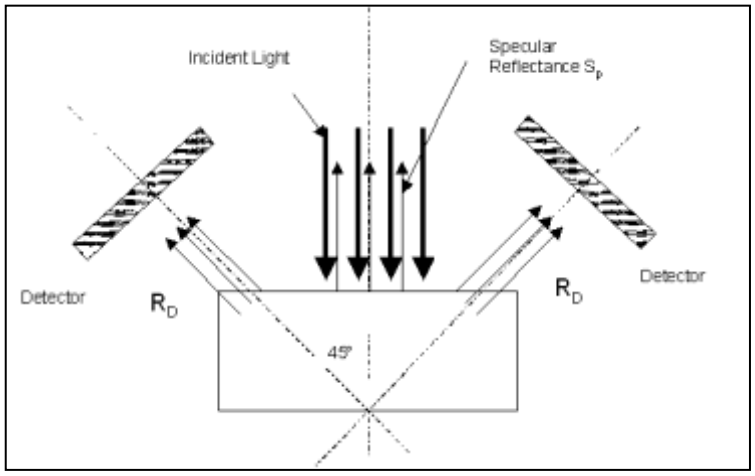
Application Note 177. Calibration Development for Ground and Whole Animal Feed Pellets Using the MultiScan Series 4000 FTNIR Spectrometer

Introduction:

Near Infrared Reflectance spectroscopy is best performed in the 1900 to 2500nm region of the electromagnetic spectrum. Within this spectral region, Protein (N-H), Moisture (O-H) and Fat (C-H) absorb NIR energy. Using 0 – 45 degree illumination and detection optics, as shown in figure 1, provides a means of collecting NIR spectra from samples such as ground meals and ground pellets used in the stock feed industry. Using a Fourier Transform (FTNIR) spectrometer to collect diffuse reflectance spectra from meals and pellets provides a very accurate and precise means of developing NIR calibrations for a wide range of chemical components in the meal and pellets, including: Crude Protein, Moisture, Fat, Fibre as well as derived calibrations for Digestible Energy, Metabolisable Energy and Ash.



This study reports the results of developing calibrations for ground and whole stock feed pellets for protein and moisture using the MultiScan Series 4000 FTNIR Spectrometer.

Figure 1. Diffuse Reflectance Optics

Procedure:

32 samples of whole feed pellets were provided for this study. A portion of each sample was ground using a laboratory grinder that uses two abrasive surfaces to fracture the pellets into a powder. The ground portions were used for protein and moisture analysis using Dumas and Oven Drying methods. The unground sample portions and the remaining ground sample portions were used for the NIR calibration.

Ground Samples:

Approximately 50 grams of sample were loaded into the flat dish used in the Series 4000 FTNIR Spectrometer. The dish is a 105 mm diameter x 5mm deep plastic ring with a 2mm quartz glass window on the bottom side. The sample was flattened using a flat piece of plastic however the powder was not compressed. The sample dish was loaded into the sample compartment of the S4000 so that light from the spectrometer illuminates the bottom of the sample dish and the diffusely reflected light is collected by the detector optics at 45 degrees to the illuminating beam. This configuration ensures the optimum amount of diffusely reflected energy is collected off the sample, yet minimises the specular reflection of the glass window.

Each sample was repacked and scanned a second time to collect another 10 spectra. As such a total of 20 scans were collected for each of the 32 samples to give approximately 640 spectra. These spectra were stored in the PC's memory and then imported into NTAS's Calibration routine where Partial Least Squares Regression (PLS) calibrations were developed.

Whole Pellets:
Approximately 80 grams of unground pellets were poured into the flat dish used in the Series 4000 FTNIR Spectrometer. This dish is 105mm diameter and 15mm deep so that the pellets can be easily loaded. The sample dish was loaded into the sample compartment of the S4000 and 10 scans were collected for each sample. The dish was reloaded and scanned a second time so that 20 spectra were collected for each sample.

