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Measurement Reproducibility – A Case Study of the Long-Term Performance of an FT Near-Infrared Analyzer

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Key Words

- Antaris
- FT-NIR
- Method Transfer
- Stability

Abstract

In this study we report on the long-term stability of the Thermo Scientific Antaris™ FT-NIR analyzer. A set of NIST-traceable photometric linearity standards was run 60 times over a period of almost three years on the same instrument. We will discuss the results including the implications of this analyzer's remarkable stability for method transfer and process applications.

Introduction

One of the most important criteria for an analyzer is long term stability, i.e., the ability of the instrument to measure the same values over long periods of time from the same standards. If the measured values tend to drift over time, then frequent re-calibration as well as re-validation efforts will be required to keep the instrument performing to specification. For Near-IR methods, with hundreds of standards, this becomes a costly and time-consuming operation. This study describes the performance of a single Antaris FT-NIR analyzer measuring known absorbance values of a series of NIST-traceable photometric linearity standards over the course of three years.

The Antaris Method Development system (Figure 1) is a dedicated, FT-Near IR analyzer that uses the 4000 cm^{-1} to 12000 cm^{-1} spectral region for QA/QC applications in the pharmaceutical, chemical, pulp and paper, food and polymer industries. Instrument qualification and method validation are accomplished using an internal validation wheel (Figure 2) that contains a NIST-traceable polystyrene standard as well as five NIST-traceable photometric linearity standards. The analyzer is gasket-sealed to prevent premature aging of or damage to the standards.

Figure 1

Figure 2

NIST photometric linearity standards at nominal transmission levels of 2%, 10%, 20%, 40% and 80% were run on a single Antaris FT-NIR analyzer (serial number AFA0000197) from December 21, 2000 through November 6, 2003. The standard samples were fixed on the instrument's internal validation wheel and the spectra were collected with the transmission module. The absorbance values of these standards were recorded at USP-recommended wavelengths of 5000 cm^{-1} (2000 nm), 6250 cm^{-1} (1600 nm), and 8333 cm^{-1} (1200 nm). Duplicate measurements were taken each time and the average value was recorded. Average values were recorded 60 times over the indicated time period. Data collection consisted of 16 co-added scans at 8 cm^{-1} resolution resulting in a collection time of approximately 8 seconds.

The photometric linearity test that was run in this experiment is a recommended Near-IR performance test for measuring Y-axis repeatability. The United States Pharmacopoeia (USP) has addressed the operational qualification of Near-IR instrumentation in Chapter <1119> using this test at the wavelengths listed above. Fourier transform technology has the added advantage of an internal HeNe laser for X-axis (frequency) calibration. The Antaris FT-NIR analyzer uses the laser to achieve frequency accuracy of better than 0.001 cm^{-1} measured at the 4332 cm^{-1} peak of polystyrene.

Long-term stability bears heavily on method transfer, the ability to transfer chemometric methods from one instrument to another without the need for re-calibration. If the measured values of a known standard change over time, then the method will have to be re-validated or even re-calibrated, nullifying many of the benefits of Near IR. Instrument design features of the Antaris analyzer such as pinned-in-place optics, a dynamically-aligned interferometer, and automatic, internal background collection all contribute to an exceptionally stable optical bench resulting in the same measured values year in and year out. Such remarkable stability contributes to seamless method transferability from instrument to instrument.

Results and Discussion

The data shown in Figure 3 plots the absorbance values of the NIST standards at three wavelengths over almost three years. The resulting 15 data sets demonstrate the extraordinary long-term stability of the Antaris FT-NIR analyzer. If the measured values were changing significantly over time, there would be a pronounced change in the absorbance values shown in Figure 3 from date to date. In this case, the data shows that these absorbance values do not exhibit any significant changes over time.

Table 1 shows the average, standard deviation and percent standard deviation for the predicted numbers across all standards and all wavelengths. The percent standard deviation varies between 0.11% and 0.73% depending on the percent transmission of the standard.

