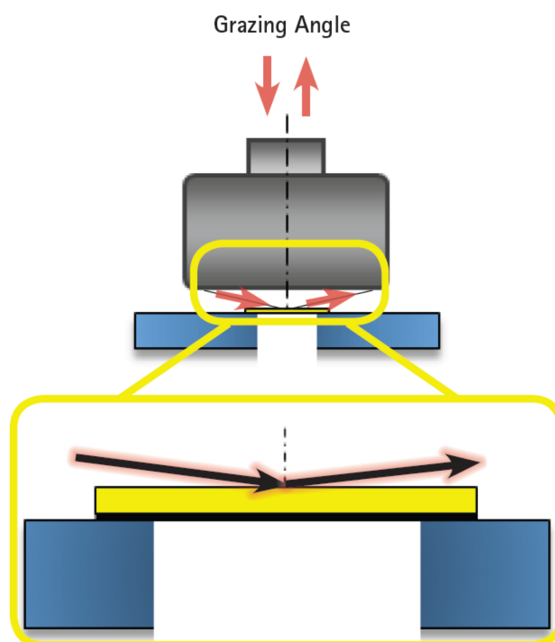

Figure 3d. Grazing angle analysis

Figure 3. For analyzing challenging samples, simply adjust the measurement mode on the versatile Agilent Cary 610 FTIR. Modes include: transmission, reflection, ATR and grazing angle reflection analysis

Micro-ATR analysis using Agilent's Cary 610 FTIR is very common due to the simplicity of the technique as well as its numerous advantages derived from the hardware and software. It is a non-destructive method that typically requires little or no sample preparation. In most experiments the sample is simply raised into contact with a crystal element. This accessory has been specifically designed to be robust, easy-to-use, and is ideal for a multi-user environment. The slide-on design enables easy cleaning and visual inspection of the crystal between samples and the software allows users to monitor for contamination. In addition, the design does not require the purchase of a separate dedicated objective (as is the case for other microscope systems).

Agilent offers the complete range of slide-on micro-ATR crystal types, such as germanium and diamond crystal sampling accessories, to match all types of samples. The selection of crystal type is based on the scope of applications as each crystal has specific advantages. Germanium is the most common general-purpose material as it has a high refractive index (making it suitable for a wider range of material types), it provides excellent throughput, and is relatively inexpensive. Diamond has distinct advantages for the analysis of hard solids and is the most physically and chemically robust material. Figure 4 highlights the simplicity of using the Cary 610 FTIR's germanium ATR for QC during the manufacturing of a polymer tube product.

The slide-on ATR accessory allows users to harness the Cary 610 FTIR microscope's performance and sensitivity advantages while maintaining very simple sample preparation protocols. It is an ideal tool for surface analysis as the depth of penetration of infrared radiation into a sample is very small (typically less than one micrometer).

A system's flexibility is equally evident from the selection of components that are available, such as the infrared detectors.

The Cary 610 FTIR microscope can be configured with high-sensitivity narrow band (10000–700 cm^{-1}), mid band (10000–600 cm^{-1}) and broad band (8000–450 cm^{-1}) MCT detectors for mid-IR analysis or indium antimonide (11000–1800 cm^{-1}) and silicon (25000-8600 cm^{-1}) detectors for near-IR and visible analysis.

Notably, the system provides the ability to upgrade the Cary 610 FTIR to the Agilent Cary 620 FTIR in the user's laboratory. The Cary 620 FTIR is a focal plane array-based infrared chemical imaging system, which allows for the simultaneous collection of hundreds to thousands of spatially-resolved infrared spectra from a large two-dimensional area and provides a chemical 'snap-shot' of a sample, without the need for a motorized stage to move the sample. It provides the ultimate in experimental control, is the most sensitive, and is the fastest available chemical imaging system. The microscope's stage can also provide the system with significant experimental flexibility. The stage can be configured either as a standard manual stage that allows for high precision positioning or as a motorized version, which enables infrared mapping functionality (as discussed in detail below). The sample focus can be adjusted both manually and automatically (through coarse, fine, and high-precision adjustments), as selected by the user. Moreover, Agilent's FTIR microscopes have the largest working distance (25 mm) and the widest assortment of standard and customized sample holders and liquid cells fit on the stage. The microscope's stage is also available with precision heating and cooling sample stages that allow for the dynamic investigation of samples across an extensive temperature range (-196 °C to 1500 °C).

