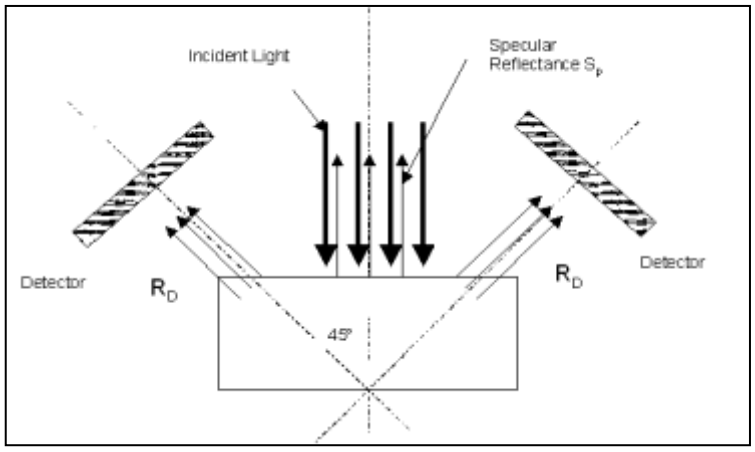
Application Note 184. Milk Analysis using the MultiScan Series 4000 FTNIR Spectrometer

Introduction:

The measurement of opaque liquids such as milk can be made using Near Infrared Reflectance spectroscopy in the 1000 to 2500nm region of the electromagnetic spectrum. The milk solids reflect sufficient light to be able to detect fat, protein, lactose and water content. From these SNF(Solid Non Fat) and TS (Total Soilds) can be calculated.

Within the 1000-2500nm spectral region, Protein (N-H), Moisture (O-H), Fat (C-H) and Lactose(C-O-H) absorb NIR energy at many wavelength. However above 1900nm, the water band absorbs so much light that measurements of protein, fat and lactose are best performed below 1900nm. The 1st and 2nd overtone regions, ie, 1200-1800nm, provide sufficient sensitivity for measuring fat, protein and lactose in milk, cream and condensed milk.

A Fourier Transform (FTNIR) spectrometer using 0 – 45 degree illumination and detection optics, as shown in figure 1, provides a means of collecting NIR spectra from samples such as milk.



This study reports the results of developing calibrations for pasteurised/homogenised milk for fat, protein, lactose, SNF and TS using the MultiScan Series 4000 FTNIR Spectrometer.

Figure 1. Diffuse Reflectance

Procedure:

10 samples of commercially packaged milk with fat content between 1% and 5% were sourced from a local supermarket. The milk containers were kept refrigerated before use. The containers were vigourously mixed and 100mls of milk from each container were placed into a 120ml plastic specimen tubes. The tubes were heated in a microwave oven for 10 seconds, removed and shaken and then heated again for 10 seconds. The sample temperatures were checked at between 40 and 45^{0}C.

Spectra were collected using the MultiScan Series 4000 FTNIR Spectrometer, see figure 2a, at a 16cm-1 resolution. Each spectrum was the average of 10 subscans. 5 spectra were collected as the liquid cell was rotated in the light beam of the Series 4000. Scan times were typically 20 seconds.

The liquid cell, shown in figure 2b, was filled with 40ml of the heated milk and 5 scans were collected for each sample as the liquid cell was rotated in the light beam. Each sample was reloaded 5 times. As such there were a total of 250 spectra collected.

