Application Note

No. 050TR0203-E

Iodine Value Measurement of Fat with FT-NIR System with Peltier Thermostatic Cell Holder

Introduction

Iodine value is used as an index for unsaturation degree of fat for evaluating the nature of fatty oil and bio-diesel. Iodine value can be generally obtained by wet method employing redox reaction as described in ASTM, JIS and so on, which, however, has a drawback that the operation is complicated. In order to solve the problem, vibrational spectroscopic method such as IR and Raman spectroscopy for Iodine value estimation has been proposed. (Refer to 050TR0125 FT/IR Application Data.) To further improve the measurement accuracy of Iodine value, an Iodine value measurement system consisting of an air-cooled Peltier thermostated sample cell holder and FT-NIR (Fourier Transform Near Infrared Spectrometers) has been developed. A calibration curve model was generated with PLS method applied to NIR spectra of fatty oil obtained with the system, and Iodine values obtained with a conventional wet method, and it was intended to show the effectiveness of the system by applying the calibration curve model to some actual samples.

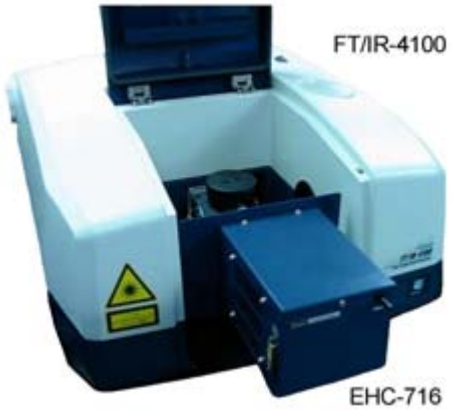

Figure 1: FT-NIR system with thermostated cell holder for Iodine value measurement (customized system)

Experimental

Firstly, 24 kinds of fatty oil having different Iodine value were measured twice and NIR spectra were obtained to make a calibration curve model each time. As a next step, a calibration curve model was generated with PLS method applied to the obtained spectra and the Iodine values obtained with wet method. Finally, the Iodine values of 5 kinds of the actual samples were measured utilizing this calibration curve model.

Experimental Conditions

Instrument:	FT/IR-4100
Light source:	High-intensity ceramic source
Detector:	DLATGS
Accumulations:	16
Resolution:	8 cm^{-1}
Temperature control:	EHC-716 Air-cooled Peltier method (customized)
Temperature setting:	60°C (The samples were solid at room temperature and the temperature was set at higher one than melting point)
Photometric mode:	Transmittance
Cell:	Quartz cell of 5 mm light pathlength

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Results and Discussion

Figure 2 shows NIR spectra of 24 kinds of fatty oil. A calibration curve model was generated with PLS by using the data in the wavenumber range from 4500 to 4800 cm^{-1} for the calculation. Figure 3 shows the calibration curve model which gives good correlation with the true value (obtained by wet analysis method) and evaluated value (obtained by spectral analysis). The correlation factor is 0.9995. The results of evaluating the quantitation reproducibility (n=30) of 5 kinds of fatty oil with different Iodine values using this model are shown in Table 1. The average value and the standard deviation obtained from the spectra are presented in the table. The values in parentheses are Iodine values obtained by wet analysis method. The error between the results obtained with the two analysis methods is less than 2% and the standard deviation of the reproducibility is less than 0.173, which can be considered as a good result. The over-all results show that the system consisting of FT-NIR spectrometer with high wavenumber accuracy and a Peltier thermostated cell holder to control the temperature of the sample is very effective for measuring Iodine value because it can provide almost the same quality data in the Iodine value measurement as the one by wet analysis method, in a quicker, easier and nondestructive way.

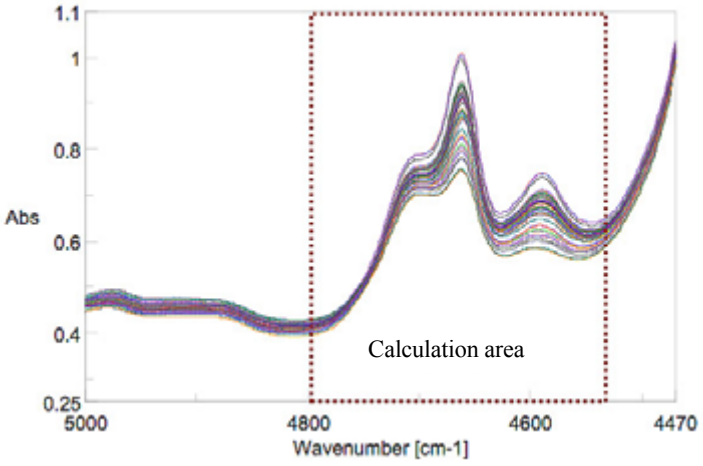

Figure 2: NIR spectra of 24 kinds of fatty oil

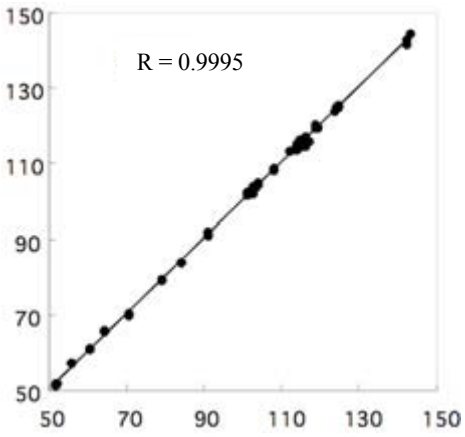

Figure 3: PLS calibration curve model

Table 1: Evaluation result of the actual samples

	A (102.6)	B (115.7)	C (56.3)	D (123.8)	E (116.7)
Average	103.5	115.6	56.4	123.7	119.0
Standard deviation	0.141	0.173	0.135	0.171	0.161

Values in （ ）: Iodine values obtained by wet analysis method (Standard fatty oil analysis method established by Japan Oil Chemists' Society: Wijs-Cyclohexane method)