

Varian FT-IR Spectrometers

Spectral Range Coverage for Unrivalled Performance

Advantage statement: By offering a range of sources, beamsplitters and detectors, and designing the Varian 600-IR series to maximize optical throughput, Varian delivers the highest performing FT-IR instruments on the market. The range of components allows the spectrometer to be properly configured so that the spectral range of all components overlap. This provides the ability to obtain the highest quality of data at the highest sensitivity.

Introduction

Varian Fourier transform infrared (FT-IR) spectrometers offer great flexibility in their design, allowing them to be configured to cover a wide spectral range with a selection of sources, beamsplitters, and detectors. The ability to tailor a spectrometer's components may, however, lead to some confusion or indecision on how to obtain the best configuration for a specific application.

This advantage note describes the spectral range coverage that can be achieved separately by sources, beamsplitters, and detectors, and details how these components can be combined to generate the spectrometer's working spectral profile.

Sources

The source of infrared radiation is the first component to consider when configuring a spectrometer. Sources available for the Varian 600-IR series FT-IR spectrometers are shown in Figure 1, along with their usable spectral ranges.

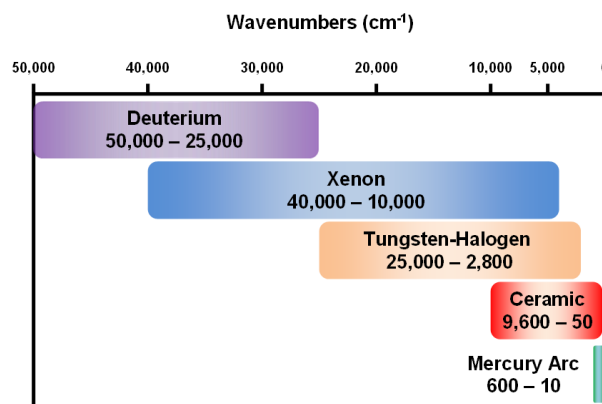


Figure 1. The sources available for the Varian 600-IR series FT-IR spectrometers provide for comprehensive infrared coverage; their typical spectral ranges are listed in wavenumbers above.

The deuterium and xenon sources are most commonly used in UV-Vis operation, while the tungsten-halogen source is used to emit near-IR radiation, and the ceramic source is useful for mid-IR and some far-IR measurements. For higher energy in the far-IR region, a mercury source can also be used. The peak performance of these sources is typically in the center of their respective ranges. Not all sources are available for all spectrometer models.



Varian FT-IR Spectrometers

Beamsplitters

The Varian 600-IR series FT-IR spectrometers have several available beamsplitter options. The beamsplitter is an essential component of a spectrometer's interferometer. An ideal beamsplitter transmits 50% of the incident radiation to a fixed mirror, and reflects the remaining 50% to a moving mirror. Each beamsplitter offered by Varian is optimized for a specific spectral range, as displayed in Figure 2.

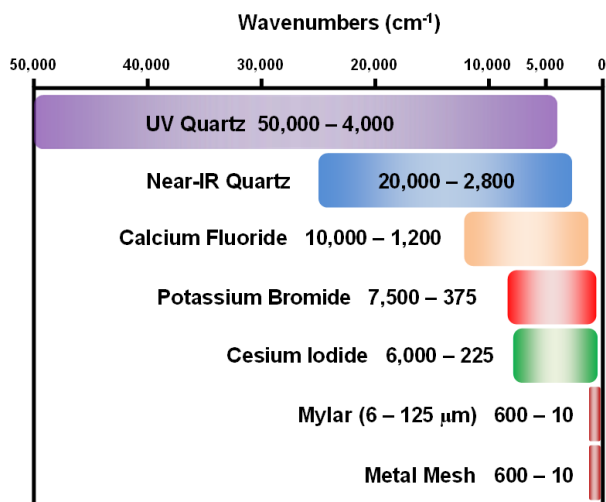


Figure 2. Beamsplitters available for the Varian 600-IR series FT-IR spectrometers with their typical spectral ranges in wavenumbers. Not all beamsplitters are available for all models.*

The UV quartz beamsplitter is commonly selected for UV-Vis applications, yet it does provide coverage into the near-IR region and hence can be used to cover a spectral range from the near-IR to the ultraviolet. As the name suggests, the UV quartz beamsplitter is made from UV-grade quartz (which is not an anhydrous material), so hydroxyl absorbance bands are present in the near-IR range. These absorbance bands may limit the effectiveness of the UV quartz beamsplitter in the near-IR, as shown in Figure 3. This figure displays the single beam spectrum for the UV quartz beamsplitter overlaid with that of the near-IR quartz beamsplitter.

