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

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Several applications utilizing the near-IR spectral region are emerging in analytical and research laboratories. An FT-IR system capable of easy, dependable access to this region will be important to the success of these new applications. This note describes the use of a single beamsplitter to collect data in both the mid- and near-IR spectral regions. Specific applications include measurements with FT-Raman and near-IR fiber optics accessories.

A proprietary version of the Nicolet KBr beamsplitter has been developed to span the spectral region from 11,000 to 375 cm^{-1}. The new XT-KBr™ beamsplitter provides good performance and eliminates the need to change beamsplitters when collecting a sample with important features occurring in both the mid- and near-IR spectral regions. The IR data presented were collected using a Magna-IR® spectrometer equipped with a dual Ever-Glo mid-IR and quartz-halogen white light source. Detectors and beamsplitters are listed with each measurement. The Raman data were collected using the Magna-IR FT-Raman Accessory Module with an InGaAs room temperature detector.

Figure 1 shows single beam spectra of the XT-KBr and standard KBr beamsplitters using common Y axis scaling. These two single beam spectra were collected using identical parameters at 4 cm^{-1} resolution using the dTGS detector and white light source. The XT-KBr beamsplitter shows significantly greater energy beyond 2,000 cm^{-1} vs. the standard KBr beamsplitter. The reflective germanium coating on the standard KBr beamsplitter absorbs strongly

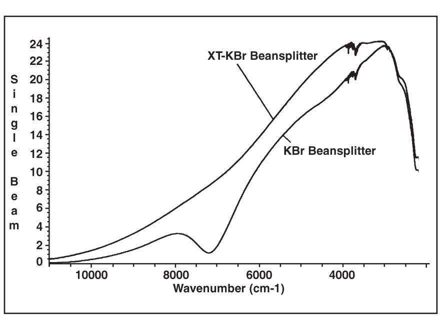

Figure 1

at about 7,400 cm^{-1} and prevents its use in the near-IR spectral region, whereas the XT-KBr beamsplitter provides a transparent energy window to 11,000 cm^{-1}.

Near-IR FT-Raman is becoming an important tool in providing additional structural information for a wide variety of laboratory samples. The advantages of FT-Raman include:

1. No sample preparation.
2. No atmospheric CO_2 and H_2O problems.
3. Structural elucidation complimentary to FT-IR.
4. Samples may be housed in glass.
5. Raman spectral data to 100 cm^{-1}.

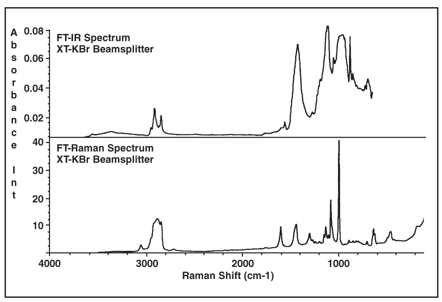

Figure 2

Figure 2 shows FT-IR and FT-Raman spectra of a powdered laundry detergent. The FT-IR data was collected using a horizontal ATR accessory and dTGS detector with the sample pressed against the ZnSe crystal. The sample for the FT-Raman spectrum was placed into a NMR tube and measured directly using an InGaAs detector. Both of these 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 is made from OMNIC® software with changeover from FT-IR to FT-Raman occurring within seconds.

In the past a near-IR beamsplitter such as CaF_2 would have to be installed for the FT-Raman experiment. How does the XT-KBr beamsplitter perform vs. a near-IR beamsplitter?

Figure 3 shows the single beam spectra for the XT-KBr and CaF_2 beamsplitters using a dTGS detector and white light source.

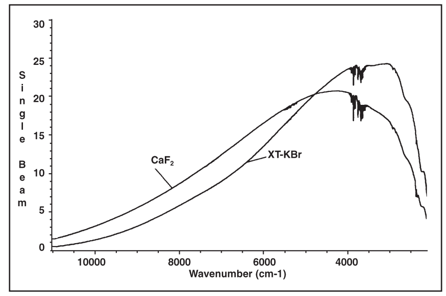

Figure 3

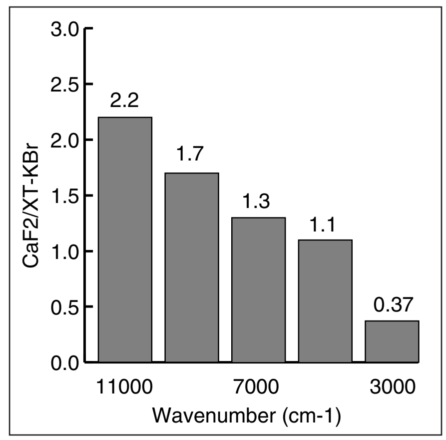

Figure 4

We can see higher near-IR energy beyond 5,000 cm^{-1} with the CaF_2 beamsplitter. We can further compare the performance by measuring the 100% lines from this combination of optical elements. The figure 4 histogram shows the relative signal-to-noise ratios (SNR) of CaF_2 vs. XT-KBr. This chart confirms the higher performance of CaF_2 above 5,000 cm^{-1}. At 11,000 cm^{-1} CaF_2 performs about 2.2 times better than XT-KBr. However, the Raman shifted spectrum occurs in the near-IR from 9,298-5668 cm^{-1} (3,600-100 cm^{-1} Raman shift) where the performance difference is lower. A direct comparison of the signal-to-noise ratio (SNR) of cyclohexane spectra with FT-Raman using XT-KBr and CaF_2 beamsplitters shows an advantage of 1.6 times better for CaF_2.

