

Application News

No. A471

Spectrophotometric Analysis

High Resolution Analysis of Carbon Monoxide (CO) Gas

■ Introduction

FTIR is being used for analysis of gases in various industries, such as the gas manufacturing industry where it is used for production management, and for gas monitoring in such fields as chemical manufacturing and semiconductor manufacturing, etc. When conducting gas analysis by FTIR, the resolution and gas cell path length must be selected depending on the concentration and peak shape of the gas component of interest. Here, using the IRTracer-100, we introduce an example of high resolution measurement of the low-molecular weight gas, carbon monoxide (CO).

■ Advantage of High Resolution

To compare spectral differences due to resolution, we measured the infrared spectra of water vapor at resolutions of 0.25 cm⁻¹, 0.5 cm⁻¹ and 1 cm⁻¹, respectively. The analytical conditions used are shown in Table 1. Fig. 1 shows an enlarged view of the infrared spectra in the vicinity of 1620 cm⁻¹.

As can be seen here, the peaks that were not separated at 0.5 cm⁻¹ and 1 cm⁻¹ were clearly separated when measured with a resolution of 0.25 cm⁻¹.

By conducting measurement at high resolution, the spectral peak intensity is noticeably increased, making it easier to distinguish between two peaks that are closely adjacent to one another. This is especially useful in component identification in cases where the mixed gas spectra include similarly shaped, overlapping peaks.

Further, since the lower the molecular weight of the gas, the more pronounced the rotation and vibration of the spectrum, it is more effective to conduct measurement of low-molecular weight gases at high resolution.

Table 1 Instrument and Analytical Conditions

Instrument	: IRTracer-100
Resolution	: 0.25, 0.5, 1.0 cm ⁻¹
Accumulation	: 30
Apodization	: Happ-Genzel
Detector	: DLATGS
Accessories	: Gas Cell (10 cm) / NaCl Window

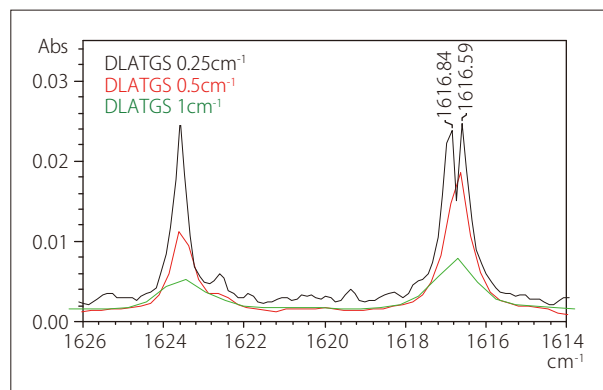


Fig. 1 Overlaid Spectra of Water Vapor Measured at Different Resolutions

■ Quantitation of Carbon Monoxide (CO) Gas

Using CO as an example of a low-molecular weight gas, we measured the spectra of CO gas samples at different concentrations (95 ppm, 191 ppm, 1207 ppm, 2415 ppm), and then generated a calibration curve. In this case, the resolution of 0.25 cm⁻¹ was used. The other analytical conditions were as shown in Table 1. For the calibration curve, we used the height of the peak in the vicinity of 2170 cm⁻¹, and primary linear calculations were conducted using the multi-point calibration curve method.

Fig. 2 shows the overlaid spectra of the CO gasses at their respective concentrations, and Fig. 3 shows the calibration curve. The correlation coefficient $r = 0.999$ for the calibration curve indicated excellent linearity.

Use of a 10 cm gas cell in the quantitation of carbon monoxide gas using a resolution of 0.25 cm⁻¹ and a DLATGS detector makes quantitation possible over a wide range of concentrations, from tens to thousands of ppm. When the 0.5 cm⁻¹ resolution is used, the lower limit of quantitation will increase due to the lower peak intensity, assuming that the noise remains constant. In actuality, the resolution and number of accumulations must be determined while monitoring the state of the obtained spectrum in order to obtain the optimum lower limit of quantitation.

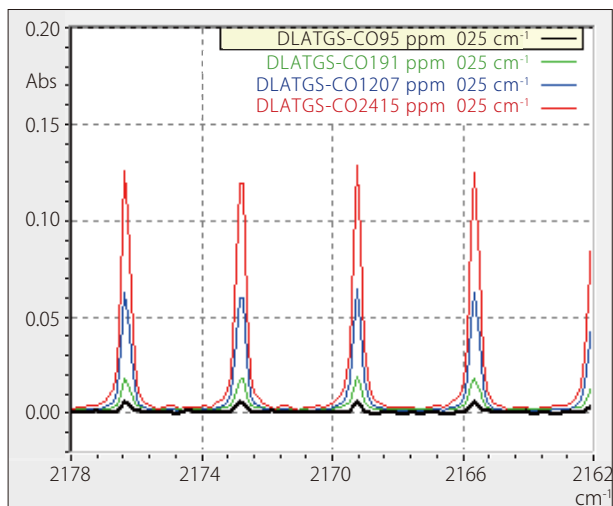


Fig. 2 Overlaid Spectra of CO at Four Concentrations (95, 191, 1207, 2415 ppm)