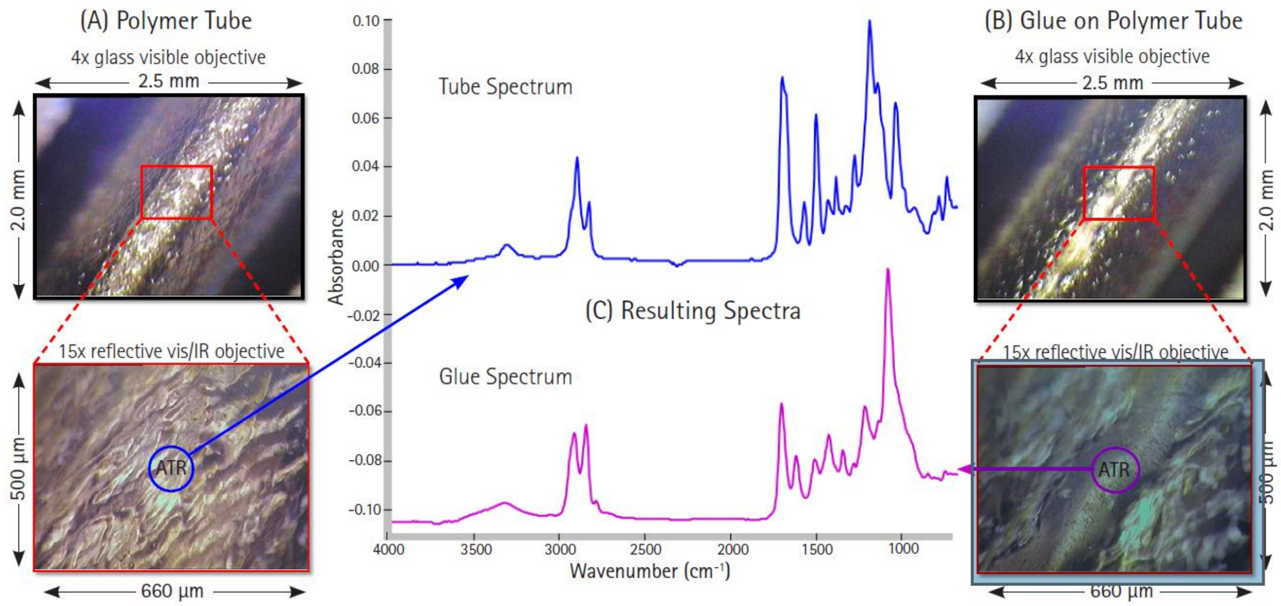

Figure 4. Visible images from the micro-ATR analysis of (A) a polymer tube material, (B) an adhesive glue product that had overflowed onto the polymer tube, and (C) the resulting infrared spectra obtained in only 5 seconds. Sampling areas of interest were visually identified then simply raised into contact with a germanium slide-on ATR (using the microscope's stage) in order to obtain high quality spectral data that allows for QC and product troubleshooting in the polymer and materials markets

Ease of Use Features

Simplicity is built-into the Cary 610 FTIR microscope's hardware and software to provide the user with full control for increased efficiency. The convenience of the microscope stems in part from the hardware features that have been implemented. The unique control panel provides access to all common actions for spectral collection, including: dynamic control of the aperture, the visual illumination, the ability to change from transmission to reflection and ATR analysis with one click, and the ability to collect visual images and infrared spectra to be used as backgrounds or sample scans. The microscope also incorporates internal purge connectors, and the microscope's stage and substage condenser have purge collars to ensure the highest quality spectral data by minimizing the inclusion of carbon dioxide and water vapor around the sample holder.

