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 Hemp Powder. Using a Fourier Transform (FTNIR) spectrometer to collect diffuse reflectance spectra from powdered samples provides a very accurate and precise means of developing NIR calibrations for a number of parameter, including: Protein, Moisture, Fat and Ash.
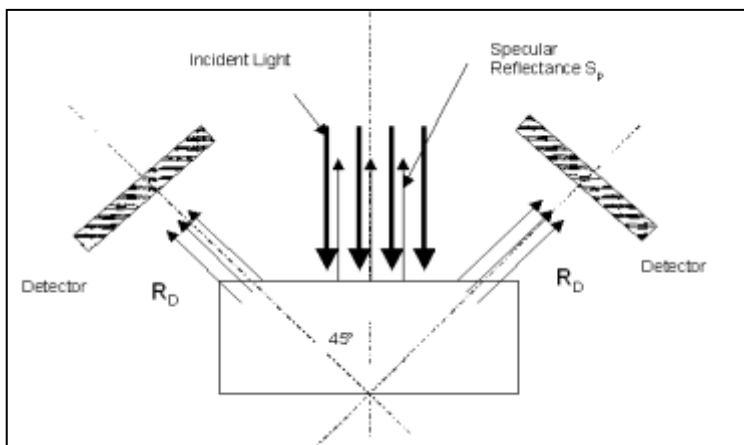

This study reports the results of developing a calibration for protein in Hemp Powder using the MultiScan Series 4000 FTNIR Spectrometer.

Figure 1. Diffuse Reflectance

Procedure:

20 samples of hemp powder were scanned using the MultiScan Series 4000 FTNIR Spectrometer in Diffuse Reflectance mode from 1000 to 2500nm. Each sample was loaded into a 5mm sample dish and levelled out using a scrapper. The sample dish was placed into the Rotating Sample Holder and the scan initiated. A Teflon Powder Reference disk is fitted into the Rotating Sample Holder so that a 100% reference scan is collected before each set of sample scans. The dish is then rotated into 10 separate locations and 10 spectra are collected and averaged for each portion of the sample dish. The lab protein values for each parameter are entered and the 10 spectra are stored in memory.

The spectra and lab data were imported into NTAS (NIR Technology Analysis Software) where a Partial Least Squares regression was used to develop a calibration for Protein.

7 further samples of Hemp Powder were provided as a Validation Set. One sample, no. 23, was very different from the others and had a protein level of 37.83 where as the other samples had protein values ranging from 59 – 67%.

Results:

Figure 2, shows the NIR spectra of the 27 samples of hemp powder.

Figure 2. FTNIR Spectra of Hemp Powder

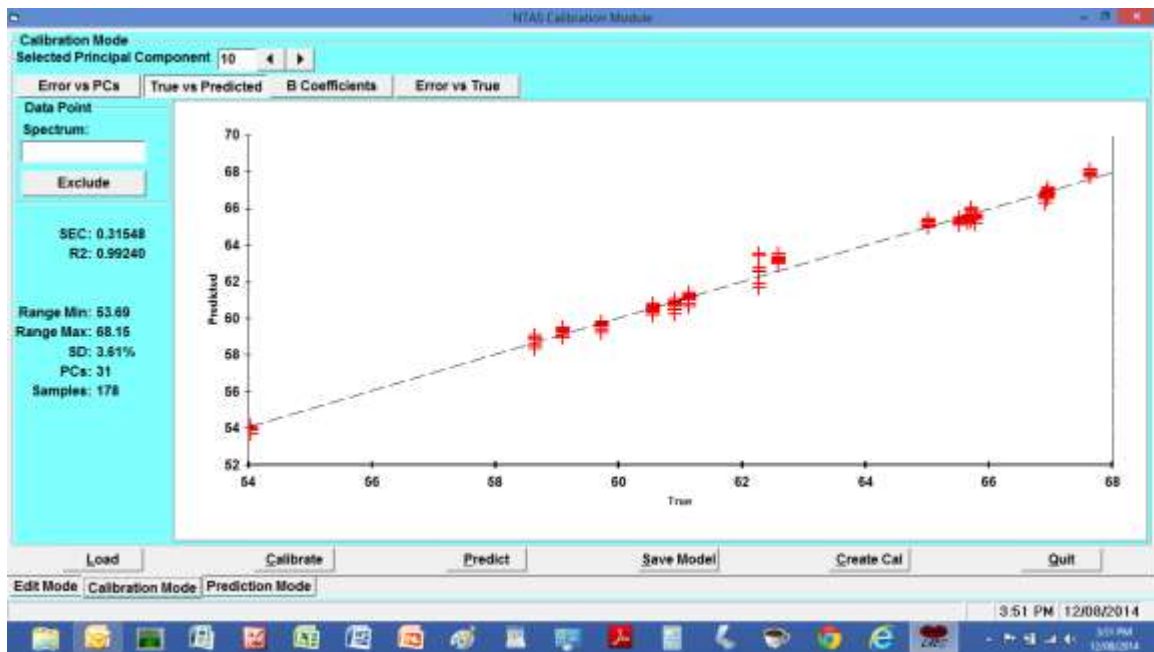Figures 3 show the calibration plot for protein in the Hemp Powder.

