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Application News

No.A468

Spectrophotometric Analysis

Quantitation of Fatty Oil in Cutting Fluid by Single Reflection ATR Spectroscopy

– Measurement of Fatty Oil According to JIS K 2241 –

Cutting fluid is a type of lubricating oil that is used to suppress the generation of heat or friction when cutting metal, and it is distinguished as either water-insoluble or water-soluble depending on its composition. If it is water-insoluble, it is further classified into four types according to its fatty oil content, total sulfur content, and corrosiveness to copper.
In JIS K 2241 Cutting Fluid (Supplement 1), two types of fatty oil content tests are described for water-insoluble cutting fluid analysis, the saponification test method and the infrared spectroscopy method. The test method using an infrared spectrophotometer specifies that a calibration curve be generated using standard samples consisting of paraffin solvent spiked with rapeseed oil, and that quantitation of the carbonyl group in the cutting fluid be conducted. This is the same method as the analysis of fatty acid methyl esters (FAME) in biodiesel, which is described in EN 14078 and ASTM D7371. Here, we introduce the determination of fatty oil content using an infrared spectrophotometer in accordance with JIS K 2241.

Measurement of Fatty Oil by Single Reflection ATR Spectroscopy

JIS K 2241 specifies that the sample absorbance be measured by Fourier transform infrared spectroscopy using the attenuated total reflection (ATR) technique, and that the fatty oil content be determined using a calibration curve generated beforehand. Here, measurement was conducted using the sample compartment-integrated MIRacle10 single reflection total attenuated reflectance (ATR) accessory (diamond prism / support element ZnSe), as shown in Fig. 1.
First, to generate the calibration curve, commercially available rapeseed oil was added to liquid paraffin to prepare standards at concentrations of 5.0, 7.5, 10.0, 12.5 and 15.0 % (mass fraction %) according to the JIS procedure. The standard oil samples with different concentrations were thinly coated on the ATR prism, and the spectra were measured. The measurement conditions are shown in Table 1, and the overlaid standard sample spectra are shown in Fig. 2. Also, Fig. 3 shows the expanded spectra of the standard samples from 1600 to 1900 cm^{-1}.

Fig. 1 IRAffinity-1 with Mounted MIRacle10 ATR Accessory

Table 1 Instruments and Analytical Conditions

Instrument	: IRAffinity-1 MIRacle10 (diamond/ZnSe)
Resolution	: 4cm^{-1}
Accumulation	: 20
Apodization	: Happ-Genzel
Detector	: DLATGS

