Application Note: F01

Analysis of Breakfast Cereal Using the SpectraStar 2400 NIR Spectrometer

Introduction

Near-infrared (NIR) technology has been used in the food, feed, and agriculture industries for over 50 years as a way to analyze for properties such as moisture, protein, fat, fiber, ash, amino acids, and more. NIR testing is fast (analysis in seconds), accurate, safe, usually nondestructive and requires minimal sample preparation with no reagents. NIR is extremely flexible and can be configured for the analysis of solids, liquids, oils, slurries, and suspensions. Accuracy is often equivalent to the wet chemical methods that it replaces. Its precision is almost always better.

Developed as a technique for predicting the chemical composition of a variety of unknown samples, near infrared (NIR) uses diffusely reflected light in the 800 to 2500 nanometer (nm) range to make a determination. Specifically, NIR light affects the molecular C-H, N-H, and O-H bonds. These bonds are directly related to the sample constituents of interest, such as fat, protein, moisture, fiber, starch, sugar, and amino acids, to name a few. Response to these bonds can be found throughout the NIR spectrum, but the primary combination bands for all of these properties, found above 1900 nm, are the most sensitive and generally provide the most accurate calibrations.

When NIR light hits a sample, part of the light is absorbed and part is diffusely reflected. The amount of absorbed light, at a particular wavelength, is directly proportional to the concentration of the constituent of interest. In other words, the more NIR light being absorbed at a particular wavelength, the greater the constituent (moisture, fat, protein, etc.) level in the sample.

A series of standard samples of known concentration, analyzed using a high accuracy reference method is scanned to measure the absorbance values at wavelengths throughout the NIR region. A calibration is then developed by using one of various mathematical models to correlate the reference lab values to the amount of absorbed NIR energy. The calibration can then be used to predict the constituent concentration of unknown samples.

In this report, the analysis of breakfast cereal is described.

Experimental

Instrumentation

All measurements were performed using a SpectraStar 2400 NIR spectrometer, equipped with a static drawer. The SpectraStar 2400 is a scanning monochromator-based NIR system that scans the optimum wavelength range of 1200-2400nm in 1nm steps. The SpectraStar 2400 utilizes an extended range InGaAs detector for enhanced stability and improved signal to noise ratio.

All calibration development and data management was performed using the CalStar software. CalStar is a Windows™ based software program that combines an intuitive, easy to use data management scheme along with the flexibility of using multiple calibration types, such as multiple linear regression (MLR) and partial least squares (PLS) to manage NIR data and develop calibrations.

All samples were analyzed by using the powder sample cup for the SpectraStar 2400. The powder cup provides consistent compaction for ground and powdered samples. Consistency in sample handling is crucial to accurate NIR measurements.

Sample Preparation

Samples were allowed to cool to room temperature and ground using a Retsch-type grinder. Samples were stirred and mixed before loading into the Unity powder cup.

Calibration Samples

Approximately 100 breakfast cereal samples were used in the development of moisture, fat, and sugar calibrations. The calibrations were developed for a specific type of breakfast cereal. The following table shows the moisture, fat, and sugar ranges of the calibration samples, along with the wet chemistry methods used to analyze them. As a secondary technique, NIR instruments are calibrated against a primary method. Performance of the NIR will never be better than the repeatability of the wet chemistry method, which can be determined by calculating the pooled standard deviation of a set of blind duplicates. As some wet chemistry methods are better than others, care should be taken when choosing a primary method or comparing NIR performance.

Property	Range	Wet Chemistry Method
Moisture	1.0 – 3.5%	16 Hour Vacuum Oven
Fat	1.05 – 6.6%	Acid Hydrolysis
Sugar	18 – 41%	HPLC

The error of the vacuum oven, calculated by taking the pooled standard deviation of blind duplicates, is approximately 0.06 – 0.12, depending on the method, operator, etc. The error of the acid hydrolysis method is approximately 0.15-0.30% and the error of the HPLC is approximately 0.7-0.85%.

Results and Discussion

Calibration Development

Figure 1 shows the calibration set of spectra measured using the SpectraStar 2400. Figure 2 shows the calibration set of spectra using an absorbance and first derivative transformation. All calibration were developed used an absorbance and first derivative transformation.

