Optimized FTIR Spectrometer design for optical performance

Technical Overview

Advantage statement

The design of Agilent Cary 600 FTIR Series spectrometers focuses on performance. This is achieved through innovative optics design, with an emphasis on optical throughput. The result is an FTIR series that offers unrivalled sensitivity and performance. A larger footprint is required, but this is a negligible compromise for the customer, given the vast increase in productivity and profitability achieved by quickly producing results of the highest available accuracy and precision.

Introduction

There are a number of different Fourier transform infrared (FTIR) spectrometers on the market, each of which is designed to achieve different goals. Some spectrometers are tailored to have a small size, to minimize their footprint in the laboratory, while others are designed for a low price, to reduce the purchase cost. Moreover, some spectrometers are designed for optical performance to allow the user to make measurements with high sensitivity and obtain the highest quality data.

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Each of these design approaches involves trade-offs that affect the ultimate performance of the spectrometer. A design focused on reducing the spectrometer's footprint requires the use of small optical components and leads to lower optical throughput and lower sensitivity. Designing a spectrometer to simply attain a lower cost requires the use of materials that may compromize its ruggedness and durability. Designing for performance offers the most advantages to the spectroscopist, however it typically requires the selection of components and materials that carry a higher cost, and results in a larger footprint. All Agilent FTIR spectrometers are designed for the last mentioned purpose — to maximize optical throughput and deliver the highest performing instruments to the market.

This paper will provide details about the design of Agilent FTIR spectrometers, describe how these design choices affect performance and benefit the user, and compare these design decisions to those of lower performance spectrometers.

Infrared source design and optical configuration

The start of a spectrometer's optical path is the infrared source. Most FTIR spectrometers use furnace igniters as their source of infrared radiation. These igniters are commercially available and manufactured in large quantities, thus they are relatively inexpensive to buy. They are composed of ceramic wires or rods that are resistively heated to a temperature of around 1500 K. At this high temperature, the ceramic elements emit infrared radiation as described by the classical black-body calculation shown in Figure 1.

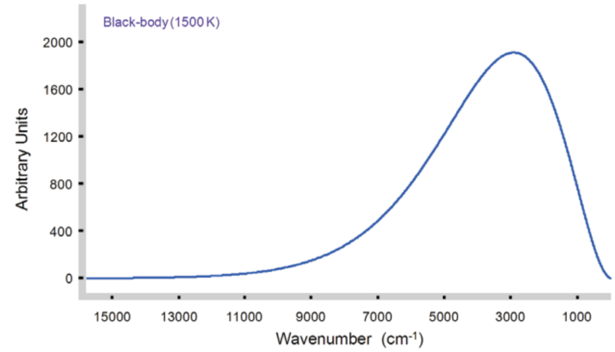

Figure 1. Theoretical emission curve of a black-body emitter calculated per unit wavenumber at 1500 K

Infrared sources can be divided into two categories; they are either relatively small and air-cooled, or larger and water-cooled. As the name suggests, the excess heat generated by an air-cooled source can be dissipated into the air that surrounds the source in the spectrometer. A water-cooled source draws more electrical power, and generates so much excess heat that it must be surrounded by a water-cooled metal envelope. The larger source image generated by the water-cooled source leads to higher total infrared emission, which can then be collimated through the interferometer and re-imaged on the sample. Until recently, a water-cooled source allowed more infrared power to be delivered to the sample than a common air-cooled source. The disadvantage of a water-cooled source is the extra burden that it puts on utilities to support the FTIR spectrometer; namely, a cooled water recirculator.

An ideal design would provide for an air-cooled source to have the same infrared output and image size as a water-cooled source. This can be achieved through optimized source optics designed to incorporate a retro-reflector behind the source element, as illustrated in Figure 2.

