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Application News

IRXross™ Fourier Transform Infrared Spectrophotometer

High Sensitivity Measurement of Ultra-Trace Amount of Silicone Oil by IRXross

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User Benefits

- IRXross enables measurement of trace samples dissolved in volatile solvents.
- It is also possible to measure samples with lower concentrations by using multiple reflection ATR.
- Although IRXross is a middle class FTIR, measurement of extremely weak peaks is also possible because its S/N ratio is on the same level as the high-end class.

■ Introduction

The IRXross, a new Fourier transform infrared spectrophotometer (FTIR) recently released by Shimadzu Corporation, has the highest sensitivity and resolution in its class, and also enables high speed scanning at a maximum of 20 spectra/sec. The IRXross also offers excellent operability supported by Shimadzu original analysis navigation program, IR Pilot™.
Focusing on the high sensitivity of the IRXross, this article introduces a quantitative analysis of a sample dissolved in a volatile solvent.
Fig. 1 shows the appearance of the instrument. To realize high performance, the IRXross uses the same stable interferometer as the IRTracer™-100. As a result, the S/N ratio is greatly improved in comparison with conventional devices, enabling measurement of extremely weak peaks.

Fig. 1 Appearance of IRXross

■ Transmission Measurement of Silicone Dissolved in n-Hexane

Oils are virtually insoluble in water, ethanol, and other polar solvents, and must be dissolved in a nonpolar solvent such as n-hexane or toluene. Using this property, it is possible to extract the oil adhering to a product using a nonpolar solvent and check the adhering oil by analysis by FTIR. However, qualitative/quantitative analysis of minute quantities of oil is difficult by the transmission method employing a liquid cell. Fig. 2 shows the data measured using a fixed thickness cell (window plate: KBr) with an optical path length of 0.1 mm for a sample prepared by dissolving 100 ppm of polydimethylsiloxane (PDMS), which is one type of silicone oil, in n-hexane (simultaneously showing the spectra for n-hexane and only silicone oil). The spectrum of the PDMS dissolved in n-hexane is almost identical with the peak for n-hexane, and no peaks originating from PDMS can be distinguished. Although not shown here, it was also impossible to detect peaks originating from PDMS by calculating the difference spectrum.

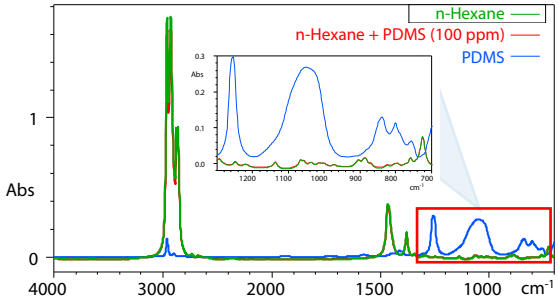

Fig. 2 PDMS in n-Hexane Measured Using Fixed Thickness Cell

■ Condensation ATR Method

This section introduces trace measurement using the condensation method. In the condensation method, as shown in Fig. 3, a small amount of a liquid solution is dripped on the ATR prism, and the solvent is evaporated. Since the solute remains on the prism, the target substance (solute) can be measured unaffected by the spectrum of the solvent. Quantitative analysis is also possible by maintaining a constant amount of drip.

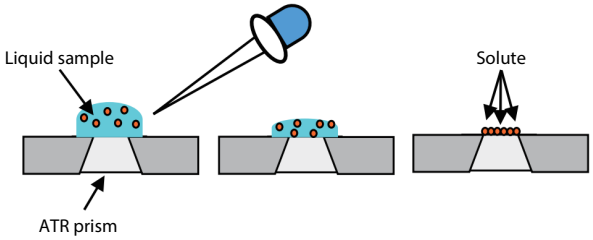

Fig. 3 Schematic Diagram of Condensation Method

■ ATR Measurement of Silicone Dissolved in n-Hexane

PDMS (100 ppm) dissolved in n-hexane, which is used in the above-mentioned transmission method, was measured using a 9-reflection multiple reflection ATR (attenuated total reflectance) accessory. Use of the 9-reflection ATR prism enabled measurement with higher sensitivity than with the widely-used single-reflection type. Fig. 4 shows the appearance of the ATR accessory, and Table 1 shows the measurement conditions. The solvent drip amount was set to 20 µL.

Table 1 Measurement Conditions

Instruments	: IRXross Fourier transform infrared spectrophotometer (KBr window plate) MicromATR (9-reflection diamond disk)
Resolution	: 4 cm^{-1}
Accumulation	: 100 times
Apodization function	: SqrTriangle
Detector	: DLATGS

Fig. 4 Appearance of MicromATR

PDMS dissolved at a concentration of 100 ppm in n-hexane was dripped on the ATR prism, and the ATR spectrum was measured. Fig. 5 and Fig. 6 show the measurement results. Here, the peaks around 2900 cm^{-1} and 1460 cm^{-1} originate from the n-hexane solvent. With the passage of time, the peaks of the n-hexane decrease, and peaks originating from PDMS can be seen at around 1260 cm^{-1} and 1100 cm^{-1}. Although it had been difficult to detect PDMS by the transmission method, it was possible to detect peaks originating from PDMS by evaporating the volatile solvent n-hexane.