Unlike other FTIR microscopes, all of Agilent's systems have binoculars (fixed or tiltable) and an internally-mounted camera to allow for the acquisition of both high-quality pictures and videos (in color or black-and-white) in real-time through the system's software. The video camera allows users to automatically acquire an image of a sample prior to infrared spectral collection. In addition, the microscope system allows for the simultaneous viewing of the sample in visible mode through the binoculars and internal visible camera, allowing users to monitor the sample through the software while positioning it on the stage. The Cary 610 FTIR is also equipped with an ultra-bright white LED for visual illumination of samples, which ensures fast setup and the highest quality sample visualization, even with (thick, dark or poorly reflective materials), for increased confidence in results.

The versatility of Resolutions Pro software and its ease-of-use, make FTIR microscopy accessible to users of all levels of experience. It provides the ability to create application-specific methods to simplify common experiments. A method can be summarized as a compilation of all spectral and instrument parameters (such as the number of scans, number of background scans, spectral resolution, scan type, wavenumber range and apodization) that are required to collect an infrared spectrum. A method can also include post-collection operations such as: baseline correction, derivatization, peak picking, spectral searching and quantitative predictions. Users do not need to input values for individual parameters or have expertise in FTIR microscopy in order to collect high-quality spectra. In Resolutions Pro software, the user selects the appropriate method with a single click of the mouse and all parameters load simultaneously. Methods can be created for any application to simplify routine experiments, and can be modified and saved to suit standard operating protocols. As in conventional FTIR analysis, the software provides complete access to all raw data for fast data reprocessing and generation of infrared spectra with different pre-processing, eliminating the need to recollect data, saving valuable time and money. The software is also designed to display visible images that are associated with spectra and to maintain an un-editable record of all spectral collection and processing parameters, thereby eliminating the possibility of losing data of interest. The software also has simple-to-use microscope performance tests included as standard, allowing users to test the performance of their system at desired intervals, thereby increasing overall confidence in the results.

FTIR Mapping

Infrared mapping is an extension of FTIR microscopy, that involves combining a single-element detector microscope with a motorized sample stage to allow for raster scanning and automation of FTIR microscope experiments. The use of mapping in spectroscopic applications allows for multiple infrared spectra to be sequentially acquired from different spatially-resolved points on the same sample and provides both spectral and spatial information, thereby enabling the study of within-sample chemical heterogeneity. FTIR mapping is an excellent means of probing a sample's chemistry as it can be used to visualize the relative distribution of specific components across a sample area of several centimeters.

The Cary 610 FTIR microscope allows for fully automated mapping analysis of large sample areas in transmission, reflection, and ATR modes. Using Resolutions Pro, mapping experiments are extremely flexible. Users can either select specific spectral collection locations themselves or use one of several grid mapping templates that can be customized to a sample, saved and re-applied at a later time. The user has the ability to define, among other parameters, the field to be mapped, the spacing between spectral acquisition locations, as well as the aperture size. Figure 5 is an example of an infrared mapping experiment in which a user defined the locations of spectral acquisition.

The output of a mapping experiment is a series of correlated visual images that indicate the location of each spectral collection and the resulting infrared spectra (represented by hollow green circles on the visual images in the software). Selecting a single green circle in the software will display the corresponding spectrum and will fill in the circle. Selecting multiple green circles within the large visible image (as shown in Figure 5) will display multiple spectra simultaneously to facilitate interpretation. Resolutions Pro software has many features that facilitate the interpretation of mapping experiments, such as the ability to create chemical contour maps overlaid on visual images of the sample(s). This feature allows users to visualize the spatial distribution of a specific functional group (for example, a carbonyl) or of multiple spectral peaks (for example, a range of 1400-1250 cm^{-1}) across the corresponding mapped sample area to examine chemical heterogeneity in 2-D or 3-D views. Chemical maps are color-coded for intensity and the output is based on a peak's spectral intensity. The software also allows for spectral comparisons or spectral searching/matching to in-house or commercial databases in order to extract additional structural information. In the case of the 100 yuan Chinese note in Figure 5, this allowed for the confirmation that the inks and printing materials had the correct chemical composition.