Although the measured SNR for FT-Raman spectra is reduced using the XT-KBr, the spectral quality shown in figure 5 is excellent for either choice of beamsplitter. The vast majority of FT-Raman sampling will be easily performed using the XT-KBr beamsplitter.

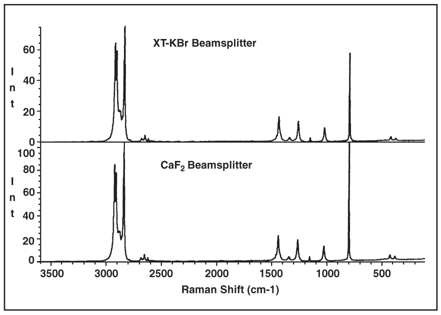

Figure 5

Near-IR fiber optic sampling is another emerging Fourier transform technology. Advantages include:

1. No sample preparation.
2. Remote fiber optics sampling.
3. Reflective samples may be housed in glass containers.
4. Very fast analysis times (10 seconds is typical).

The use of a fixed beamsplitter FT spectrometer for near-IR and mid-IR sampling provides sampling speed and ease of multi-range spectroscopy. Near-IR fiber optics sampling accessories are available for mounting into the main or auxiliary Magna-IR sample compartments or onto one of its external beam ports. With the use of a multi-range beamsplitter, a change from mid-IR to near-IR is accomplished through computerized selection of source and detector with only seconds of time elapsed and no system purge loss.

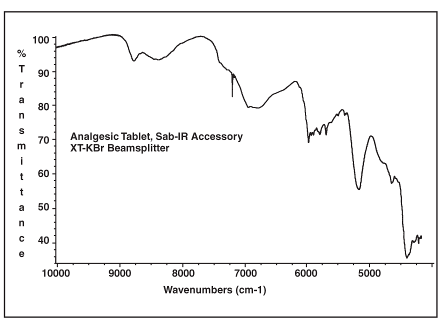

Figure 6

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

The use of the multi-range XT-KBr beamsplitter makes the speed and easy sampling of near-IR readily available in a single spectrometer with a single beamsplitter.

As a mid-IR beamsplitter XT-KBr performs very well. Figure 7 shows the single beam spectra for standard vs. XT-KBr beamsplitters using the Ever-Glo mid-IR source and dTGS detector.

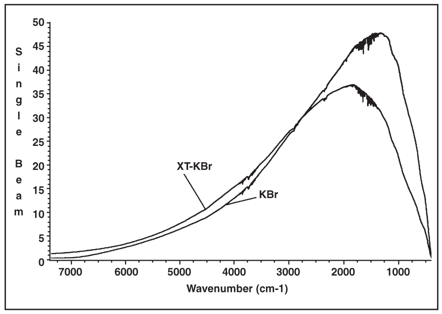

Figure 7

From these single beam energy curves we can see that above 2,600 cm^{-1} XT-KBr has greater energy than KBr. Below 2,600 cm^{-1} KBr demonstrates higher energy. A numerical comparison of the SNR ratios for these two beamsplitters in the mid-IR spectral region is shown in figure 8.

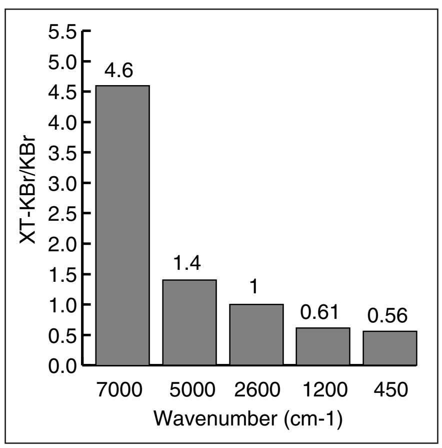

Figure 8

The SNR ratio values of figure 8 are computed from the SNR of the XT-KBr beamsplitter vs. the KBr beamsplitter. At 2,600 cm^{-1} the SNR using either beamsplitter is identical. At 7,000 cm^{-1} the SNR using the XT-KBr beamsplitter is 4.6 times better than KBr. At 450 cm^{-1} the SNR produced from the KBr beamsplitter is about two times better than XT-KBr. The SNR of Magna-IR spectrometers is generally significantly greater than most applications require and for that reason the XT-KBr beamsplitter will provide sufficient performance for mid-IR spectroscopy below 2,600 cm^{-1} and better performance above 2,600 cm^{-1}.

CONCLUSIONS

The XT-KBr beamsplitter provides the ability to collect mid and near-IR spectral data from 11,000 to 375 cm^{-1}. Although its performance is slightly reduced above 5,000 cm^{-1} in the near-IR vs. CaF_2 and slightly reduced below 2,600 cm^{-1} in the mid-IR vs. KBr, all routine mid-IR and near-IR applications are easily measured with the XT-KBr beamsplitter. The ease-of-use and time savings provided by the use of a single beamsplitter to address two spectral ranges is a significant advantage.

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