

Gas Analysis System using Full Vacuum Type FT/IR (Trace amount of H₂O in N₂ gas)

<Introduction>

The analysis of low concentration gases using long pathlength gas cells has been reported previously, however, it can be difficult to accurately quantitate gases at low concentrations when the absorption peaks of the target gas overlap with the peaks of atmospheric water vapor or, when the target gas itself is H₂O or CO₂, which are present in the atmosphere. This is because even with a vacuum FT-IR instrument, it was not possible to evacuate the sample chamber with the long pathlength cell present. The full vacuum gas analysis system introduced in this application note has a special gas cell integrated into the sample chamber, allowing the entire light path to remain under full vacuum with the gas cell in place. This full vacuum model makes it possible to quantitate concentrations of H₂O even at the 0.2 ppm level.

<Instrument and Measurement>

For the measurement, there are separate vacuum lines for evacuation of the FT-IR instrument and the gas cell, with independent evacuation capability for the separate components. Adding a vacuum gauge to the gas cell makes it possible to control extremely low concentrations of the gas samples.

<Cell specification>

Cell type:	Multi-pass 'White' cell
Pathlength :	10 meters
Cell body:	Stainless steel
Cell inner surface:	Electrochemically polished
Mirror material:	Stainless steel
Mirror surface:	Gold coated
O-ring :	Viton
Window:	CaF ₂
Heating:	Possible, max. 100°C
Cell capacity:	Approx. 2 L
Gas in/output port:	1/4 inch VCR



Figure 1. Full vacuum type FT-IR gas analysis system (10 meter cell)

<Measurement example>

The quantitation of a trace amount of water vapor (H₂O) within CO₂ in gas cylinder was attempted. Water vapor with a concentration of 15.0 ppm was diluted by monitoring the pressure gauge during dilution of the standard samples with concentrations of 1.5, 3.0, 4.5, 6.0, 7.6 and 9.12 ppm. Figure 2 shows the IR spectra of water vapor for each concentration. The lowest detection limit calculated from Signal to Noise ratio was around 0.2 - 0.3 ppm. The calibration curve in Figure 3 was created using the absorption peak at 1734 cm⁻¹. As seen, the precise quantitation of low concentration can be done in this system.

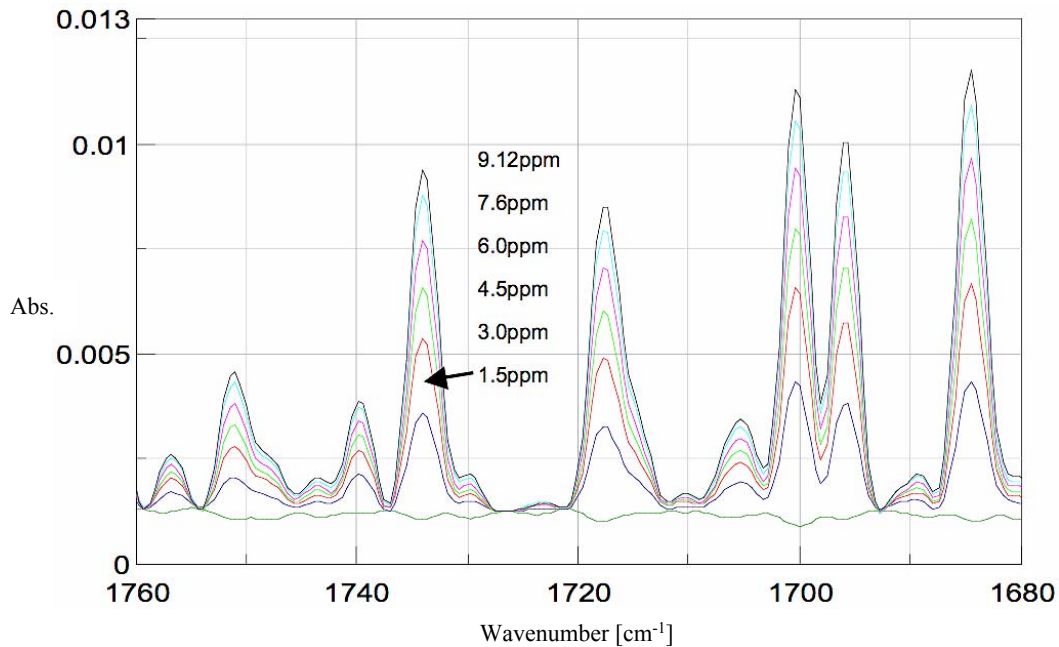


Figure 2. Expanded region for spectra of low concentration H₂O

<Condition>

Instrument:	FT/IR-6300
Accumulations :	100
Resolution :	2 cm ⁻¹
Zero filling:	ON
Apodization:	Cosine
Gain:	Auto (1)
Aperture:	1.8 mm
Scan speed:	Auto (4 mm/sec.)
Light source:	Standard
Detector:	MCT

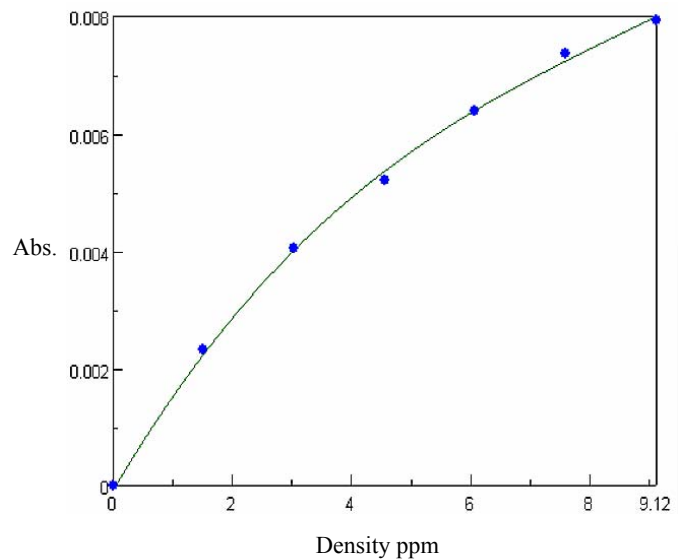


Figure 3. H₂O calibration curve