

Application News

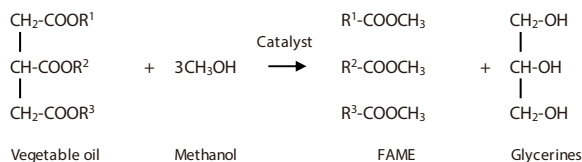
No. A600

Spectrophotometric Analysis

Quantitative Analysis of FAME (Fatty Acid Methyl Ester) in Diesel Fuel by FTIR

Bio Diesel Fuel (BDF) is a general term for fuels for diesel engines in which bio-derived oils are used as the raw material. BDF, which is a form of biomass energy derived from biological sources, has attracted attention from the viewpoint of prevention of global warming, and efforts are being made to use BDF as a fuel for automobiles and railways.

One BDF, as shown in Fig. 1, is a biofuel which is produced by transesterification of oil raw material with methanol in the presence of a catalyst such as sodium hydroxide, removal of glycerines, and conversion to fatty acid methyl ester (FAME).



R¹ to R³: Chain hydrocarbon groups

Fig. 1 Formation of FAME

FAME has a number of advantages, in that it does not form sulfur oxides (SO_x) when burned, it can be mixed with petroleum at arbitrary ratios, and it has a high flash point and lubricating ability.

The properties of FAME under a condition in which it is not mixed with diesel fuel are provided in the European standard EN14214⁽¹⁾, and the upper limit of FAME that can be mixed with diesel fuel is set at 7 wt% in the EN590:2009⁽²⁾, which is a quality standard for diesel fuels. Standardization has also been studied in Japan referring to the European standards, and the upper limit of FAME that can be mixed with diesel fuel was set at 5 wt%⁽³⁾.

This article introduces a quantitative analysis method for FAME in diesel fuel by the transmission method of the Fourier transform infrared spectrometer (FTIR).

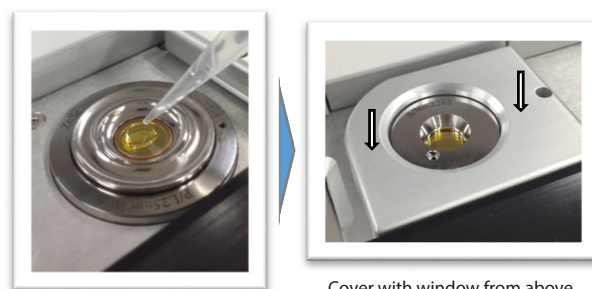
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Measurement Method

In the transmission measurements, the Pearl™ horizontal type liquid FTIR transmission accessory (Specac) shown in Fig. 2 was used. Pearl™ is an easy-to-use accessory product that prevents entry of air bubbles, as liquid samples can be held in a horizontal position, and can be cleaned more easily than the liquid cells that are generally used. Sample setting is extremely simple, requiring only dripping a liquid sample followed by covering with the window (see Fig. 3).



Fig. 2 Pearl™ Horizontal Type Liquid FTIR Transmission Accessory



Drip liquid sample

Cover with window from above

Fig. 3 Sample Setting Procedure

FAME Quantitation Method

In accordance with the test method for measuring the concentration of FAME with an FTIR provided in EN14078⁽⁴⁾, samples with concentrations of 0.1, 0.2, 0.3, 0.4, 0.6, and 1.0 vol% were prepared by diluting FAME (content: 90%) in diesel fuel with cyclohexane. As shown in Fig. 4, absorption by the carbonyl group of esters can be confirmed at around 1,750 cm⁻¹ in the spectrum of FAME. Here, quantitation of FAME was done using the peak height value of 1,747.82 cm⁻¹.

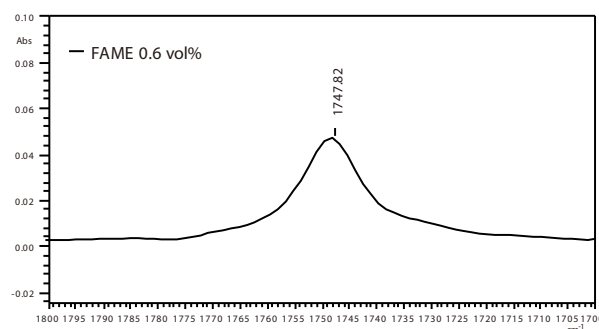


Fig. 4 Peak of Carbonyl Group Used in Quantitation of FAME

■ Preparation of Calibration Curve

After a background measurement by injecting cyclohexane in the Pearl™, samples with FAME concentrations of 0.1, 0.2, 0.3, 0.4, 0.6, and 1.0 vol% were measured. As provided in EN14078, a calibration curve was prepared by drawing baselines at 1,670 cm⁻¹ and 1,820 cm⁻¹ in the infrared spectra obtained by those measurements, and obtaining the height (corrected height) from the baselines to the peak top of 1,747.82 cm⁻¹. Satisfactory results were obtained, as the coefficient of correlation was 0.999. The measurement conditions, calibration curve, and absorbance of the samples are shown below in Table 1, Fig. 5, and Table 2, respectively.

