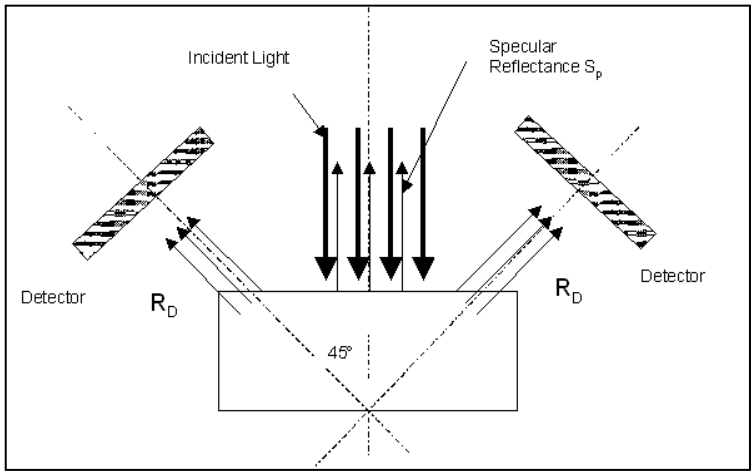
Application Note 170. Calibration Development for Stock Feed Pellets Using the MultiScan Series 4000 FTNIR Spectrometer

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 stock feed pellets for their nutrient profile using the MultiScan Series 4000 FTNIR Spectrometer.

Figure 1. Diffuse Reflectance Optics

Procedure:

19 samples of stock feed pellets were provided with nutrient data provided 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 ground using a laboratory grinder that uses two abrasive surfaces to fracture the pellets into a fine powder.

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.

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)

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.

Results:

Figure 2. shows the NIR spectra of the 19 samples of ground pellets.

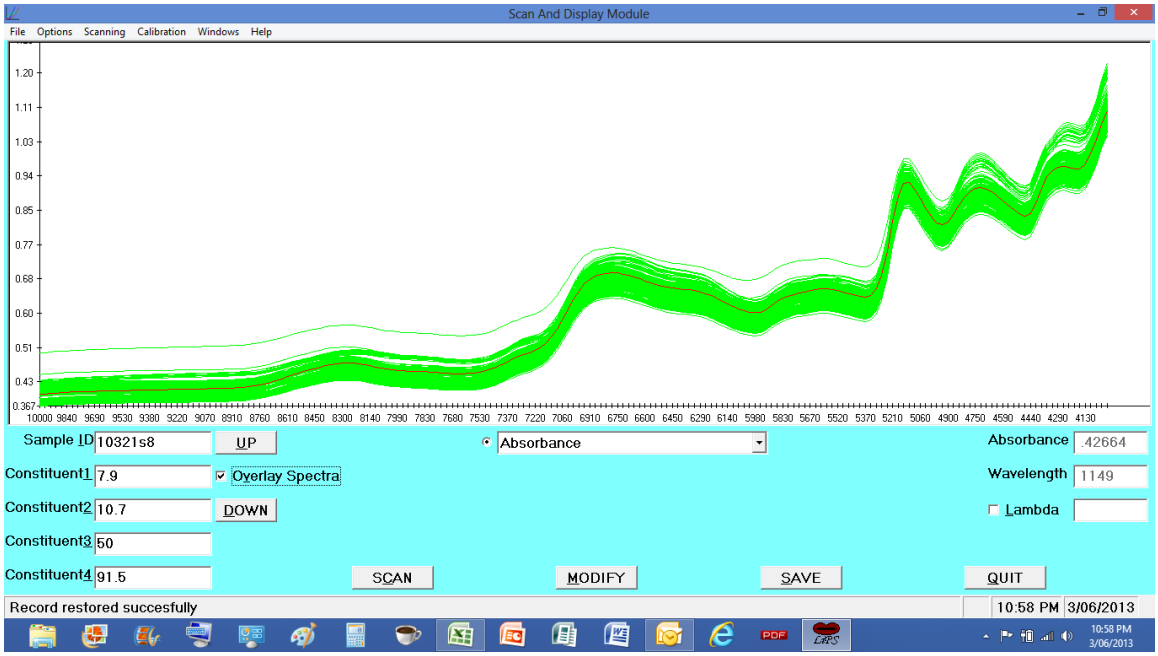


Figure 2. Diffuse Reflectance Spectra of Ground Pellets

Figures 3 through 7 show the calibration data for Crude Protein. Moisture, Fat, Digestible Energy, Metabolizable Energy and Ash.

