Application Note 173. NIR Calibration Development for Bone Meal using the 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 2120nm), Moisture (O-H, 1940nm) and Fat (C-H, 2350nm) 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.

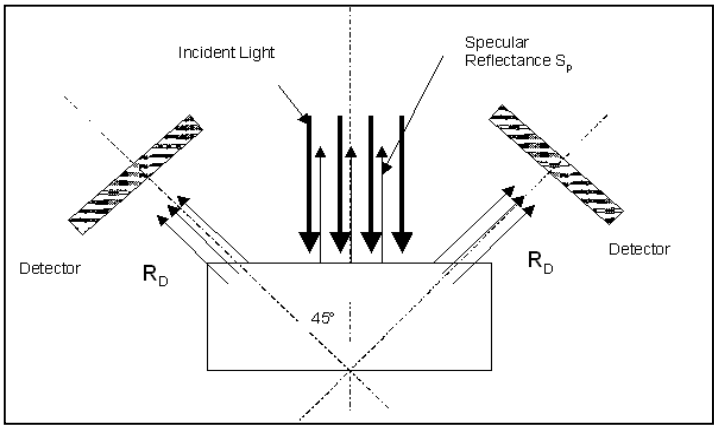

Figure 1. Diffuse Reflectance

This study reports the results of developing calibrations for Bone Meal for protein and moisture using the MultiScan Series 4000 FTNIR Spectrometer.

Procedure:

500 spectra were collected from 30 samples of Bone Meal across the wavelength range 1000 to 2500nm in Diffuse Reflectance using the Series 4000 FTNIR Spectrometer. Figure 2 shows the NIR spectra of these samples. Note that there are two groups of spectra. This is due to the presence of low and high ash content meals. High ash samples are less optically dense than Low ash samples. As such the spectra are displaced between the two groups.

