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Performance Features of an Extended Range Beamsplitter for Mid- and Near-IR Spectroscopy

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Key Words

- Smart NIR Integrating Sphere
- Smart UpDRIFT
- Fiber Optics
- FT-Raman
- Mid-Infrared
- Near-Infrared

The multitude of samples facing the modern analytical laboratory makes versatility in the instrumentation a critical concern. The typical laboratory FT-IR spectrometer is equipped with mid-infrared capabilities (MIR), operating from 4000 to 400 cm^{-1}. However, many applications utilizing the near-IR (NIR) spectral region, from about 10,000 to 4000 cm^{-1} are also critical, including raw material identification, moisture analysis and FT-Raman spectroscopy. The MIR and NIR ranges are typically covered by two instruments, or one instrument with a change in beam splitter. This technical note describes the use of a Thermo Scientific XT-KBr™ beamsplitter to collect both MIR and NIR data. Application data showing the trade-offs in performance is also presented.

The XT-KBr beamsplitter covers the spectral range from 11,000 to 375 cm^{-1}. The detectors and sources need to be selected correctly for optimal performance. The wide range eliminates the need to change the beamsplitter when studying a sample with important MIR and NIR features, or when swapping between MIR and NIR applications. Both of the Nicolet™ series spectrometers can be equipped with the XT-KBr beamsplitter, though the Thermo Scientific Nicolet 6700/8700 spectrometers are better equipped to deal with the multiple ranges through multiple-installed sources and detector, selectable by software.

The data shown were collected using a Nicolet 6700 spectrometer equipped with both the Thermo Scientific Ever-Glo™ ETC™ MIR source and the quartz-halogen NIR source. Raman data were collected using the Thermo Scientific Nicolet NXR FT-Raman Module with an InGaAs room temperature detector. Other conditions were as noted in the experiment description.

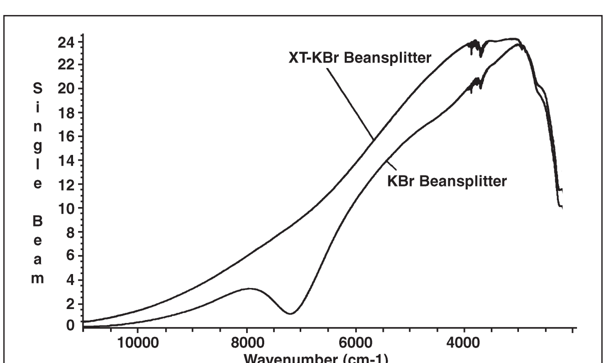

Figure 1

Figure 1 shows single beam (non-ratio) spectra of the XT-KBr and standard KBr beamsplitters using a common scale, collected using identical parameters at 4 cm^{-1} resolution, a DTGS detector and white light source. The XT-KBr beamsplitter shows significantly greater energy beyond 2000 cm^{-1} versus. the KBr beamsplitter. The reflective germanium coating on the KBr beamsplitter absorbs strongly at about 7400 cm^{-1}, limiting its use in the NIR, whereas the XT-KBr beamsplitter provides a transparent energy window to 11,000 cm^{-1}.

NIR FT-Raman has become an important tool in providing additional structural information for a wide variety of laboratory samples. The technique has value to analytical labs because it permits analysis with several advantages:

- No sample preparation
- No atmospheric CO_2 and H_2O problems
- Structural elucidation complimentary to FT-IR
- Samples may be housed in glass or plastic
- Spectral data can be obtained down to 100 cm^{-1} (Raman shift)
- Low potential for fluorescence (relative to dispersive Raman)

Figure 2 shows FT-IR and FT-Raman spectra of a powdered laundry detergent. The FT-IR data was collected using a Thermo Scientific Smart™ Performer accessory with the sample pressed against the ZnSe crystal, and a DTGS detector. The sample for the FT-Raman spectrum was placed into an NMR tube. Both spectra were measured using the XT-KBr beamsplitter. No manual change of optical components was required for the measurements. Computerized selection of the FT-Raman accessory can be made from the OMNIC™ spectroscopy software, with changeover from FT-IR to FT-Raman occurring within seconds.