Figure 2a MultiScan Series 4000 FTNIR Spectrometer

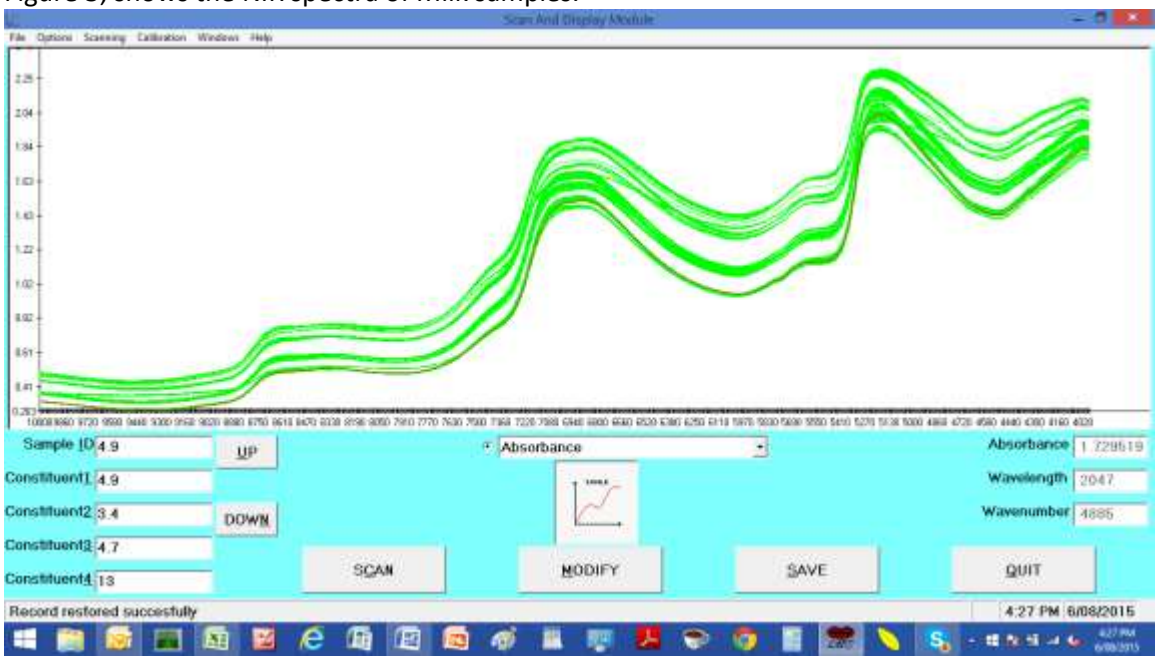
Figure 2b Liquid Cell

The fat, protein and lactose values were taken from the nutritional labels off the packaging. SNF was computed as the sum of the protein and lactose, where as TS was computed as the sum of the protein, fat, lactose and mineral content.

The constituent values were entered into the spectral file using MicroSoft Excel and saved as a .csv file. The spectral file was imported into NTAS(NIR Technology Analysis Software) where a Partial Least Squares Regression was performed between the spectra and the constituent values in order to develop calibration models for each parameter.

Results:

Figure 3, shows the NIR spectra of Milk samples.



Figures 4 show the Calibration Plots for Fat in Milk.

