Application Note: 50822

Incoming Analysis of Sugars and Polysaccharides

Key Words

- Diffuse Reflectance
- Discriminant Analysis
- NIR
- Polysaccharides
- Sugars

Confirming the identity of incoming raw materials is becoming increasingly more important in the pharmaceutical, food, and specialty chemicals industries. Many of the traditional analysis methods involve lengthy wet chemical or chromatographic approaches. These analytical techniques are often time and resource intensive. Near infrared spectroscopy can offer a quick and reliable method for verifying the identity of bulk materials. Often the samples can 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 of glucose, lactose, sucrose, and cornstarch. The samples were run in small glass vials using the Nicolet™ Antaris™ FT-NIR analyzer, a dedicated near infrared (NIR) Fourier transform infrared analyzer that covers the NIR region from 12000 cm^{-1} to 4000 cm^{-1}. The calibrated method was designed to be used at multiple plant sites and was developed using calibration data collected from three separate Nicolet Antaris analyzers. Combining the data from different spectrometers to create a single calibration model allows an evaluation of spectrometer-to-spectrometer transferability of a single calibration.

Figure 1

The Integrating Sphere module was used to measure the spectra of the powdered samples directly through the bottom of the glass sample bottles (Figure 1). The sample bottle is simply placed on the horizontal sapphire window and the diffuse reflectance spectrum is measured. The data was collected at 4 cm^{-1} resolution with a collection time of 60 seconds per sample. Examples of the sample spectra are shown in Figure 2.

The identity of incoming raw materials can be determined in a variety of ways. In this particular case, the TQ Analyst™ software classification methodology was selected to provide identification and also to provide purity information. The goal of a classification method is to create a cluster or group for each compound or class type using some mathematical manipulation of the spectral data. In this way, the spectrum of an unknown can be used to determine which class or group the unknown belongs to. In this type of method, multiple spectra of each compound type are obtained. These multiple spectra should encompass the variations that are expected in the samples encountered in real plant environments. These sources of variation would include normal and acceptable variations in purity, particle size, and powder packing. The overall shape of the spectra is very reproducible from spectrometer to spectrometer but there are shifts in the spectral baseline, which are related to variations in powder packing. These variations become part of the variation incorporated into the classification model.

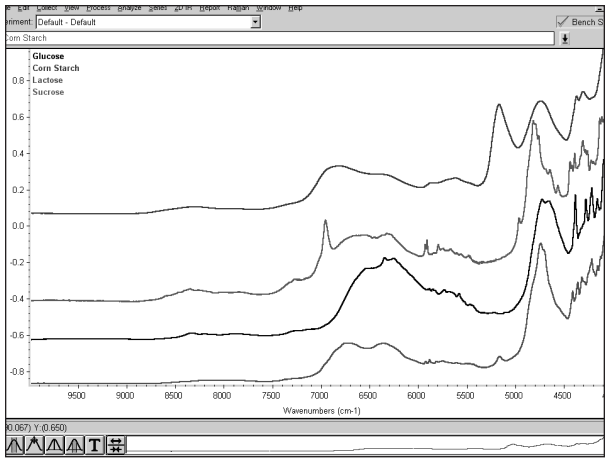

Figure 2

A number of different mathematical algorithms can be used to develop classification models. The classification approach used in TQ Analyst software involves the use of 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 spectra that can be used to approximate any of the original spectra through linear combinations. In the classification method development, the Principle Component Spectra (PCS) are used to develop the classification model. In this experiment each sample was analyzed twice on each of three spectrometers resulting in a total of 24 spectra. The 24 spectra were then reduced to five PCS spectra and were used to create the classification model.

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Two spectral regions were selected for the method. These regions are shown in Figures 3 and 4. These particular regions were chosen because of the significant differences in 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.