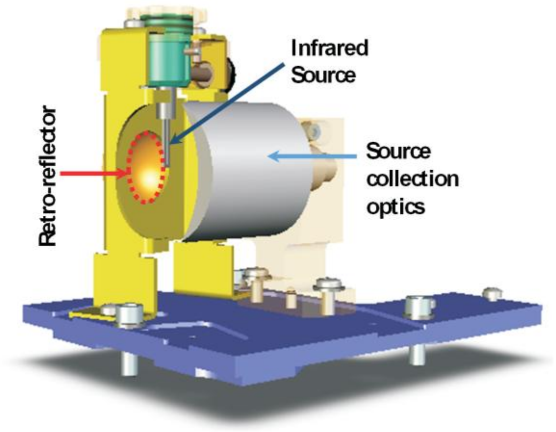

Figure 2. Optical source design used in the Agilent Cary 600 FTIR series spectrometers

The retro-reflector reflects the inverted image of the source back onto the source with a slight offset that effectively doubles the size of the hot source image. Figure 3 illustrates the source output without and with a retro-reflector. The Agilent Cary 600 FTIR spectrometer series incorporates this novel source design, and provides the power output of a water-cooled source, with the simplicity of an air-cooled source.

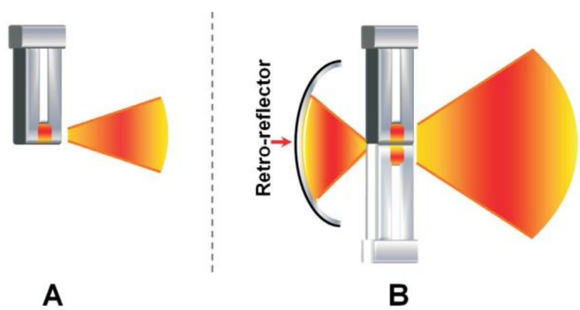

Figure 3. Air-cooled infrared source without (A) and with (B) retro-reflection. The use of retro-reflection optimizes the air-cooled infrared source's output by creating an inverted source image

Another fundamental aspect of source optics design is the collection efficiency (or optical speed, f) of the source mirror. The f-number is defined by the diameter of the source mirror, and its distance from the source. Lower f-number optics collect more light than higher f-number optics. The f-number of the source optics in Agilent FTIR spectrometers is 0.7. Conversely, the f-number of the source optics of most other spectrometers is typically twice that value or even larger. Thus an Agilent FTIR spectrometer will collect more infrared energy from the source.

A further enhancement in the Agilent Cary 600 FTIR Series source design is the ability to achieve variable power at the sample by allowing the user to operate the source at two selectable source settings: normal and boost. The higher power source setting leads to higher source temperatures, and shift the peaks of the black-body emission curves to higher wavenumbers in the spectrum. The higher source power option results in a greater emission of IR energy and increases the sensitivity for low throughput measurements such as when using IR-microscopy or hyphenated techniques.

Interferometer design

The component in the spectrometer that has the most significant affect on optical performance is the interferometer. Agilent uses a dynamically-aligned Michelson design (composed of a fixed plane mirror and moving plane mirror with respect to the beamsplitter), with an incident angle of 30 ° as measured from normal on the beamsplitter (compared to the traditional 45 ° incident angle). A shallow angle of incidence leads to a larger projected image of the beamsplitter, as shown in Figure 4. The optical throughput of the interferometer is proportional to the area of the projected beamsplitter image. By using a shallow angle of incidence such as 30 °, Agilent's instruments attain a 22% higher optical throughput relative to those with a 45 ° angle.

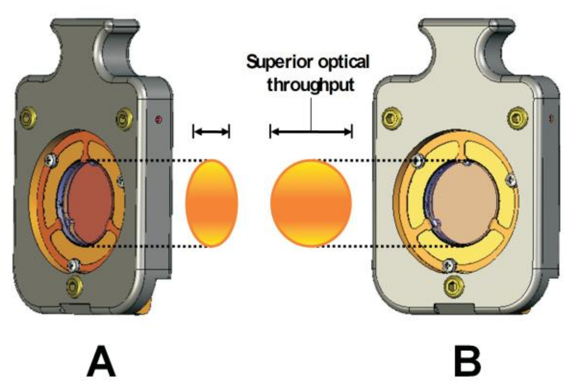

Figure 4. Projected beamsplitter image at (A) an angle of incidence of 45 °, and (B), at Agilent's angle of 30 °