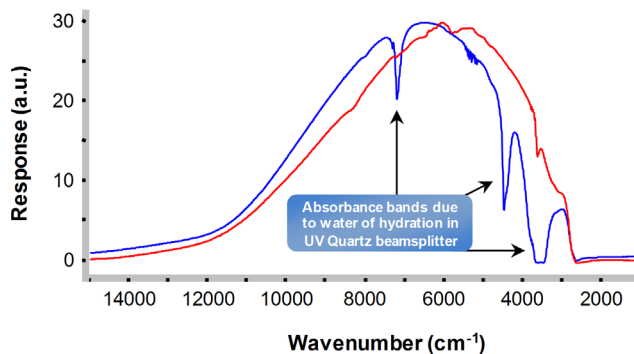


Figure 3. Single beam spectra for the UV quartz beamsplitter (blue) and the near-IR quartz beamsplitter (red) collected using a tungsten-halogen source and PbSe detector.

The near-IR quartz beamsplitter does not possess strong hydroxyl absorbance bands in its spectrum, as it is manufactured from anhydrous quartz to optimize its performance in the near-IR region.

The calcium fluoride (CaF₂) beamsplitter is commonly used for near-IR analyses as well, and it will function as effectively as the near-IR quartz beamsplitter in the near-IR region to ~10,000 cm⁻¹. The benefit of using the CaF₂ beamsplitter is that it provides coverage into the mid-IR region (down to 1,200 cm⁻¹), so one beamsplitter covers a significant part of the mid-IR region and the most commonly analyzed region of the near-IR.

Potassium bromide (KBr) is the most common material used in beamsplitters, as it provides good coverage in the mid-IR as well as significant coverage of a portion of the near-IR region. The overall performance of the KBr beamsplitter in the near-IR region is, however, severely limited when compared to that of the near-IR quartz or CaF₂ beamsplitters, as demonstrated in Figure 4.

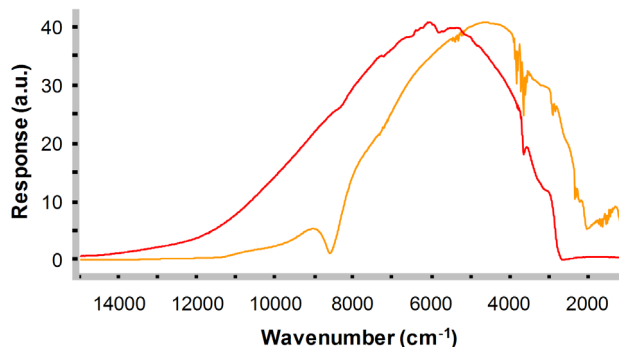


Figure 4. Single beam spectra for the KBr beamsplitter (orange) and the near-IR quartz beamsplitter (red) collected using the tungsten-halogen source and PbSe detector. These spectra demonstrate that the near-IR quartz beamsplitter provides better spectral coverage than the KBr beamsplitter across the near-IR region.

Varian FT-IR Spectrometers

It is therefore recommended that one of the near-IR beamsplitters be used for quantitative measurements in the near-IR region. The cesium iodide (CsI) beamsplitter is also commonly used for mid-IR measurements (due to its coverage from $\sim 6,000$ to 225 cm^{-1}), however, its near-IR coverage is limited compared to that of the KBr beamsplitter. Figure 5 displays the overlay of the spectra obtained from the CsI and KBr beamsplitters.