Calibration:
NTAS (NIR Technology Analysis Software) is used through the built-in touch screen PC to operate the Series 4000 FTNIR Spectrometer. The Scan and Display routine was used to collect the NIR spectra for each sample the dish is rotated on a platform which holds a metallic reference disc that is illuminated in the same manner as the sample and is used to collect the 100% reference scan needed to compute the absorbance spectrum for the sample. As the dish is rotated into 10 individual locations around the outer perimeter of the sample, the sample scans are collected. The absorbance spectrum for each of these 10 sample scans is computed using the equation;

Absorbance = Log (100% Scan/ Sample Scan)

Results:

Figure 2. shows the NIR spectra of the 32 samples of ground pellets.

Figure 2. Diffuse Reflectance Spectra of Ground Pellets

Figures 3 and 4 show the calibration data for Protein and Moisture

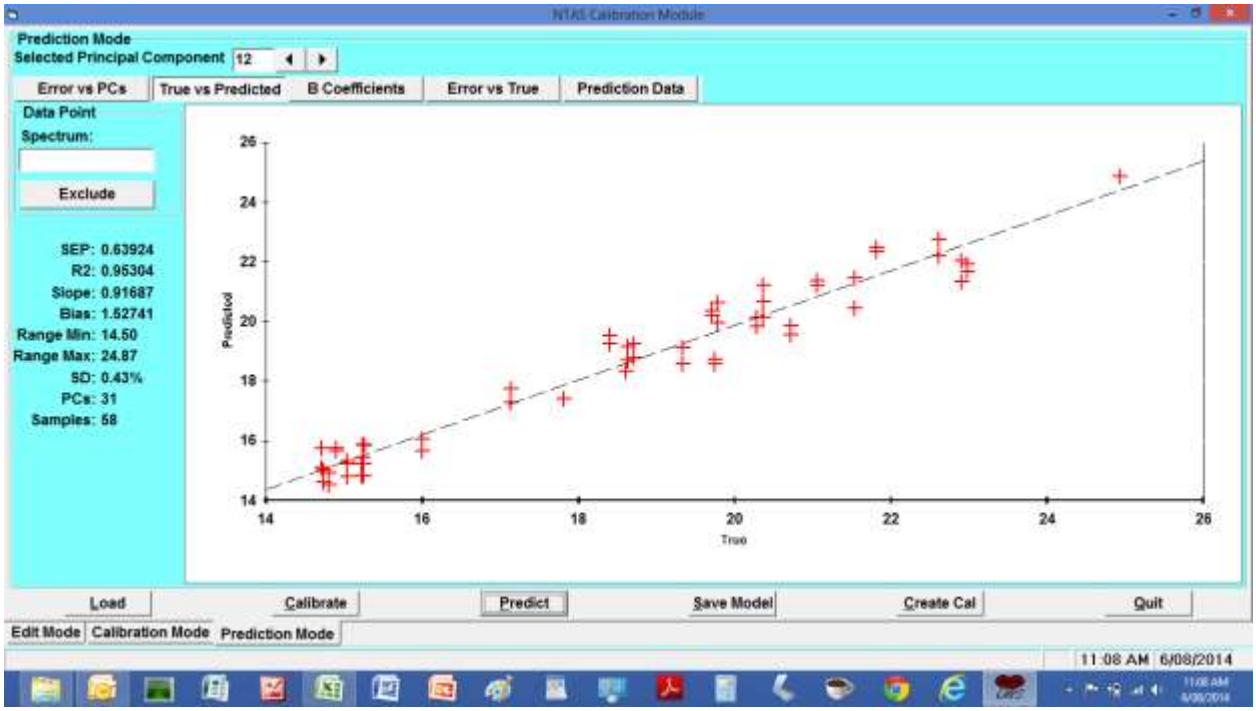


Figure 3. Protein Calibration

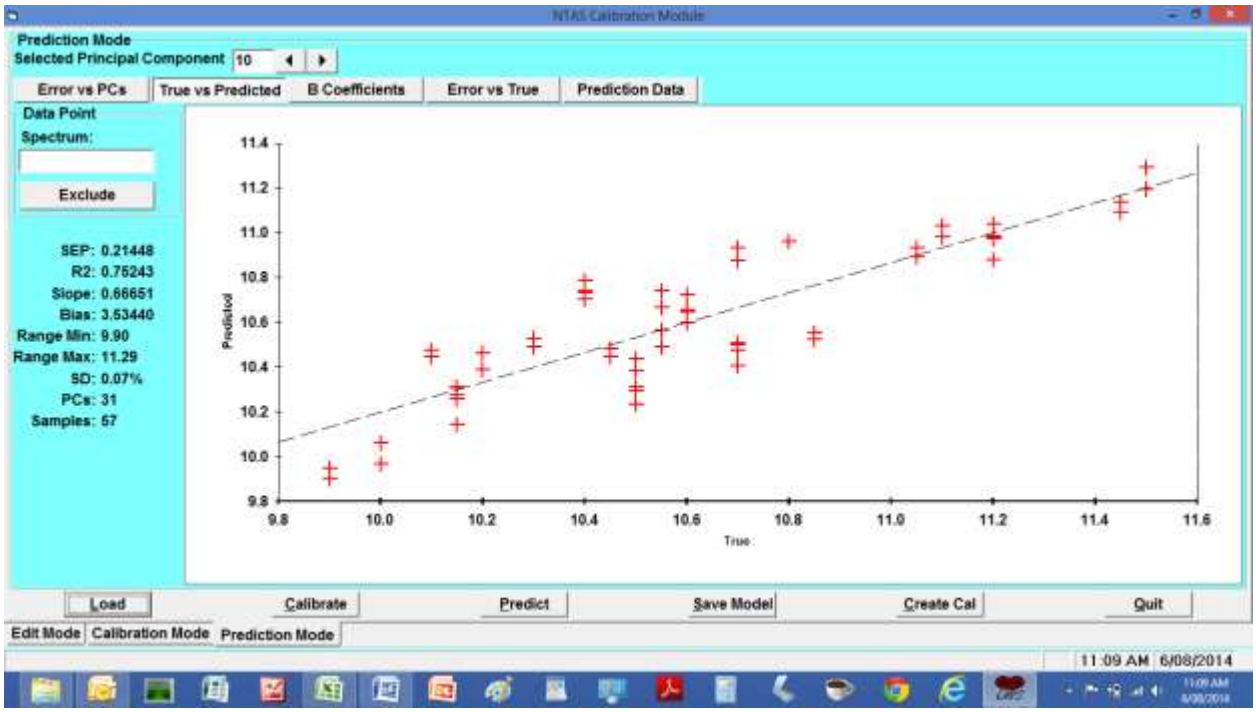


Figure 4. Moisture Calibration

Figure 5. shows the Diffuse Reflectance Spectra of Whole Pellets

Figures 6 and 7 show the calibration plots for protein and moisture in Whole Pellets.

