

# Verifying the Performance of the Integrating Sphere Module on the Thermo Scientific Antaris FT-NIR Analyzer

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## Abstract

Many important application areas for Fourier transform near-infrared (FT-NIR) analysis involve the acquisition of diffuse reflectance spectra directly from powdered or solid samples. Thermo Scientific Antaris™ FT-NIR analyzers offer diffuse reflectance capabilities. The diffuse reflectance module of the Antaris system is based on an optimized internal integrating sphere concept. In this report, we will describe a series of tests performed on the Antaris Solid Sampling System to verify key performance features that are important in a number of reflectance applications.

## Introduction

A major reason for the increased popularity of FT-NIR spectroscopy is the large number of samples that can be rapidly identified or analyzed with little or no sample preparation. High-quality spectra can be acquired directly from solid samples and from powders in glass or plastic containers by diffuse reflectance spectroscopy. We developed the Antaris FT NIR System (Figure 1) to provide an easy way to rapidly analyze solid and powdered materials.

A major component of the diffuse reflectance sampling system is the gold-coated integrating sphere that collects the reflected energy from a sample placed on the integrating sphere window. The analyzer has been designed to provide a large, open area around the protective sapphire window so large samples or special accessories can be easily positioned at the focal point above the integrating sphere. A drawing of the integrating sphere module is shown in Figure 2. An important feature of this design is the computer-controlled diffuse gold reference reflector, which can be automatically positioned in front of the window to acquire a background spectrum. This internal positioning not only eliminates damage to

the reference but also allows background spectra to be acquired without moving the sample.

Four major performance areas having significant effects on an analysis are 1) instrument sensitivity, 2) wavelength accuracy, 3) spectral resolution and 4) instrument stability. In this report, we will describe a series of tests performed on the Antaris FT-NIR analyzer integrating sphere module to determine its performance in each of these four areas.

## Experimental

All spectra for this study were acquired by placing the samples directly upon the integrating sphere module of an Antaris FT-NIR analyzer. An example of an appropriate glass sample vial is shown in Figure 1. The sensitivity test was performed at 8  $\text{cm}^{-1}$  resolution, and the precision test was performed at 4  $\text{cm}^{-1}$  resolution. The resolution test was performed with the National Institute of Standards and Technology (NIST) SRM 1920a reflectance standard at various resolutions. The NIST reflectance standard is a powdered mixture of heavy metal oxides mounted in a special holder (Figure 1). All backgrounds were collected using the internal gold flag.

## Results and Discussion

The instrument sensitivity was determined by first collecting a background spectrum with the gold flag in the beam and then collecting a 30-second sample spectrum without moving the reference flag. The root-mean-square (RMS) noise on the resulting 8  $\text{cm}^{-1}$  resolution spectrum was excellent with less than 10 micro-absorbance units in the 4500  $\text{cm}^{-1}$  and 6000  $\text{cm}^{-1}$  spectral regions. This corresponds to a signal-to-noise ratio of 100,000 to 1 for a diffuse reflectance spectrum with features that approach one in  $\log(1/R)$  units. The wavelength accuracy was verified by calculating the locations of several peaks in the water vapor spectrum and comparing them to the values reported in the HITRAN 1996 reference table. The reference spectrum of atmospheric water vapor used for this measurement is shown in Figure 3. This spectrum was obtained by introducing moist air into the instrument.

At 4  $\text{cm}^{-1}$  resolution, the differences between the measured peaks and the corresponding values reported in the HITRAN database were less than 0.3  $\text{cm}^{-1}$  (0.1 nm). The instrument resolution was verified by measuring spectra acquired from the NIST SRM 1920a reflectance standard at spectral resolutions between 2  $\text{cm}^{-1}$  and 32  $\text{cm}^{-1}$ . The five spectra are shown in Figure 4.



Figure 1: The Antaris Solid Sampling System with internal integrating sphere module

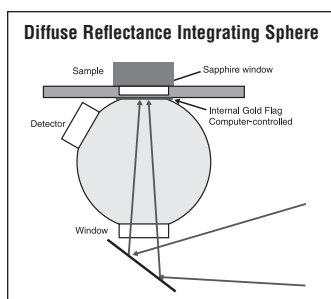


Figure 2: Schematic drawing of the internal integrating sphere module

### Key Words

- Antaris
- FT-NIR
- Integrating Sphere
- Reflectance

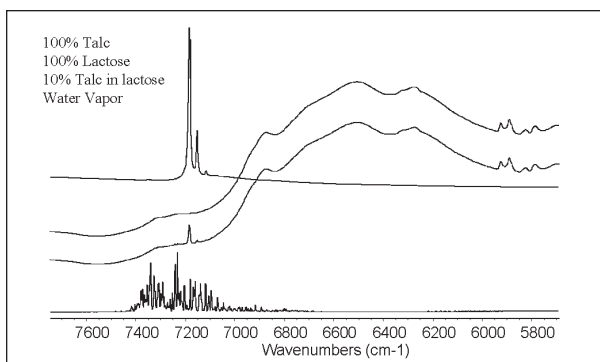


Figure 3: Spectra acquired from samples of talc, lactose, a talc/lactose mixture and water vapor

