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Verification of Wavelength Accuracy in an FT-NIR Spectrometer

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Key Words

- **FT-IR**
- **NIR**
- **Wavelength Accuracy**

Abstract

The success of many FT-NIR analyses is often limited by the wavelength accuracy of the instrument. The objective of this work was to determine the wavelength stability of the Thermo Scientific Antaris™ FT-NIR spectrometer over time and to evaluate the wavelength accuracy between several systems. Spectra were acquired from atmospheric water vapor, NIST SRM 2035 glass, and a polystyrene reference mounted in the validation wheel of the instrument. The effects of resolution and beam path optics on line shapes were also evaluated.

Introduction

The development of sophisticated statistical analysis techniques and reliable software packages has resulted in an increased use of near infrared spectroscopy in many material analysis or identification applications. While many of these chemometric methods can compensate for model non-linearities in the measured intensity values, small shifts in the wavelength positions of the spectral data points can create a much greater problem. For this reason it is very important that the instrument has excellent long-term wavelength stability. FT-NIR systems that use an internal HeNe laser for referencing generally provide excellent wavelength precision. However, it is more difficult to ensure that high wavelength accuracy is maintained among different instruments. In this paper we will describe a three-step process for verifying the wavelength accuracy of a FT-NIR spectrometer. The first step involves the acquisition of a high resolution spectrum of atmospheric water vapor, which can be used to verify the basic wavelength accuracy of the optical system with a very high degree of precision. In the second step, a spectrum acquired from the NIST SRM 2035 standard reference material is used to verify the actual spectral quality under normal operating conditions. In the third step, a spectrum is acquired from a sample of polystyrene plastic mounted on a computer-controlled wheel within the spectrometer. The spectrum acquired from this sample can be used for everyday performance verification.

In this paper, the results of a series of tests performed on several different instruments to determine the level of wavelength accuracy will be reported. Because the Antaris spectrometer can be configured with three different optical paths for transmission, reflectance, or fiber optic applications, measurements were made for each sampling configuration. Each beam path is computer-controlled and has an optimized detector. The internal polystyrene reference can be used with each of the optical configurations to verify performance.

Experiment

All spectra were acquired with an Antaris FT-NIR spectrometer configured with a tungsten halogen source, a CaF_2 beamsplitter, and indium gallium arsenide (InGaAs) detectors. High resolution spectra of atmospheric water vapor were acquired at 2 cm^{-1} resolution with two levels of zero filling. Norton-Beer weak apodization was used instead of Boxcar to reduce the side lobes while providing reasonably narrow line shapes. The spectra from the polystyrene reference and the NIST SRM 2035 standard were acquired at 8 cm^{-1} resolution with one level of zero filling using the Norton-Beer-Medium apodization function. Sixty-four scans were averaged to improve the signal-to-noise. The results of the tests were stored in an Excel accessible file for further analysis.

Results and Discussion

The high resolution spectrum of atmospheric water vapor contains sharp peaks with good signal-to-noise in the 4000 cm^{-1}, 5400 cm^{-1}, 7300 cm^{-1}, and 8800 cm^{-1} spectral regions. An expansion of the peaks in the middle of the NIR region is shown in Figure 1. While these peaks are good for determining the wavelength accuracy of the basic instrument, many NIR applications do not require 2 cm^{-1} resolution, so a more appropriate reference material is needed. Figure 2 contains the NIR spectra from the NIST SRM 2035 standard and a sample of 0.75mm (30 mil) thick polystyrene plastic.

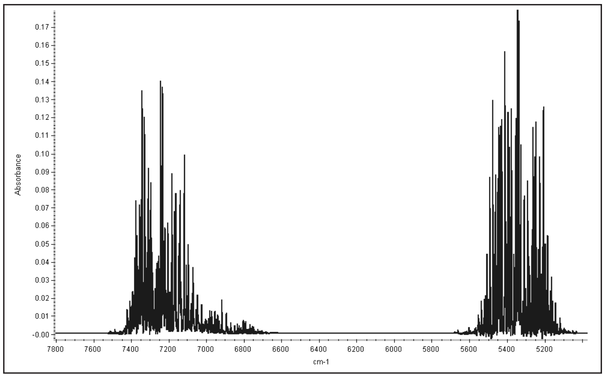

Figure 1: High resolution spectrum of atmospheric water vapor showing the rotational bands

