Application Note 174. Calibration Development of Free Fatty Acids in Vegetable Oils Using the MultiScan Series 4000 FTNIR Spectrometer

Introduction:

Near Infrared Transmission spectroscopy is best performed in the 1000 to 2500nm region of the electromagnetic spectrum. Within this spectral region, Protein (N-H), Moisture (O-H) and Fat (C-H) absorb NIR energy. Figure 1, shows a double pass Transmission optics sampling system that provides a means of collecting NIR spectra from liquid samples such as Vegetable Oils. Using a Fourier Transform (FTNIR) spectrometer to collect transmission spectra from oils provides a very accurate and precise means of developing NIR calibrations for Free Fatty Acids, Iodine Values and Peroxide Values in oils.

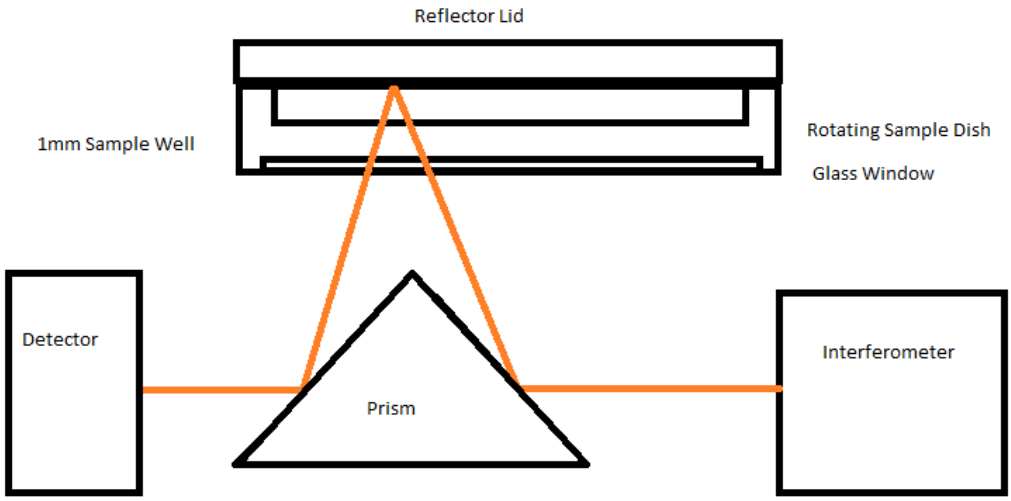


Figure 1. Transmission Cell Optics

This study reports the results of developing a calibration for Free Fatty Acids in Vegetable Oils including Olive Oil, Canola Oil, Sunflower Seed Oil, Mustard Seed Oil and Cod Liver Oil using the MultiScan Series 4000 FTNIR Spectrometer.

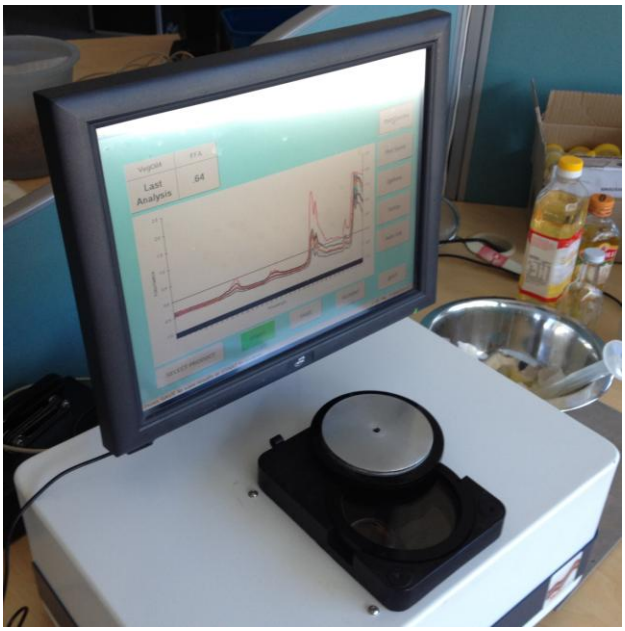
Procedure:

18 samples of vegetable oils were procured from several olive growers and local supermarkets. The samples included:

6 Samples of Olive Oil from 2013
3 Samples of Olive Oil from 2007
1 Bertolli Classic Olive Oil
1 Bertolli Extra Virgin Olive Oil
2 Crisco Canola Oil
1 Simply Canola Oil
1 Homebrand Sunflower Oil
1 Generic Canola Oil
1 Mustard Seed Oil: Imported
1 Cod Liver Oil

Each sample of oil was mixed with petroleum ether and allowed to stand for 30 minutes. The supernatant was collected and the FFA content was determined by titrating the supernatant with a 0.123M NaOH solution using a Phenolphthalein indictor.

