Application Note 175. Calibration Development for Ground and Whole Stock Feed Pellets Using the MultiScan Series 4000 FTNIR Spectrometer

NIR Technology Systems

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 ground and whole stock feed 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 and Ash.
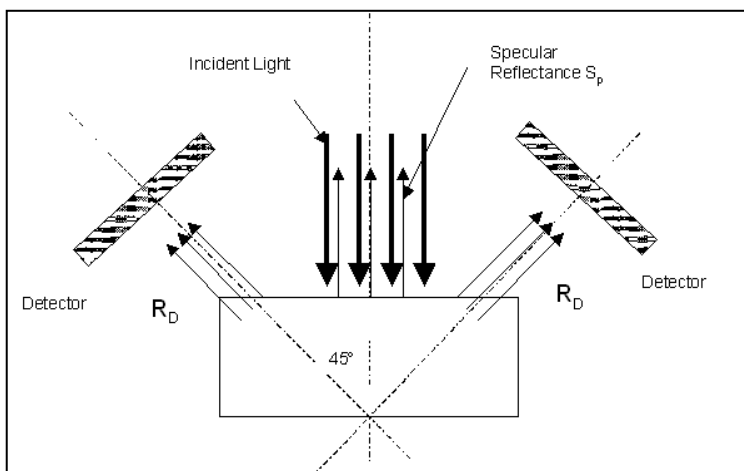

This study compares the results of developing calibrations for whole and ground stock feed pellets using the MultiScan Series 4000 FTNIR Spectrometer.

Figure 1. Diffuse Reflectance Optics

Procedure:

19 samples of stock feed pellets were supplied along with nutrient data measured by a third party testing laboratory, the nutrient data was determined by another NIR analyser with calibrations developed over many years and thousands of samples. The 19 samples were scanned as whole pellets and as ground powders. Figure 2 shows images of both sample types.

Ground Pellets Scans: Approximately 50 grams of ground pellets 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 powder was flatted using a Perspex disk however the sample 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.

Whole Pellets Scans: Approximately 30 grams of Whole Pellets were loaded into a flat dish similar to that used for powders but 15mm deep. The sample was shaken on the benchtop in order to distribute the pellets evenly. The dish was loaded into the Series 4000 FTNIR and diffuse reflectance spectra collected from the bottom side of the dish.

Calibration: NTAS (NIR Technology Analysis Software) running on the built-in touch screen PC, was used to operate the Series 4000 FTNIR Spectrometer. The Scan and Display routine was used to collect the NIR spectra for each sample. The sample dish was rotated on a platform so that multiple, ie, 10 spectra, can be collected for each sample. A powdered Teflon reference disc is mounted on the rotating platform 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)

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 19 samples. 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 for both the Ground Pellets and the Whole Pellets..

Results:
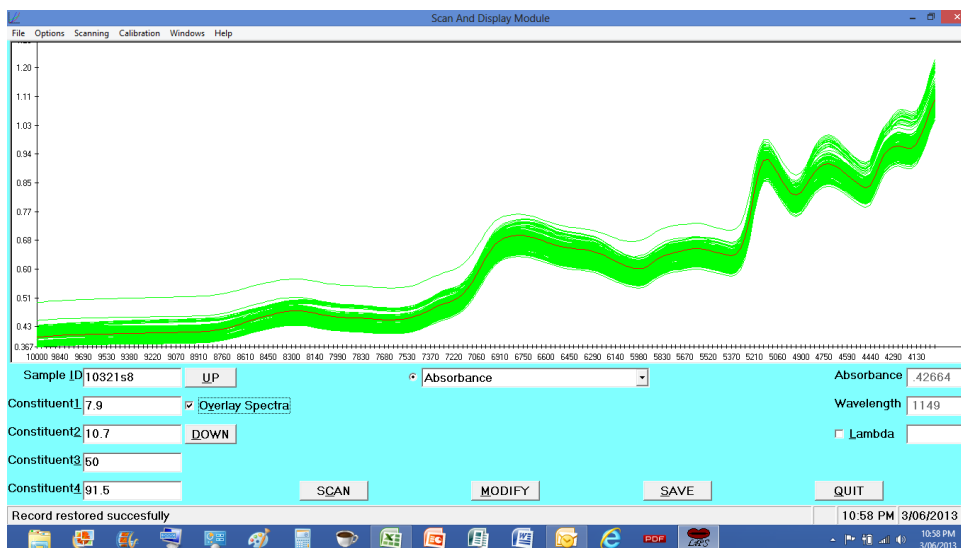Figure 3 and 4. shows the NIR spectra of the 19 samples of ground and whole pellets.