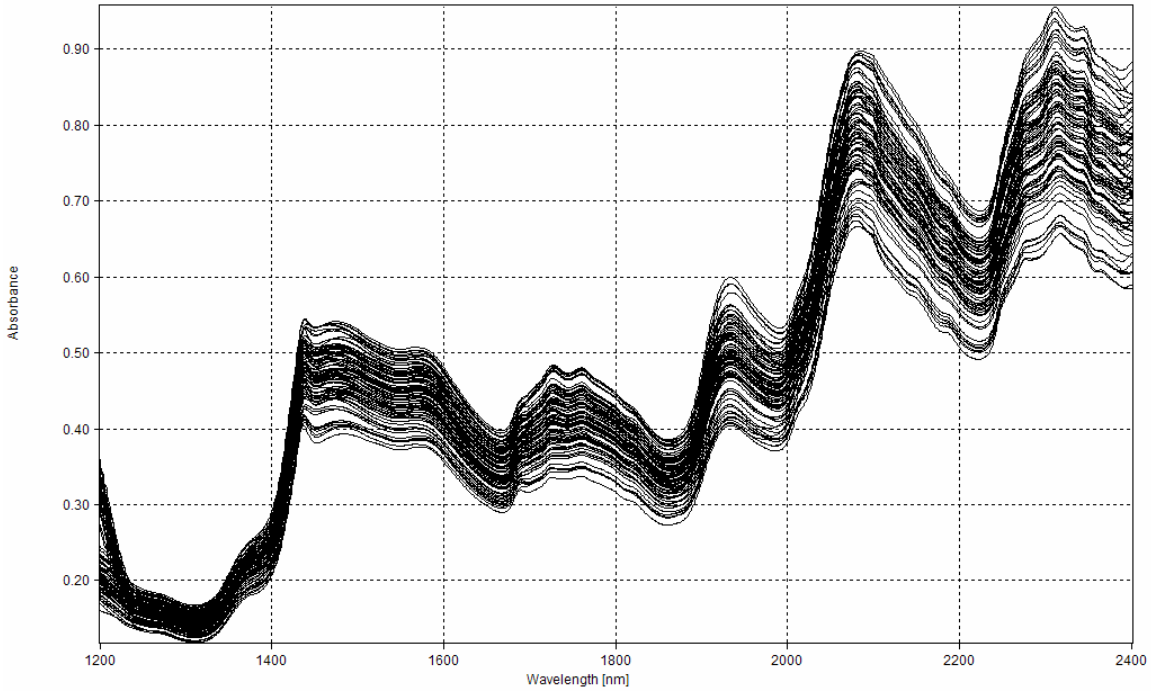
Partial Least Squares (PLS) calibrations were developed for moisture, fat, and sugar using the breakfast cereal samples. The following table shows the multiple correlation coefficient and standard error of cross validation for the calibrations. The multiple correlation coefficient is the agreement between the wet chemistry result and the NIR result. Perfect correlation is equal to 1. The standard error of cross validation is the performance that can be expected when using the calibration for routine analysis. Typical error for a vacuum oven is approximately 0.06 – 0.12%.

Property	Multiple Correlation Coefficient	Standard Error of Prediction
Moisture	0.994	0.087
Fat	0.993	0.212
Sugar	0.992	0.70

The results above are calculated using calibrations developed for a specific breakfast cereal. This calibration may be applied to a similar cereal product with a bias and/or skew adjustment or a global calibration can be developed by combining the data. The way in which NIR calibrations are developed will usually depend on the accuracy needed for the application.