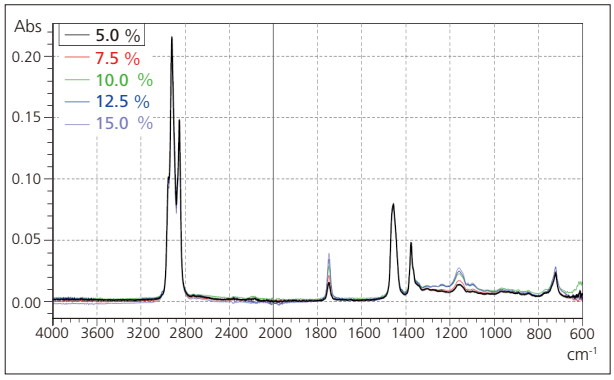

Fig. 2 Spectra of Standard Samples

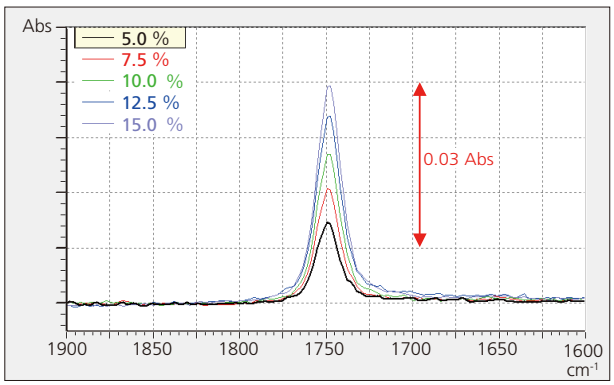

Fig. 3 Expanded Spectra of Standard Samples

New Quantitation Program of LabSolutions IR

The quantitative measurement program that is included in the standard LabSolutions IR software permits simple calibration curve generation and quantitation of unknown samples. By loading the previously measured spectrum files and setting the calibration curve parameters, including component names, unit, and quantitation method, etc. beforehand, the calibration curve can be generated automatically. An example of the calibration curve parameters is shown in Fig. 4.
JIS K 2241 specifies that "Absorbance is the value expressed by the difference between the peak top (point at which peak is maximum between 1740 and 1750 cm^{-1}) in the spectrum and the baseline (a location where the baseline is stable on both sides between 1740 and 1750 cm^{-1})." Therefore, we generated a calibration curve using a peak in the vicinity of 1749 cm^{-1} and the baseline between 1712 and 1780 cm^{-1} for the corrected peak height.
The generated calibration curve is shown in Fig. 5. The correlation coefficient was greater than 0.9998, indicating excellent results.

	Parameter	Value
1	Substance	Fatty oil content
2	Unit	%
3	MSC	None
4	Conversion process	None
5	Quantitation method	Peak height
6	Name	Peak1
7	Expression	PeakHeight 1712.000, 1749.000, 1780.000, BC: ON
8	Calibration curve conditions	Multi-point calibration curve
9	Order	1st order
10	Origin	Not used
11	Correlation coefficient	r

Fig. 4 Calibration Curve Parameters

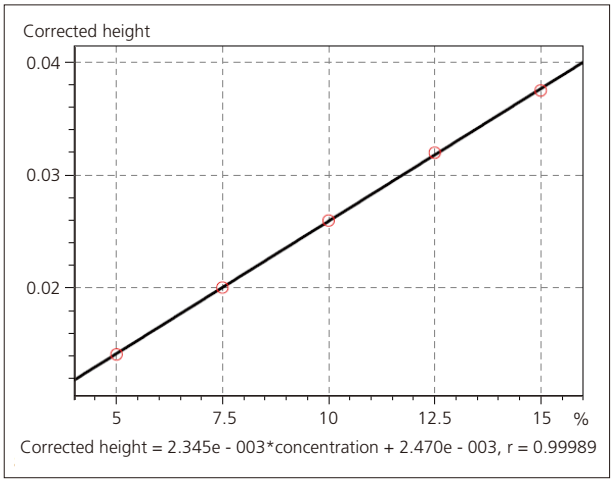

Fig. 5 Calibration Curve of Fatty Oil Contained in Standard Samples by Single Reflection ATR Spectroscopy

After generating the calibration curve, we conducted quantitation of an unknown sample. Either an unknown sample can be measured, or the spectrum file of a previously measured sample can be loaded into the software.
Here, we analyzed a sample of commercially available water-insoluble cutting fluid. An expanded region of the unknown sample spectrum is shown in Fig. 6. The fatty oil content of water-insoluble cutting fluid measured here was calculated to be 10.7 %.

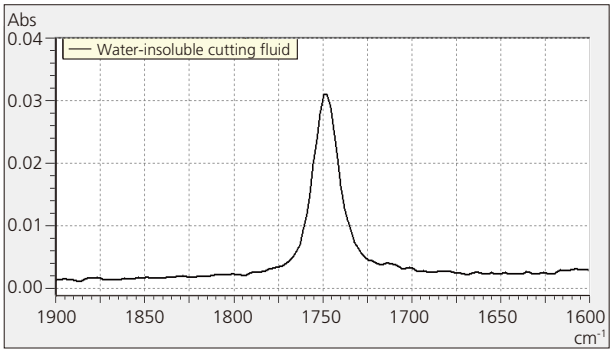

Fig. 6 Peak of Unknown Sample

Conclusion

The single reflection ATR method was used to measure the fatty oil content of water-insoluble cutting fluid according to JIS K 2241 Cutting Fluid (Supplement 1), and quantitation was conducted using a calibration curve with good linearity with a correlation coefficient greater than 0.9998. This technique is effective for quality control of cutting fluids and their waste.
Use of the LabSolutions IR quantitative measurement program makes it easy to generate and store calibration curves, and conduct quantitative analysis of unknown samples. In addition to the quantitation of fatty oil in cutting fluid, a wide variety of quantitative analyses are supported.
Also, for details regarding fatty acid oil testing of cutting fluid, please refer to JIS K 2241 Cutting Fluid (Supplement 1).

[Reference]
JIS K 2241:2007 Cutting Fluid (Supplement 1)

First Edition: Sep. 2013

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