SHIMADZU APPLICATION NEWS

LAAN-A-FT-E011

● SPECTROPHOTOMETRIC ANALYSIS

No.A381

# Analysis of Iodine Value with NIR Heated Transmission Cell and PLS Method

Near infrared (NIR) spectrometry is a simple and quick quantitative method, when used in combination with multivariate analysis methods such as multiple regression calculation and the PLS method. The NIR method draws much attention and is utilized in many fields such as foods, pharmaceuticals and petrochemicals.
Here we introduce examples of analysis of iodine values in fats and oils, using the NIR heated transmission cell and PLS quantitative calculation introduced in Shimadzu Application News No. A365.

## ■ Iodine Value

Iodine is absorbed by fats and oils containing unsaturated fatty acids and opens the double bonds as shown in Fig.1. The iodine value expresses the volume (g) of iodine that is absorbed by 100g of sample material. The larger the amount of unsaturated fatty acids is and the higher the degree of unsaturation in the fatty acids in the sample material is, the higher the iodine value is. In this way, the iodine value is the measure for indicating the degree of unsaturation in fats and oils.

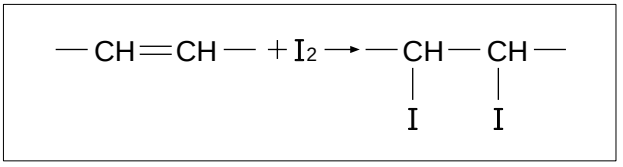


**Fig.1 Reaction of Double Bond with Iodine**

## ■ NIR Spectra of Cooking Oils and Fats

The measurement results of four different kinds of cooking oil with different iodine values using the NIR heated transmission cell are shown in Fig.2. The temperature of the cell was kept at 60˚C to analyze fats and oils with melting temperature higher than the room temperature. The iodine values of the fats and oils used were 133.4, 116.5, 91.5, 80.9 and 52.0. Fig.3 shows the difference spectra (the spectrum of 52.0 iodine value is subtracted from those of other samples). From the measurement results we can see clearly the difference in the iodine values in the NIR spectra. It is important to note that absorption is strong in the 5800cm$^{-1}$ area and the peak is saturated.

**Table 1 Analytical Conditions**

| | |
|---|---|
| Resolution | : 8cm$^{-1}$ |
| Accumulation | : 40 |
| Detector | : InGaAs |
| Cell Temperature | : 60˚C |

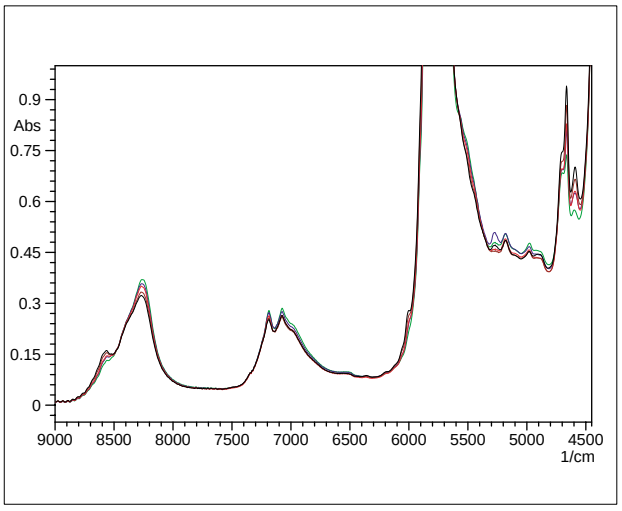


**Fig.2 NIR Spectrum of Oils and Fats**
**Iodine value: black 133.4, brown 116.5, red 91.5, blue 80.9 and green 52.0**

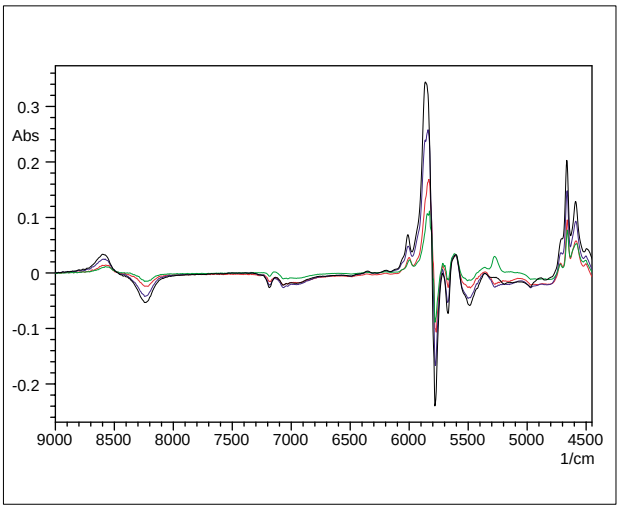


**Fig.3 Spectrum Subtraction**
**black 133.4 - 52.0, blue 116.5 - 52.0, red 91.5 - 52.0 and green 80.9 - 52.0**

## ■ Quantitative Analysis of Iodine Value with PLS

The Iodine value of 22 different types of cooking oils was measured and the PLS method was used for quantitative calculation. The 9000 to 5860cm$^{-1}$ and 5750 to 4500cm$^{-1}$ ranges where the peaks are not saturated were used for quantitative calculation. The calculation results are shown in Table 2. The plotting of the true values (input values) and the predictive values (calculation results) is shown in Fig.4. We can see good results in the iodine value range from 52.0 to 133.4.

