FTIR chemical imaging using focal plane array–based systems

Technical Overview

Advantage statement

The Agilent Cary 620 FTIR imaging microscope is in a class of its own. Designed for ultimate performance, it provides comprehensive pixel resolution and field of view options, combined with the highest sensitivity and fastest collection times. These core capabilities of the Cary 620 FTIR allow you to solve your analytical challenges in a variety of application areas, including polymers and materials, biological and biomedical, or chemicals and petrochemicals quickly, accurately and easily.

Introduction

Focal Plane Array[1] (FPA) detectors allow for the simultaneous acquisition of an n by n number of spatially resolved spectra (where n = 16, 32, 64 or 128 in the detector array), as each pixel provides an independent infrared spectrum. By simultaneously acquiring thousands of spectra within minutes, FPA detectors provide information about the identification and concentration of specific compounds and their distribution in the measured field of view. See Figure 1.

The advantages of using a Agilent chemical imaging system over other commercially available FPA–based systems include:

- largest field of view (from 4 to 16 times larger areas)
- a calibrated live infrared image for optimum micro–ATR contact and better sample viewing without damaging the surface of the sample
- superior sensitivity and analytical performance
- the greatest degree of experimental flexibility and overall simplicity
- powerful and robust software

All of these advantages are required for advanced infrared imaging experiments.

Agilent Technologies

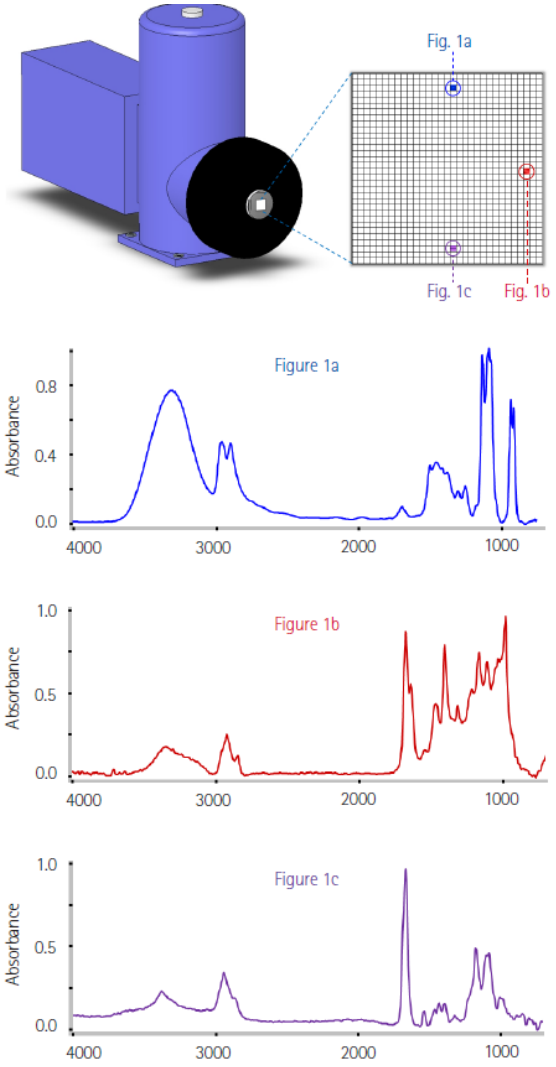


Figure 1. illustrates the identification of three distinct chemical species (Figs. 1a, 1b and 1c) through their corresponding infrared spectra at three spatially different locations within the same image

Agilent provides the complete solution for chemical imaging — the Cary 620 FTIR for micro–imaging and the Large Sample accessory (LS) for macro–imaging.

Options include:

For the 620–IR microscope:

- micro–imaging
- micro–imaging with field expanding optics (FEOs)
- micro–imaging with mosaicing
- micro–imaging with an attenuated total reflectance (ATR) accessory

For the LS accessory:

- macro–imaging
- macro–imaging with an ATR accessory

Key benefits

Largest field of view: significant time saving

The optical configuration of Agilent's Cary 620 FTIR imaging microscope provides advantages for the rapid investigation of large areas of analysis in a short period of time. The field of view (FOV) of an FTIR imaging system is the area that is measured by the focal plane array during one analysis. It is governed by the overall magnification of the system and the size of the imaging detector.

Each optical configuration provides a unique FOV and pixel resolution, creating distinct advantages for a range of applications. For example, a larger FOV is particularly beneficial for applications involving defect analysis or those where the visible appearance may not provide an accurate guide to the required area of infrared analysis. In these cases, fast, high quality, high spatial resolution chemical images may become vital to a practical solution by increasing the chances of finding defects within the area of analysis.

