Optimizing Optical Bench Performance for Near-IR and Visible Spectroscopy

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KEY WORDS

Near-IR
Visible
Multi-Range

The overall performance of an optical bench for use in the near-infrared and visible spectral regions depends considerably on the system optical components and data collection parameters. The selection of beamsplitter, detector and sampling parameters will be based upon the application to be solved, including spectral range, time and sensitivity requirements. This note will describe various component combinations that can be used to obtain spectra in the near-IR and visible spectral ranges as well as discuss the sampling parameters necessary to optimize the data produced.

EXPERIMENTAL

All spectra were obtained using a Nicolet Nexus™ FT-IR spectrometer equipped with a computerized dual quartz-halogen, near-IR/visible source and Ever-Glo™ mid-IR source. The detectors used in this experiment include tri-glycine sulfate (TGS), mercuric cadmium telluride (MCT-A and MCT-B), lead selenide (PbSe), indium antimonide (InSb) and silicon (Si). Calcium fluoride (CaF_2) and quartz beamsplitters were used to produce the spectra. Sampling parameters vary for each of the configurations and are noted with the spectral figures.

The spectral range addressed in this note is shown in Figure 1. Single beam energy curves for each of the combinations of beamsplitter and detector with full scale display, show spectral range coverage from 27,000-2000 cm^{-1}. These data include portions of the UV, visible, near-IR and mid-IR spectral ranges. The single beam spectra of each of the configurations are discussed in greater detail in the following sections. Performance characteristics of the various optical components will be made by comparing single beam and ratioed spectra. While the energy comparisons from the single beam spectra is generally an indication of spectral performance, the proof of performance is made by comparing the ratio of the signal-to-noise ratios (SNR) of the different combinations of optical elements.

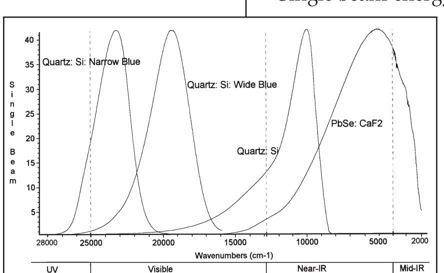

Figure 1

NEAR-INFRARED REGION

The near-IR is generally considered to span the spectral range from 12,800 to 4,000 cm^{-1}. The spectral absorbances in this region are generally overtones and combination bands from absorbances in the mid-IR region. A variety of beamsplitters, sources and detectors may be utilized to cover this spectral region.

COMPARISON OF SOURCES

Extending available near-IR range to higher wavenumbers can be achieved by using a near-IR/visible source rather than a mid-IR source. The single beam spectra shown in Figure 2 were collected using a CaF_2 beamsplitter with DTGS detector and the near-IR/visible and mid/far-IR sources. Both spectra were collected at 4 cm^{-1} resolution with 32 scans and a mirror velocity of 0.6329 cm/sec.

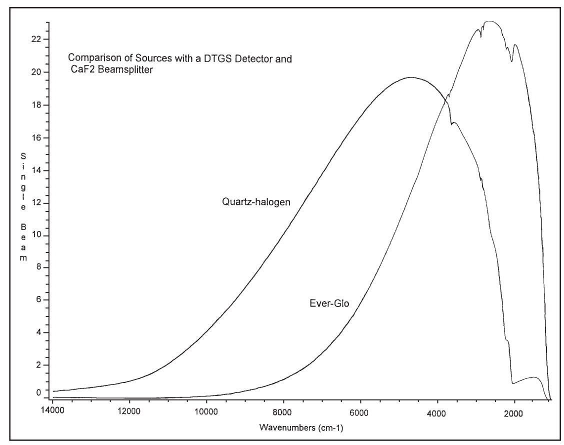

Figure 2

Figure 2 shows the added spectral range and greater intensity of the quartz-halogen source in the near-IR. Above 4,000 cm^{-1} the energy output of the quartz-halogen source is greater than that of the mid/far-IR source. Table 1 compares the ratio of signal-to-noise ratio (SNR) of the quartz-halogen versus mid-IR sources using the CaF_2 beamsplitter and DTGS detector. We can see that the quartz-halogen source gains the advantage in sensitivity at about 4000 cm^{-1} and is therefore generally used for all near-IR and visible applications.