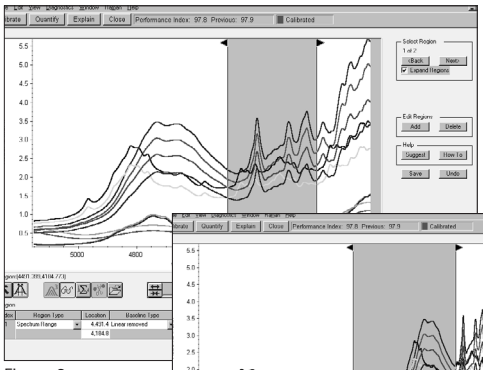


Figure 3

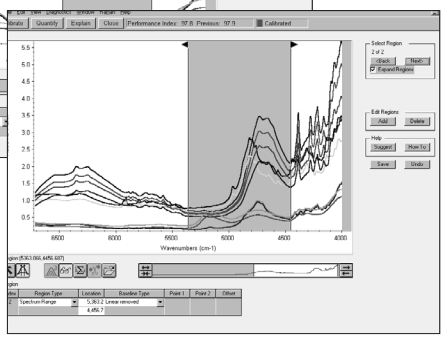


Figure 4

The results of the calibration are shown in Figure 5. The plot at the top of the figure shows a two dimensional representation of the class clusters. A good calibration will show tight, compound class specific clusters that are separated from the other class clusters. The results are explained in detail in the table at the bottom of the figure. In this table, the distance of each calibration/validation spectrum from the center of the nearest and next nearest class is indicated. 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. This is an indication that the method should be reliable and will correctly classify future spectra.

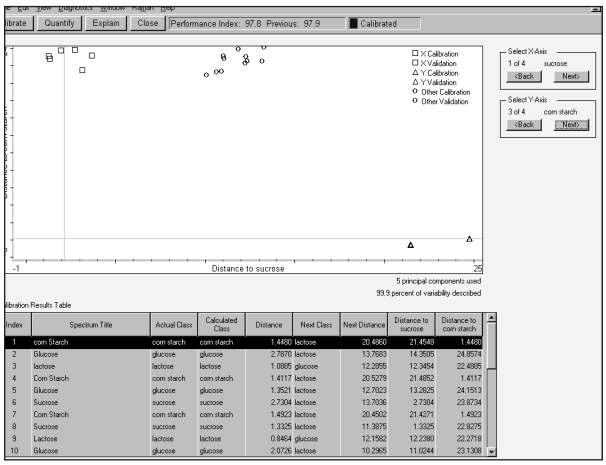
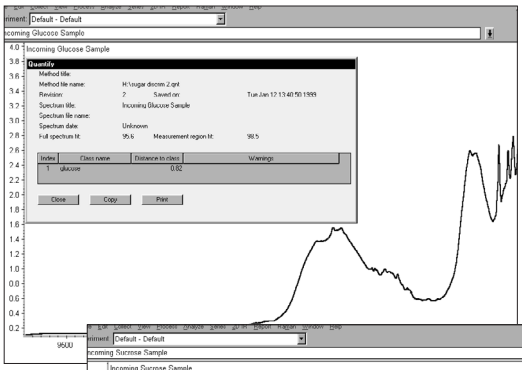


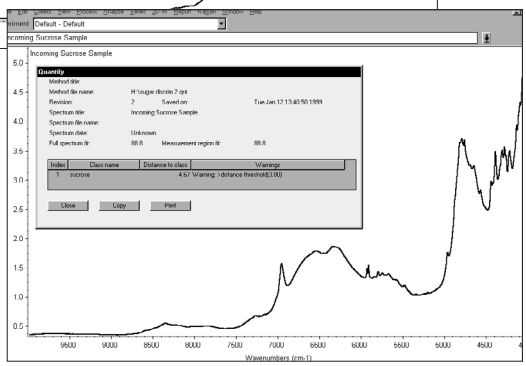
Figure 5

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 Figure 6 and Figure 7. The sample that was used to develop the results for Figure 7 was sucrose 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.

NIR spectroscopy offers ease-of-sampling benefits such as the analyses of powders in glass bottles. This ease-of-use coupled with the methods development tools provided by TQ Analyst software make the Nicolet Antaris FT-NIR analyzer a powerful quality control problem-solving tool.



Figures 6



Figures 7

In addition to these offices, Thermo Electron Corporation maintains a network of representative organizations throughout the world.

Australia
+61 2 9898 1244
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 532 4752
China
+86 10 5850 3588
France
+33 1 60 92 48 00
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