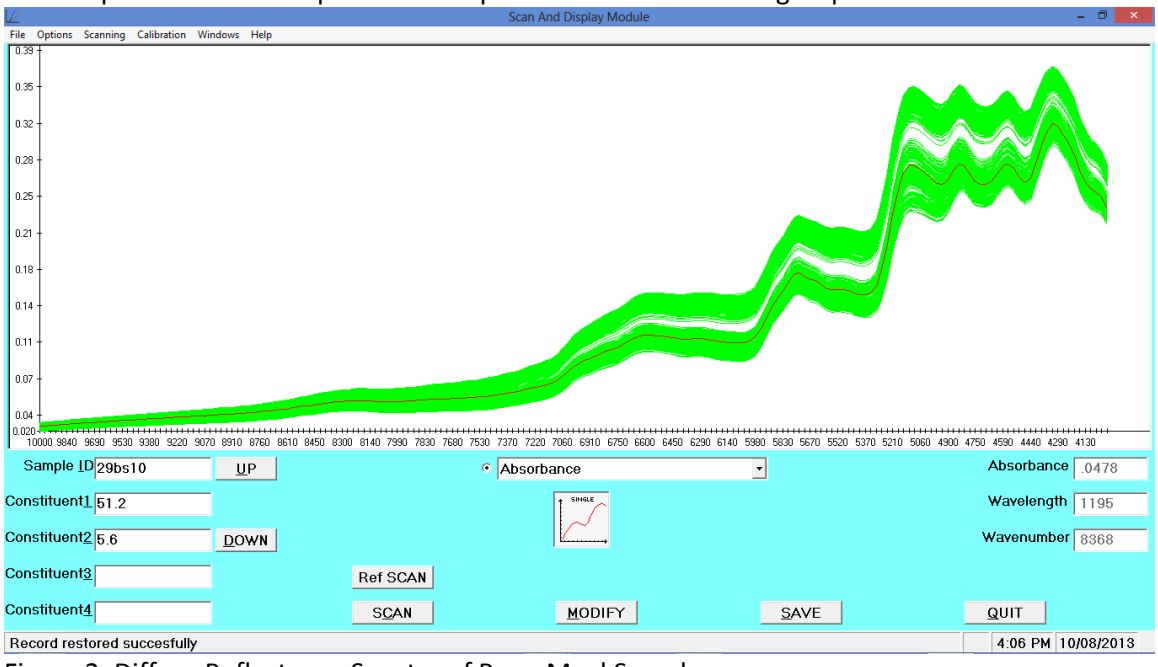

Figure 2. Diffuse Reflectance Spectra of Bone Meal Samples

Approximately 40 grams of bone meal was poured into a 5mm deep dish. The meal was scrapped across the dish with a flat blade in order to evenly fill the dish. The excess meal was scrapped off the dish and the blade used to flatten the top surface. The meal was not compressed. The dish was placed into the Series 4000's rotating sample dish holder and the scan initiated. The dish holder has a Teflon Powder Reference disk fitted to one end of the dish. The 100% reference scan is stored in memory and then the dish is rotated to the first scan position. This process is repeated for 10 portions of the sample. 10 scans are collected and averaged from each of the 10 sample portions. The average scan of each portion was stored in memory. The sample dish was removed, emptied and refilled with another sample taken from the same meal container.

Each of the 30 samples had been analysed in duplicate for protein using a VELP Scientifica, NDA701 Dumas Analyser and for moisture using the Oven Drying method. The protein and moisture values for each meal sample were recorded with the spectral data.

The 500 spectra of the bone meal samples were imported into NTAS (NIR Technology Analysis Software) where Partial Least Squares Regression was applied to develop calibrations for protein and moisture.

Results:

Figure 3. shows the results of the protein calibration for the 500 blood meal spectra.

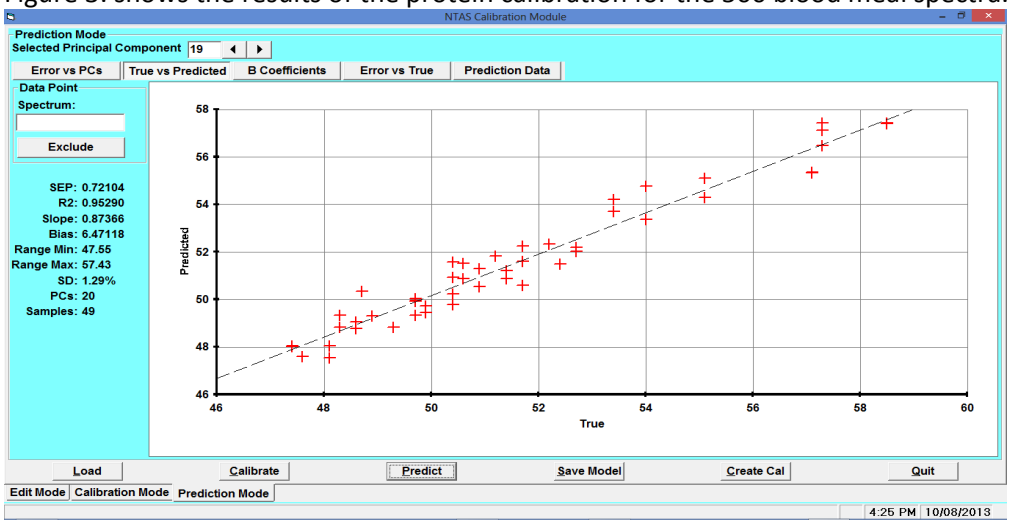

Figure 3. Protein Calibration Plot.

Figure 4. shows the results of the moisture calibration for the 500 bone meal spectra.