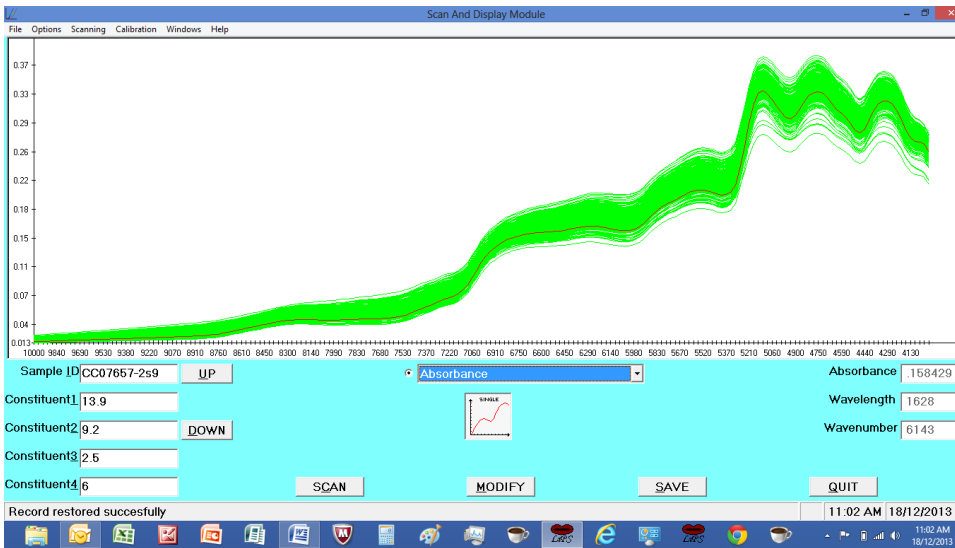Figure 3. Diffuse Reflectance Spectra of Ground Pellets

Figure 4. NIR Spectra of Whole Pellets

Figures 5 through 10 show the calibration data for Crude Protein. Moisture and Ash.
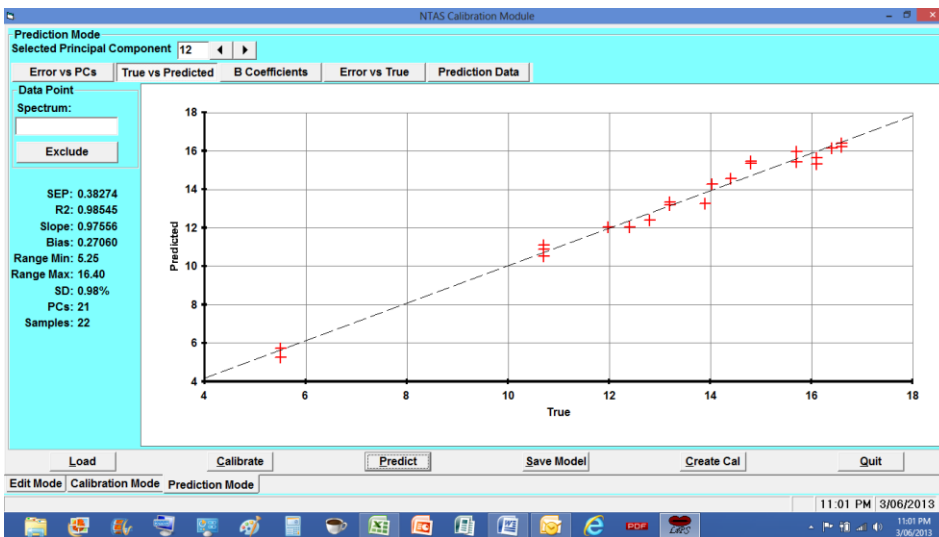

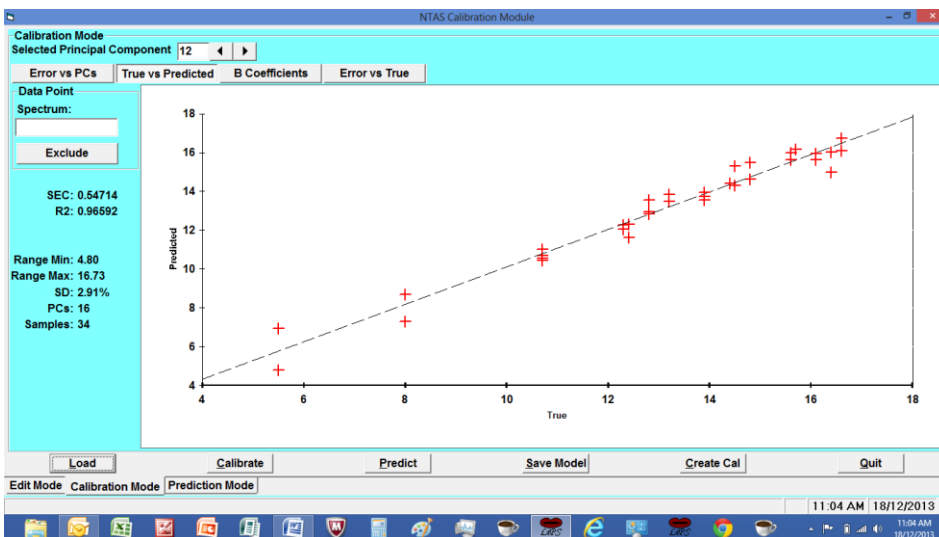Figure 5. Protein Calibration for Ground Pellets