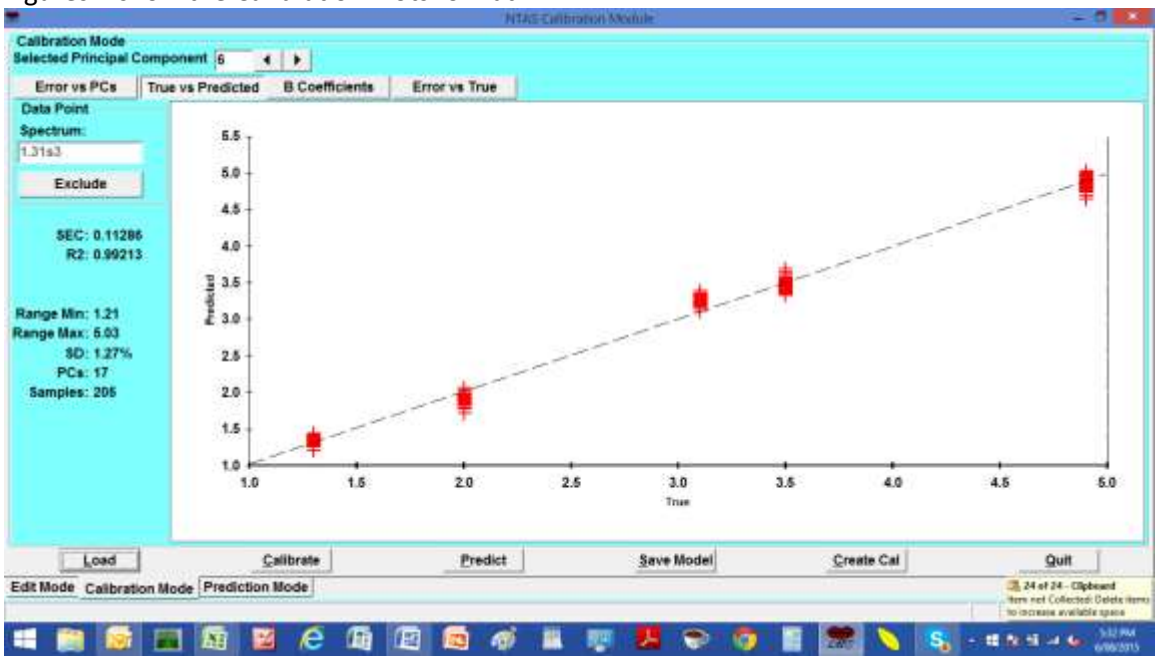


Figure 5 shows the calibration plot for Protein in Milk

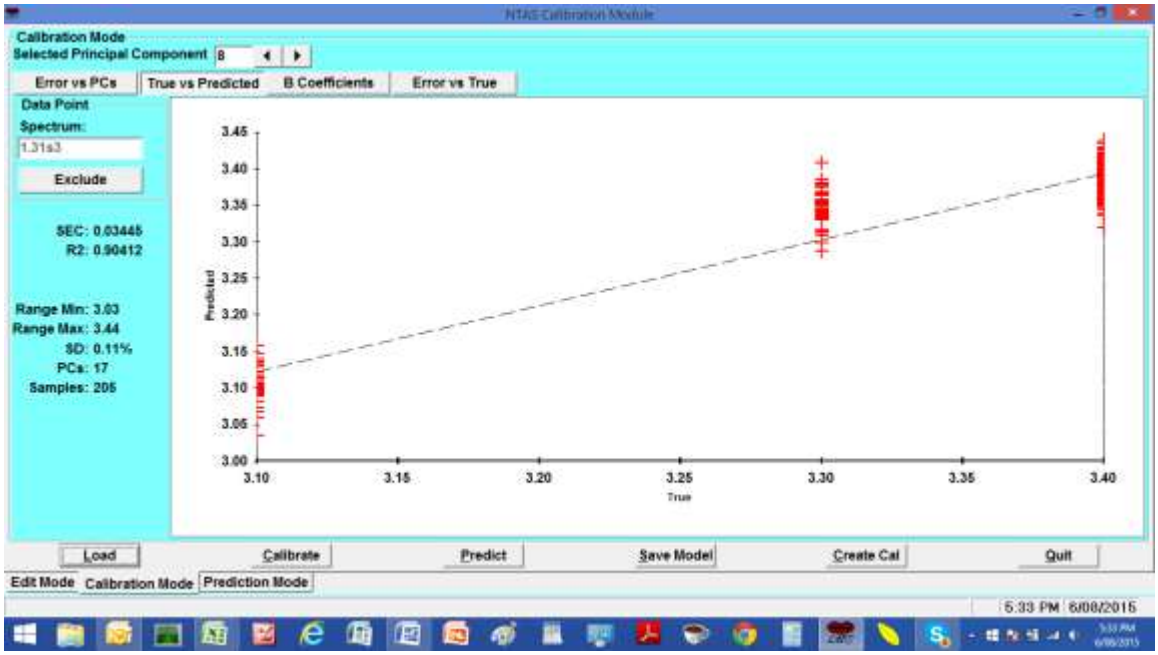


Figure 6. shows the calibration plot for Lactose in Milk.

