

### Introduction:

In the conversion of tryglycerides to glycerin and then methyl esters, there is a residual level of mono, di and tri glycerides remaining unreacted. These are generally at very low levels, less than .1%. at higher levels the glycerides can cause build up in the injectors in the engine. As such ,measuring these unreacted glycerides is an important issue for producers of biodeisel.

This study was undertaken to assess the possibility of rapidly measuring glycerides in biodiesel either with an on line analyser or an at line analyser.

### Description:

20 samples of biodiesel were scanned using the NIT-38 Near Infrared Transmission Analyser and a 30mm pathlength cuvette. The samples were at room temperature, eg, 22C, however there was no attempt at controlling the sample temperature.

The samples were poured into the cuvette and placed into the NIT-38. 5 scans of each sample were collected across the wavelength range, 720-1100nm. The spectra were saved in the memory of the NIT-38 and then uploaded into a PC using NTAS(NIR Technology Analysis Software). Figure 1 shows the NIT spectra of these samples.

The spectra were imported in Microsoft Excel where the reference data for Mono, Di and Tri Glycerides and Free Glycerol were manually entered and Total Glycerides was calculated as the sum of the individuals. Since the glyceride levels are very low, the reference values were multiplied by 100. This was do so that values less than .01 would not be rounded down to 0.000, since the NTAS software generally describes numbers to two decimal places. The edited spectra were then imported into NTAS, Calibration Creation routine. A Partial Least Squares Regression was run for each of the four glyceride components. Figures 2 through 4 show the calibration plots of the Mon, Di and Tri Glyceride components. There was insufficient data to run a regression analysis for Free Glycerol.