In addition to the 30 ° interferometer design, Agilent offers two types of moving mirror bearings in its interferometers; a lubricated glass (mechanical) bearing and a cylindrical air bearing. An important objective in bearing design is the elimination of velocity errors (non-constant velocity of the moving mirror during data collection). Velocity errors lead to errors in the time base of the data collection and produce significant noise in the computed spectrum. While velocity errors can be reduced through the use of dynamic alignment, minimization of velocity errors is essential to the performance of an FTIR spectrometer.

In a mechanical bearing interferometer, friction has the largest impact on smooth motion. Velocity errors are smaller when the bearing carries lower mass, thus frictional bearings are generally limited in the size and mass of the mirrors they carry. In the Agilent mechanical bearing spectrometers, the moving mirror has a diameter of 38 mm which provides for a smaller mass but also a smaller clear aperture.

The cylindrical air bearing has no friction in the moving parts of the bearing and can carry more mass than the mechanical bearing. The benefits of the air bearing include minimal friction, reduced velocity errors, and higher optical throughput due to the larger mirror mounted on the air bearing. The mirror on the Agilent air bearing interferometer has a diameter of 57 mm. Since the optical throughput of the interferometer is proportional to the projected area of the beamsplitter, the optical throughput of the air bearing interferometer is approximately three times that of the mechanical bearing interferometer.

Infrared power at the sample

The type of source and the interferometer design are critically important for maximizing optical throughput; however these are only part of the complete picture. A key indicator of the actual performance of the whole optical system is given by the infrared power at the sample focus. This measurement is an accurate prediction of the ultimate light throughput of the spectrometer.

Infrared power is measured at the beam focus in the sample compartment with an 'infrared power meter' (Coherent Inc., Santa Clara, CA) which is set for measurement at 2000 cm^{-1}. Figure 5 shows the power measured in the sample compartment of the Agilent mechanical bearing and air bearing instruments. As the figure demonstrates, the Agilent air bearing spectrometers (Cary 670/680 FTIR) typically deliver >160 mW of infrared power, while the Cary 660 FTIR mechanical bearing spectrometer typically delivers >50 mW of infrared power at the sample.

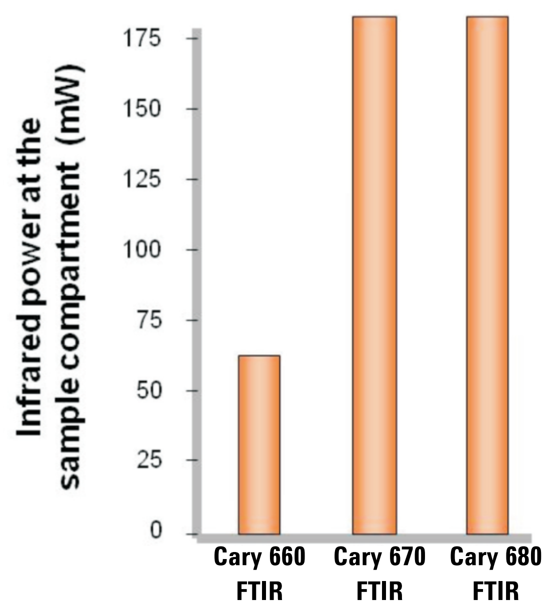

Figure 5. A typical example of the infrared power at the sample focus for Agilent FTIR spectrometers

The power level of the Agilent air bearing spectrometers is unmatched by any other spectrometer on the market. The Agilent Cary 640 FTIR and 660 FTIR spectrometers deliver better optical performance than any instrument in their respective classes, and their optical performance is only surpassed by the Agilent Cary 670 FTIR and 680 FTIR. These high power levels that are achieved for each of Agilent's FTIR spectrometers will directly translate into better spectrometer signal-to-noise (S/N) performance under real-world measurement conditions.