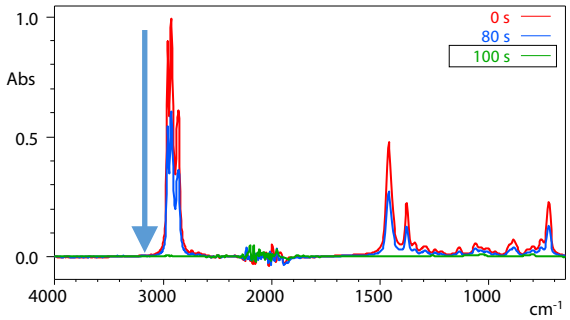

Fig. 5 Spectrum Change of PDMS (100 ppm) Dissolved in n-Hexane with Evaporation Time

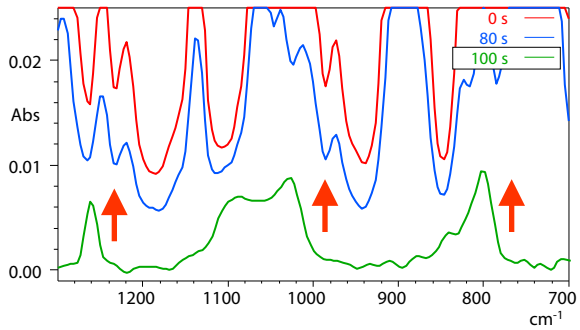

Fig. 6 Enlarged View of 1300 cm^{-1} to 700 cm^{-1} Region in Fig. 5

■ Quantitative Analysis of Low Concentration Silicone Dissolved in n-Hexane by ATR Method

20 µL of samples containing 1 ppm to 250 ppm of PDMS dissolved in n-hexane was dripped on the ATR prism, and the samples were measured after confirming evaporation of the n-hexane. Fig. 7 shows the measurement results. Peaks originating from PDMS could be obtained at all concentrations.

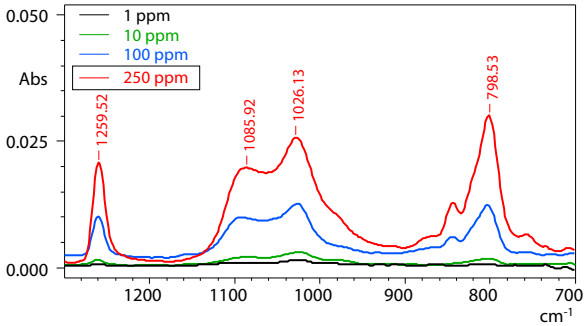

Fig. 7 Spectra of PDMS (1 ppm to 250 ppm) Dissolved in n-Hexane

A calibration curve was prepared using the intensity (corrected height) of the peak at 1260 cm^{-1}, which had a comparatively sharp shape unaffected by residual absorption due to n-hexane. Table 2 shows the corrected height at each concentration, and Fig. 8 shows the calibration curve prepared using these values. The results showed a coefficient of correlation of $r^2 = 0.999$ or more, demonstrating that quantitation is possible by the 9-reflection condensation ATR method, even with low concentration samples.

Table 2 Corrected Height of 1260 cm^{-1} Peak for Each Concentration

PDMS concentration (ppm)	Corrected height (Abs)
1	0.0002
10	0.0009
100	0.0079
250	0.0194

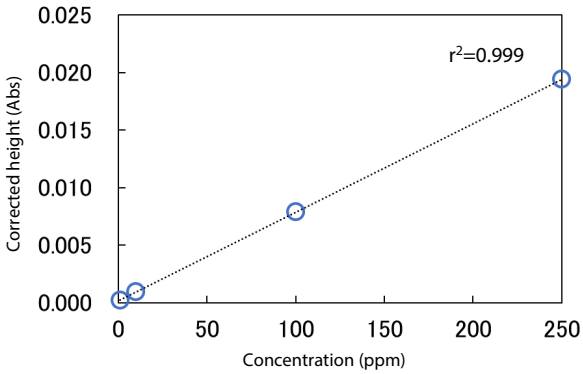

Fig. 8 Calibration Curve Obtained from Spectra of PDMS (1 ppm to 250 ppm) Dissolved in n-Hexane

Although this quantitative analysis was carried out by the 9-reflection ATR method, it should be noted that quantitation accuracy may be reduced by outflow of the dripped sample from the prism when using the single-reflection ATR method.

■ Conclusion

Low concentration PDMS dissolved in n-hexane was measured using the new IRXross FTIR. It is possible to measure the solute unaffected by the solvent by using condensation ATR method. Measurement of lower concentration samples is also possible if the multiple reflection ATR method is used.

Because the IRXross FTIR has the highest S/N ratio in its class, even extremely small peaks can be measured.

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