The FOV (i.e. the sampling area that is simultaneously analyzed) is of prime importance in the selection of an infrared imaging system because it is critical to the system's overall productivity. Figure 2a shows the sample area that is covered in one scan at high pixel resolution mode with Agilent's 128 × 128 FPA detector (corresponding to 16,384 spectra), while Figure 2b shows the sample area that can be covered with a 128 × 128 FPA detector by another FPA–based system under the same collection conditions. With Agilent's chemical imaging system, you can see more due to the superior FOV.

Larger fields of view provide more chemical information. Agilent's Cary 620 FTIR systems have four times the FOV of other commercial available systems, giving users four times better productivity.

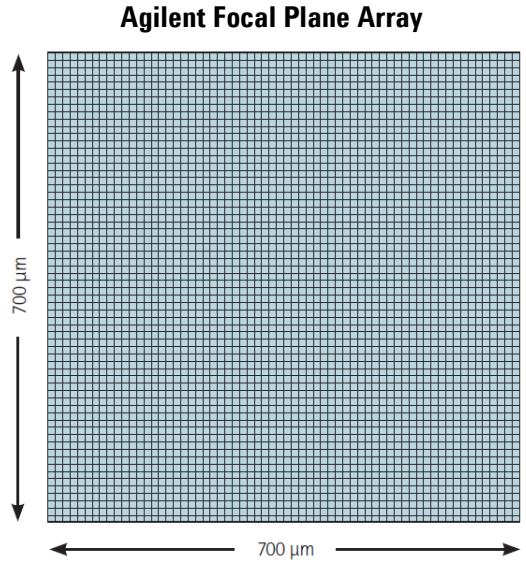


Figure 2a. Up to 700 x 700 microns measured in one single scan in high spatial resolution mode using Agilent's 128 x 128 FPA detector

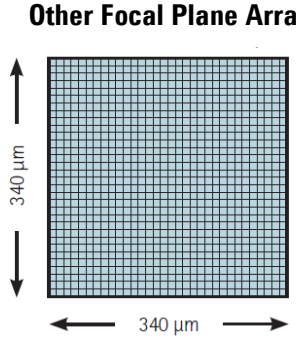


Figure 2b. Up to ~340 x 340 microns measured in one single scan in high spatial resolution mode using other 128 x 128 FPA detectors

Agilent's FOV advantage equally applies to micro–imaging with an ATR accessory, saving significant analysis time. For example, in one scan Agilent's 64 × 64 FPA detector in micro–ATR imaging mode will cover an area of 70 × 70 µm, while other FPA–based systems can only collect data from ~35 × 35 µm.

Some experiments may not need a high pixel resolution mode of 1.1 µm (micro–ATR mode) or 5.5 µm (reflection or transmission) for image acquisition. Rather, they may benefit from the use of field expanding optics (FEOs) which quadruple the field of view and enable data collection in a medium pixel resolution mode (11 µm).

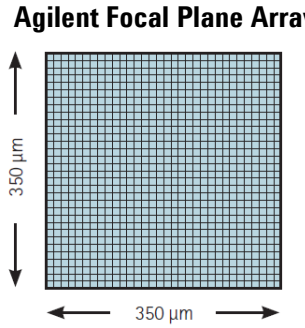


Figure 3a. When not using FEOs, Agilent's 64 x 64 FPA simultaneously measures 350 x 350 µm

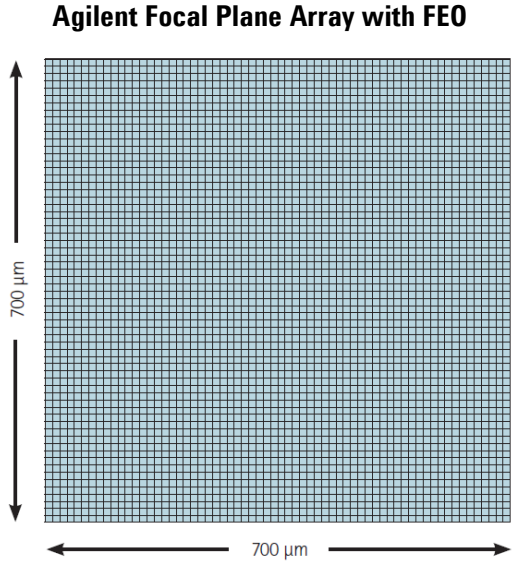


Figure 3b. Using FEOs, a 64 x 64 FPA measures 700 x 700 µm, significantly increasing the area of analysis

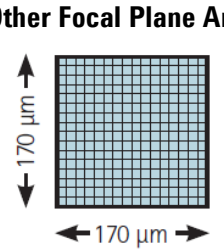


Figure 3c. Lack of FEOs means other 64 x 64 FPAs can only analyze ~ 170 x 170 µm

Exclusively available in Agilent's imaging system, FEOs are a convenient user–selectable method of quadrupling the area of analysis — this effectively provides a 16 times larger area of analysis than other equivalently sized FPA–based systems.