Signal-to-Noise performance

The infrared power measurements in Figure 6 can be used to predict the sensitivity of the spectrometers. A common specification that is reported by FTIR spectrometer manufacturers is the open beam signal-to-noise ratio. This S/N ratio value is obtained by sequentially measuring two open beam (single beam) spectra using a deuterated L-alanine doped triglycine sulphate (DLaTGS) detector, then ratioing one spectrum to the other to obtain a baseline at 100% transmission. The peak-to-peak value in a region of the spectrum (typically around 2000 cm^{-1}) is measured as the noise, and divided into 100 (for 100% T) as the signal. This test can be useful as a basis for comparing the sensitivity of spectrometers as long as scan conditions, beam attenuation conditions, and post-collection data processing algorithms are identical for all spectrometers. Unfortunately, this is not always the case between various FTIR spectrometer manufacturers.

Typical 5 second open beam signal-to-noise ratio values for the Agilent FTIR spectrometer series, obtained using a DLaTGS detector, are listed in Table 1. These values attest to the state-of-the-art performance of these spectrometers. The tabulated values for open beam measurements of the Cary 670/680 FTIR spectrometers were recorded with beam attenuation. Attenuation of the IR beam inside the spectrometer is important due to the fact that the power incident on the DLaTGS detector in an open beam configuration without attenuation would saturate the detector and detector electronics. The Cary 670/680 FTIR air bearing spectrometers produce a very large amount of IR power.

Table 1. Typical signal-to-noise ratio values for Agilent FTIR spectrometers

Signal-to-noise ratio	Spectrometer		
	Cary 660 FTIR	Cary 670 FTIR	Cary 680 FTIR
5 s open beam	10,000:1	12,000:1	12,000:1
5 s with ATR (typical)	2,500–3,500:1 p-p	7,000–10,000:1 p-p	7,000–10,000 p-p

Typically their beam energy must be attenuated by a factor of 4 (75%), to only 25% of the original open beam energy, in order to run the 100% line S/N test. It is for this reason that the open beam specifications for the Agilent air bearing spectrometers (Cary 670/680 FTIR) are similar in numerical value to the Cary 660 FTIR mechanical bearing spectrometer in Table 1. Thus the common method of reporting the S/N measurements does not demonstrate the true performance of the Agilent air bearing spectrometers. A more relevant point is that most research spectrometers are typically used in conjunction with energy inefficient sampling techniques, or used to measure intractable samples (and not merely used to measure air as is done in the common S/N test).

A more appropriate method of evaluating the S/N specifications of these spectrometers is to measure the 100% line noise with an energy-robbing accessory, such as a single bounce diamond attenuated total reflectance (ATR) accessory. These accessories have become very popular recently due the ease of sampling they allow. By performing the S/N test under real-world conditions (such as when using accessories that limit the IR throughput to ~8—15%) we can quantify the actual performance of a spectrometer. When a single bounce ATR accessory is used in the Agilent Cary 670 FTIR or 680 FTIR spectrometers, an analyst can remove the attenuation that is used for transmission measurements and direct the full power of the spectrometer to the accessory. Under these conditions, the Agilent spectrometer's detector is illuminated to a similar level as in other spectrometers in an open beam configuration. The Agilent spectrometers can achieve similar or better S/N performance with the accessory in place, as other spectrometers in open beam measurements. The S/N performance of the Agilent spectrometers with the Pike Miracle diamond ATR accessory is also listed in Table 1.

A range is listed, as the S/N may depend on the variation between ATR accessories, as well as the alignment of the ATR within the spectrometer. These values demonstrate that the S/N performance with the accessory in place is better than open beam results obtained in most other commercially available spectrometers from other manufacturers.

Conclusion

Agilent FTIR spectrometers offer unparalleled sensitivity and optical performance, due to innovative optics and an emphasis on optical throughput in all aspects of their design. Designing for performance may slightly increase the footprint, however, the benefit of exceptional performance by far outweighs any such perceived compromise. Agilent customers can always be assured of achieving their results with the highest accuracy and precision, in the shortest measurement time possible, thereby maximizing their productivity and profitability.

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