SHIMADZU APPLICATION NEWS

LA103-E000

● SPECTROPHOTOMETRIC ANALYSIS

No.A308

Liquid Sample Analysis with the MIRacle Single-reflection ATR Accessory

This Application News presents examples of liquid-sample analysis using the MIRacle single-reflection ATR accessory that was introduced in Application News A304.
The liquid-film method and multiple-reflection ATR (horizontal ATR) method are frequently used for liquid-sample analysis. Each method offers its own merit: the liquid-film method allows adjustment of the absorption intensity (sensitivity) with the cell thickness; and the multiple-reflection ATR method allows easy cleaning after sampling analysis.
However, neither of these methods can achieve measurement without peak saturation across the full wavelength range: the liquid-film method requires adjustment of the cell thickness and the multiple-reflection method requires adjustment of the drip-volume (contact area) onto the prism.
Conversely, as only one reflection is required using the MIRacle single-reflection ATR accessory, good spectra can be obtained without peak saturation simply by dripping the sample onto the prism.

■ Outline of the ATR Sampling Accessory

A photograph of the MIRacle accessory is shown in Fig. 1. As described in Application News A304, the accessory comprises four major parts: base unit, ATR prism plate, liquid sample adapter plate, and pressure clamper with micrometer. However, as liquid samples can be simply dripped onto the prism, pressing the sample against the prism is not required and the pressure clamper with micrometer can be removed.

Fig.1 MIRacle sampling accessory

Table 1 Analytical conditions

Resolution	:	4cm^{-1}
Accumulation	:	100
Detector	:	DLATGS

■ ATR Spectra of Liquid Samples

Fig. 2 shows the spectra for water, mayonnaise, and vegetable oil measured using a ZnSe prism. A good spectrum without peak saturation was obtained for each sample. The analytical conditions are listed in Table 1.

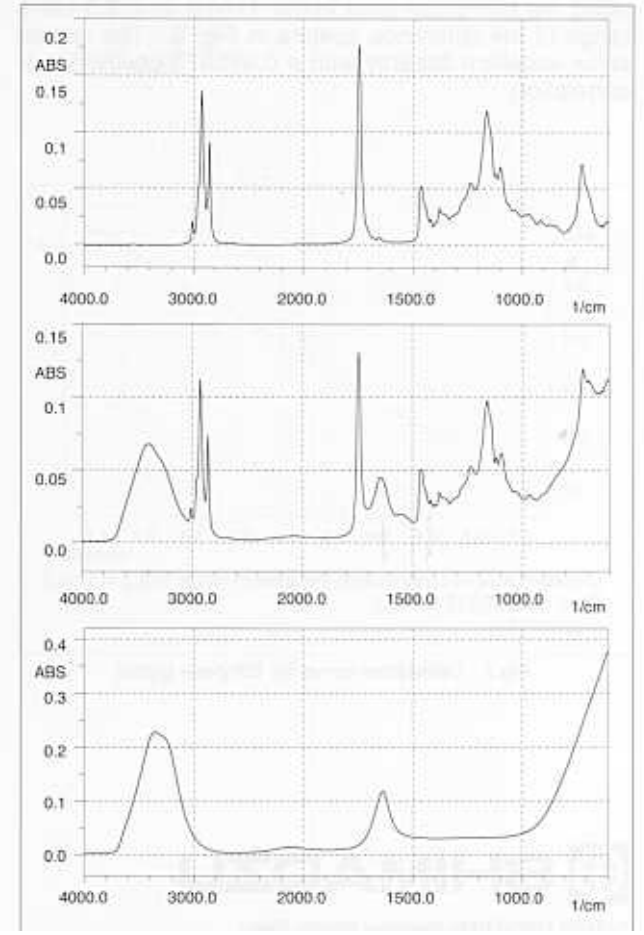

Fig.2 Spectra of vegetable oil, mayonnaise, and water (top to bottom)

■ Analysis of Lubricating Oil

Antioxidants and rustproofing agents are added to maintain the performance of mineral-oil-based lubricants over a long period. Their type and concentration differ according to application, grade, and manufacturer. A vacuum pump lubricating oil was compared with paraffin oil using a ZnSe prism. The analytical conditions are shown in Table 1. The results are shown in Fig. 3.

The concentration of additives is normally about 1%, resulting in extremely small absorption peaks. However, small peaks thought to be due to additives were confirmed in the range 1300 to 750cm-1.

Fig. 4 shows enlargements of the Fig. 3 spectra plus the spectrum of the antioxidant tris (2,4-di-tert-butylphenyl) phosphite. This analysis indicates that the lubricating oil contains a phosphite-based substance.