Figure 3a shows how Agilent's 64 × 64 FPA detector, operated under 'normal (high pixel resolution)' mode, will generate a chemical image from an area of 350 × 350 µm. Figure 3b shows how the same 64 × 64 FPA detector, operated under 'Field Expansion (medium pixel resolution)' mode, will generate a chemical image from an area of 700 × 700 µm in which each FPA pixel has a pixel resolution of 11 × 11 µm. The area of analysis covered in the same amount of time with a 64 × 64 FPA of other FPA–based systems is 16 times smaller as shown in Figure 3c.

FEOs are included as standard with Agilent's imaging systems. They are not available on other FPA–based imaging systems and allow users to analyze larger samples in significantly less time, while also increasing the signal–to–noise performance.

A summary of the most common dimensions of FPA detectors used in micro–imaging applications is shown in Table 1. By increasing the number of pixels in a focal plane array detector, the field of view increases as illustrated in the table.

Table 1. Common dimensions and area of analysis of FPA–based imaging systems

FPA Type	Array Size	Number of Spectra in One Scan	Area of Analysis (µm) in transmission or reflection mode in one scan	
			High Resolution	Medium Resolution (with FEOs)
Agilent FPA	16 x 16	256	88 x 88	175 x 175
Agilent FPA	32 x 32	1024	175 x 175	350 x 350
Agilent FPA	64 x 64	4096	350 x 350	700 x 700
Agilent FPA	128 x 128	16384	700 x 700	N/A
Other FPA	16 x 16	256	N/A	N/A
Other FPA	32 x 32	1024	85 x 85	N/A
Other FPA	64 x 64	4096	170 x 170	N/A
Other FPA	128 x 128	16384	340 x 340	N/A

Better Micro–ATR analysis and sample viewing

Agilent's micro–imaging with an ATR (Attenuated Total Reflectance) accessory is another very powerful mode of image collection with distinct advantages.

Micro–ATR imaging provides a significant enhancement in spatial resolution, over and above the diffraction limited spatial resolution in air. A significant benefit of ATR imaging is the fact samples may be presented 'as is', often requiring little or no sample preparation.

Agilent is the only FPA supplier to conduct a calibration on the imaging detector. This simplifies the step of making contact between a sample and the ATR crystal, and allows users to visualize the sample in the infrared before data collection and at the exact moment of ATR contact with the sample. This ensures that the appropriate pressure is applied to the sample, ensuring no deformation or damage in the case of sensitive samples. The ability to monitor the live infrared image in real–time provides direct and immediate feedback on the quality of crystal contact, which is the single most important experimental control point in ATR analysis. This feature is particularly important for the analysis of soft and precious samples, as well as those that may not be perfectly flat. Other FPA–based systems use a coarse 'pressure alert' feature which has the drawback of only providing indirect feedback through monitoring the pressure applied, without providing any information on the quality of contact. This can compromise data quality, lead to sample damage, and result in increased collection times.