Figure 3. Protein Calibration Plot

Figure 4. shows the prediction plot for the protein in Hemp Powder.

Figure 5. Shows the same data excluding sample 23, which was noted to be a different material for the other samples.
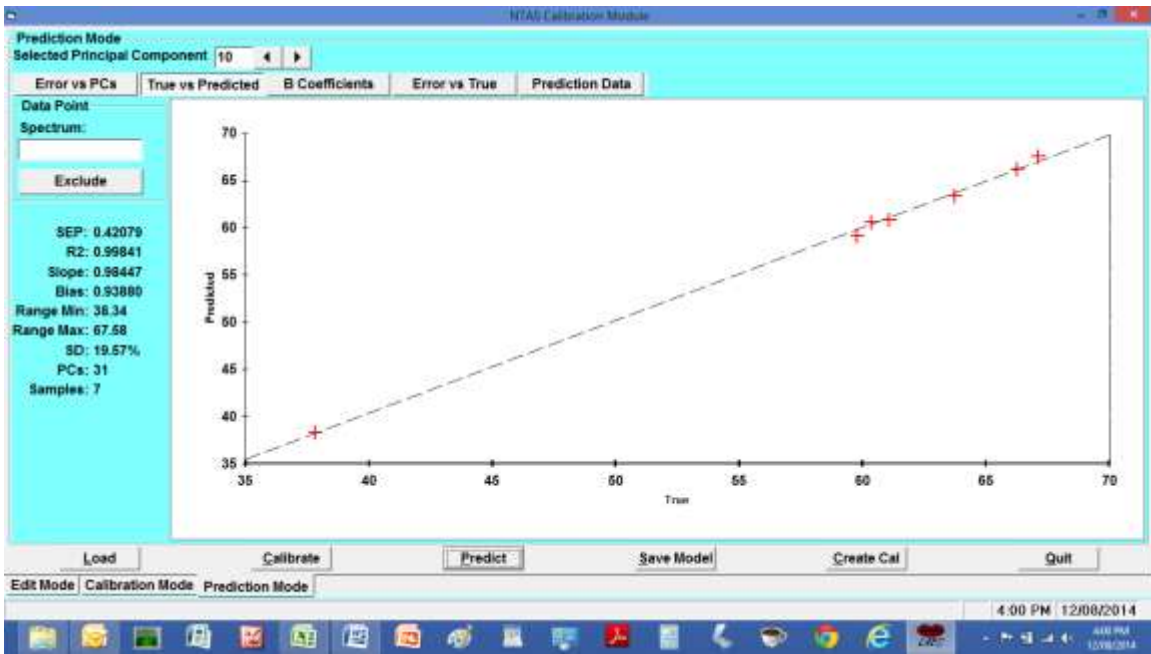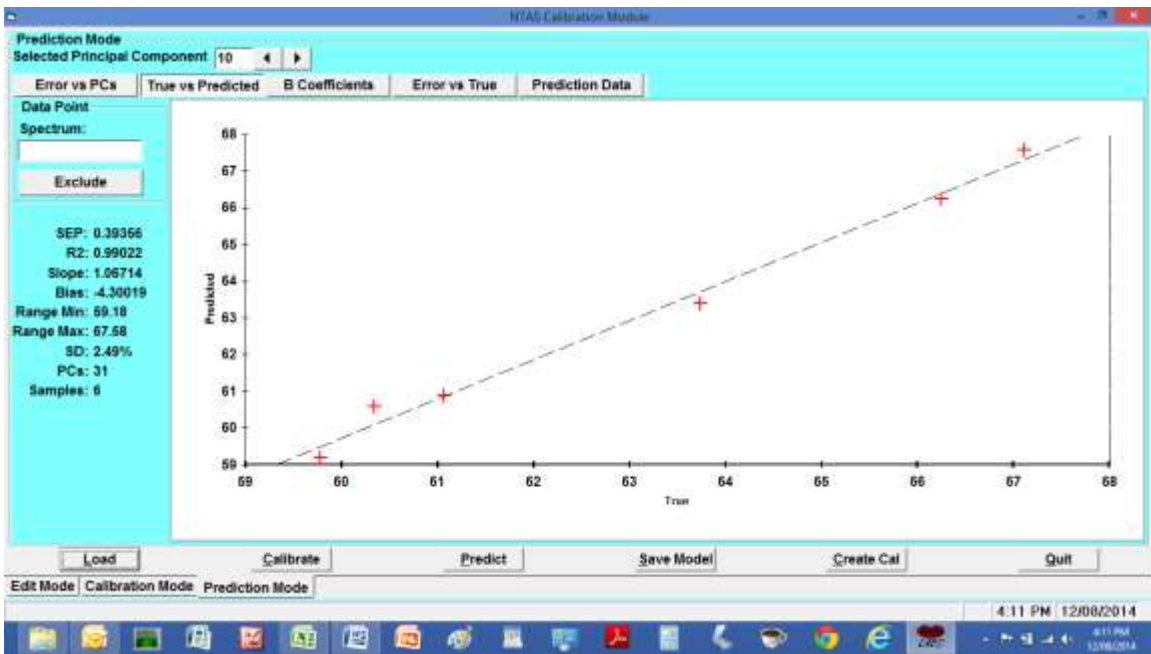
Figure 4. Protein Prediction Plot

Figure 5. Protein Prediction Plot (Excluding Sample 23)

Table 1. sumamrises the calibration and prediction statistics.

	SEC	R^2	SEP	R^2
Protein(Including Sample 23)	0.31%	.992	.42	.98
Protein (excluding Sample 23)			.39	.990

Discussion:

The measurement of protein using NIR in very high protein samples such as the Hemp Powder provided for this study is a new application. Similar products such as whey powder, soybean protein powder and meat meal are very well established NIR methods and since the NIR spectra of the Hemp Powder is very similar to these other high protein powdered materials, it is not unexpected that a calibration can be easily developed.

There are two things to note in the data presented above.

1) Sample 23 was well outside the range for protein as compared to the other samples, yet when included in the prediction set, the predicted protein was very close to the lab value as shown below;

H21As	60.34	60.60
H22As	63.74	63.39
H23As	37.83	38.34
H24As	61.07	60.89
H25As	66.25	66.25
H26As	59.78	59.18
H27As	67.10	67.58

2) Even though the calibration was developed on a relatively small set of samples, the 7 Validation Samples were accurately predicted.

The conclusion from this study is that the MultiScan Series 4000 FTNIR can be easily calibrated for measuring protein in Hemp Powder. From our experience in NIR method development, it would also be possible to measure moisture, fat and ash in this product.