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Use of Short Path Cell FTIR for High-Speed, Near Real-Time Analysis of Gases

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Introduction

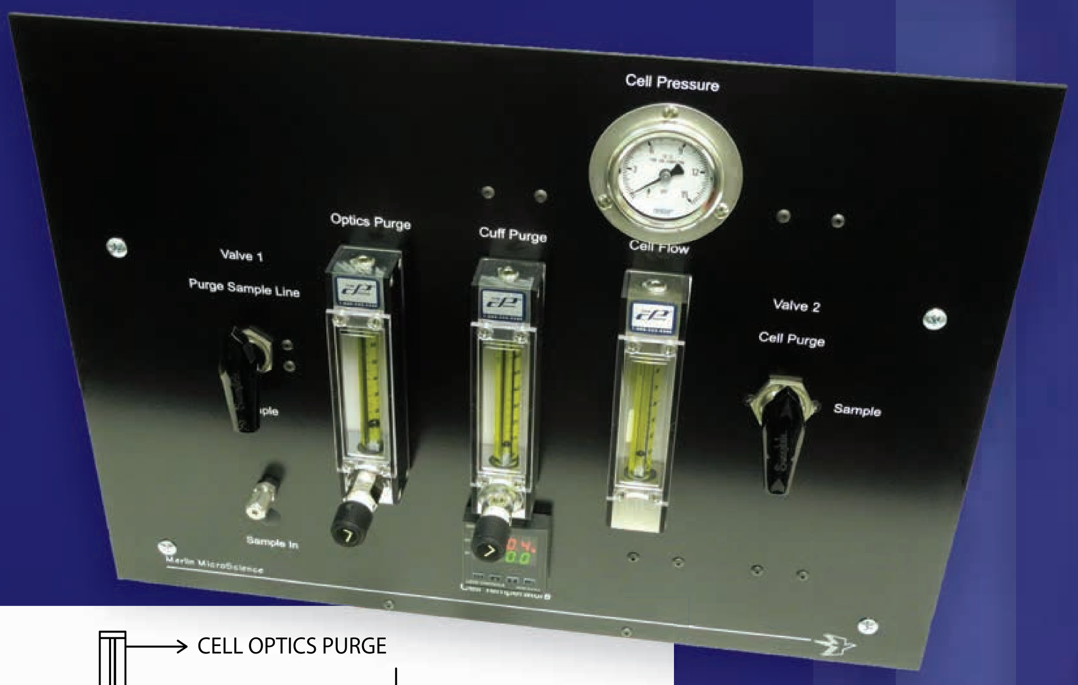
The FTIR (Fourier transform infrared) spectrometer is a powerful tool for the analysis of trace impurities in high purity gases. In both benign and aggressive matrices, FTIR spectroscopy is often the best or only choice for the detection of impurities like moisture (water), acids such as hydrogen chloride, hydrogen fluoride, hydrogen bromide, and other species not amenable to chromatography.

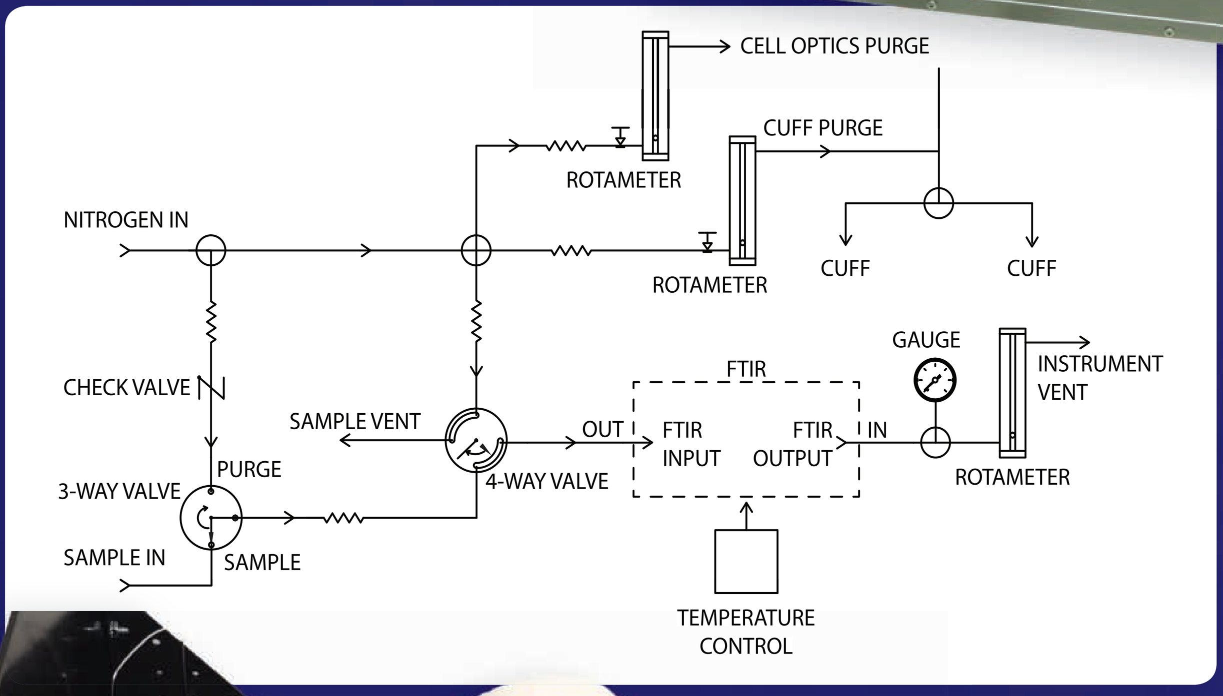
In addition, the FTIR spectrometer's ability to analyze gas mixtures that would require complex or multiple chromatographic techniques is often more convenient. Simply changing the instrument's gas cell is all that is required to cover a wide range of analyses.