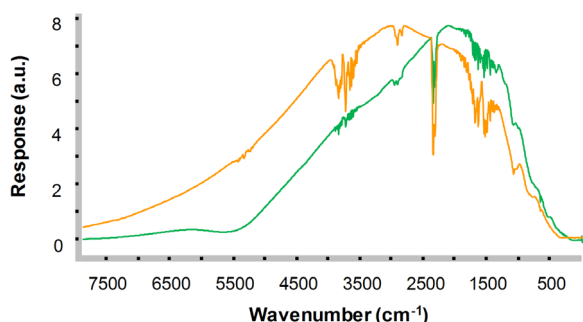


Figure 5. Single beam spectra for the CsI beamsplitter (green) and the KBr beamsplitter (orange) collected using a ceramic source and DLATGS detector.

The Mylar™ and metal mesh beamsplitters are used in far-IR configurations. The spectral coverage of Mylar beamsplitters is based on internal reflection within the Mylar film, with the thicker films producing more internal reflections and broader spectral coverage. One limitation of using thicker Mylar films is that they produce nodes within their spectral ranges where their modulation efficiency is low, as demonstrated in Figure 6.

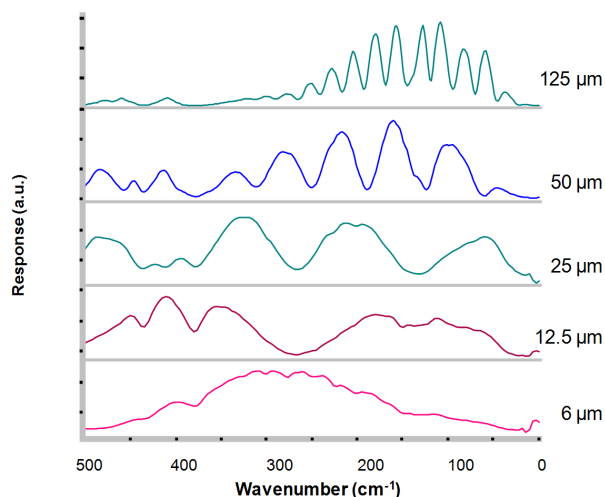


Figure 6. Single beam spectra for the 6, 12.5, 25, 50 and 125 μm Mylar beamsplitters (bottom to top, respectively) in the far-IR spectral region. Spectra have been stacked for clarity.

Multiple Mylar beamsplitters are usually used in combination, starting with the 6 μm beamsplitter to obtain the broadest spectral coverage without nodes in the spectrum, and then selecting a thicker beamsplitter to extend coverage to lower wavenumbers if necessary.

An alternative to multiple Mylar beamsplitters in the far-IR region is the metal mesh beamsplitter. This beamsplitter will provide coverage through the entire far-IR region, however it has a lower efficiency than any of the Mylar beamsplitters for measurements above $\sim 50\text{ cm}^{-1}$. Figure 7 displays a single beam spectrum of a metal mesh beamsplitter overlaid with that obtained from a 6 μm Mylar beamsplitter, plotted on the same scale to demonstrate the relative efficiency of each beamsplitter.

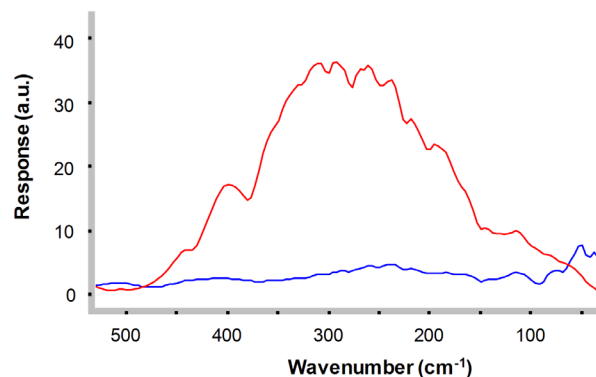


Figure 7. Single beam spectra of the 6 μm Mylar beamsplitter (red) overlaid with that of the metal mesh beamsplitter (blue).

Selection of a beamsplitter is primarily driven by the customer's application and desired range of experiments. Varian provides a comprehensive range of beamsplitters to address all infrared spectroscopic needs.

Detectors

Choosing a detector depends on the desired spectral region of analysis as well as on the detector's relative sensitivity, speed, and signal processing characteristics. Hence, the discussion of detector selection for the Varian 600-IR FT-IR spectrometer series is more complex than that of sources or beamsplitters. To avoid unnecessary confusion, the following discussion of detector selection has been divided into four sections based on the relative spectral region.