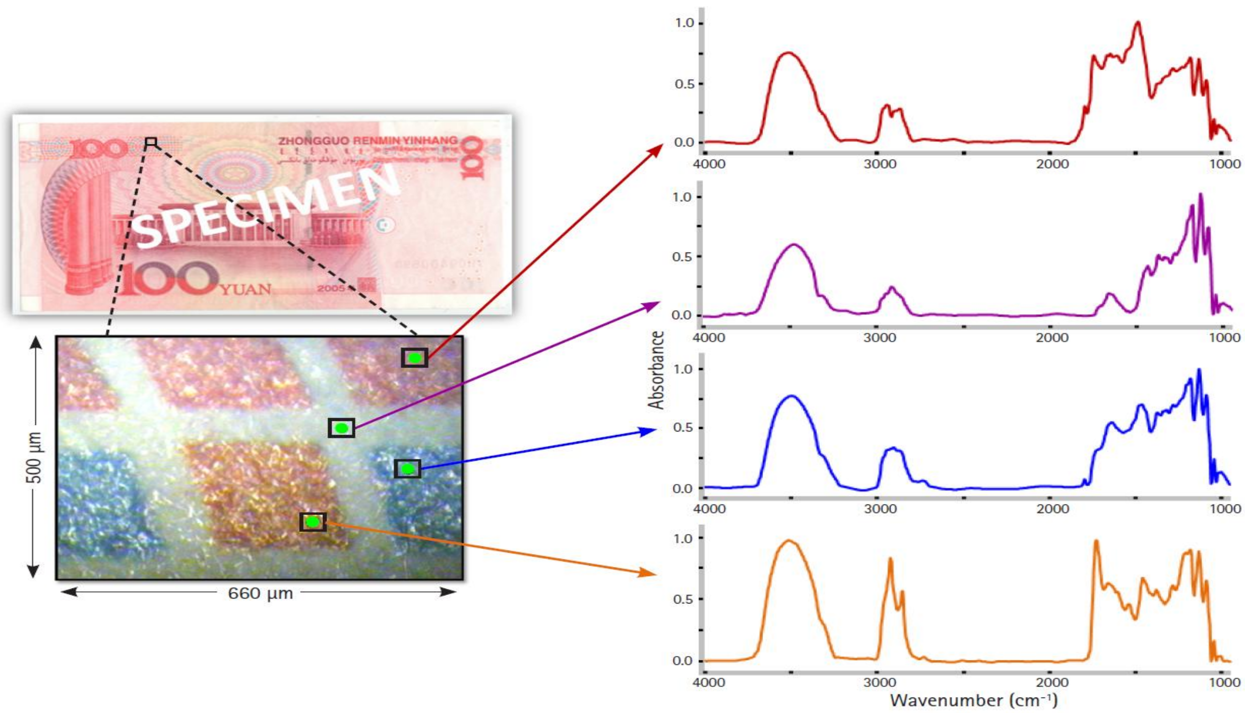

Figure 5. Visual images of a 100 yuan Chinese note and FTIR spectra resulting from the infrared mapping of different areas on the bill. The chemical composition of the inks and printing materials were investigated using a spatial resolution of ~35 µm (designated by the black squares that contain green circles on the zoomed visible image). The mapping feature allowed for the unattended collection of dozens of spectra from user-defined locations across several inches of the bill in order to confirm that the currency was not counterfeit. Applications extend across the forensics, chemicals and polymers markets

Summary

The Agilent Cary 610 FTIR microscope is the highest performing, most versatile and simplest to use FTIR microscope available on the market. It covers a wide spectral range and offers the complete solution from single point analysis, to dual-single point analysis, to infrared mapping, allowing users to customize the exact configuration for their needs.

References

1. Agilent Technologies, Inc. 2008. Optimized FTIR spectrometer design for optical performance. Technical Overview SI-1335. Available at: http://www.agilent.com

2. Agilent Technologies, Inc. 2008. Highest available signal-to-noise performance, delivering superior sensitivity and analytical performance. Technical Overview SI-1353.
Available at: http://www.agilent.com

www.agilent.com/chem

Published March, 2011
Publication Number SI-02288

Agilent Technologies