

Application News

No. A547

Spectrophotometric Analysis

Quantitative Analysis of Fatty Oil Content in Cutting Fluid Using IR Pilot

The Shimadzu IRSpirit launched recently is a compact Fourier transform infrared spectrophotometer characterized by the highest signal to noise ratio and the highest maximum resolution in its class (Fig. 1). The dimensions of the body are W390 × D250 × H210 mm and less than A3 in size. The IRSpirit adopts a unique design which allows not only lateral installation but also longitudinal installation so that it can be installed in a narrow space in width. The IRSpirit also has another feature that it can mount the existing accessories such as a single reflection type attenuated total reflection attachment and a diffuse reflection attachment and the commercially available accessories in addition to the accessories for the transmission measurement such as a demountable cell and a KBr pellet holder.

A dedicated program named IR Pilot to assist the analysis, which allows four measurement items - identification test, contaminant analysis, quantitative analysis and film thickness determination - to be easily operated, is equipped in the LabSolutions IR software which controls the IRSpirit and executes data manipulations. By using the IR Pilot, successive operations of measurement, analysis and print can be orderly performed following the on-screen instructions. The IR Pilot is comprised of twenty-three kinds of the dedicated program of which the often-used program up to four kinds can be registered in its main menu.

This article introduces the determination of fatty oil content in cutting fluid using the IR Plot.

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Fig. 1 External View of IRSpirit compact FTIR

■ Determination of Fatty Oil Content in Cutting Fluid

JIS K 2241 Cutting Fluid (Supplement 1) describes the test methods for fatty oil content and total sulfur content in water-insoluble cutting fluid and total sulfur content in water-soluble cutting fluid. In these test methods, fatty oil content in water-insoluble cutting fluid is determined by the saponification test method or the infrared spectroscopy test method.

This article introduces the determination of fatty oil content in cutting fluid using the IR Plot following Application News No. A468A "Quantitation of Fatty Oil in Cutting Fluid by Single Reflection ATR Spectroscopy - Measurement of Fatty Oil According to JIS K 2241 -".

■ Calibration Curve for Fatty Oil Content

The standard samples with concentrations of 5.0, 7.5, 10.0, 12.5 and 15.0 % (mass fraction %) were prepared by adding commercially available cooking oil to liquid paraffin to generate a calibration curve using "Quantitation" program in the LabSolutions IR software. The IRSpirit mounting the QATR-S single reflection ATR attachment was used for the measurement. Infrared spectra of the standard samples were measured after dropping them on a diamond prism. The calibration curve was generated using the peak intensities at 1749.17 cm⁻¹. Fig. 2 shows their infrared spectra expanded in the range from 1900 cm⁻¹ to 1600 cm⁻¹. The obtained calibration curve is shown in Fig. 3.

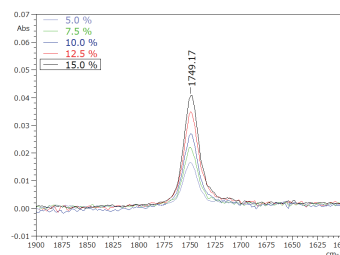


Fig. 2 Peaks of Standard Samples at 1749.17 cm⁻¹

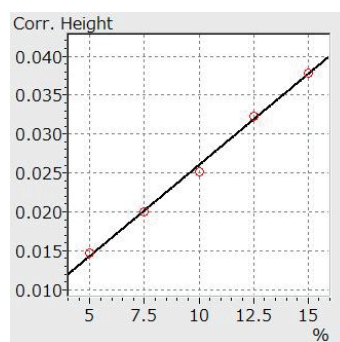


Fig. 3 Calibration Curve

In Fig. 3, the abscissa and the ordinate represent the concentration and the peak intensity after baseline correction at 1749.17 cm⁻¹, respectively. The correlation coefficient of the calibration curve was 0.998 with high correlation between them.

■ Quantitative Analysis Using IR Pilot

Fatty oil content in unused and used cutting fluid was determined using the quantitation program in the IR Plot. After starting the IR Plot, "Execute" button is pressed. Then "Spectrum" program in the LabSolutions IR is displayed on the screen and the IRSpirit is automatically initialized. When "Main Menu" shown in Fig. 4 is displayed, "Quantitative Analysis" surrounded with a blue box is selected. ATR spectroscopy and a diamond prism were selected as the measurement method and a using ATR prism, respectively.