Table 1 Measurement Conditions

Instrument	: IRSpirit™-T (KBr window plate) Fourier transform infrared spectrometer Pearl™ horizontal type liquid FTIR transmission accessory (optical pass length: 25 μm)
Resolution	: 4 cm ⁻¹
Cumulative number	: 20
Apodization function	: Sqr-Triangle
Detector	: DLATGS

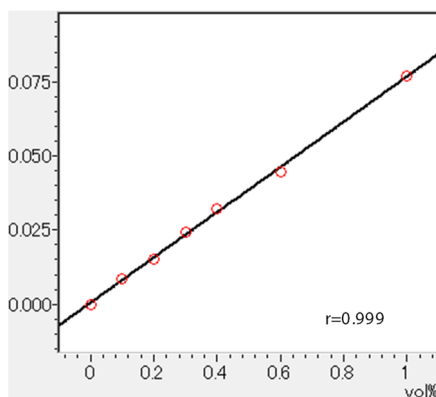


Fig. 5 Calibration Curve

Table 2 Absorbance of Samples

FAME concentration [vol%]	Absorbance Abs
0.1	0.008
0.2	0.015
0.3	0.024
0.4	0.032
0.6	0.045
1.0	0.077

■ Confirmation of Measurement Error

In EN14078, the allowable error in two measurements of the same sample using the same analytical instrument is 0.029 vol% for diluted samples. Here, two measurements were carried out, and the measurement error was confirmed. As a typical result, Fig. 6 shows the 1st and 2nd infrared spectra of a sample with a FAME concentration of 0.6 vol%, and Table 3 shows the measurement error. The measurement error was 0.003 vol%, which satisfies the allowable value.

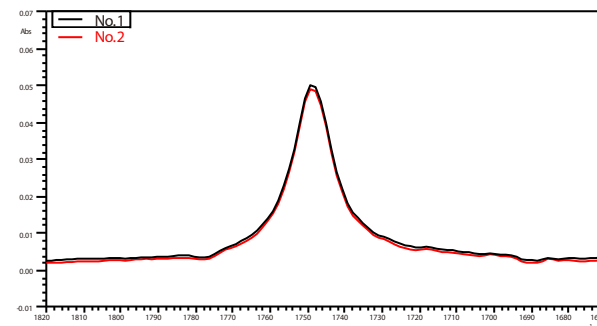


Fig. 6 Infrared Spectra of 1st and 2nd Measurements

Table 3 Measurement Error

	Absorbance Abs	FAME concentration [vol%]
1 st measurement	0.047	0.593
2 nd measurement	0.046	0.590
Measurement error	0.001	0.003

■ Conclusion

A quantitative analysis of FAME in diesel fuel was carried out by using a Pearl™ horizontal type liquid FTIR transmission accessory. A calibration curve was prepared from the peak height of around 1,750 cm⁻¹, and a satisfactory coefficient of correlation of 0.999 was obtained. Two repeated measurements were made, and a measurement error of 0.003 vol% was obtained, confirming that this method satisfies the allowable measurement error of 0.029 vol% for diluted samples provided in the standard. Pearl™ is an FTIR accessory that is simple to use and can be cleaned more easily than conventional liquid cells. These results show that transmission measurement by FTIR is an effective technique for quantitative analysis of FAME.

<References>

- (1) EN14214: Liquid petroleum products - Fatty acid methyl esters (FAME) for use in diesel engines and heating applications - Requirements and test methods
- (2) EN590: Automotive fuels - Diesel - Requirements and test methods
- (3) Website of the Agency for Natural Resources and Energy, Ministry of Economy, Trade and Industry (METI): "Securing the Quality of Petroleum Products"
https://www.enecho.meti.go.jp/category/resources_and_fuel/distribution/hinnkakuhou/ (as of July 16, 2019)
- (4) EN14078: Liquid petroleum products - Determination of fatty acid methyl ester (FAME) content in middle distillates - Infrared spectrometry method

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