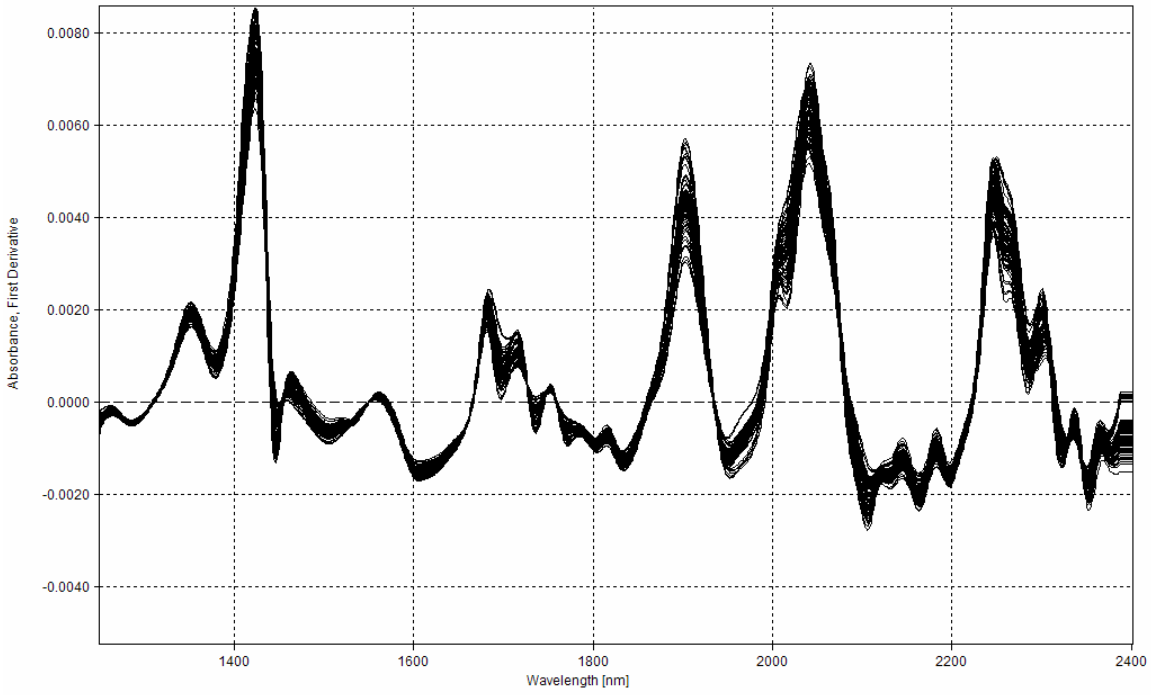
Figure 3 shows the predicted (NIR) vs. Actual (lab) plots of the moisture and sugar calibrations.

Figure 1



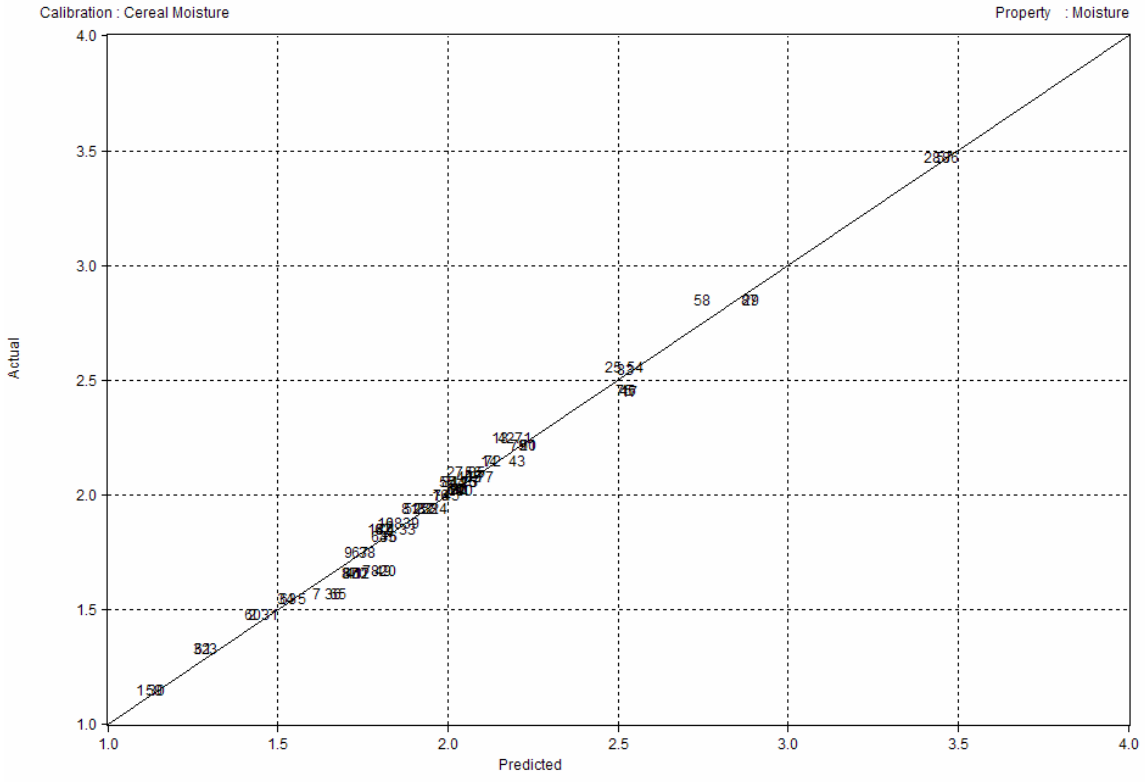
Absorbance vs. Wavelength Plot of Breakfast Cereal Calibration Spectra