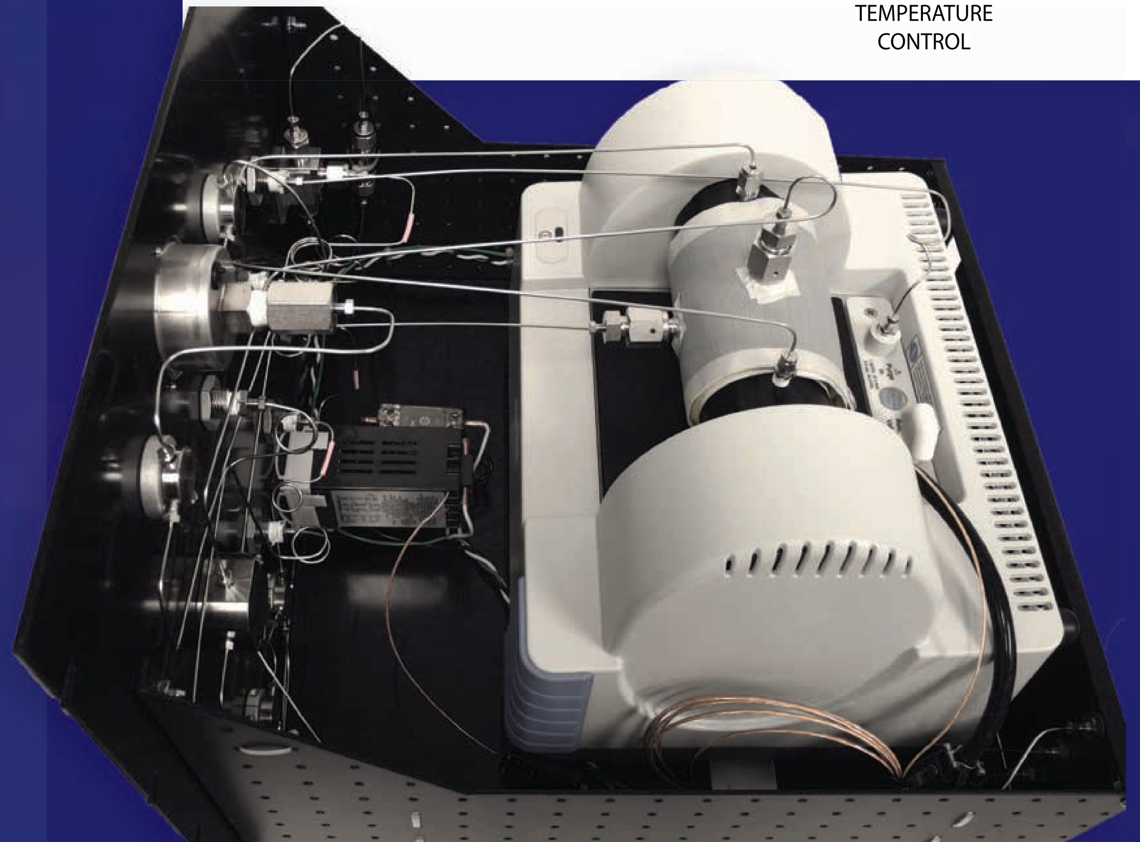
Speed and the ability to monitor results on a continuing and near real time basis is an advantage over conventional chromatographic solutions. We have been able to develop this with our software macros.

