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Incoming analysis of sugars and polysaccharides

Introduction

Confirming the identity of incoming raw materials is becoming increasingly important in the pharmaceutical, food, and specialty chemicals industries. Many of the traditional analysis methods involve lengthy wet chemistry or chromatographic approaches, making them time and resource intensive. Near-infrared spectroscopy (NIR) can offer a quick and reliable method for verifying the identity of bulk materials. The samples can often be analyzed directly in a small sampling vial or bag within a matter of seconds.

This application note describes an experimental approach for identifying and determining the similarity of lots containing glucose, lactose, sucrose, and cornstarch. The samples were run in small glass vials using the Thermo Scientific™ Antaris™ FT-NIR Analyzer, a dedicated near-infrared Fourier-transform analyzer that covers the region from 12000 cm-1 to 4000 cm-1. (Note: a newer model, the Antaris II FT-NIR Analyzer, is now available and builds on the capabilities of the first generation instrument used in this application note.) The calibrated method was designed to be used at multiple plant sites and was developed using calibration data from three separate Antaris Analyzers. The data from different spectrometers was combined to create a single calibration model to evaluate the spectrometer-to-spectrometer transferability of a single calibration.

Experiment

The Integrating Sphere Module was used to measure the spectra of the powdered samples directly through the bottoms of glass sample bottles (Figure 1), which were simply placed on the horizontal sapphire window where the diffuse reflectance spectrum was measured. The data was collected at 4 cm-1 resolution with a collection time of 60 seconds per sample. Example spectra are shown in Figure 2.
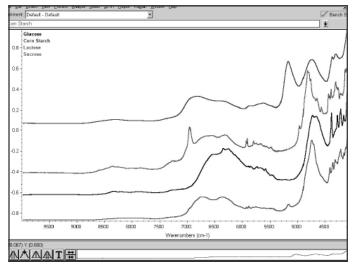

Figure 2. Example spectra of polysaccharide samples collected with the Integration Sphere Module on the Antaris FT-NIR Analyzer.

Figure 1. Powder analysis with the Antaris FT-NIR Analyzer.

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The identity of incoming raw materials can be determined in a variety of ways. In this particular case, a classification methodology in Thermo Scientific TQ Analyst™ Software was used to provide identification and purity information. The goal of this method was to create a cluster/group for each compound or class type through mathematical manipulation of the spectral data. These categories can then be used to classify an unknown spectrum. Multiple spectra of each compound type are obtained to account for typical variations encountered in real plant environments such as variations in purity and particle size. Additionally, while the overall shape of the spectra is generally reproducible from spectrometer to spectrometer, there are typically shifts in the spectral baseline related to variations in powder packing; these are also incorporated into the classification model.

A number of different mathematical algorithms can be used to develop classification models. The classification approach used in TQ Analyst Software uses principle component analysis (PCA). It is beyond the scope of this application note to detail the workings of PCA; simply put, it is a way to reduce a relatively large number of spectra down to a few that can be used to approximate any of the original spectra through linear combinations. The principle component spectra (PCS) are used to develop the classification model. In this experiment, each sample was analyzed twice on three different spectrometers, resulting in a total of 24 spectra, which were then reduced to five PCS that were used to create the classification model.
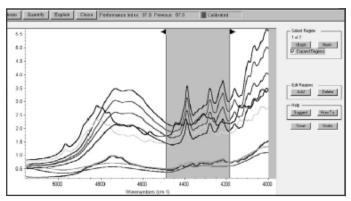

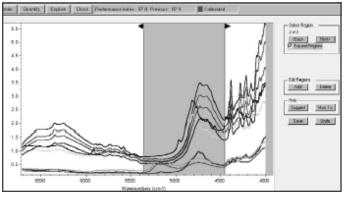Figure 3. One of two spectral regions used to develop the classification model.

Figure 4. One of two spectral regions used to develop the classification model.

Results

Two spectral regions were selected for the method (Figures 3 and 4) because they show significantly different spectral features among the compound types. TQ Analyst Software provides many options for spectral data pre-processing, including baseline treatment. For this experiment, a linear baseline correction was performed across each region.
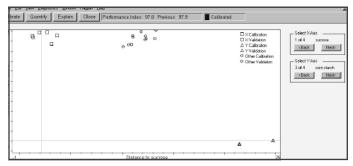

Figure 5. Linear baseline correction of spectral regions identified in Figures 3 and 4.

The results of the calibration are shown in Figure 5. The plot at the top of the figure shows a 2D representation of the class clusters. A good calibration will show tight, compound-class-specific clusters that are separated from the other class clusters. The table at the bottom of the figure shows the distance of each calibration/validation spectrum from the center of the nearest and next nearest class. From this information we find that all of the calibration/validation spectra were correctly classified. Also, for each sample (calibration and validation), the distance to the next nearest class center is many times larger than the distance to the expected class, indicating that the method should reliably and correctly classify future spectra.
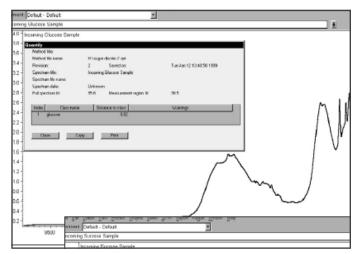

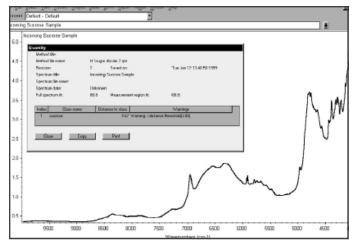Figure 6. Typical spectrum of a sucrose sample.

Figure 7. Spectrum of a sucrose sample contaminated with glucose.

For classification methods, a "distance to class" threshold can be set to indicate when the identified class for an unknown may be in error. Examples of typical outputs are shown in Figures 6 and 7. Figure 7 shows a sucrose sample contaminated with a small amount of glucose. In this example, the distance to the nearest class (sucrose) is larger than the allowable distance threshold, and the sample is flagged as being a potential problem.

Conclusions

NIR spectroscopy offers ease-of-sampling benefits such as the analysis of powders in glass bottles. Coupled with the method-development tools provided by TQ Analyst Software, this make the Antaris FT-NIR Analyzer a powerful tool for quality control.

Note: The data in this application note was collected using an older model Antaris FT-NIR Analyzer. Currently, Thermo Fisher Scientific offers an improved model, the Thermo Scientific Antaris II FT-NIR Analyzer, which offers superior performance over its predecessor model.

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