Conclusion

The Antaris FT-NIR analyzer exhibits superior stability characteristics over long periods of time, contributing in large part to the overall ruggedness of the instrument. This stability is central to the success of transferring chemometric methods from one system to another which, in turn, allows the customer to avoid time consuming re-calibration. In addition, low drift/high stability is desirable for instruments that are used in line for the most demanding types of industrial applications.

	2% NIST STANDARD (8333 cm^{-1})	2% NIST STANDARD (6250 cm^{-1})	2% NIST STANDARD (5000 cm^{-1})
Average	1.7380	1.4737	1.3457
St. Dev.	0.0020	0.0020	0.0016
% St. Dev.	0.11	0.13	0.12

	10% NIST STANDARD (8333 cm^{-1})	10% NIST STANDARD (6250 cm^{-1})	10% NIST STANDARD (5000 cm^{-1})
Average	1.1217	1.0520	1.0185
St. Dev.	0.0017	0.0017	0.0016
% St. Dev.	0.15	0.16	0.16

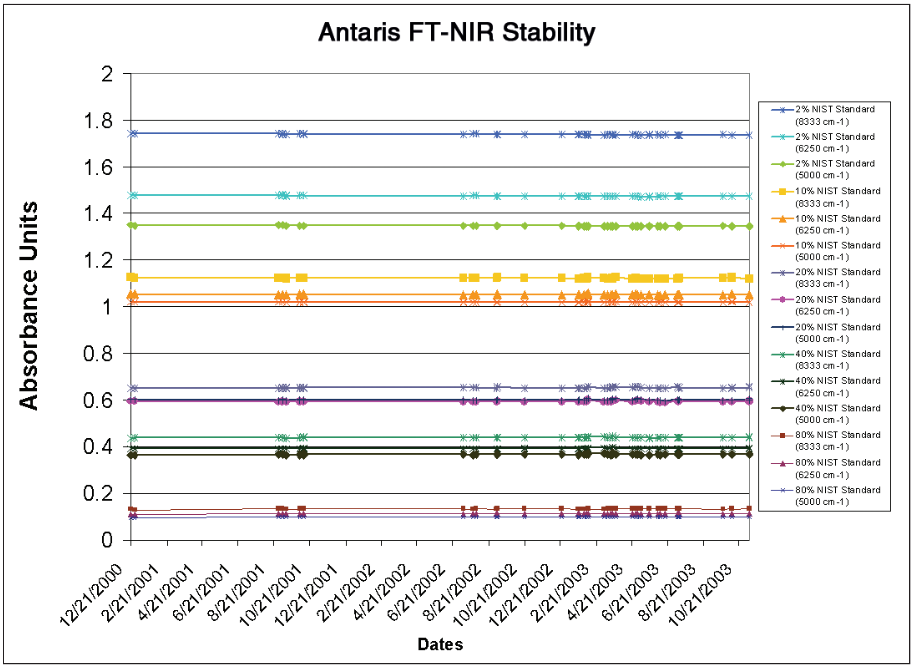
	20% NIST STANDARD (8333 cm^{-1})	20% NIST STANDARD (6250 cm^{-1})	20% NIST STANDARD (5000 cm^{-1})
Average	0.6522	0.5935	0.6005
St. Dev.	0.0027	0.0026	0.0026
% St. Dev.	0.42	0.44	0.43

	40% NIST STANDARD (8333 cm^{-1})	40% NIST STANDARD (6250 cm^{-1})	40% NIST STANDARD (5000 cm^{-1})
Average	0.4389	0.3918	0.3666
St. Dev.	0.0018	0.0018	0.0017
% St. Dev.	0.42	0.45	0.47

	80% NIST STANDARD (8333 cm^{-1})	80% NIST STANDARD (6250 cm^{-1})	80% NIST STANDARD (5000 cm^{-1})
Average	0.1344	0.1122	0.0991
St. Dev.	0.0008	0.0008	0.0007
% St. Dev.	0.60	0.67	0.73

Table 1

Figure 3



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