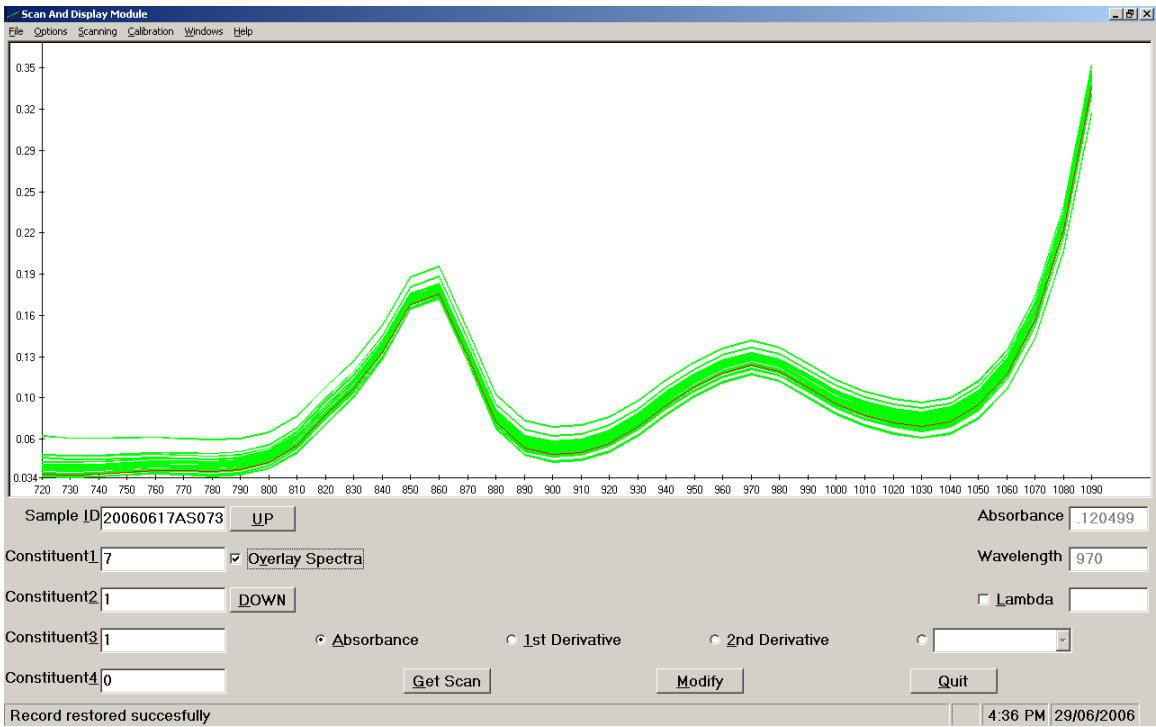


Figure 1. NIT Spectra of Biodiesel

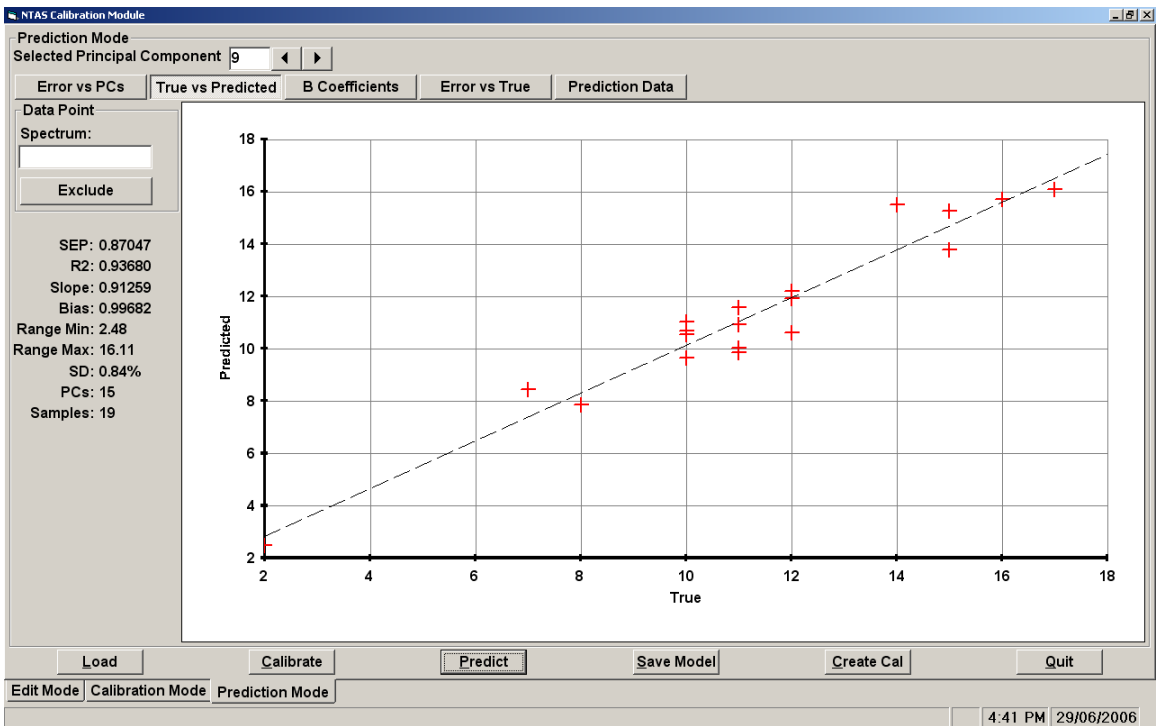


Figure 2. Plot of NIT Calibration for Mono Glyceride in Biodiesel

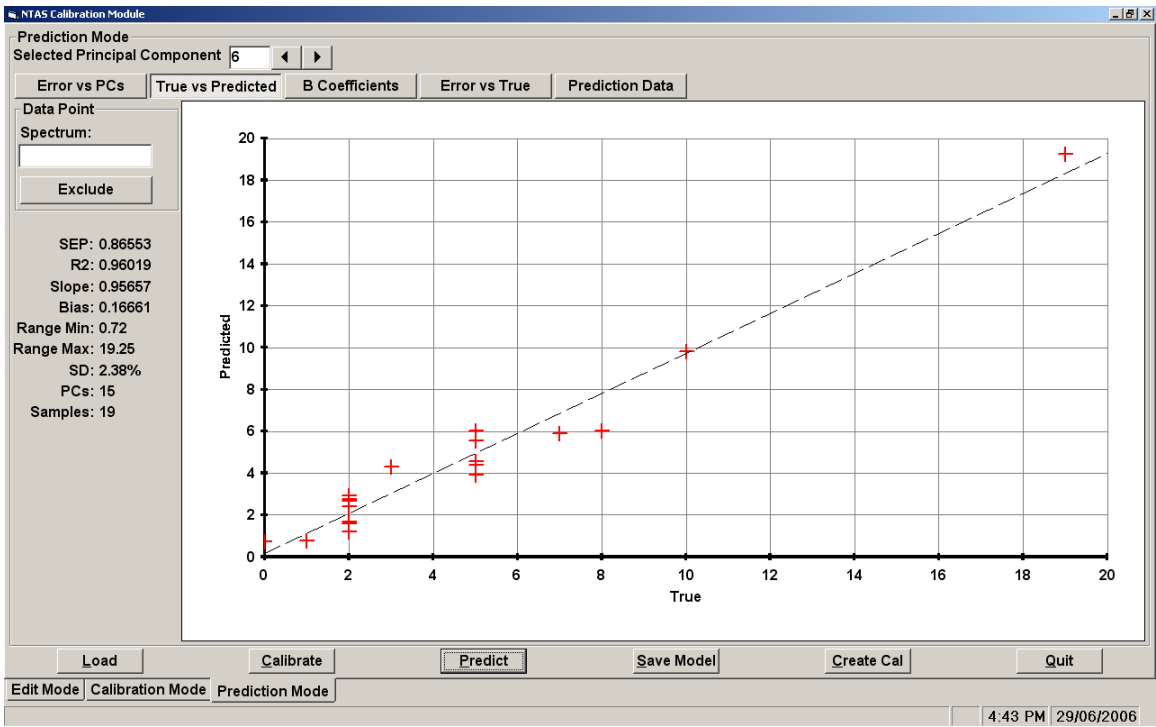


Figure 3. Plot of NIT Calibration of Di Glyceride in Biodiesel

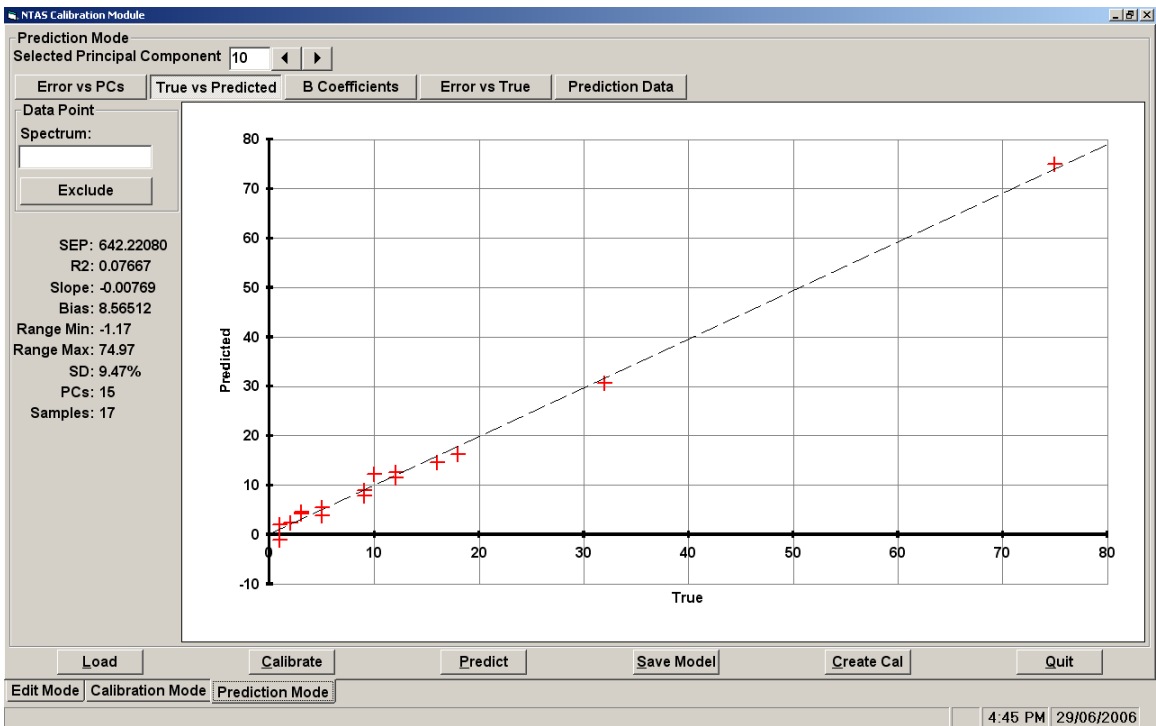


Figure 4. Plot of Calibration for Tri Glyceride in Biodiesel

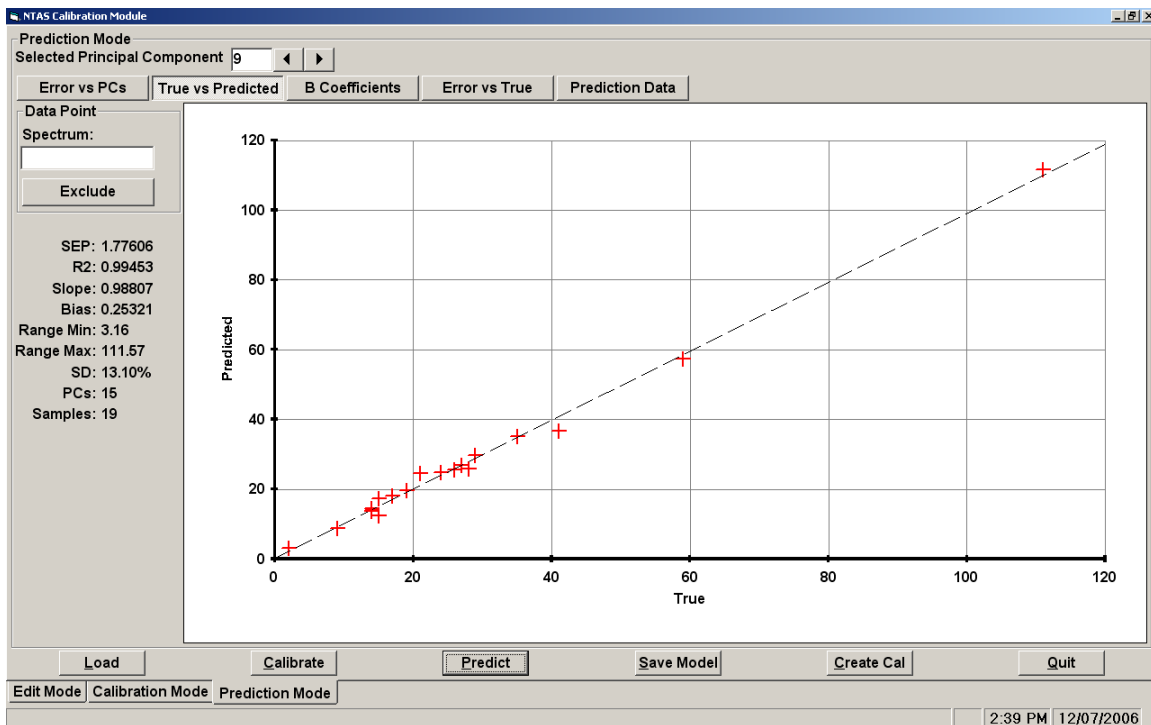


Figure 5. Calibration Plot of Total Glycerides in Biodiesel

### Discussion:

The NIT spectra of the biodiesel samples was collected in approximately 30 seconds per sample. There was minimal cleaning required of the sample cell, ie, rinsing with Heptane and wiping with a tissue, such that it is considered that the sampling procedure would be within the ability of the plant operators. Likewise the operation of the instrument was shown to be very simple and only required two key strokes.

The calibration plots for Mono, Di, Tri and Total Glycerides appears to be very good. The Standard Error of Calibration were;

Component	SEC	R <sup>2</sup>
Mono	0.009	.94
Di	0.009	.96
Tri	0.020	.98
Total	0.018	.99

There is some concern over the size of the concentration factors used in the calibration models. This is most likely due to the low concentration levels, however further assessment is required to prove that the calibration models have not been over fitted. This can be done by analyzing another 10 samples using these calibrations. By analyzing each sample several times, then an estimate of the accuracy and precision of the NIT technique could be determined.