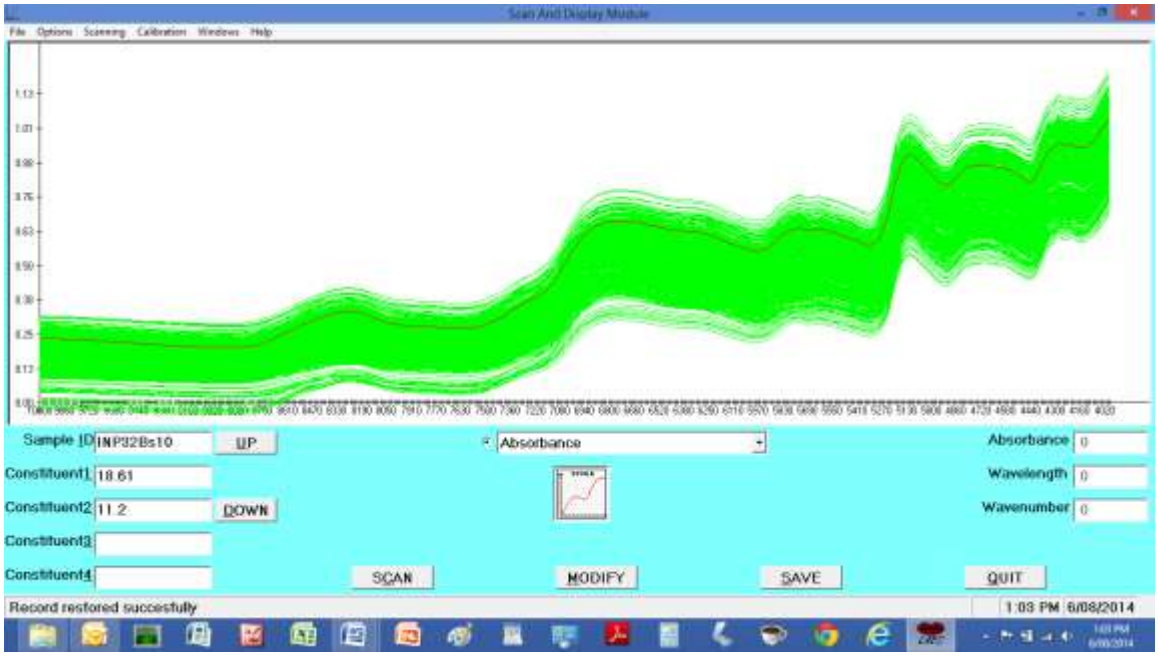


Figure 5. Whole Pellets Spectra

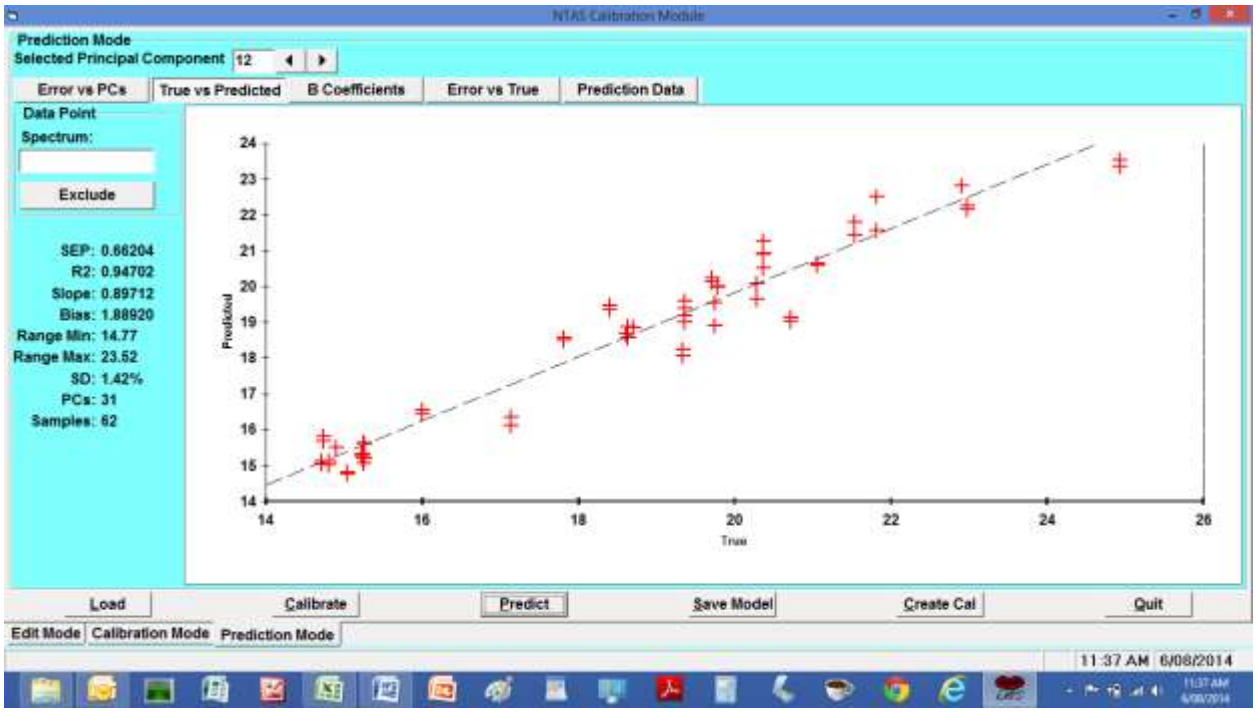


Figure 6. Whole Pellets, Protein Calibration Plot

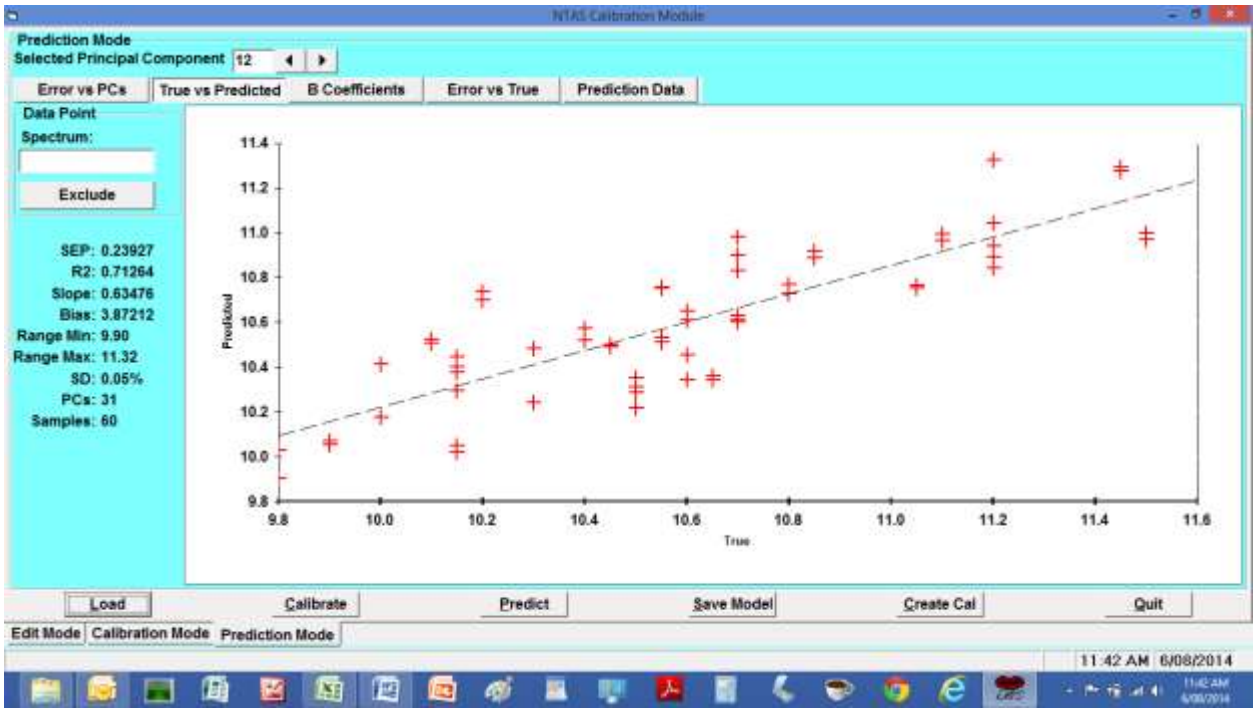


Figure 7. Whole Pellets, Moisture Calibration Plot

Discussion:

NIR Spectroscopy is well established for use in the animal feed industry as a rapid means of measuring protein, moisture, fat and other nutrients. In most applications, the samples are ground before analyses. Grinding takes time and packing the sample cup can introduce variability from operator to operator. As such measuring whole animal feed pellets reduces the time for analysis and should reduce the variability between operators.

This study is based on a set of 32 animal feed pellets which include whole wheat and barley grains. The sample matrices are very different and the inclusion of whole grains introduces another level of complexity. Nonetheless, the difference in calibration data between ground and whole pellets is negligible as shown in table 1.

Sample	Protein SEC	R^2	Moisture SEC	R^2
Ground Pellets	.63%	.96	.66	.95
Whole Pellets	.21	.76*	.23	.70*

** Note that the low correlations for moisture are due to the narrow range of moisture in the samples.*

Based on the data shown in this study, it is concluded that the Series 4000 FTNIR Spectrometer using a rotating dish and collecting diffuse reflectance spectra from beneath the sample, provides a rapid and accurate method of measuring small animal feed pellets. It cannot be concluded that larger pellets will demonstrate the same accuracy since the larger pellets would present a less consistent sample than the small pellets.

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