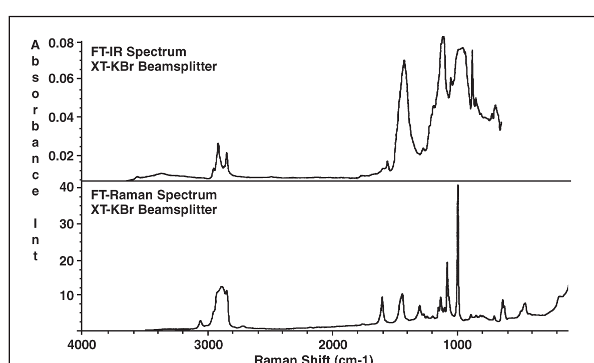

Figure 2

Normally, an NIR beamsplitter such as CaF_2 would have to be installed for the FT-Raman experiment. Comparison of the performance of the CaF_2 and XT-KBr beamsplitter options is shown in Figure 3. These single beam spectra were collected using a DTGS detector and white light source. The CaF_2 beamsplitter clearly provides higher NIR energy beyond 5000 cm^{-1}. Figure 4 shows a histogram of the relative signal-to-noise ratios (SNR) of CaF_2 vs. XT-KBr obtained from the 100% lines. This chart confirms the higher performance of CaF_2 above 5000 cm^{-1}.

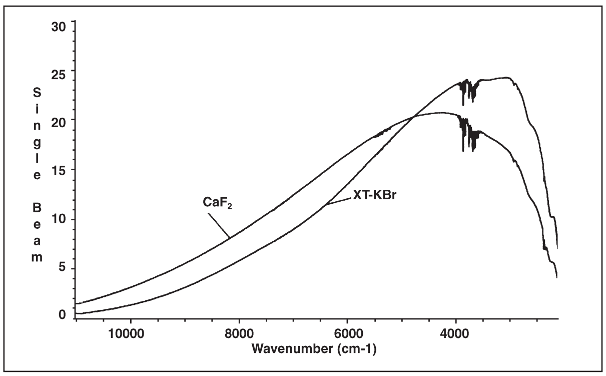

Figure 3

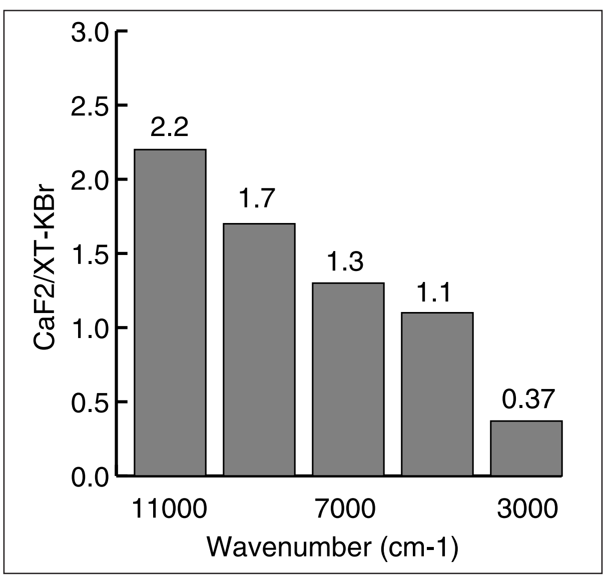

Figure 4

In the NIR range for the Raman shifted spectrum (9298-5668 cm^{-1}, representing a 3600-100 cm^{-1} Raman shift), the signal-to-noise ratio for a cyclohexane spectrum is about 1.6 times better with the CaF_2 beamsplitter. However, the spectral quality shown in figure 5 is excellent for either choice of beamsplitter. The vast majority of FT-Raman sampling can easily be performed using the XT-KBr beamsplitter.

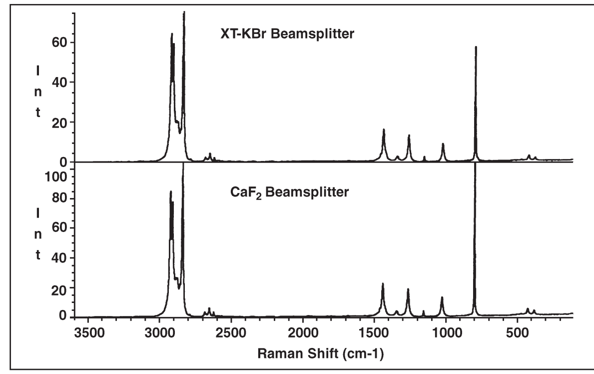

Figure 5

NIR fiber optic sampling is another technology for simplification of laboratory data collection. The sampling advantages include:

- No sample preparation
- Remote sampling
- Reflective samples may be housed in glass containers
- Very fast analysis times (10 seconds is typical)

NIR fiber optics sampling accessories are available both coupled to a sample compartment fiber-launcher or for mounting onto the face of the Nicolet 6700 FT-IR. There are also two accessories – the Smart NIR UpDRIFT and the Smart NIR Integrating Sphere – which fit into the sample compartment and provide enhanced NIR sampling capabilities.

