Verifying the Performance of the Fiber Optic Reflectance Probe on the Antaris™ FT-NIR Analyzer

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Key Words

Antaris
Fiber Optic
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Abstract

The ability to obtain high-quality sample spectra remotely through several meters of low-OH optical fiber is one of the significant advantages of Fourier transform near-infrared (FT-NIR) spectroscopy. The Antaris, by Thermo Nicolet Industrial Solutions, was designed to provide the highest possible performance with the SabIR™ fiber optic reflectance probe.

Introduction

A major reason for the increased popularity of FT-NIR spectroscopy is the ability to acquire high-quality spectra from remote samples by means of fiber optics. Fiber optics are often used to ruggedly mount sample probes directly into a process reactor or other manufacturing system. Fiber optics can also be used with a reflectance probe to provide a convenient way to manually check the quality of materials by placing the probe in direct contact with the sample. If the probe is also designed to provide a remote trigger and an indicator signal, various lots of material can be rapidly and accurately screened. The Antaris FT-NIR analyzer has an optimized fiber optic port that focuses the light from the spectrometer onto a fiber optic connector and then focuses the return near-infrared (NIR) signal onto a high sensitivity InGaAs detector. Thermo Nicolet Industrial Solutions offers the SabIR reflectance probe, shown in Figure 1, a rugged diffuse reflectance probe for use with the Antaris instrument.

Figure 1: Picture of the Antaris Fiber Port and SabIR reflectance probe

Figure 1 also shows the electronic connector that interfaces the SabIR probe head to the instrument. This interface allows both remote start-of-scan and the indicator lights to report the outcome of the analysis

Experimental

The SabIR probe consists of a bifurcated, randomly mixed bundle of low-OH optical fibers mounted in a stainless steel probe head with an angled sapphire window on the tip of the probe. Half of the fibers bring the light from the spectrometer to the window and the second half returns the reflected light back to the detector.

A mixture of lactose and talc was used to test the stability and precision of the fiber optic probe. These data were obtained at 2 cm^{-1} resolution to test the effects of resolution on analysis results. Several basic tests used for traditional reflectance analysis were carried out to compare the performance of the SabIR with the Integrating Sphere on the Antaris. All spectra for this study were acquired using a Thermo Nicolet Industrial Solutions Antaris FT-NIR analyzer configured with a CaF_2 beamsplitter, a Tungsten-Halogen source and an InGaAs detector.

Results and Discussion

Spectral noise and spectral response are two basic spectral measurements that provide easy verification of overall performance. Background spectra were acquired for both the SabIR fiber optic probe and the integrating sphere with a diffuse gold reference positioned over the sapphire window. The spectral noise was calculated for each sampling system by acquiring a second spectrum from the gold reference. The RMS noise in the 6000 cm^{-1} spectral region was less than 20 micro-absorbance units with both reflectance techniques. Because the polystyrene validation sample is mounted in the spectrometer module before the sampling systems, a reference spectrum can be obtained for each sampling technique by moving the polystyrene into the beam while leaving the gold reference on top of the window. A comparison of the polystyrene spectrum acquired with the SabIR probe and the one from the integrating sphere is shown in Figure 2. A spectral subtraction of the two spectra acquired with different detectors reveals that the residual artifacts are less than +/- 2 milli-absorbance units. This result provides evidence that any spectral difference in the reflectance measurements is caused by sampling and not spectrometer linearity. The second two spectra in Figure 2 show the true reflectance spectra from a sample of white polystyrene plastic placed on top of the probe window. While these spectra are quite similar there are differences due to the expanding beam and acceptance angle of the fiber optic probe. This results in a wavelength dependent difference of the pathlength of the light reflected in the sample. Because of this difference, care should be taken in transferring a method that was developed on an integrating sphere to the SabIR probe.

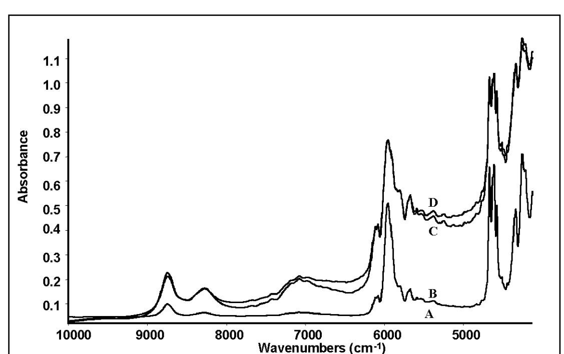

Figure 2: Comparison of polystyrene spectra. A) Reference spectrum acquired with the SabIR. B) Reference spectrum acquired with the Integrating Sphere. C) Sample spectrum with the SabIR. D) Sample spectrum with the Integrating Sphere

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To verify the wavelength accuracy of the SabIR probe we chose the KTA-1920x reference, a combination of talc with heavy metal oxides used in the NIST SRM 1920a sample. High-resolution spectra of pure talc and this reference material are shown in figure 3. Another important performance consideration with a fiber optic probe is sampling precision. For this test a simple quantitative analysis method using the classical least squares algorithm was created to determine the % talc in a lactose/talc mixture. One hundred repetitive measurements were made on a sample over a 12-hour time period. A spectrum of the mixture is shown at the bottom of figure 3.

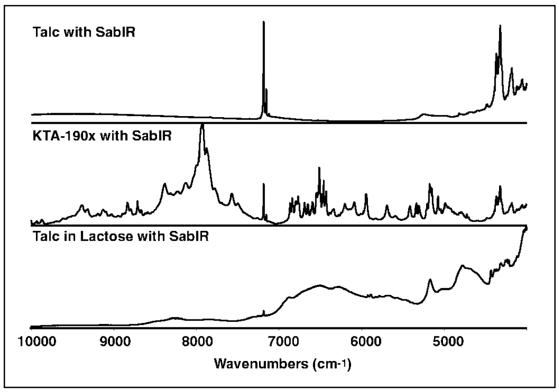

Figure 3: High-resolution spectra from samples containing talc

A chart of the repeat measurements is shown in figure 4. No attempt was made to control the temperature in the room and the optical fiber was allowed to swing freely (The minimum bend radius of the optic fiber allows excellent flexibility and sensitivity). A single background was acquired before the test so that we could check for any effects from instrument drift. No trend can be observed in the results in figure 4 and the standard deviation of 0.037 is quite good.

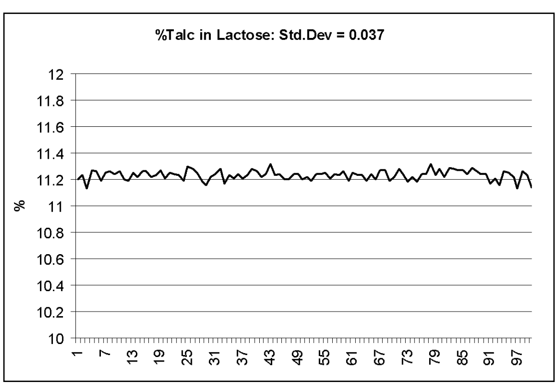

Figure 4: Results of 100 repeat measurements of a mixture of lactose and talc over 12 hours

Conclusion

We have described a number of tests to verify the performance of a new reflectance fiber optic probe and compared its spectra with similar spectra acquired with an integrating sphere. Although slight differences were observed in comparative spectra, the performance of the SabIR was very similar to the integrating sphere. Overall wavelength accuracy based on the spectrum from the KTA-1920x reference is good and the spectral response is also good with a spectral noise level similar to the integrating sphere. We observed no significant drift over time with the optical probe. We believe that the SabIR probe can be reliably used to acquire spectra directly from containers or packages.

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