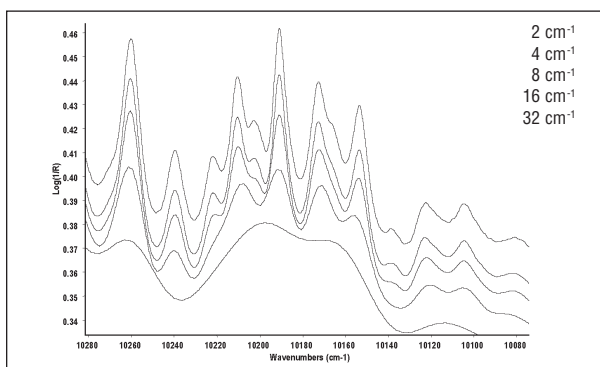


Figure 4: Spectra acquired from NIST SRM 1920a at different resolutions (2-32  $\text{cm}^{-1}$ )

The choice of resolution may not be important for samples with broad peaks, but many of the active pharmaceutical materials have spectra with sharp peaks. It is important to remember that while higher resolution may improve the specificity of the analysis with an FT-NIR system, the spectral noise also increases with improved resolution. For many applications, a resolution of  $4 \text{ cm}^{-1}$  or  $8 \text{ cm}^{-1}$  provides the best compromise between sensitivity and specificity.

A good example of an application where higher resolution is appropriate is the analysis of talc in lactose. Talc is found in numerous pharmaceutical tablets and has a very sharp peak in the  $7200 \text{ cm}^{-1}$  region of the NIR spectrum. To test the long-term stability of the analyzer with the integrating sphere module, repetitive measurements were made on a powder mixture of talc in lactose. A sample was prepared by adding approximately 10% talc to a vial containing powdered lactose. Spectra were then acquired from the 10% mixture after thorough mixing and from the two pure starting materials. The sample vials were placed on the integrating sphere and the spectra were measured directly through the bottom of the vials. These spectra are shown in Figure 3.

The Thermo Scientific TQ Analyst™ method development software was used to create a simple classical least squares (CLS) method to determine the % talc in the sample. Once the TQ Analyst method was tested, a workflow was developed in the Thermo Scientific RESULT™ software Integration mode to acquire a spectrum every 15 minutes and determine the % talc. The purpose of this repetitive test was to measure any changes in the calculated results with no movement or change of sample. Normally, a new background would be acquired before each sample spectrum using the automated reference, but for this test, the option of collecting one background for each workflow run was chosen. By only using one background for the entire test, the effect of instrument stability on the measured concentration can be observed.

Figure 5 shows a graph of % talc as a function of time over a 24-hour period in a laboratory with no special temperature control. Virtually no drift can be observed over the 24-hour period. The standard deviation for the 100 repeat measurements was 0.029%, with a mean value of 9.997%.

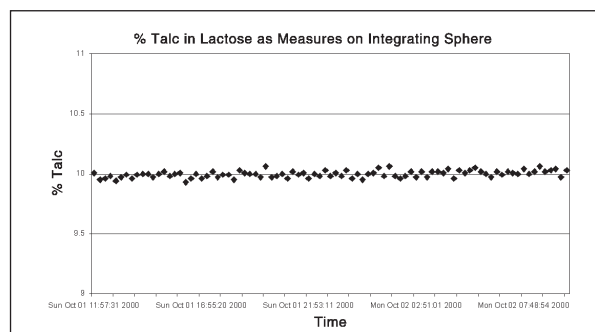


Figure 5: Overnight precision test for talc mixture with integrating sphere module

## Conclusions

The goal of this study was to evaluate the performance of the integrating sphere module on the Antaris FT-NIR analyzer. The sensitivity of the system was determined by measuring the background noise of the system with the gold reference as the sample. The wavelength accuracy of the system was determined by comparing the measured peak locations of water vapor with the values reported in the HITRAN database. The spectral resolution was determined by measuring the spectrum from the NIST SRM 1920a reflectance standard at various resolution settings. The instrument precision was determined by making repetitive measurements on a sample over a 24-hour period. The results of these tests demonstrate the level of performance that can be expected for diffuse reflectance applications. Similar tests have been performed on more than 20 systems with similar results. This proven instrument performance provides a very powerful solution for many diffuse reflectance applications.

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