The Thermo Scientific Nicolet SabIR™ NIR fiber optic sampling accessory was used to measure the spectrum of an analgesic tablet using the XT-KBr beamsplitter. The spectrum shown in figure 6 was produced in 20 seconds at 16 cm^{-1} resolution.

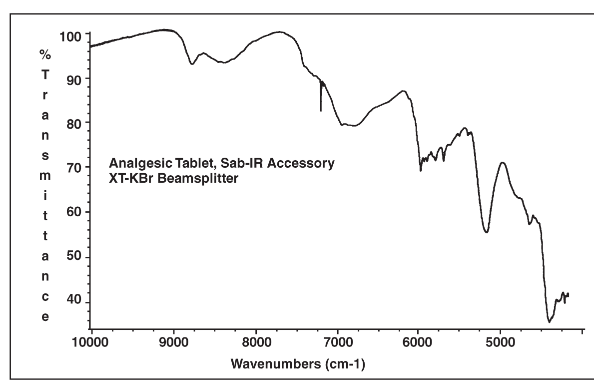

Figure 6

The XT-KBr beamsplitter also performs very well in the MIR. Figure 7 compares single beam spectra for the KBr and XT-KBr beamsplitters using the Ever-Glo ETC MIR source and DTGS detector (common scaled). These curves show that above 2600 cm^{-1}, the XT-KBr has greater throughput than KBr. Below 2600 cm^{-1}, the situation is reversed.

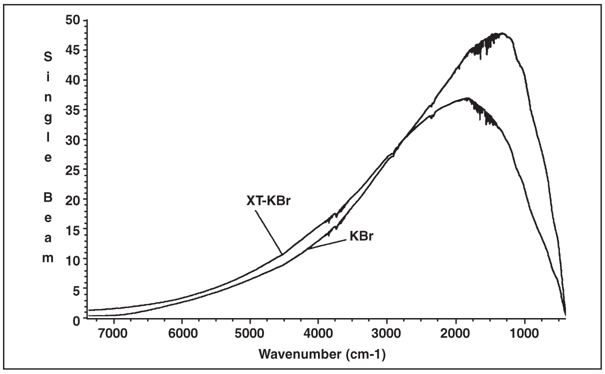

Figure 7

A numerical comparison of the MIR signal-to-noise ratios (SNR) for the two beamsplitters is shown in figure 8. At 2600 cm^{-1}, the SNR is identical. At 7000 cm^{-1}, the XT-KBr beamsplitter outperforms KBr by 4.6 times. At 450 cm^{-1}, the KBr beamsplitter outperforms the XT-KBr by about 2 times. The SNR of the Nicolet series of FT-IR spectrometers is significantly greater than most applications require, so the XT-KBr beamsplitter will provide reasonable performance for MIR spectroscopy over its full operating range.

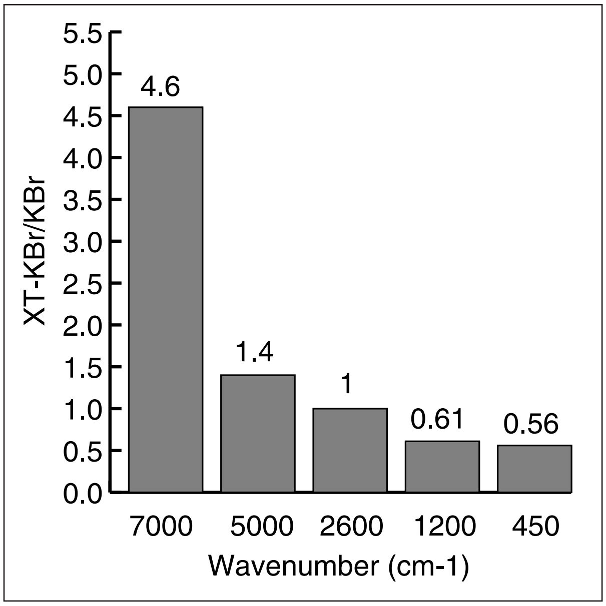

Figure 8

Conclusions

The XT-KBr beamsplitter provides the ability to collect mid and near-IR spectral data from 11,000 to 375 cm^{-1}. As with any spectroscopic solution, there are performance compromises over subsets of this range, but most routine mid-IR and near-IR applications can be easily done with the XT-KBr beamsplitter. The simplicity and time savings provided by the use of a single beamsplitter to address two spectral ranges is a significant advantage.

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