RANGE (cm^{-1})	RATIO OF SNR VISIBLE/MID-IR SOURCES
3100-3000	0.64
4000-3900	0.95
4100-4000	1.04
6100-6000	2.39
8100-8000	6.51

Table 1

COMPARISON OF BEAMSPLITTERS

Quartz and CaF_2 beamsplitters are commonly used to study the near-infrared spectral region. Two factors must be taken into account when choosing a beamsplitter to solve the near-IR application: the spectral region of interest and the efficiency of the beamsplitter in that region.

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Figure 3 shows the comparison of spectral range and performance for the CaF_2 and quartz beamsplitters. Both spectra were collected using the DTGS detector at 4 cm^{-1} resolution using 32 scans with a mirror velocity of 0.6329 cm/sec.

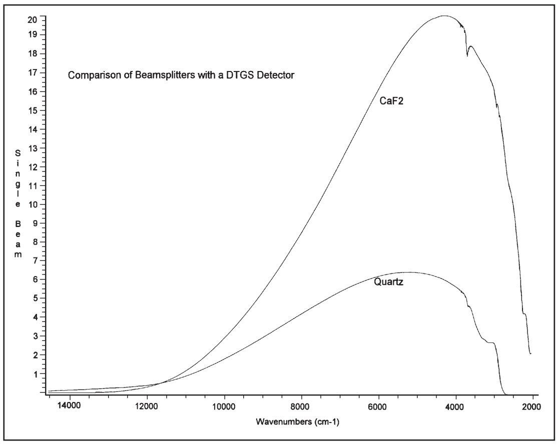

Figure 3

These data show CaF_2 provides significantly greater single beam energy below 10,000 cm^{-1}. At 11,600 cm^{-1} the single beam energy curves are equivalent, and above 11,600 cm^{-1} the quartz beamsplitter provides greater single beam energy. The ratio of the quartz/CaF_2 SNRs for several areas of this spectral region verify the advantages of the quartz beamsplitter above 11,600 and the CaF_2 beamsplitter below 11,600 as reported in Table 2.

RANGE (cm^{-1})	RATIO OF SNR (CaF_2/QUARTZ)
5100-5000	2.52
8100-8000	2.15
11,600-11,500	1.14
11,700-11,600	0.86
12,500-12,400	0.49

Table 2

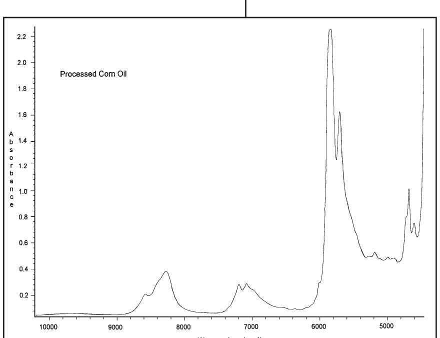

Figure 4

A common use for near-IR spectroscopy is in the agriculture and food industry. Edible oil analysis is just one of the many applications that are currently studied by NIR. A near-IR spectrum of processed corn oil is shown in Figure 4. The corn oil was sampled in the transmission mode using a 0.5 cm quartz cuvette at 4 cm^{-1} resolution using 128 scans with the DTGS detector and CaF_2 beamsplitter.

Choice Of Detectors

Selection of the detector is dependent upon the desired spectral range, the sensitivity required for sample measurement and any time requirements. A variety of detectors were studied to cover the near-infrared region and to meet individual application requirements.