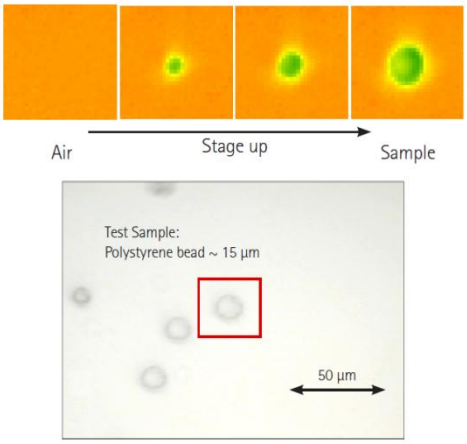


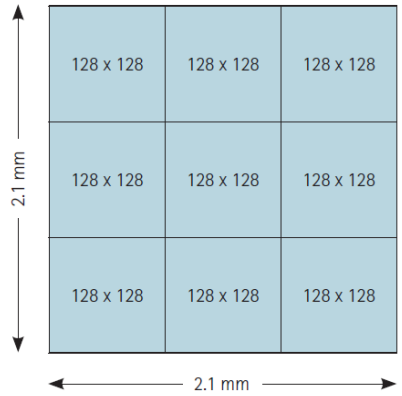
Figure 4. In addition to covering an area four times larger than other FPA–based systems, Agilent's micro–ATR is the only one that provides a live calibrated image to allow users to visually monitor the contact between their sample and the ATR crystal in real–time. In this example, the microscope's sample stage is raised to bring a 15 µm polystyrene bead into contact with the ATR. Selected images captured during the real–time contact monitoring show how Agilent's powerful calibration software simplifies micro–ATR imaging analysis.

Superior sensitivity and analytical performance

The sensitivity afforded by an infrared imaging system is of prime importance as it is fundamental to the quality of the data and the overall utility of the technique. The sensitivity of imaging systems can be compared in terms of analytical performance based on signal–to–noise values, effective area of analysis, and overall collection time required while operating under realistic and typical conditions that a majority of users are likely to employ. The collection parameters (number of co–added scans, spectral resolution, pixel resolution, and collection area) have a significant impact on the overall time of collection and the quality of the data.

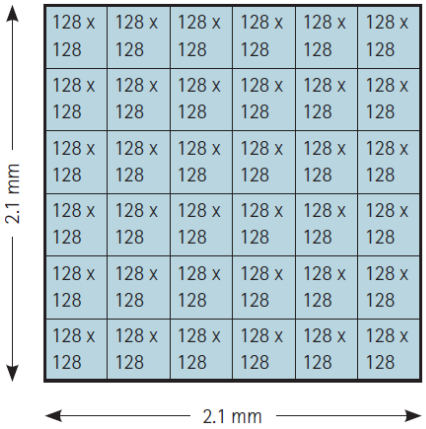
The overall analytical performance of several imaging spectrometers can be evaluated by measuring an identical sample under fixed parameters such as a fixed area of ~2 × 2 mm. Using real–world conditions (16 scans, 4 cm^{-1} resolution, high pixel resolution) to analyze the relatively small area of 2.1 × 2.1 mm, Agilent's 128 × 128 FPA–based system only requires ~7 mins., whereas other 128 x 128 FPA–based systems require ~28-30 mins. under equivalent conditions. This simple comparison, shown in Figure 5, highlights the clear advantage of using Agilent's chemical imaging system when acquiring spectra from a large area of analysis at a high spatial resolution. In addition, Agilent is the exclusive supplier of 3rd generation FPA detectors, which possess superior performance characteristics that make them more robust than other FPA detectors.

Agilent FPA System



9 tiles required = 3 x 3 mosaic, Total collection time is ~ 7 minutes

Other FPA System



36 tiles required = 6 x 6 mosaic, Total collection time is ~ 28-30 minutes

Figure 5. Agilent's chemical imaging systems exhibit superior analytical performance combined with the highest data quality, allowing for large area analysis at a high spatial resolution in a shorter period of time. Advantageously, Agilent's mosaic software can collect data from areas that are both n × n and n × m in size (i.e. square or rectangular areas), allowing users to fully customize their spectral acquisition areas to their individual requirements.

Analysis flexibility and simplicity

Agilent is unique among chemical imaging system suppliers to have developed an infinity–corrected microscope providing features that cater for the unique needs of an imaging microscope. 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. The design ensures maximal versatility, enabling the integration of an extensive range of 'off–the–shelf' visible high magnification objectives, such as those from Olympus, and accessories such as polarizer filters or trinoculars for the fitting of high resolution dedicated visible cameras. In addition, Agilent's systems offer a comprehensive range of detector and sampling options including the 'Large Sample Microscope Objective' for use with infinitely large samples that do not fit under the microscope objective. Essentially, there is no upper limit on sample size and may include samples such as helmets, helicopter blades, automotive materials and components such as steering wheels and bumper bars, liquid crystal displays, pharmaceutical packaging materials, artwork and paintings, etc.

Additional flexibility in Agilent's FPA–FTIR imaging spectrometers is imparted through selectable hardware options and pixel resolution modes, including: ultra–high (ATR analysis 1.1 µm), high (5.5 µm), medium (11 µm), low (22 µm) and even larger sizes with macro–imaging (e.g. >40 µm), as shown in Figure 6.