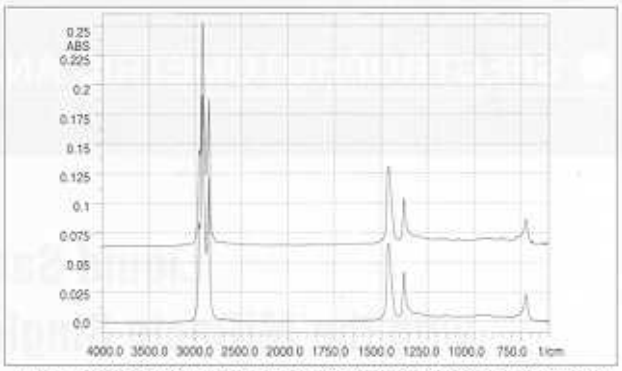

Fig.3 Spectra of lubricating oil (top) and paraffin oil (bottom)

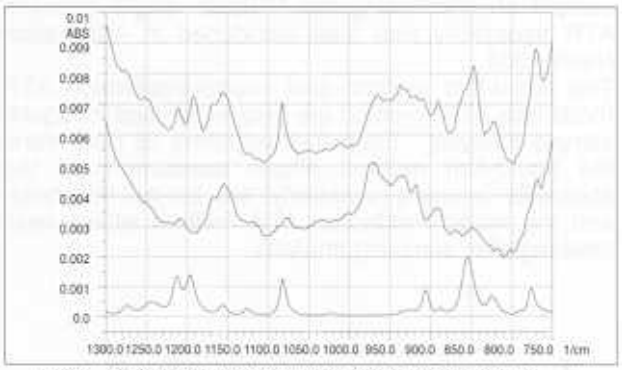

Fig.4 Spectra of lubricating oil, paraffin oil, and tris (2,4-di-tert-butylphenyl) phosphite (top to bottom)

■ Application of Quantitative Analysis

Like horizontal ATR, which is commonly used for the analysis of liquid samples, MIRacle can be used for quantitative analysis. Fig. 5 shows the analysis results for ethylene glycol solutions with 0%, 0.5%, 1%, 3%, 5%, and 10% concentrations. Fig. 6 shows the difference spectra of the analysis results for each solution concentration and the analysis results for pure water (0%). Analysis was conducted with a ZnSe prism and the analytical conditions shown in Table 1. The calibration curve shown in Fig. 7 was generated using the correction area in the 1124.3 to 975.3 cm-1 range of the difference spectra in Fig. 6. The results show excellent linearity with a 0.999978 coefficient of correlation.

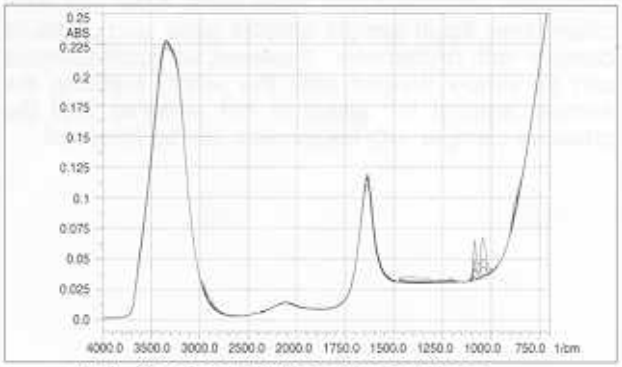

Fig.5 Spectra of ethylene glycol solutions (0.5%, 1%, 3%, 5%, and 10% concentration)

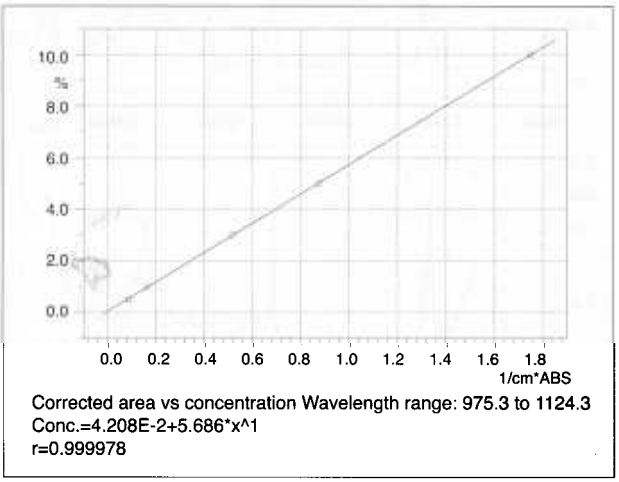

Fig.7 Calibration curve for Ethylene glycol

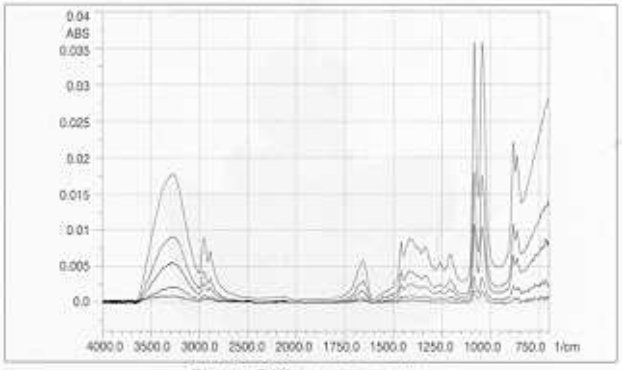

Fig.6 Difference spectra

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