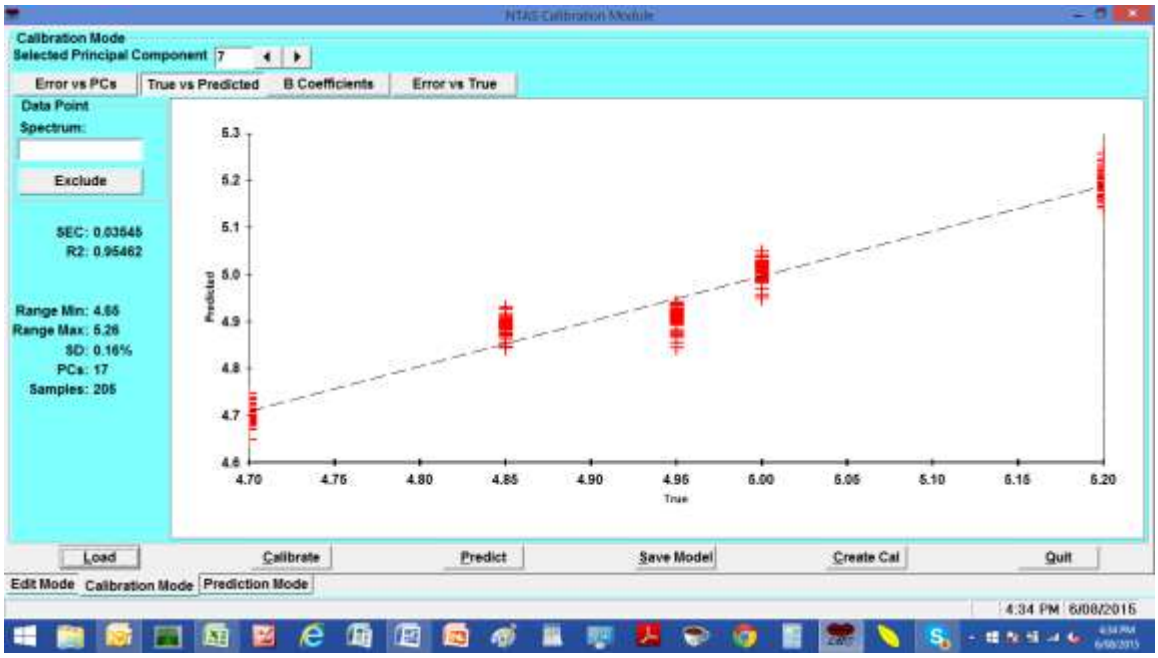


Figure 7 shows the calibration plot for TS.

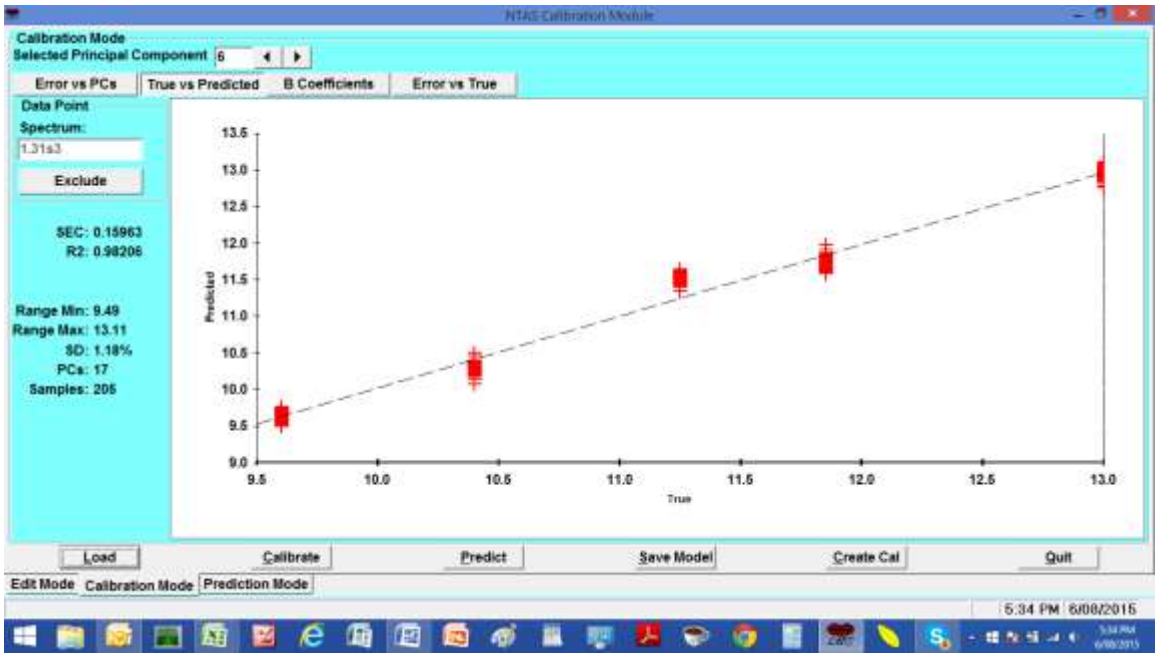
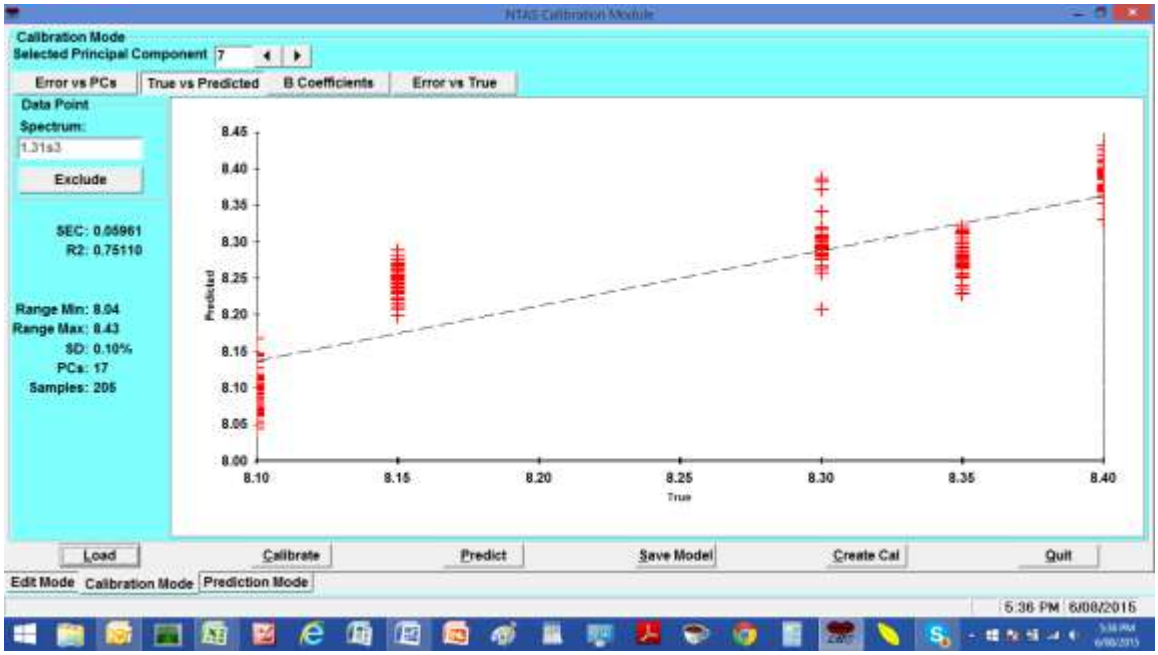