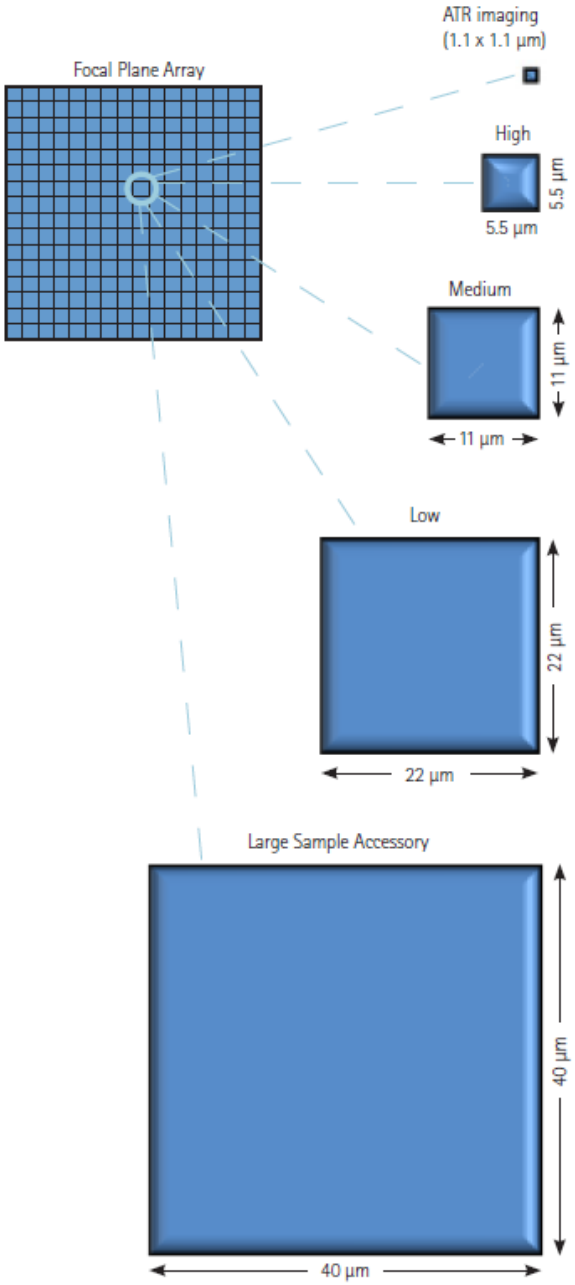


Figure 6. The comprehensive range of pixel resolution options of Agilent's chemical imaging system allows users to tailor the spectrometer's configuration to their application and to extract the desired chemical information

Although other FPA–based systems may report smaller pixel resolutions, the wavelengths in the mid–IR are typically much larger than the claimed resolution. For instance, given that most infrared microscopes are diffraction–limited and wavelengths in the fingerprint region of the mid–IR spectrum range from ~5 to 10 µm, the achievable spatial resolution cannot be better than this, irrespective of pixel resolution. As such, small pixel resolutions are simply 'empty magnification' of the optical design and are attained at the expense of the field of view. Agilent's chemical imaging system provides the best balance between the field of view options and the pixel size.

Powerful and robust software

Simplicity is built into the Cary 620 FTIR microscope's hardware and software to provide users with full control and increased efficiency. The versatility of the Resolutions Pro software and its ease of use make FT–IR imaging accessible to users of all levels of experience. Its robustness is evident from data acquisition through to data processing. For example, network cards can be enabled during image collections, and there are no specific restrictions with respect to the use of antivirus software. In addition, the PC can still be used during imaging data collection. The software's advanced capabilities include mosaic options to extend the field of view for unlimited image sizes and easy data exploration features such as 'Play', 'Extract' and 'Image peak' for rapid data analysis. Resolutions Pro's sophisticated post–run analysis capabilities also enable users to easily visualize the distribution of components in an image — useful as a quick check of a sample's heterogeneity. Furthermore, it provides a comprehensive range of spectral transformations, full access to data history (including raw interferogram data), and the ability to rapidly export images into many file formats for direct transfer to third–party and dedicated data interpretation packages if desired.

Summary

The Agilent Cary 620 FTIR Microscope is the highest performing, most versatile, and fastest chemical imaging system on the market. Its advanced optical design ensures maximum analytical sensitivity and flexibility.

References

[1] This product is regulated by the U.S. Department of State under the International Traffic in Arms Regulations, 22 CFR 120-130 ('ITAR'). An export license from the U.S. government is therefore required to export this product from the United States, and other ITAR restrictions apply to the shipment, use, service and other aspects of this product and the FTIR instrument in which it is used.

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