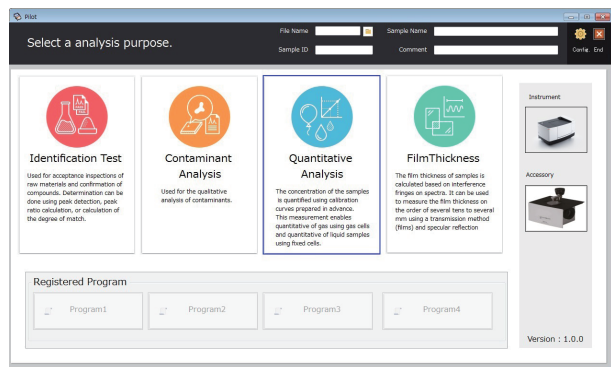


Fig. 4 Main Menu

The quantitation program needs to read in the calibration curve used for the quantitation before the measurement. The calibration curve obtained with the LabSolutions IR is used for it. In this article, two unknown samples were determined. Fig.5 show a screen to input the number of the unknown sample and that to read in the calibration curve.

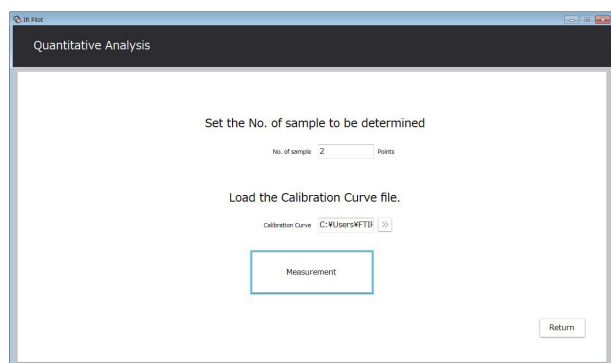


Fig. 5 Screen to Input Number of Unknown Sample and to Read in Calibration Curve

After inputting the number of the unknown sample, a background measurement and a sample measurement are performed successively. The operations such as the sample setting are conducted according to the on-screen instruction. After the measurement, the quantitation results are displayed shown in Fig. 6. The fatty oil content in unused cutting fluid shown as No. 1 and that in used cutting fluid shown as No.2 are 13.08 % and 6.84 %, respectively. After displaying the results, a screen to select the execution of the print is displayed. Fig.7 shows an example of the print. Infrared spectra of the standard samples and unknown samples, the calibration curve, the unknown sample table, the calibration curve parameters and the measurement parameters are printed.

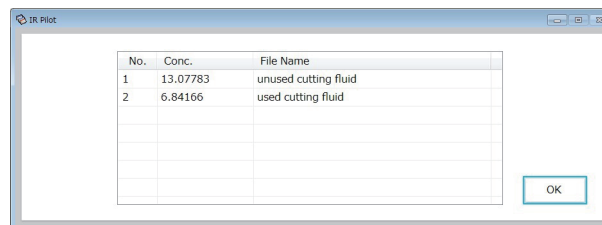


Fig. 6 Results of Quantitation

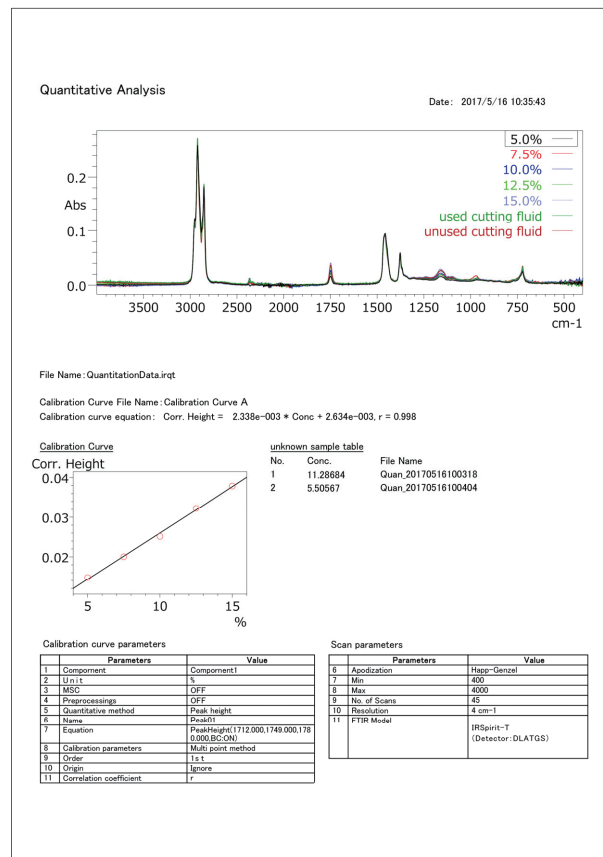


Fig. 7 Example of Print

Conclusion

The quantitative analysis using the IR Pilot facilitates successive operations of the read of a calibration curve, the background measurement, the sample measurement, the quantitative calculation and the print because they are performed only by selecting the setting in sequence. The IR Pilot allows a beginner of FTIR and a user who has no experience of the quantitative analysis using FTIR to conduct it easily with the IR Spirit. Please try to use the IR Spirit and the IR Pilot.

If you need to know the detail on the determination of fatty oil content in cutting fluid, please refer to JIS K 2241 Cutting Fluid (Supplement 1).

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