SHIMADZU

Application News

Spectrophotometric Analysis No.A401

Analysis of Biodiesel Blends by FTIR

Biodiesel fuel is a fuel for diesel engines that is derived from the fats and oils of natural life forms as the raw material. This fuel is receiving much attention as a means of helping to prevent global warming, and its practical use is being advanced in every country around the world. Biodiesel fuel is produced through the conversion of fats and oils into fatty acid methyl esters (abbreviated as FAME) by the process of methyl esterification, in which glycerin is removed. FAME has many advantages, including the fact that it does not generate sulfur oxides during combustion, can be freely mixed with petroleum oil, and has a high flash point as well as high lubricity. The European standard specifies a 5% upper limit of FAME in automotive fuel, and the Japanese standard[1)] conforms to this limit as well.
This Application News introduces the use of FTIR transmittance measurement by the horizontal ATR method to generate a FAME calibration curve for fuel mixtures with 5% or less FAME content.

■ Measurement

The FAME fuel mixture calibration curve was generated using the Shimadzu Fourier Transform Infrared Spectrophotometer IRPrestige-21 with a fixed thickness cell (NaCl window, cell length 0.5 mm) and the horizontal ATR attachment (ATR-8200H) shown in Fig. 1.
The FAME standard mixtures used for the measurement were prepared according to the European EN standard[2)]. The measurement conditions are shown in Table 3.

Fig.1 Horizontal ATR Attachment

■ Infrared Spectrum of FAME

When vegetable oil is used as the fuel for diesel cars, use of the raw fats and oils directly as the fuel causes engine problems due to high viscosity and the occurrence of deposits. Therefore, the raw fats and oils are normally converted to fatty acid methyl esters (FAME) for use as automotive fuel in an ester replacement reaction.
Fig. 2 shows overlaid spectra of FAME (derived from copra oil) and petroleum diesel measured using a horizontal ATR attachment. In the FAME spectrum, absorption due to the ester carbonyl group is clearly evident in the vicinity of 1750 cm^{-1}. Because this absorption is absent in the diesel oil, this peak can be used for quantitation of FAME in diesel oil.

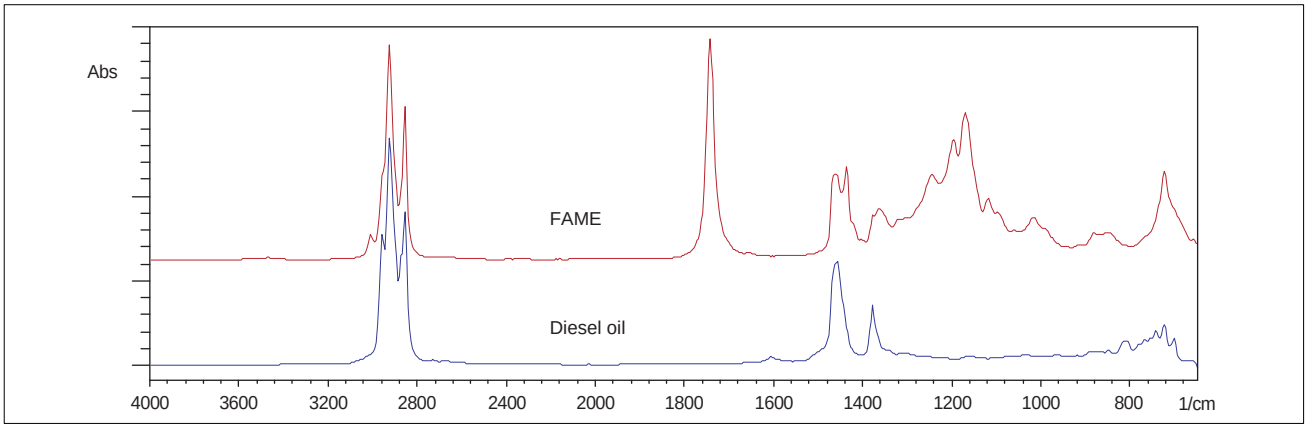

Fig.2 Infrared Spectra of FAME and Petroleum Diesel Measured with a Horizontal ATR Attachment