Figure 6. Protein Calibration for Whole Pellets
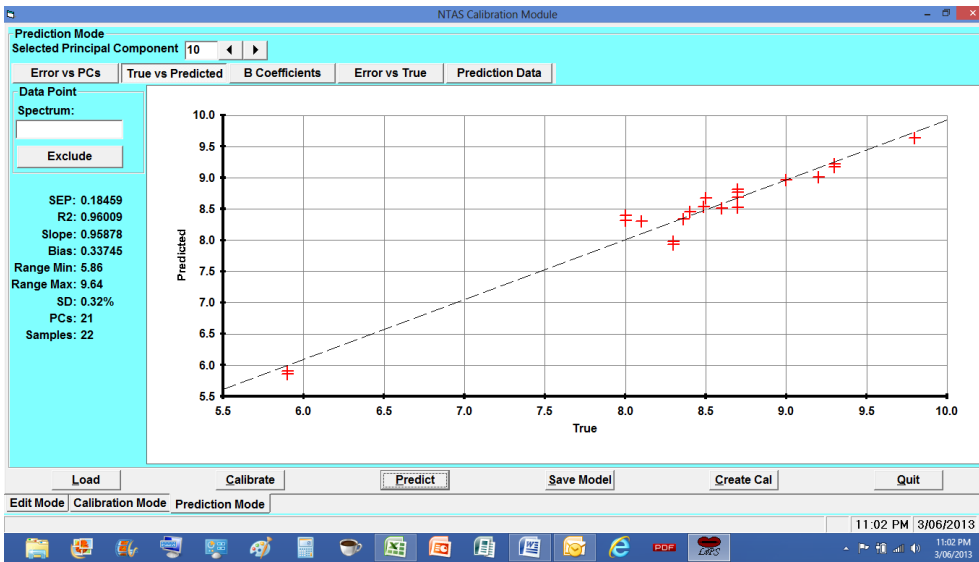
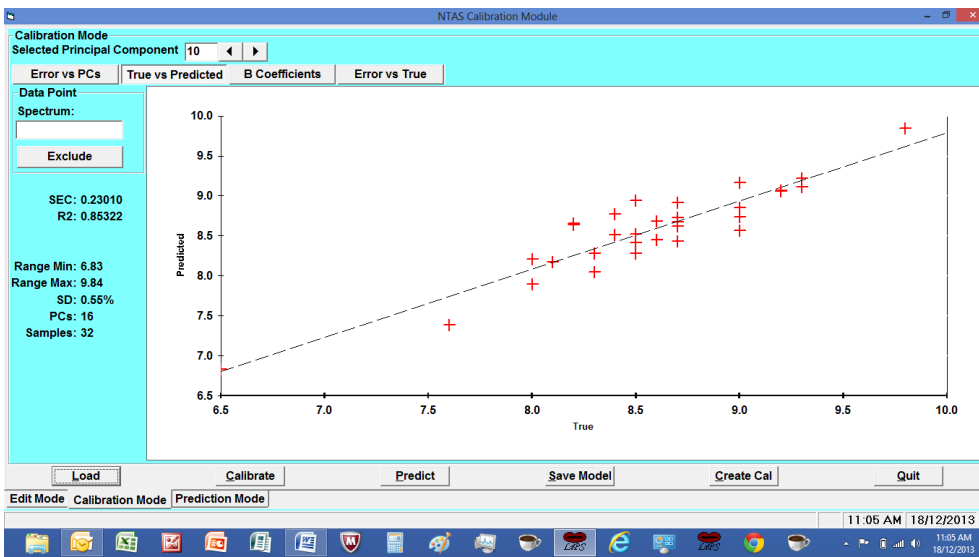Figure 7. Moisture Calibration for Ground Pellets