Figure 8 shows the calibration plot for SNF



Four samples of packaged milk were opened and analysed 8 times using the calibration models developed above. Table 1 shows the predicted results for Fat, Protein and Lactose, as the average versus the ref values and the Standard Deviation of Differences between the 8 analyses.

Table 1. Prediction data.

Sample	Ref Fat	Ave	SDD	Ref Protein	Ave	SDD	Ref Lactose	Ave	SDD
2	1.9	1.91	0.06	3.4	3.36	0.03	5.2	5.02	0.02
3	3.1	3.26	0.04	3.5	3.52	0.02	4.95	4.89	0.01
4	3.5	3.46	0.07	3.4	3.34	0.02	5	4.89	0.02
5	4.9	4.85	0.05	3.4	3.38	0.01	4.85	4.72	0.03

Discussion:

The analysis of milk has historically been performed in the Mid IR region using transmission through a sample of milk. The pathlength of the transmission cell sued in the Mid IR region is typically .1mm. Although a well proven method, the Mid IR method requires cleaning of the cell between each analysis. The maintenance and reagent costs associated with the Mid IR method have long been an issue for dairies and milk processors.

Until the introduction of FTNIR Spectrometers, NIR analysers were not capable of measuring milk with the precision and accuracy obtained with the Mid IR method. FTNIR Spectrometers provide an order of magnitude improvement in signal to noise ratio over conventional NIR analysers, and as such, the FTNIR Spectrometer can now match the Mid IR analysers, however the benefits are that the sampling is as simply as pouring the milk into a glass bottomed dish and placing the dish into the FTNIR Spectrometer for analysis. To clean the liquid cell, simply remove it from the spectrometer and rinse under warm water, then dry with a tissue.

The data collected for milk samples using the MultiScan Series 4000 FTNIR Spectrometer shows accuracy and precision in the same order as the Mid IR analysers.

NIR Technology Systems
366 Edgar Street, Condell Park, NSW, 2200, Australia
Tel: 612 9771 5444 Fax: 612 9771 5255
Email: sales@nextinstruments.net Web: www.nextinstruments.net