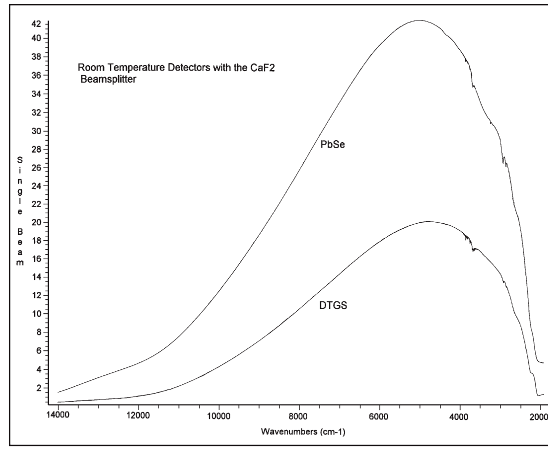

Figure 5

A comparison of the PbSe and DTGS detectors using a CaF_2 beamsplitter can be seen in Figure 5. These spectra, ordinate scaled to identical values, show similar spectral range, but significantly higher energy from the PbSe detector. The DTGS detector is generally used in the mid-IR spectral region, but is also useful for obtaining data in the near-IR. The DTGS detector provides excellent linearity over a wide concentration range, but has a lower sensitivity to infrared signal as well as a slower response time compared to cooled detectors. The PbSe detector is a commonly used detector in the near-infrared since it is very sensitive to infrared signal with no liquid nitrogen (LN_2) cooling requirement. Like the LN_2 cooled MCT and InSb detectors, the PbSe detector is a photoconductive detector with spectral response relatively independent of scanning velocity.

The ratio of the SNRs for the PbSe/DTGS detectors shows the performance advantage of the PbSe detector over this entire near-IR spectral region. These values can be seen in Table 3.

RANGE (cm^{-1})	RATIO OF SNR (PbSe/DTGS)
4100-4000	2.49
6100-6000	1.62
8100-8000	2.31
11,100-11,000	1.91

Table 3

A comparison of the single beam spectra produced with liquid nitrogen cooled detectors and a CaF_2 beamsplitter in the near-IR

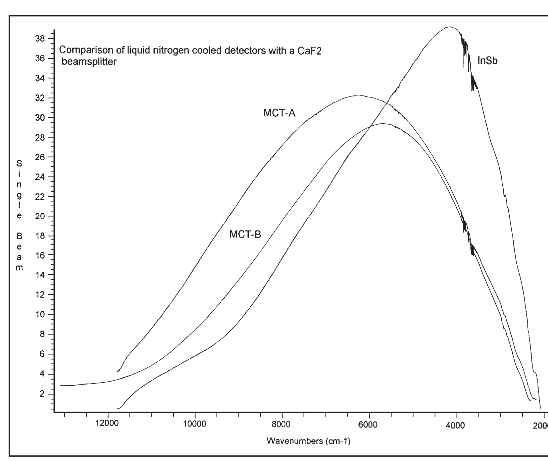

Figure 6

spectral region can be seen in Figure 6. Each of the spectra were collected at 4 cm^{-1} resolution using 32 scans with a mirror velocity of 1.2659 cm/sec and a 10% screen placed in the beam path to prevent saturation of the detector. The MCT-A (narrow-range) and InSb detectors provide higher signal strength than the MCT-B (wide-range) detector. These detectors are all very sensitive to weak infrared signals and respond quickly, so they are ideal for low throughput samples or applications where high speed data collection is required. These detectors must be cooled with liquid nitrogen (about 1L per day).

The MCT-B detector provides better linearity for quantification than either the MCT-A or InSb detectors. However the MCT-A or InSb detectors are a better choice for samples with a very low throughput. The InSb detector can only be used down to 1850 cm^{-1}, but between the cut off and 4500 cm^{-1} this detector performs almost two times better than the MCT-A detector as shown in Table 4. The MCT-A is a better choice if most spectral features are above 7000 cm^{-1} since the noise is almost twice as high for the InSb in this region. The ratio of SNRs for both MCT-A versus InSb and MCT-A versus MCT-B is provided in Table 4.

RANGE (cm^{-1})	RATIO OF SNR (MCT-A/InSb)	RATIO OF SNR (MCT-A/MCT-B)
4100-4000	0.59	6.19
5650-5550	1.22	6.51
8100-8000	1.96	7.11
10,100-10,000	4.92	13.86

Table 4

Single beam spectra produced using detectors commonly used with the quartz beamsplitter can be seen in Figure 7. Both spectra were produced at 4 cm^{-1} using 32 scans and a mirror velocity of 0.6329 cm/sec. These detectors are both very sensitive to infrared signal and are used at room temperature.