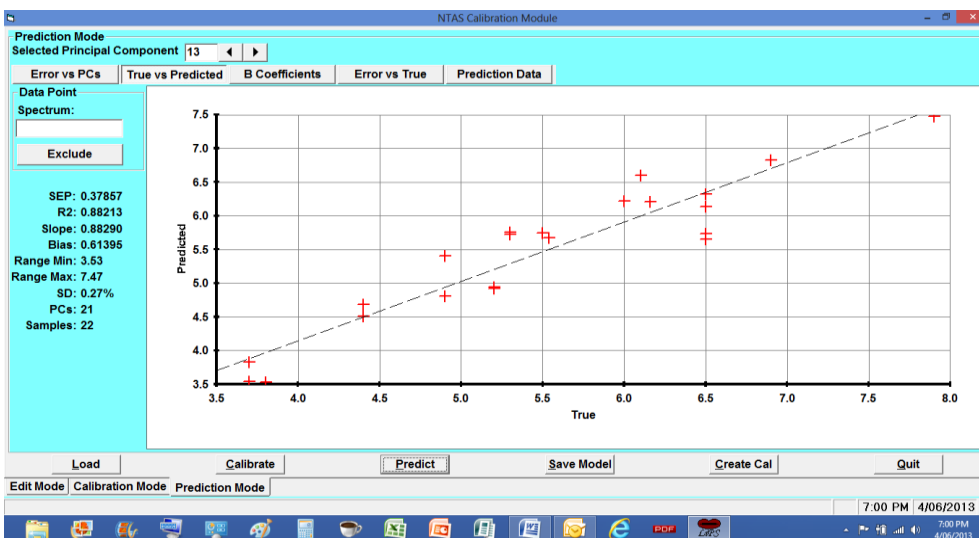
Figure 8. Moisture Calibration for Whole Pellets
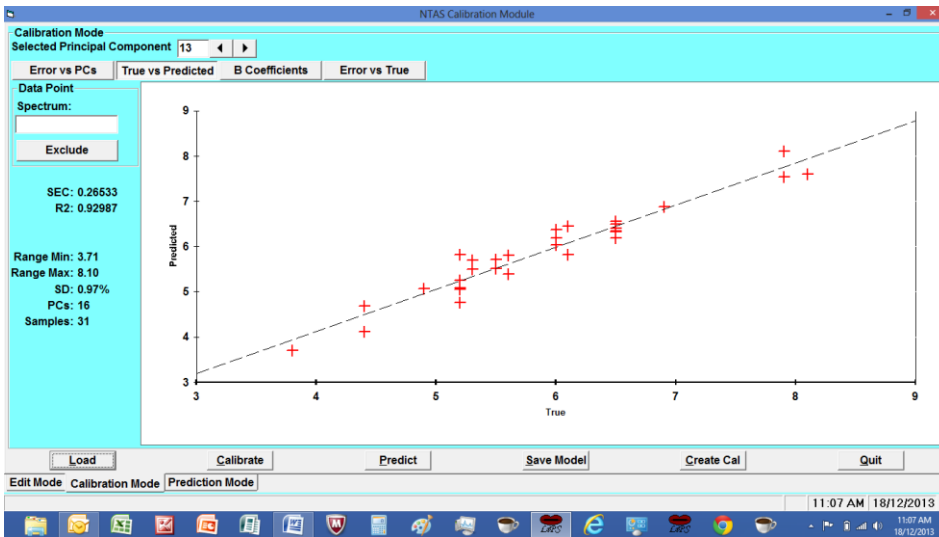

Figure 9. Ash Calibration for Ground Pellets

Figure 10. Ash Calibration for Whole Pellets

Table 1. Summary of the calibration statistics for both Ground and Whole Pellets.

Component	Ground Pellets		Whole Pellets	
	SEC	R^2	SEC	R^2
Protein	0.38	.985	0.45	.956
Moisture	0.18	.960	0.23	.853
Ash	0.38	.880	0.26	.929

Discussion:

It is to be expected that NIR spectroscopy will be suitable for the development of calibrations for Protein, Moisture and Ash in most meals and stock feeds. However the general expectation is that only powdered samples can be accurately measured using NIR spectroscopy.

Table 1 shows that there is an increase in SEC(Standard Error of Calibration) and a reduction in R^2 (Correlation Coefficient Squared) between Ground and Whole Pellets calibrations. However the differences a small and in the case of Ash the statistics are reversed.

Although the number of samples used in these calibrations is small, the fact that 20 scans for each sample were collected, then the data is considered typical of what would be expected with 50-100 samples. The larger number of samples would be beneficial in confirming the calibrations and making them more robust. However based on the data presented above, it is concluded that successful calibrations should be possible using Whole Pellets. The benefit of measuring Whole Pellets includes sample turn around, reduced human error due to sample compaction and reduced dust in the laboratory.