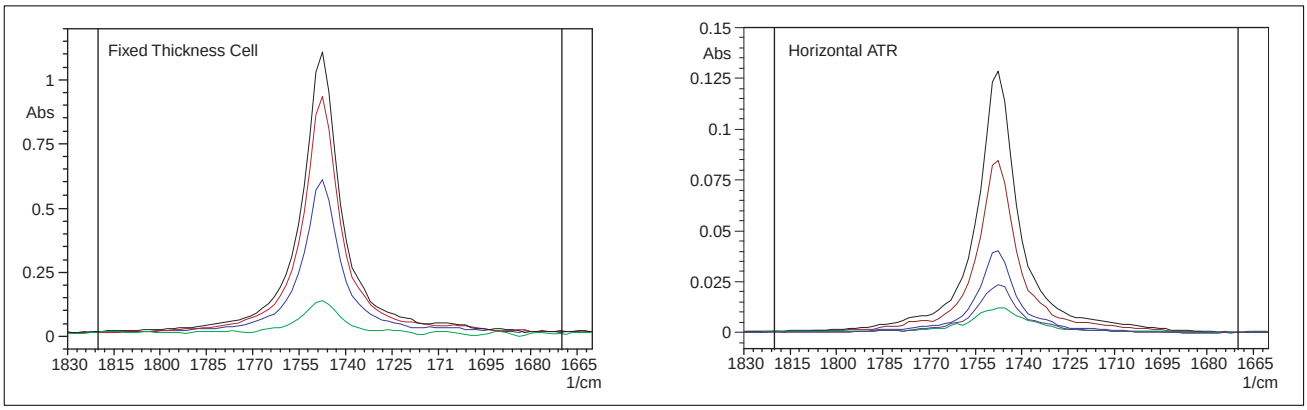

Fig.3 Peak Region Selected for Calibration of FAME

Table 1 Peak Height (Intensity) at each FAME Standard Concentration Measured with Fixed Thickness Cell

Concentration of Standard Mixture	Peak Height
	Fixed Thickness Cell (NaCl 0.5 mm)
0.1% FAME	0.125
0.2% FAME	0.290
0.4% FAME	0.593
0.6% FAME	0.919
0.7% FAME	1.091

Table 2 Peak Height (Intensity) at each FAME Standard Concentration Measured with Horizontal ATR Attachment

Concentration of Standard Mixture	Peak Height
	Horizontal ATR (ZnSe)
0.4% FAME	0.012
0.6% FAME	0.023
1.0% FAME	0.040
2.0% FAME	0.084
3.0% FAME	0.128

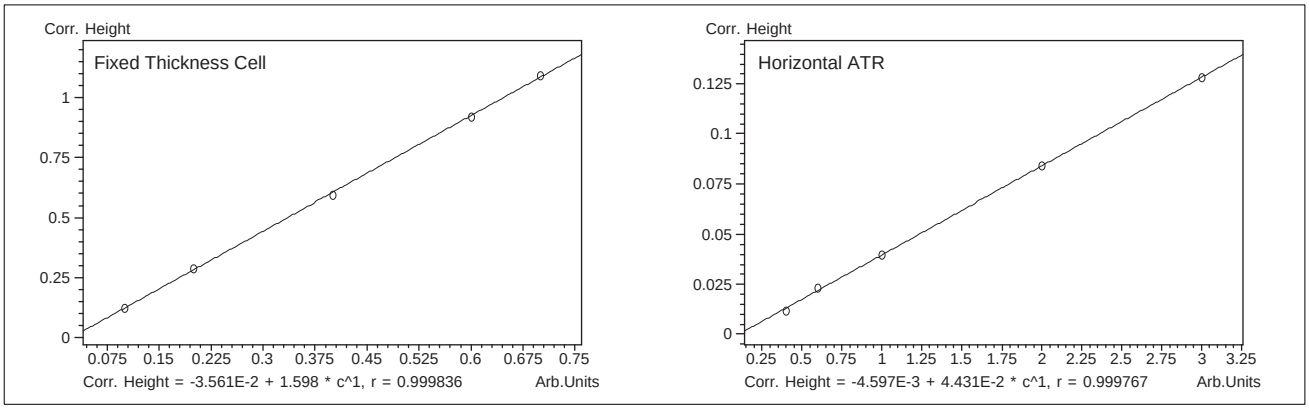

Fig.4 Calibration Curves of Peak Height versus Percentage FAME by Fixed Thickness Cell and HATR

■ Calibration Curves of FAME

Fig. 3 shows the peak of the FAME carbonyl group obtained in measurements using the fixed thickness cell and the horizontal ATR. Table 1 and Table 2 show the concentrations and peak heights of the respective standards. In both methods, excellent results were obtained, with a correlation coefficient greater than 0.9997.
These results indicate that both the fixed thickness cell and the horizontal ATR attachment are effective for quantitative analysis of FAME in diesel fuel mixtures.

Table 3 Analytical Conditions

Resolution	: 4cm^{-1}
Accumulation	: 40

■ References

1) Agency for Natural Resources and Energy “Ministerial ordinance which revises part of the law dealing with enforcement rules for ensuring the quality of volatile oils, etc. (January 15, 2007)”

2) BS EN 14214: 2003 Automotive fuels – fatty acid methyl esters (FAME) for diesel engines - requirements and test methods.

This Application News was created based on material produced by SHIMADZU (ASIA PACIFIC) PTE LTD. (SAP)

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Printed in Japan 3295-06805-10A-IK