Here we introduced an example of quantitative analysis using NIR spectrometry and the PLS method to measure iodine values of fats and oils. It is expected that the combination of NIR spectrometry and the PLS method will be used for a wider range of applications, including simultaneous quantitation of multiple substances.

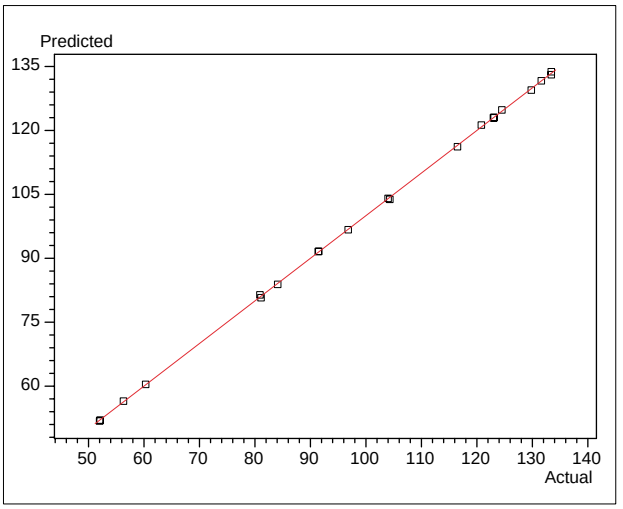


**Fig.4 Relationship between True and Predicted Iodine Values**

**Table 2 Results of Quantitative Calculation by PLS Method**

PLS Calibration Method Report

Calibration curve table : calibration curve table
Algorithm : PLS I
No. of elements : 1
Number of references : 22
Range [1] : 5860.00 - 9000.00
Range [2] : 4500.00 - 5750.00
Preprocessing :
PLS calibration method
Base line: auto zero
Data centered : yes
Elements : element 1
Factors : 6
Correlation coefficient : 0.99995
MAEP : 0.00009
SEP : 0.00961
X leverage report : 0
Y Residual error report : 0

| Reference spectra | True value | Predicted value | Difference (%) | Concentration residue | Spectral residue |
|---|---|---|---|---|---|
| 1 ¥ 1 | 123 | 122.96 | 0.032 | 0.04 | 0.033 |
| 2 ¥ 2 | 124.5 | 124.818 | -0.256 | -0.318 | 0.034 |
| 3 ¥ 3 | 104 | 104.058 | -0.055 | -0.058 | 0.076 |
| 4 ¥ 4 | 104.3 | 103.827 | 0.454 | 0.473 | 0.085 |
| 5 ¥ 5 | 116.5 | 116.186 | 0.27 | 0.314 | 0.116 |
| 7 ¥ 7 | 91.5 | 91.618 | -0.129 | -0.118 | 0.057 |
| 8 ¥ 8 | 91.4 | 91.597 | -0.215 | -0.197 | 0.12 |
| 9 ¥ 9 | 129.8 | 129.468 | 0.256 | 0.332 | 0.035 |
| 12 ¥ 12 | 96.8 | 96.707 | 0.096 | 0.093 | 0.072 |
| 13 ¥ 13 | 120.8 | 121.265 | -0.385 | -0.465 | 0.072 |
| 14 ¥ 14 | 123 | 123.039 | -0.032 | -0.039 | 0.062 |
| 15 ¥ 15 | 131.6 | 131.652 | -0.04 | -0.052 | 0.036 |
| 16 ¥ 16 | 123.1 | 123.032 | 0.056 | 0.068 | 0.047 |
| 17 ¥ 17 | 133.4 | 133.083 | 0.237 | 0.317 | 0.039 |
| 18 ¥ 18 | 133.4 | 133.766 | -0.275 | -0.366 | 0.035 |
| 19 ¥ 19 | 60.3 | 60.411 | -0.184 | -0.111 | 0.028 |
| 21 ¥ 21 | 56.3 | 56.489 | -0.335 | -0.189 | 0.014 |
| 22 ¥ 22 | 52.1 | 52.047 | 0.101 | 0.053 | 0.02 |
| 23 ¥ 23 | 52 | 51.822 | 0.343 | 0.178 | 0.023 |
| 24 ¥ 24 | 81.1 | 80.758 | 0.422 | 0.342 | 0.022 |
| 25 ¥ 25 | 84.1 | 83.883 | 0.258 | 0.217 | 0.048 |
| 26 ¥ 26 | 80.9 | 81.415 | -0.637 | -0.515 | 0.047 |

SHIMADZU

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