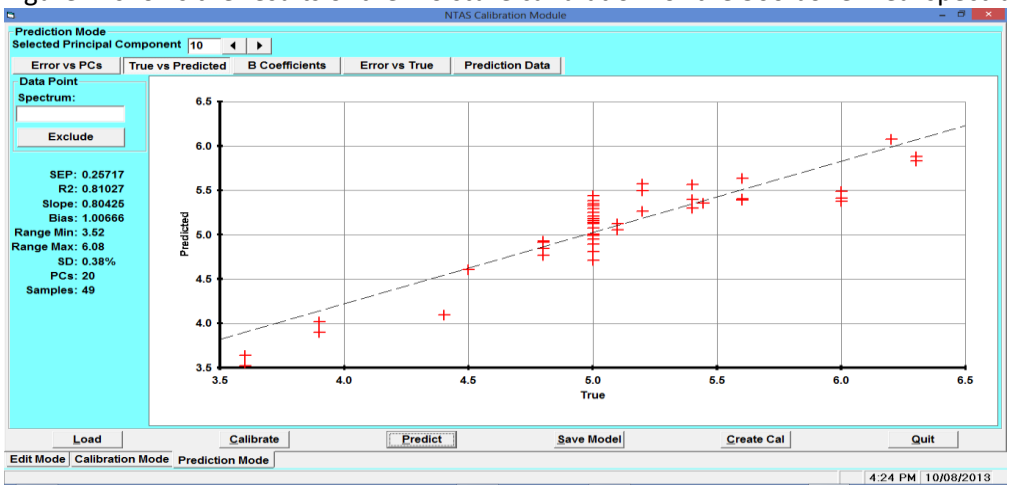

Figure 4. Moisture Calibration Plot

The spectra for the Low Ash and High Ash meal samples were separated in order to assess whether the calibrations could be improved. Figures 5 and 6 show the calibration plots for protein and moisture in Low Ash meals and figures 7 and 8 show the plots for the High Ash meals.