Ultraviolet/Visible Detectors

Varian offers multiple detectors, which operate in the UV-Vis range, including photomultiplier tube (PMT) detectors and a near-IR silicon (Si) detector, which responds into the visible region to $\sim 25,000\text{ cm}^{-1}$ as shown in Figure 8. The PMT detector assembly has a standard PMT detector socket so that any customer-supplied PMT detector (that is compliant with that socket) can be used in addition to the three described.

Varian FT-IR Spectrometers

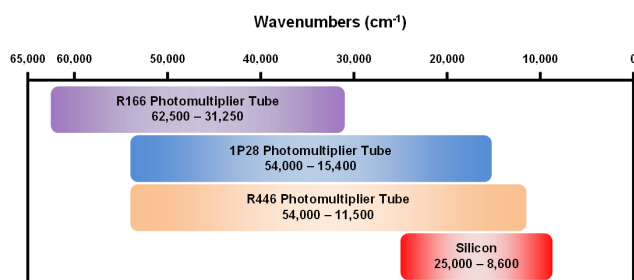


Figure 8. Typical spectral range coverage for the UV-Vis PMT detectors and the near-IR Si detector available for Varian 600-IR series FT-IR spectrometers.**

The R166 PMT is known as a 'solar blind' detector, as it responds only to UV radiation. The 1P28 covers both the UV and visible ranges, while the R446 also covers the UV-Vis range and extends into the near-IR region. The responsivity of the Si detector is highest in the near-IR region, but it can be used to acquire data in the visible region. It is preferable to use the PMT detectors in the visible region, rather than the Si detector, as they provide higher responsivity in that region.

Near-IR Detectors

The near-IR detectors available for the Varian FT-IR spectrometers are listed in Figure 9. The Si detector is primarily used to cover the short near-IR (above 9,000 cm⁻¹). The indium antimonide (InSb) detector operates at liquid nitrogen temperature and has very high sensitivity, however it is relatively easy to saturate. Therefore, the InSb detector is recommended only for near-IR measurements in conditions of very low energy flux, for example, in emission measurements or with low throughput fiber optics. The indium gallium arsenide (InGaAs) and lead selenide (PbSe) detectors both operate at ambient temperature.

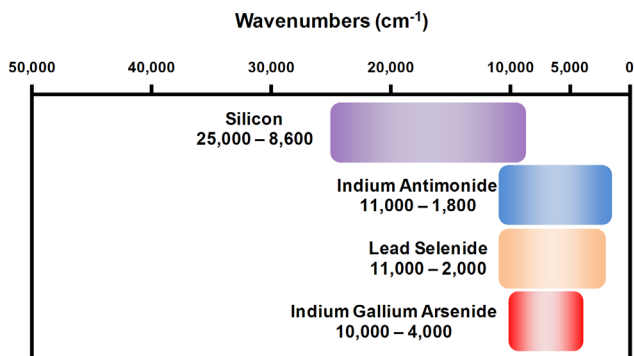


Figure 9. Typical spectral range coverage for the near-IR detectors available for Varian 600-IR series FT-IR spectrometers.

The PbSe detector is the most versatile for spectral range coverage and price. However, if maximum sensitivity is required, the InGaAs is the detector of choice.

Mid-IR Detectors

Two types of mid-IR detectors are available from Varian – deuterated L-alanine doped triglycine sulfate (DLaTGS) and mercury cadmium telluride (MCT) detectors, listed in Figure 10.

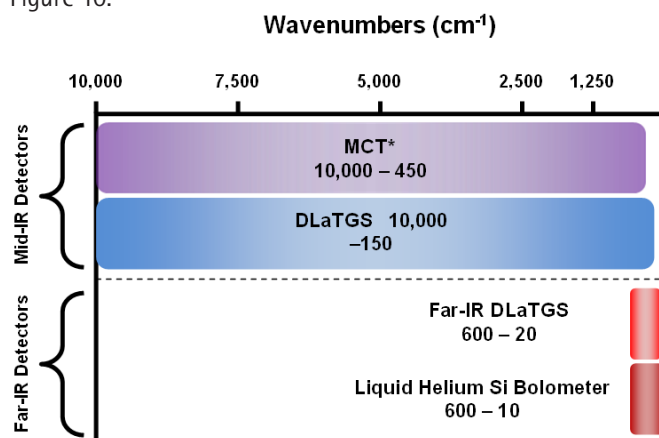


Figure 10. Typical spectral range coverage for the mid-IR and far-IR detectors available for Varian 600-IR Series FT-IR spectrometers.

