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Comparing the Performance of a Fiber Optic Probe to an Integrating Sphere

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Key Words

- FT-NIR
- Diffuse Reflectance
- Fiber Optic
- Integrating Sphere

Abstract

The performance of a fiber optic probe compared to an integrating sphere is assessed for diffuse reflectance spectra. Fundamental design differences are discussed. A repeatability study highlights the major difference in variability between the two techniques.

Introduction

Recent advances in fiber optic technology have made sweeping improvements in fiber optic probes used in near-infrared systems. The lower loss characteristics of the new fibers provide higher signal-to-noise. The flexibility of the fibers and the more robust designs of the fiber optic probe heads and cables allow routine remote sampling. Because of these advances, fiber optic analysis has become the most commonly used near-infrared technique. Nevertheless, there are important limitations of fiber optics that should be considered, especially when choosing between a fiber optic probe and an integrating sphere. Integrating spheres are typically more robust and reproducible than fiber optic probes.

Experimental

Diffuse reflectance spectra were acquired on a Thermo Scientific Antaris™ Fourier transform near-infrared (FT-NIR) Method Development Sampling System with the 2-meter Thermo Scientific SabIR™ Fiber Optic probe. Typical parameters were selected (100 scans with a resolution of 16 cm^{-1}). The same Spectralon® reference was used as the background for both the fiber optic and the integrating sphere to eliminate the choice of background as a source of variation. The sample used was the KTA-1920x wavelength standard. It is a mixture of talc and three rare earth oxides. The wavelength standard was placed in position over the integrating sphere window and a set of ten spectra was collected. For the next experiment, another set of ten spectra was collected with the SabIR placed in the remote probe stand. The final set of ten spectra was collected by keeping the wavelength standard in a fixed position and moving the fiber optic probe between each spectrum collected. RESULT™ software was used to collect the spectra and TQ Analyst™ software was used to display the spectra and produce the variance spectrum for each set of ten spectra.

Figure 1: Antaris FT-NIR Method Development System

Results and Discussion

Spectrum Response Curve

Fiber optic spectral response has significant drawbacks compared to that of the integrating sphere as can be readily seen by examining the backgrounds (Figure 2). Because near-infrared fiber optic fibers are typically silica, single beam spectra collected with the fiber optic probe have spectral features. There is a particularly strong band at 7235 cm^{-1} associated with the second OH overtone. Strong bands occurring in the background decrease the signal-to-noise level. In certain situations, the strong bands may be observed in critical experiments as artifacts, even in ratioed spectra. Silica fibers strongly absorb at lower wavenumbers and have significantly lower spectral response at 4000 cm^{-1} than the integrating spheres.

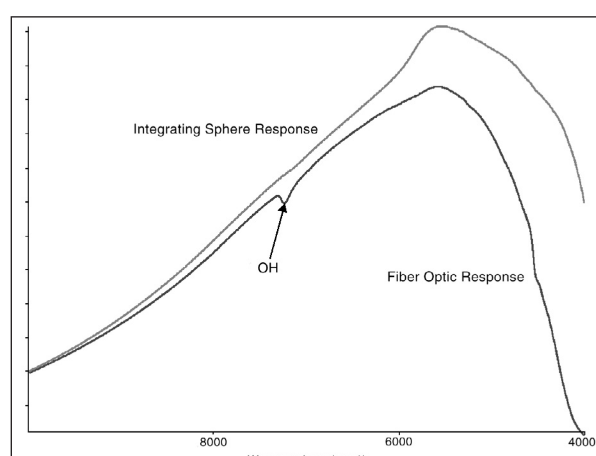

Figure 2: Near-infrared background response

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Throughput

Fiber optics channel spectral information by internally reflecting rays of near-infrared light that have an angle of incidence within the acceptance angle of the fiber. Near-infrared rays with angles greater than the acceptance angle strike the interior walls of the fiber at an angle less than the critical angle of reflection and are quickly lost. If the fiber is made bigger to accept more light, the fiber becomes inflexible giving up an advantage over an integrating sphere. As the fiber is bent and moved, the angle of the NIR ray may again be less than the critical angle and the ray is lost. This creates a change in the amount of energy that reaches the detector. Because integrating spheres collect rays reflected at any angle, integrating spheres collect more diffuse reflectance from the sample. The integrating sphere's diameter is optimized for a typical range of sample sizes to make collection of near-infrared energy even more efficient.

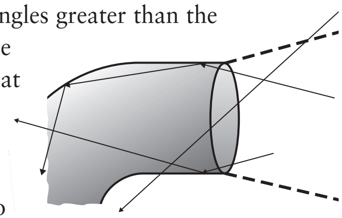

Figure 3: Fiber optic cable showing light loss

Noise Levels

In many practical situations, the integrating sphere and fiber optic modules have roughly equivalent signal-to-noise. Typically, the integrating sphere is slightly better. Because fiber optic cables attenuate the signal as a function of distance, the longer fiber selections will have even lower signal-to-noise.

Repeatability

Because integrating spheres are built into the body of the analyzer, spectral responses are more stable and repeatable. The use of a diffuse gold flag as a reference standard internal to the integrating sphere ensures that the background does not become contaminated or improperly used. The gold flag can be set to automatically collect a background before every sample even while the sample is in position. (To eliminate the choice of background as a source of variation the gold flag was not used for this study.)

When the KTA-1920x standard was measured with the integrating sphere, the ten spectra overlaid each other. Figure 4 shows ten spectra each viewed with common scale. It is difficult to see any differences. When the 1920x was measured with the fiber optic probe in the remote probe stand, the ten spectra overlaid on common scale showed significant offset (Figure 5).