Figure 2

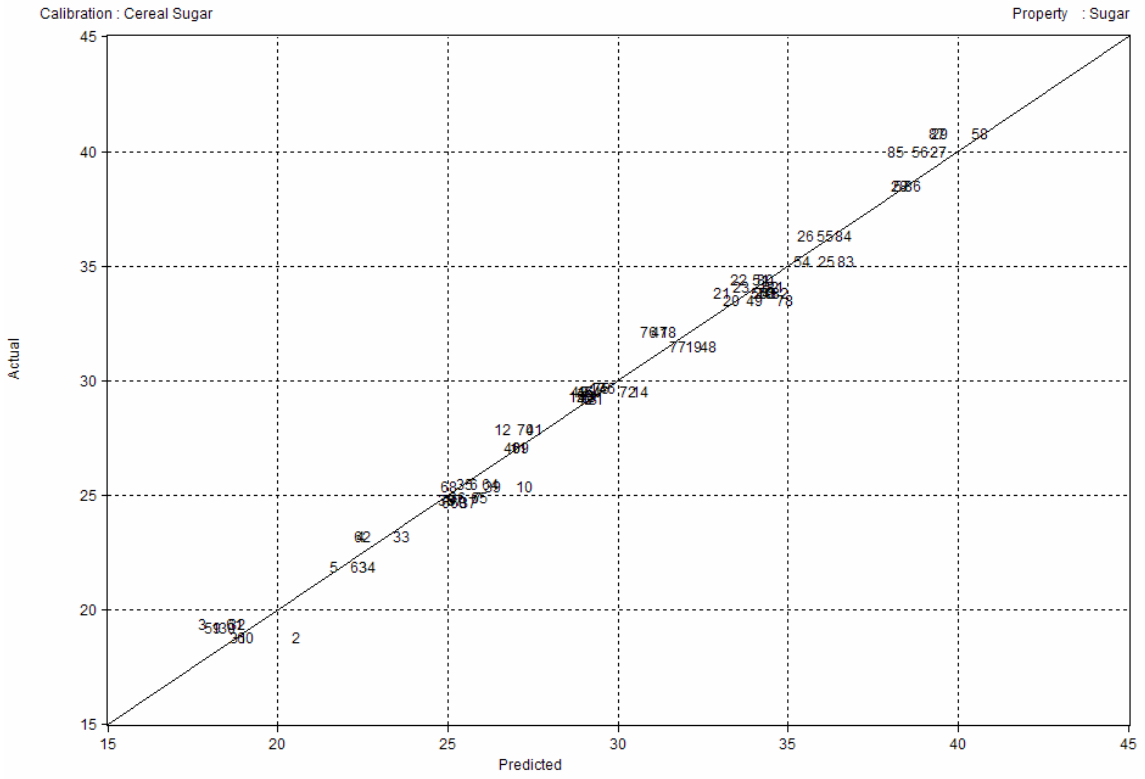


Absorbance, 1st Derivative Plot of Breakfast Cereal Calibration Spectra

Figure 3



Predicted vs. Actual Plot of Breakfast Cereal Moisture Calibration



Predicted vs. Actual Plot of Breakfast Cereal Sugar Calibration

Conclusion

NIR is an important quality tool used in the food industry. Analysis of incoming raw materials, in-process intermediates, and finished products can help to ensure product quality and provide quick financial payback. The SpectraStar 2400 will accurately analyze breakfast cereal for moisture, fat, and sugar. The SpectraStar's optimum wavelength range of 1200-2400nm covers the primary combination bands for C-H, N-H, and O-H bonds. These bonds are critical to accurately analyzing constituents such as moisture, protein, and fat. Specifically, the primary combination bands found above 1900nm are the most sensitive and generally develop the most accurate calibrations.

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