The silicon detector is most useful for data collection at higher wavenumbers out to the HeNe laser emission at 15,798 cm^{-1}. Due to the increased range achieved by these detectors, a filter must be placed in the beam path to remove interfering spectral features. A red filter is used in the near-IR region to block interference from the laser that occurs at 15,798 cm^{-1}, and to remove all information present at higher wavenumbers when a sample

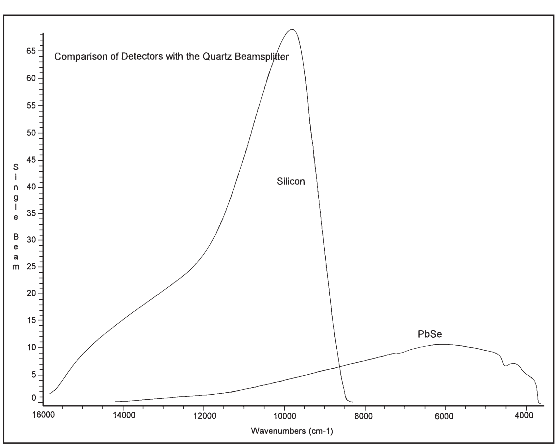

Figure 7

spacing of 1 is used to collect the data. If data is to be collected on both sides of the laser emission then a sample spacing (SSP) of 0.5 must be utilized and the red filter is not required.

The PbSe is not as sensitive to infrared signal as the silicon detector, but is a good choice at lower wavenumbers. The silicon detector provides significantly greater performance in the near-IR spectral region above 9,000 cm^{-1} as seen in Figure 7. Table 5 shows the factor of performance improvement of the silicon detector for several portions of this spectral region.

RANGE (cm^{-1})	RATIO OF SNR (SI/PbSe)
8700-8600	0.54
9100-9000	2.74
10,100-10,000	6.89
12,600-12,500	12.69

Table 5

Visible Region

The visible region provides information for spectral absorbances due to color in the sample, generally from electronic transitions within the sample molecules. A silicon detector, quartz beamsplitter and selective filters are required to collect data in the visible region. Blue filters are used in this region to both optimize the spectral result and to block any unwanted information from reaching the detector. Information filtered from the detector includes interference from the laser at 15,798 cm^{-1} along with data found at lower wavenumbers.

Figure 8 shows two single beam spectra produced using the silicon detector and quartz beamsplitter, the only difference between the two spectra is the filter placed in the beam path. The wide range blue filter is useful over a broad portion of the visible region from 25,000 cm^{-1} to 16,000 cm^{-1}, while the narrow range blue filter is used over a more selective region, from 27,000 cm^{-1} to 20,000 cm^{-1}, allowing for higher performance data to be collected in this region.

The single beam spectra show approximate visible energy comparisons using the two different blue filters. Ratios of the SNRs for the narrow blue versus wide blue filter over several regions of overlapping range of the filters are provided in Table 6. From these SNR ratios it is clear that the narrow range blue filter provides better performance above 22,700 cm^{-1} compared to the wide range blue filter.

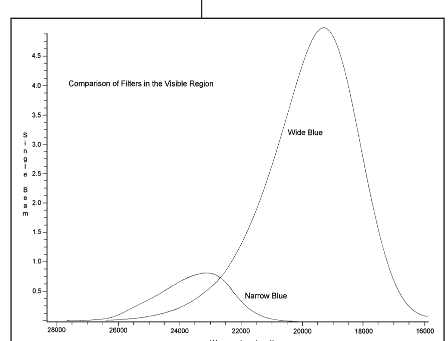

Figure 8

RANGE (cm^{-1})	RATIO OF SNR (NARROW/WIDE)
21,100-21,000	0.02
22,700-22,600	0.92
22,800-22,700	1.59
23,600-23,500	4.09
24,600-24,500	4.67

Table 6

A holmium filter sample spectrum is shown in Figure 9. This spectrum was collected using a quartz beamsplitter, Si detector, and the wide blue filter at 16 cm^{-1} resolution using 128 scans.