The DLaTGS detector is a pyroelectric detector that typically operates at ambient temperature, although it is also available with a Peltier cooler. The Peltier cooler is intended to 'cool' the detector to 20 °C in order to stabilize it, since its responsivity is temperature-dependent.

The MCT detectors are photoconductive detectors that operate at liquid nitrogen temperature. As such, the detector elements are mounted in vacuum dewars on cold fingers that extend to a liquid nitrogen reservoir. The MCT detectors are faster and more responsive than the DLaTGS detector and they are recommended for low energy measurements (where the sample or accessory limits the optical throughput) or for high speed kinetics (where the faster response of the MCT is required).

MCT detectors can be further sub-classified as narrow band or broad (wide) band. The narrow band detector has higher responsivity than the broad band detector and thus potential for higher sensitivity. However, the narrow band detector also has a more limited spectral response range, especially at the low wavenumber end of the spectrum.

Both the narrow band and broad band MCT detectors are available with a selection of pre-amplifiers – standard, linearized and DC-coupled. The function of a pre-amplifier is to boost the detector's signal and the choice of the pre-amplifier is based on the type of experiments that are to be performed. The standard pre-amplifier has the highest bandwidth (to 125 KHz) of the pre-amplifiers and is recommended for modulation measurements (for example, polarization modulation reflection absorption or vibrational circular dichroism) or for high-speed kinetic

Varian FT-IR Spectrometers

measurements. The patented linearized pre-amplifier (see Varian's Linearized MCT Detector application note, publication number SI-1336) corrects the standard MCT for its non-linear response to energy flux, allowing it to be used at much higher incident energy than the standard pre-amplifier, while maintaining photometric accuracy. The sensitivity of the linearized MCT detector can be up to ten times higher than the standard MCT detector in high-energy situations. The DC-coupled pre-amplifier is required for step-scan time-resolved spectroscopy (TRS) experiments at a time resolution to 5 microseconds. Nanosecond TRS experiments (to 10 nanoseconds time resolution) require yet another MCT detector. This MCT is a photovoltaic detector with both high bandwidth AC-coupled and DC-coupled outputs and is recommended only for nanosecond TRS experiments.

Far-IR Detectors

Two detectors (also shown in Figure 10) are available for use in the far-IR region – a far-IR DLaTGS detector and a liquid helium-cooled Si bolometer. The far-IR DLaTGS detector has the same detector element as the mid-IR DLaTGS detector, but it has a polyethylene (PE) window in place of the CsI window used on the mid-IR detector. The PE window is transparent in the far-IR region, whereas the CsI window limits the range of the mid-IR detector to 150 cm^{-1} , which precludes its use for far-IR measurements.

The liquid helium cooled bolometer detector offers higher sensitivity in the far-IR region than the DLaTGS detector (analogous to the MCT compared to the mid-IR DLaTGS), but the obvious drawbacks are its requirement for liquid helium cooling, and its expense. Only laboratories equipped to handle liquid helium should attempt to use this detector.

Conclusion

To properly configure a spectrometer, the source, beamsplitter, and detector must all operate in the same spectral range. The usable range of the spectrometer will be defined by the overlap of each component's effective spectral range. In addition, although the ranges of the components are shown as bar graphs for convenience, each component will generally have optimal performance at the center of its range and poorer performance in the extremes. Consequently, the spectrometer will operate at maximum performance when there is significant overlap between the ranges of the source, beamsplitter, and detector. It is the combination of these optical components that will provide the ability to make measurements with high sensitivity and obtain the highest quality data. By offering a comprehensive range of components and by tailoring its FT-IR spectrometers to maximize optical throughput, Varian delivers the highest performing instruments on the market.

*Availability of components varies between spectrometer models. Consult your Varian representative for further information.

**Not all detectors are available on all models. Consult your Varian representative for further information.

References

1. R. Curbelo, Techniques for correcting non-linearity in a photodetector using predefined calibration information, U.S. patent 5,262,635, November 16, 1993

