

Full Vacuum FTIR Spectrometer for Quantitative Trace Gas Analysis (H₂O in N₂ Gas)

Introduction

The analysis of low concentration gases using long path length 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 FTIR spectrometer, it was not possible to evacuate the sample chamber with the long path length cell present. The FT/IR-6000 Series 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-6800FV and the gas cell, with independent evacuation of the separate compartments. 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 amounts of water vapor (H₂O) in CO₂ in a 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 the Signal to Noise ratio was approx. 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 made with this system.

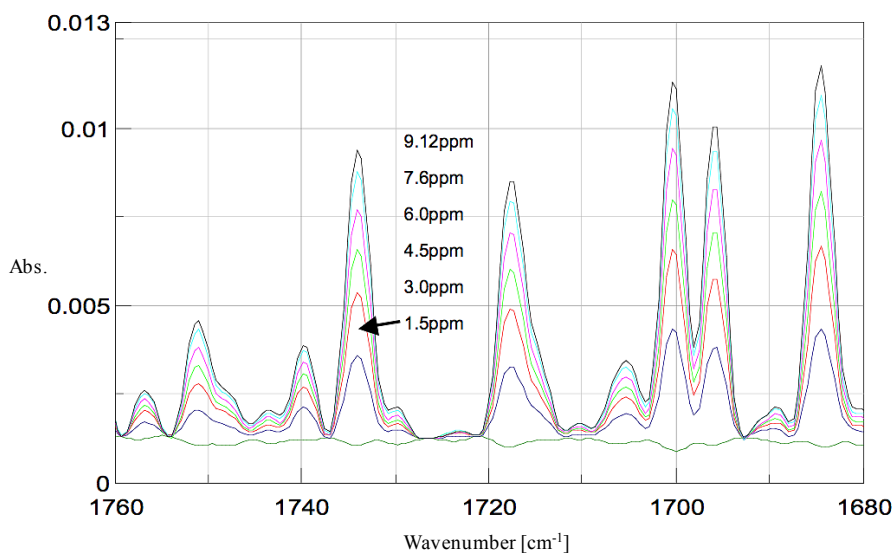


Figure 2. Expanded region of spectra with low concentration H₂O

Condition

Instrument: FT/IR-6800FV
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

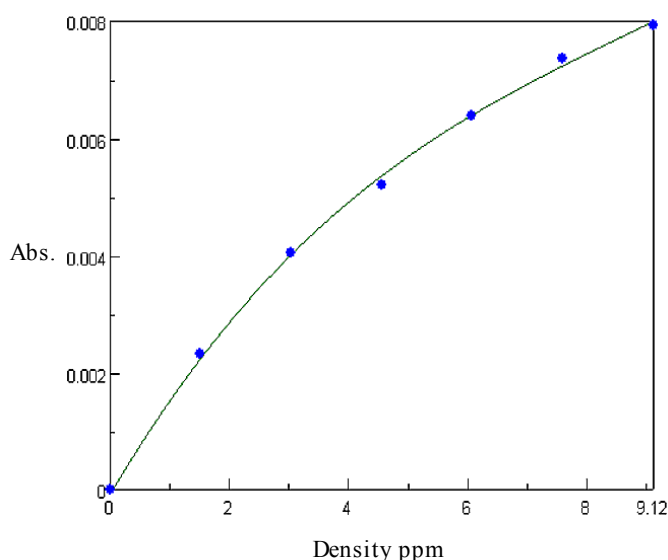


Figure 3. H₂O calibration curve