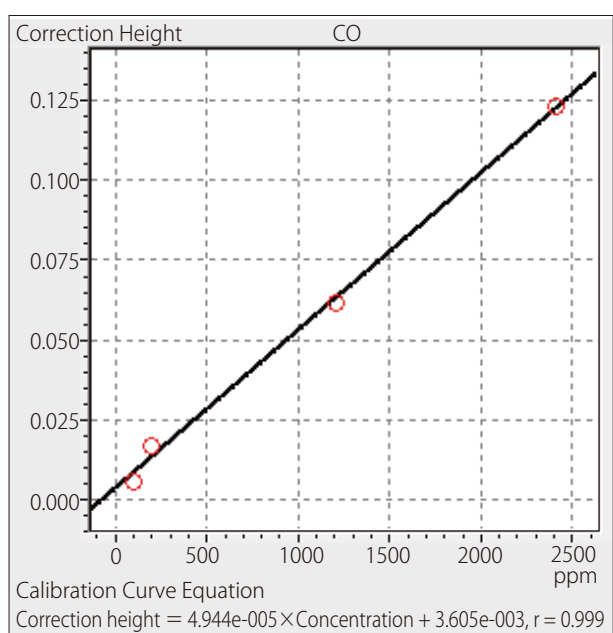


Fig. 3 Calibration Curve of CO Using Four Concentrations

■ Advantage of Optional MCT Detector

The standard detector for the IRTracer-100 is the DLATGS detector, which provides stable detection over a wide wave number range. However, for detection with higher sensitivity, the liquid nitrogen-cooled MCT detector is available as an option.

In measurement at high resolution, the aperture between the interferometer and the light source must be reduced to increase the parallelism of incident light on the interferometer. This results in a reduction in the amount of light reaching the detector. Further, in analysis of a low-concentration gas, there are cases in which a long optical path length cell is required, but as the optical path length increases, the amount of light in

the cell is reduced. Therefore, when using a gas cell with a long optical path length for high-resolution measurement, a better spectrum can be obtained with an MCT detector, which can provide high sensitivity detection with low noise.

Fig. 4 shows overlaid spectra of CO gas measured with an MCT detector and a DLATGS detector. Measurement of the 2415 ppm CO gas was conducted using a resolution of 0.25 cm⁻¹, and a gas cell having an optical path length of 10 cm. The red-trace spectrum was obtained using the DLATGS detector, while the black-trace spectrum was measured with the MCT detector. Due to the high sensitivity of the MCT detector, the light intensity used for this measurement was reduced. However, when compared with the DLATGS detector under the same conditions, a spectrum with less noise was obtained using the MCT detector. Another advantage of the MCT detector is the faster mirror speed compared to that of the DLATGS detector, due to its higher optimum frequency response, which permits a shorter measurement time.

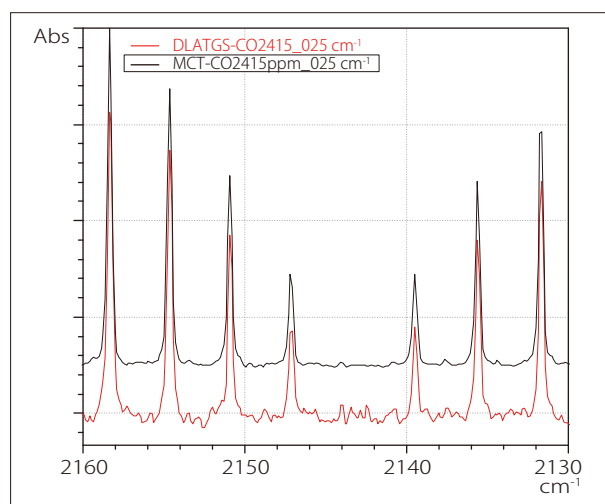


Fig. 4 Spectra of CO Measured with a DLATGS Detector and an MCT Detector

■ Summary

Here, using analysis of CO gas as an example of low-molecular weight gas analysis, we introduced the excellent performance of the IRTracer-100, with its high 0.25 cm⁻¹ resolution and quantitative performance, as well as the advantages of using the optional MCT detector.

Using the IRTracer-100 with the LabSolutions IR software offers stable measurement and simple quantitative operations.

As always, when conducting gas analysis by FTIR, it is necessary to carefully consider various conditions, including the detection limit, corrosiveness, coexisting substances, etc. Please contact your Shimadzu representative for information such as cell selection, etc.