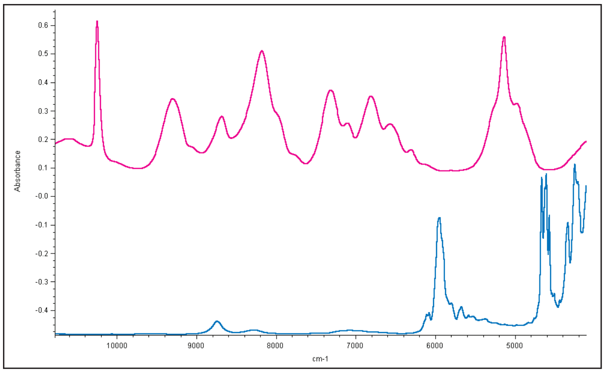

Figure 2: FT-NIR spectra from NIST SRM 2035 (top) and the internal polystyrene reference (bottom)

A peak-location algorithm was used to calculate the peak positions for a number of peaks in the high resolution water vapor spectrum, while correcting for the size of the aperture in the instrument. These peak values were compared to the corresponding values reported in the HITRAN 1996 Molecular Data Base, which contains the locations of over 20,000 lines in the high resolution water spectrum. For the 10 peaks selected to span the NIR region the average difference of the calculated peak locations to the HITRAN96 was 0.027 cm^{-1}. The peak at 7299.45 cm^{-1} has been used by several researchers to monitor the wavelength precision of NIR spectrometers.

A spectrum from the NIST SRM 2035 standard was acquired on five different instruments. The peak location of the sharp band at 10245 cm^{-1} was calculated on each system. The standard deviation was 0.08 cm^{-1}.

To measure the wavelength stability of the instrument, spectra were acquired over three days and peak maxima for a number of the peaks in the spectrum were calculated. The instrument was in a normal environment with no special humidity or temperature controls. Figure 3 shows a plot of the measured value for the peak at 7299 cm^{-1} as a function of time. For this experiment the average value was 7299.370 cm^{-1} with a standard deviation of 0.002 cm^{-1}. An examination of the data in Figure 3 suggests that the small degree of drift corresponds to changes in the room temperature and humidity as the thermostat resets over the weekend.

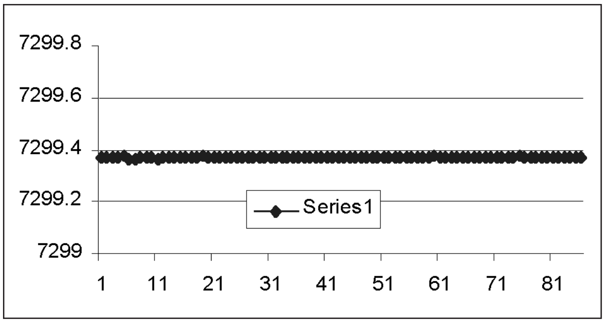

Figure 3: Wavelength stability test once per hour

Because the three sampling systems on the instrument use different optics, it is necessary to measure the wavelength accuracy on each. The internal polystyrene reference mounted in the instrument can be used to acquire a test spectrum with each of the optical paths. The location of the polystyrene peak near 4570 cm^{-1} was calculated for each spectrum and a control chart of all 18 values is shown in Figure 4. The low value for point 14 was caused by the use of an optical test fiber with a smaller diameter on this particular system. The small diameter fiber became the limiting aperture on the system resulting in a slight peak shift. Using the same test fiber on all of the systems would have eliminated this variance.

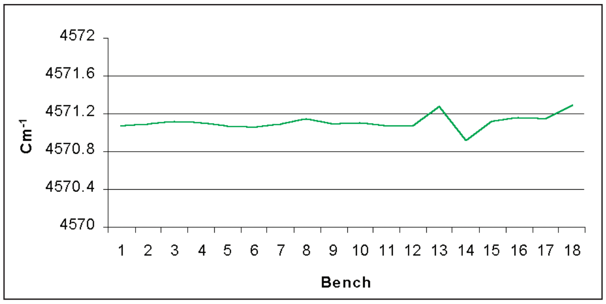

Figure 4: Polystyrene peak location on six systems

Conclusion

In this technical note, we have described three different reference materials that can be used to verify wavelength accuracy in the NIR spectral region. While the water vapor spectrum provides the highest resolution peaks, it does not represent the operating conditions normally used for NIR spectroscopy. The SRM 2035 is a certified reference that can obviously be used for instrument validation. However, it is very costly and probably cannot be integrated into the instrument. It may be necessary to use SRM 2035 in certain regulatory situations, but there is still a need for an every day method of ensuring instrument performance. We have demonstrated that polystyrene can be used as a reliable wavelength validation reference that is integrated into the instrument for ease of use, reproducibility, and protection. The results reported in this note suggest that all three techniques are appropriate in specific circumstances.

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