10 ml of oil was then poured into the S4000 Sample Dish to create a 1mm thick layer of oil. The Reflector Lid was placed over the oil sample and 10 spectra were collected by rotating the dish in the light beam. The MultiScan Series 4000 FTNIR Spectrometer was setup to scan at 16cm-1 resolution between 10000 and 4000cm-1 or 1000 and 2500nm.



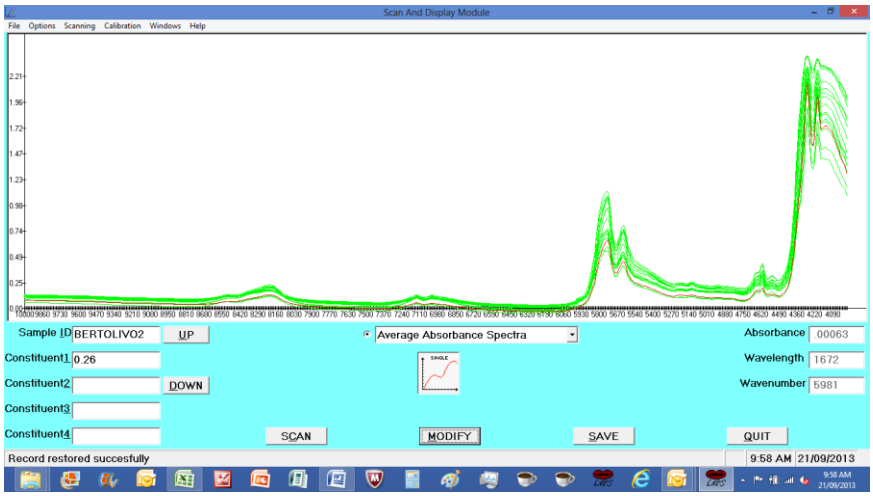
The Series 4000 is operated from a touch screen PC which runs NTAS (NIR Technology Analysis Software) in a Windows environment. The Scan and Display routine was used to collect the NIR spectra for each sample. The sample dish is rotated on a platform which holds a Powdered Teflon reference disc that is illuminated in the same manner as the sample and is used to collect the 100% reference scan which is 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)

These spectra were stored in the PC's memory and then imported into NTAS's Calibration routine where a Partial Least Squares Regression (PLS) calibration for FFA was developed.

Results:

Figure 2. shows the NIR spectra of the 19 samples of Vegetable Oils.



Figures 3 shows the calibration plot for Free Fatty Acids in Vegetable Oils.

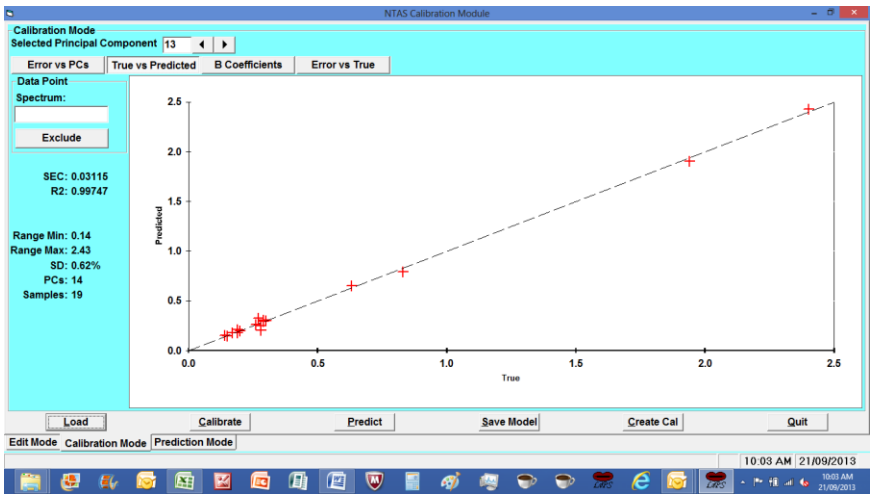


Figure 3.FFA Calibration Plot

Discussion:

It is to be expected that NIR spectroscopy will be suitable for the development of calibrations for Protein, Moisture and Fat in most grain and oil seeds. However, the measurement of Free Fatty Acids is well established. The explanation for the NIR region being sensitive to FFA is that when the Triglycerides breakdown to form fatty acids, there appears a new chemical bond for the carboxylic acid that would not occur in the triglycerides. This bond contains C-O-H as a functional group and this is observed in the NIR region.

Although this is study using a small number of samples, it demonstrates the ability of the Series 4000 FTNIR Spectrometer to measure Free Fatty Acids in a range of vegetable oils.

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