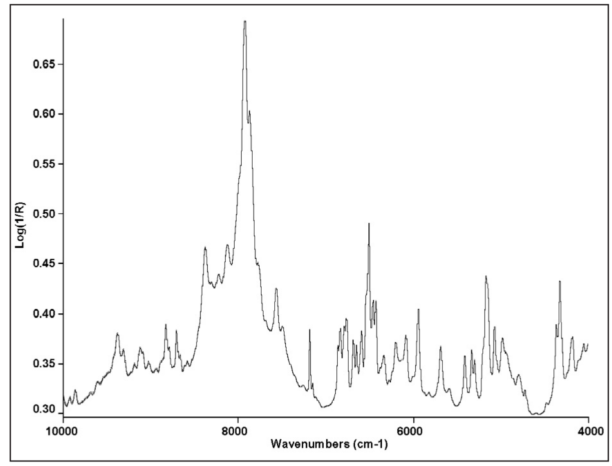

Figure 4: Integrating sphere reflection spectra of KTA-1920x standard

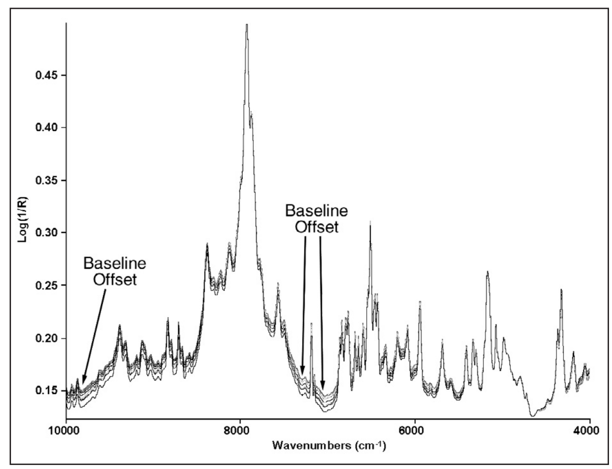

Figure 5: Fiber optic reflection spectra of KTA-1920x standard

Variance Spectra

Using variance spectra can enhance repeatability studies by calculating the standard deviation of the y-axis values at each spectral data point. The advantage of using standard deviation to describe variance is that the y-axis has the same units as the original spectra.

A variance spectrum can be calculated in RESULT software to evaluate or check the variability of a sampling presentation or method. TQ Analyst can be used to produce and save a variance spectrum from a set of spectra. The variance spectra of the integrating sphere set, the set with the SabIR in the stand and the set with the SabIR moving freely were calculated in TQ Analyst and are shown in Figure 6.

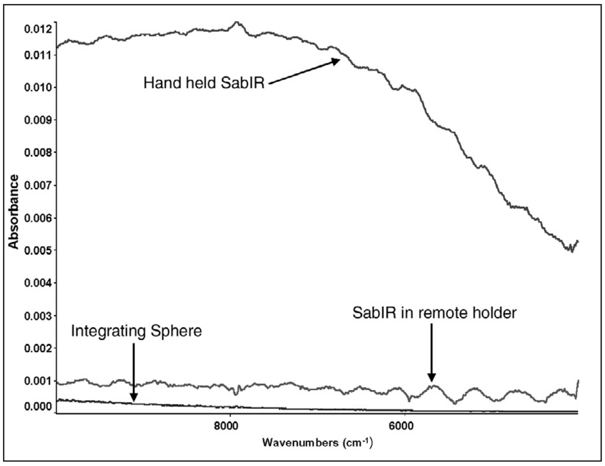

Figure 6: Variance spectra displayed on common scale

The integrating sphere variance spectrum shows very low variance (~400 micro-absorbance) for these experimental conditions. When the sample and SabIR are placed in the remote probe stand, the variance spectrum is slightly higher than the results from the integrating sphere (about 1 milli-absorbance). The wave-like feature at lower wavelengths is probably due to fringing differences from the window on the 1920x standard and the sapphire window on the SabIR caused by repositioning. This was not observed on the integrating sphere variance spectra because the 1920x sample placed on the stainless steel surface above the integrating sphere provides reproducible air gap.

The final variance spectrum was obtained by collecting 10 spectra with the sample position fixed, but the fiber probe moved between each collect, allowing the fiber bundle to swing free. The variance rises from seven milli-absorbance at lower wavenumbers to 11 milli-absorbance at 10,000 cm^{-1}. As the wavelength of the near-infrared gets shorter (at higher wavenumbers), the variation of intensity due to changes in the angle from bending the fiber increases. This was the result of holding the fiber optic probe by hand and allowing the cable to drape loosely. Other testing has shown that if the fiber bundle is even more tightly curved than in this experiment, the variance can increase by more than a factor of three due to increased light loss.

Conclusion

Fiber optic sampling is a useful tool for FT-NIR analysis. The ability to bring the analysis to the sample increases the flexibility of FT-NIR methods. It is not, however, the only choice.

Other sampling methods, like integrating sphere diffuse reflectance techniques, should be examined as well. Integrating spheres have better reproducibility and are easier to use.

With the robust design of the Antaris analyzer, it is now possible to bring the FT-NIR instrument to the sample. Placing samples in disposable glass vials simplify operation by eliminating a cleaning step required for fiber optic probes. The integrating sphere has become a viable alternative to fiber optic sampling due to the portability of the instrument, improved performance, ease of use and reproducibility.

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