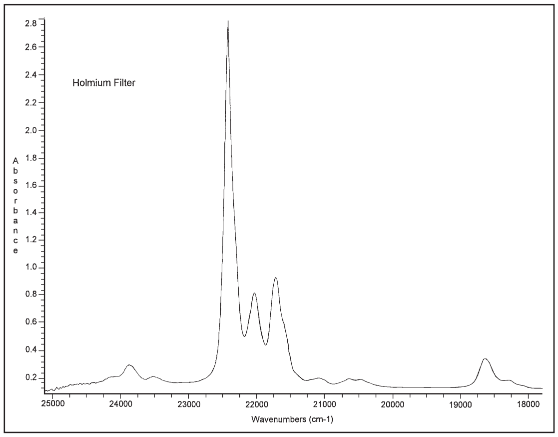

Figure 9

Combined Spectral Regions

Often we must provide spectral information spanning a wide range of the visible and near-IR spectral regions. This may be accomplished by collecting individual spectra with the appropriate combination of beamsplitters, detectors and filters. Then we add the respective data files to form the entire spectral range. In Figure 10 we show the diffuse reflectance spectrum of yttrium chloride from 26,000 to 2,000 cm^{-1}. This spectrum was produced from the combination of the following optical elements and ranges: 26,000-22,000 cm^{-1} (Si, quartz, narrow blue filter), 22,000-17,000 cm^{-1} (Si, quartz, wide blue filter), 17,000-14,000 cm^{-1} (Si, quartz, SSP=0.5), 14,000-9,000 cm^{-1}, (Si, quartz, red filter), 9,000-2,000 cm^{-1} (PbSe, CaF_2).

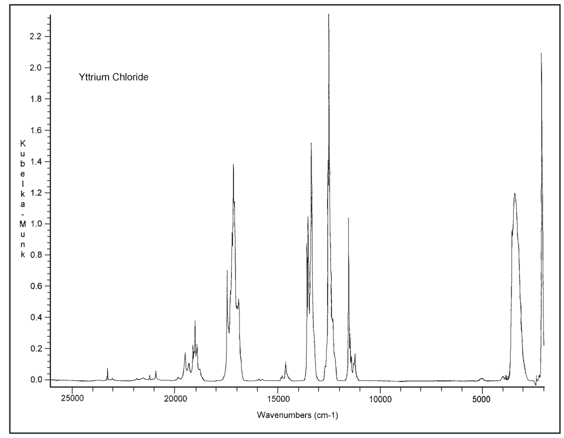

Figure 10

Conclusions

Many different configurations of optical elements are available to obtain spectra in the visible and near-IR spectral regions. The choice of a source, beamsplitter, detector and data collection parameters is made based upon the specific application and the spectral range requirement. Once a configuration is selected, optical filters may be used to further refine spectral results. Matching the system components with the application can provide benefits in SNR, spectral range and sampling speed.

Figure 11 provides a useful reference for the spectral range covered by each of the components discussed.

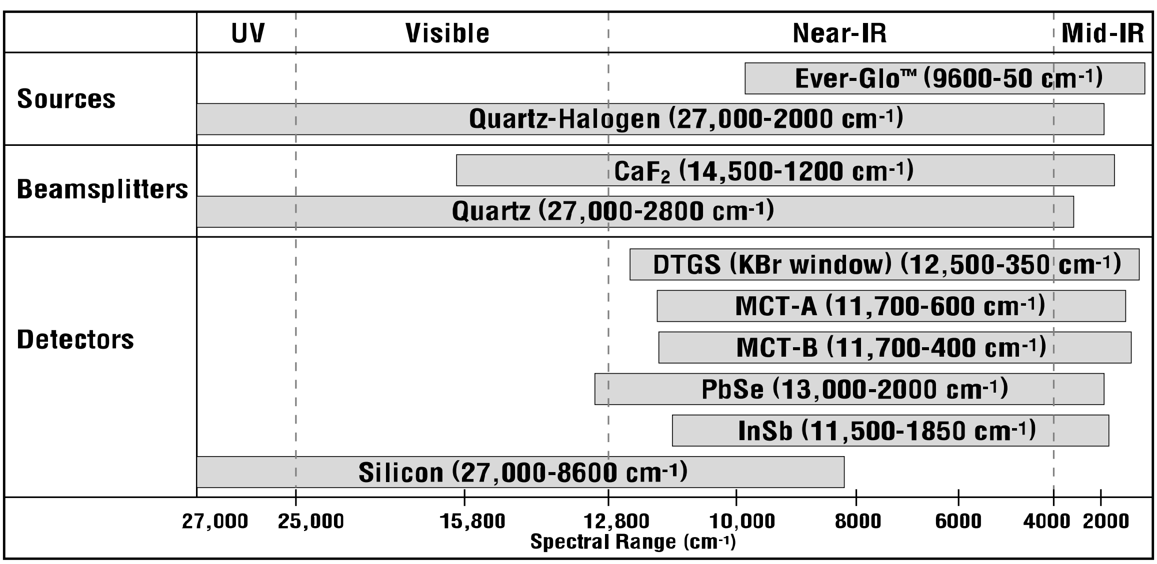

Figure 11

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