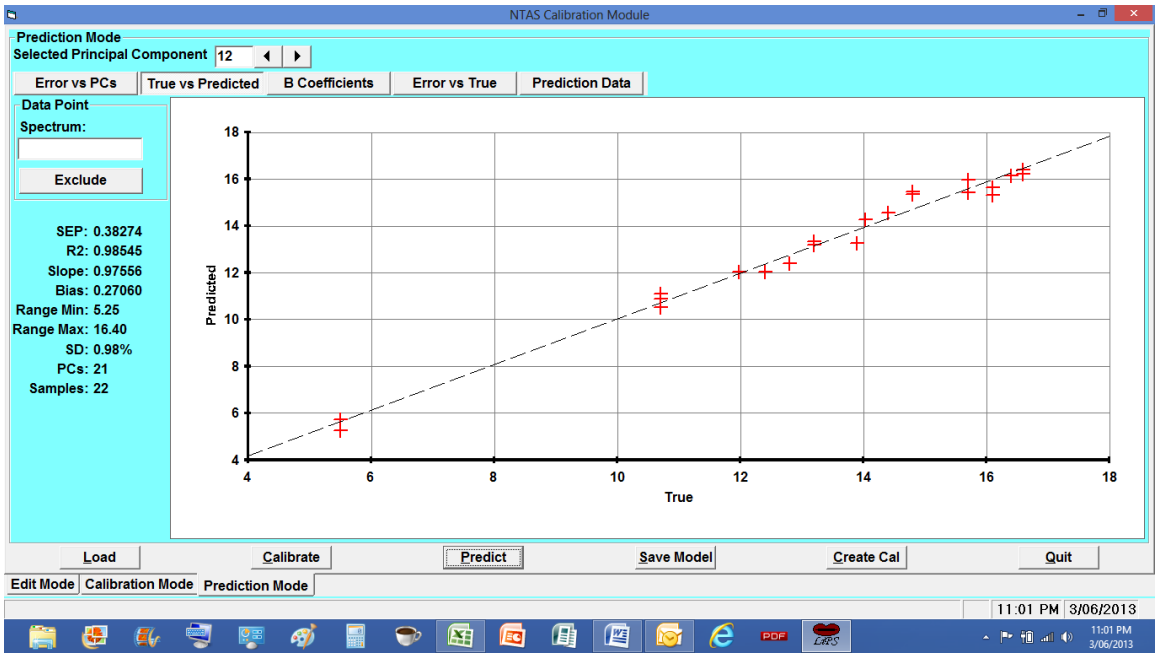


Figure 3. Protein Calibration

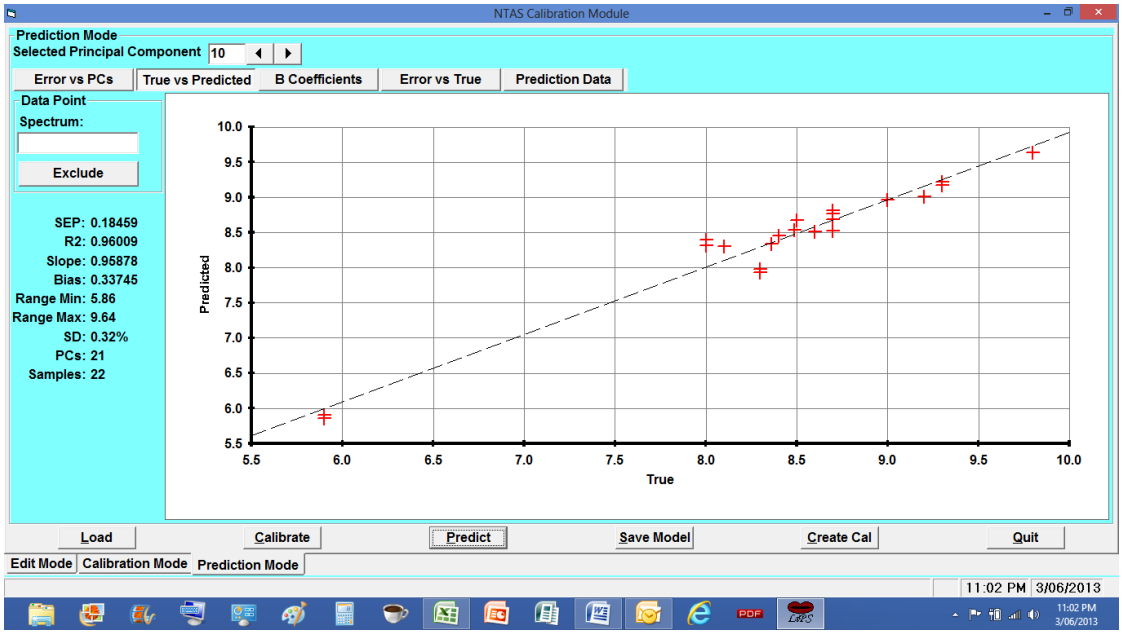


Figure 4. Moisture Calibration

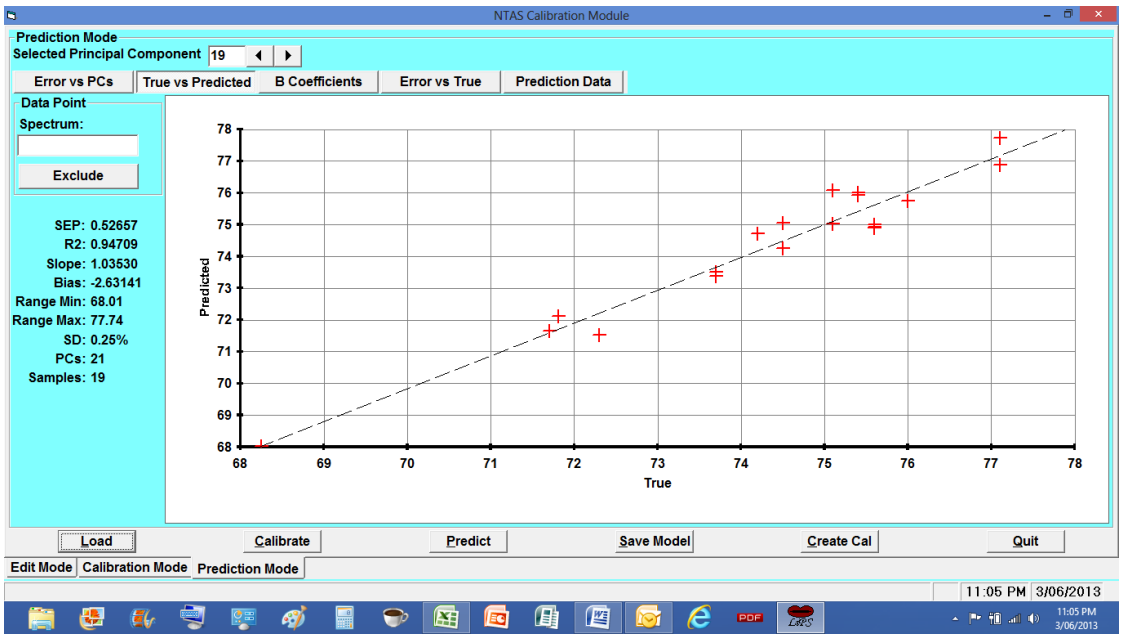


Figure 5. Digestible Energy Calibration

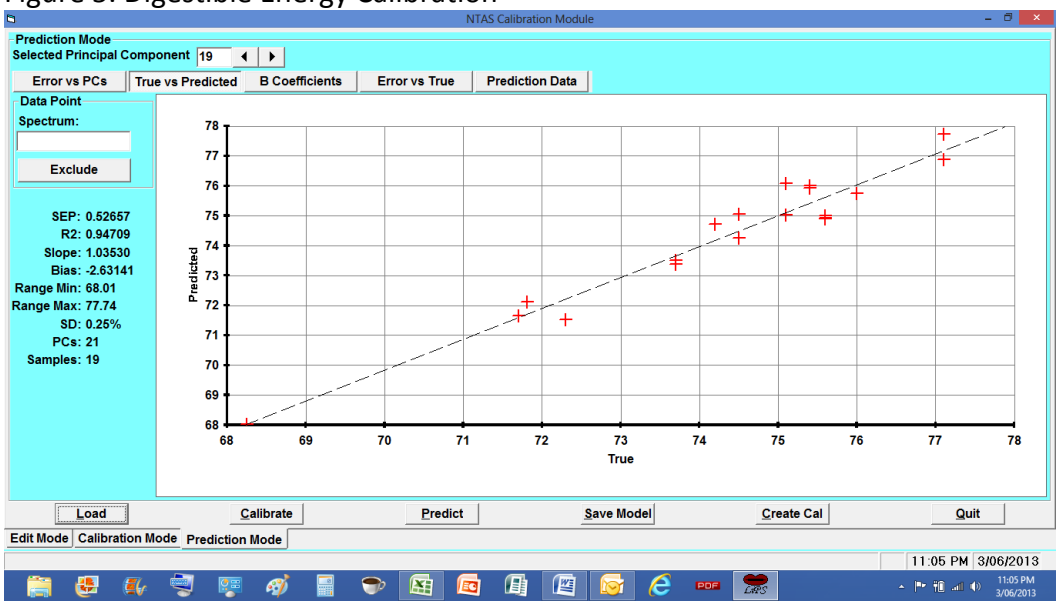


Figure 6. Metabolizable Energy

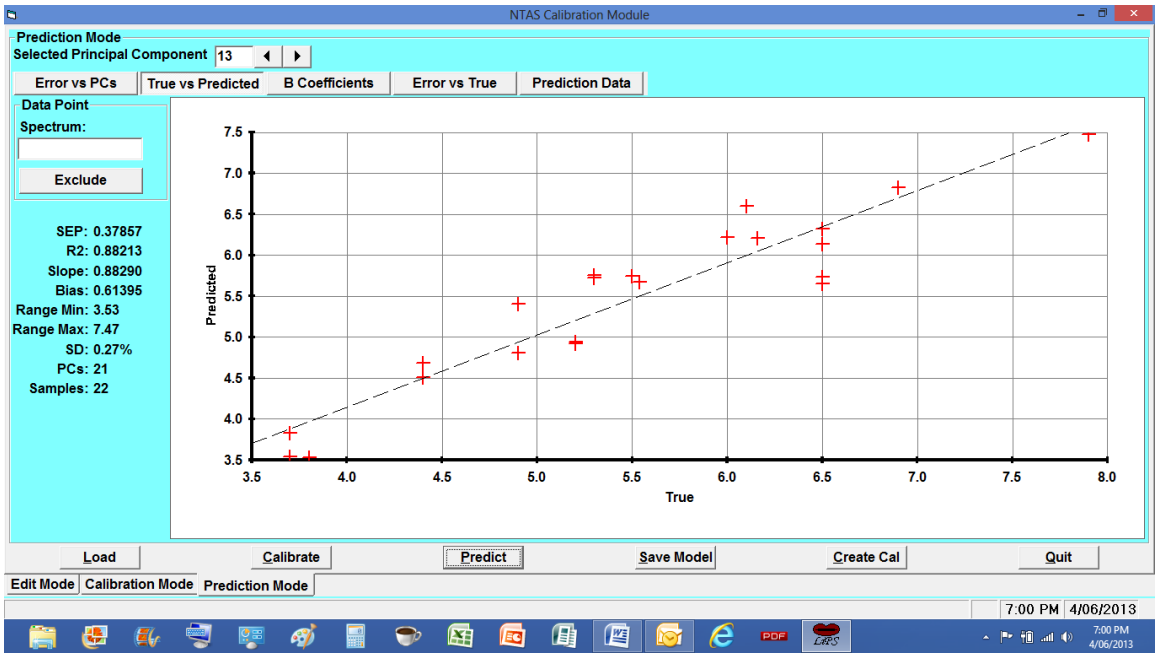


Figure 7. Ash Calibration

Discussion:

It is to be expected that NIR spectroscopy will be suitable for the development of calibrations for Protein, Moisture and Fat in most meals and stock feeds. However, nutrient levels such as Digestible Energy and Metabolisable Energy are not by their own right components that have NIR absorbance peaks and as such, it is less likely that NIR calibrations can be developed. Nonetheless the data from this study shows that both DE and ME calibrations have excellent correlation and low errors. It is generally considered that DE and ME are related to the concentration of the carbohydrates, fat and protein.

The Ash parameter shows the lowest correlation, however Ash is not a component that can be explained in terms of NIR peaks, but more as the difference between the total mass and the % Protein, Moisture, Carbohydrates and Fat. Since these four components do have NIR peaks, then the Ash calibration can be explained in terms of these other components.

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.