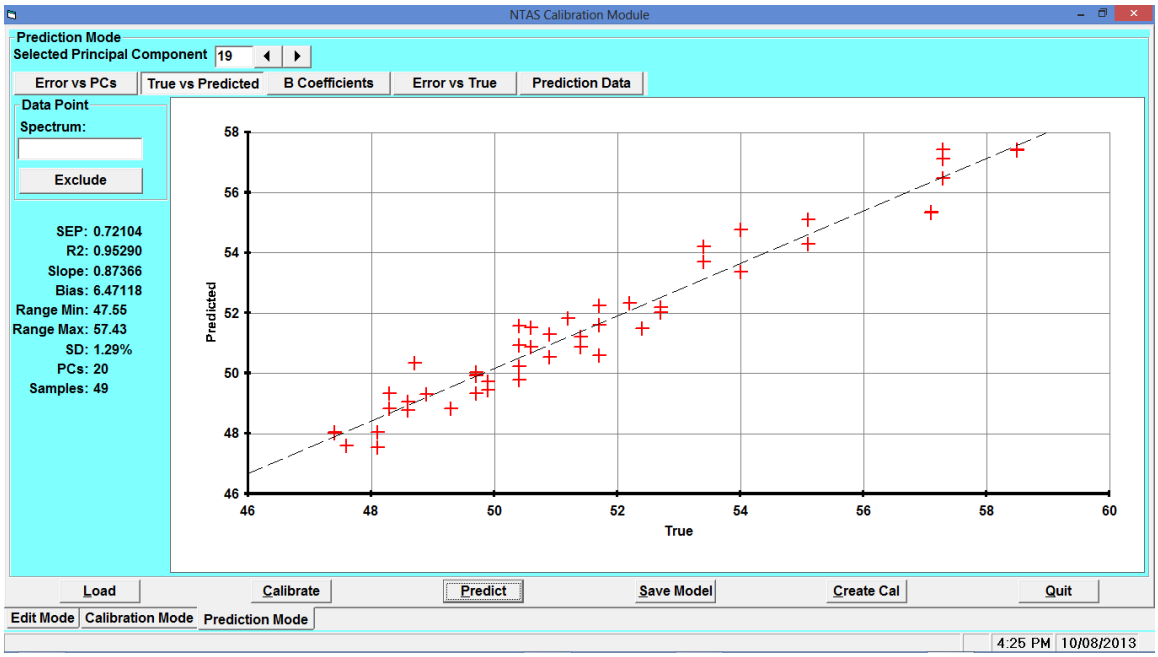

Figure 5. Protein Calibration Plot for Low Ash Meals

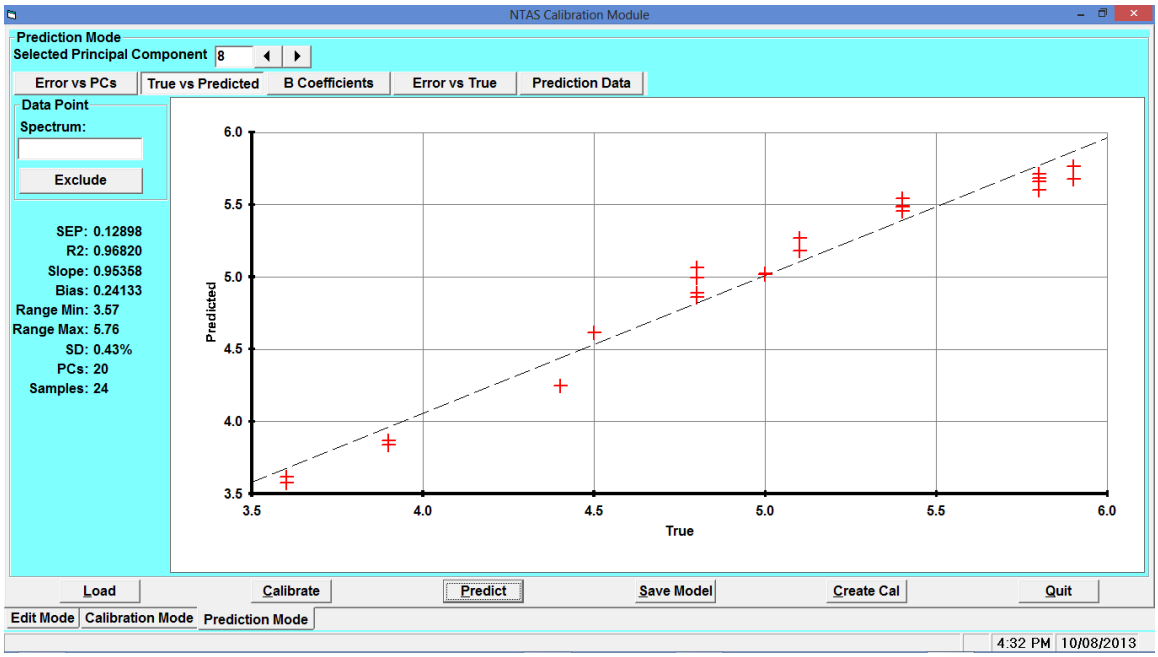

Figure 6. Moisture Calibration Plot for Low Ash Meals

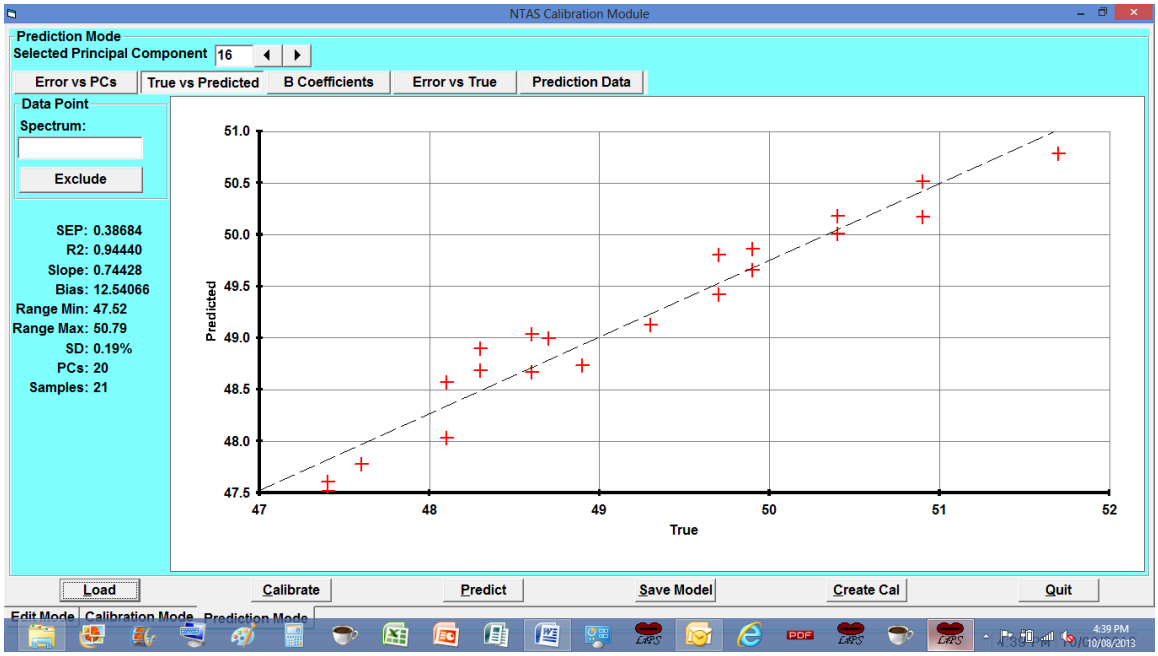

Figure 7. Protein Calibration Plot for High Ash Meals

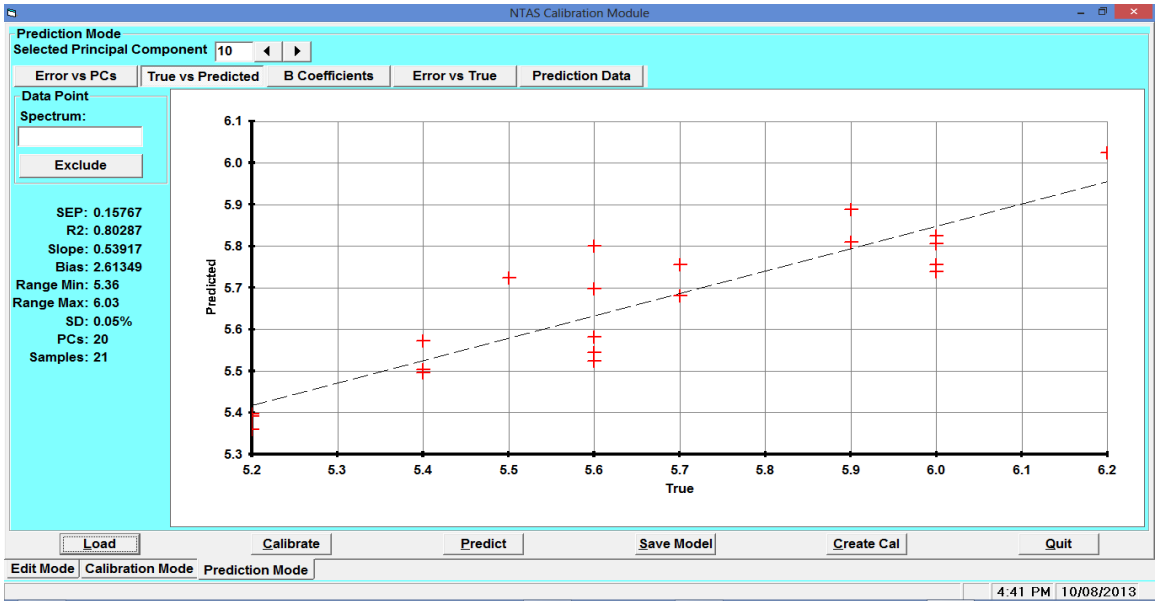

Figure 8. Moisture Calibration Plot for High Ash Meals.

Discussion:

Near Infrared Reflectance spectroscopy has been used for measuring protein and moisture in meals and animal feed pellets for decades. As such the data presented in this report is not revolutionary. Fat and Ash can also be measured using NIR spectroscopy. Unfortunately the samples of bone meal provided for this study did not have reference values for fat and ash.

The objective of this report is to demonstrate that the Series 4000 FTNIR Spectrometer provides a means of accurately measuring meal samples such as Bone Meal. The data presented in this report shows that the Series 4000 FTNIR Spectrometer can be calibrated to measure protein and moisture in Bone Meal. The study also shows that the calibrations can be significantly improved by separating Low Ash from High Ash samples.