Hardware

These figures illustrate the flow path, plumbing, and panel arrangement for the presentation of gas samples to a Thermo iS5 FTIR spectrometer utilizing cells of varying lengths (1 cm, 10 cm, or 6 m) in the sample compartment. Because most FTIRs are not dedicated to gas analysis, we have developed this flow path and panel configurations below. Naturally, materials of construction and options will vary according to the specific needs. For example; laboratory use, process use, type of gas, etc.



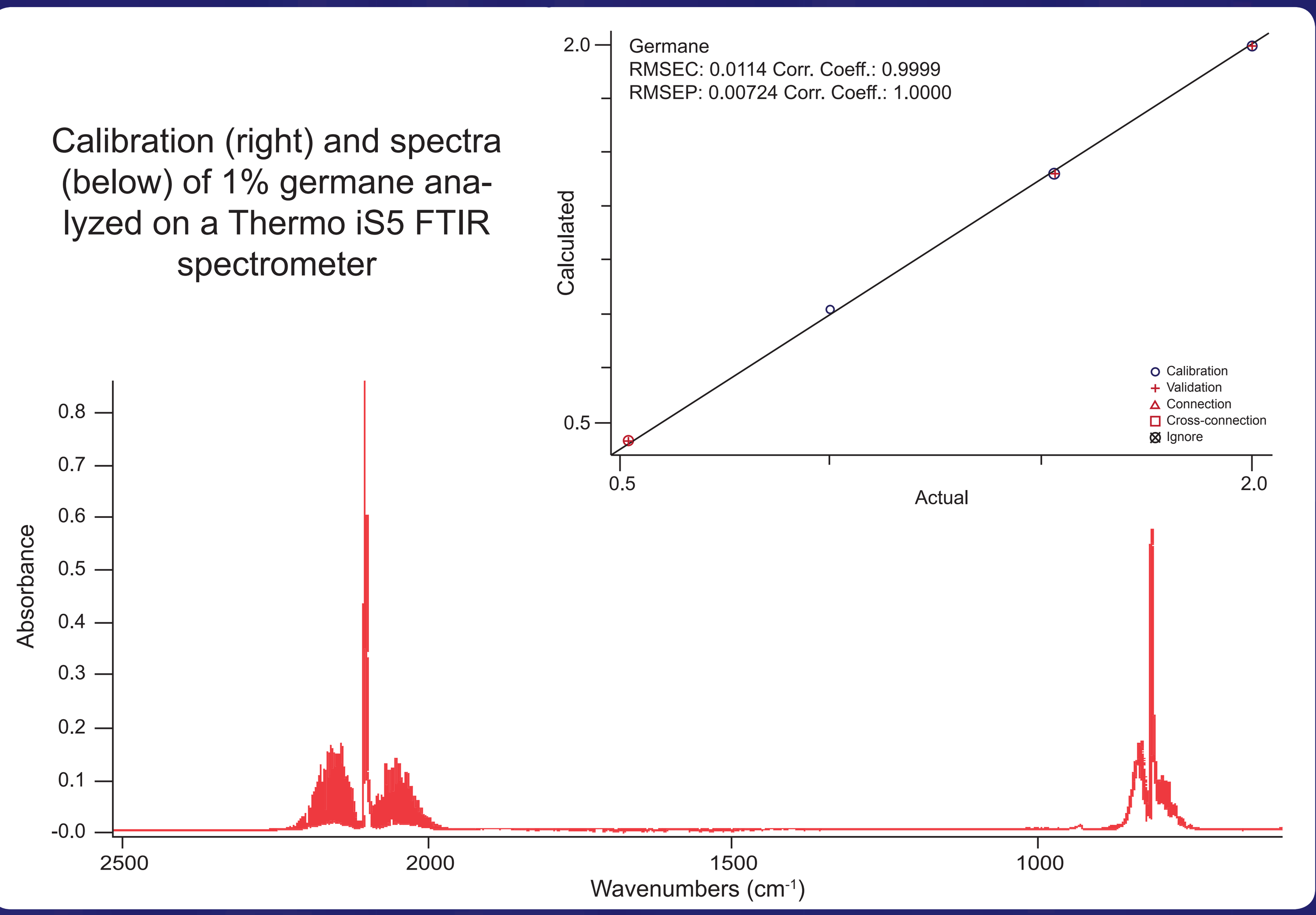




Calibration

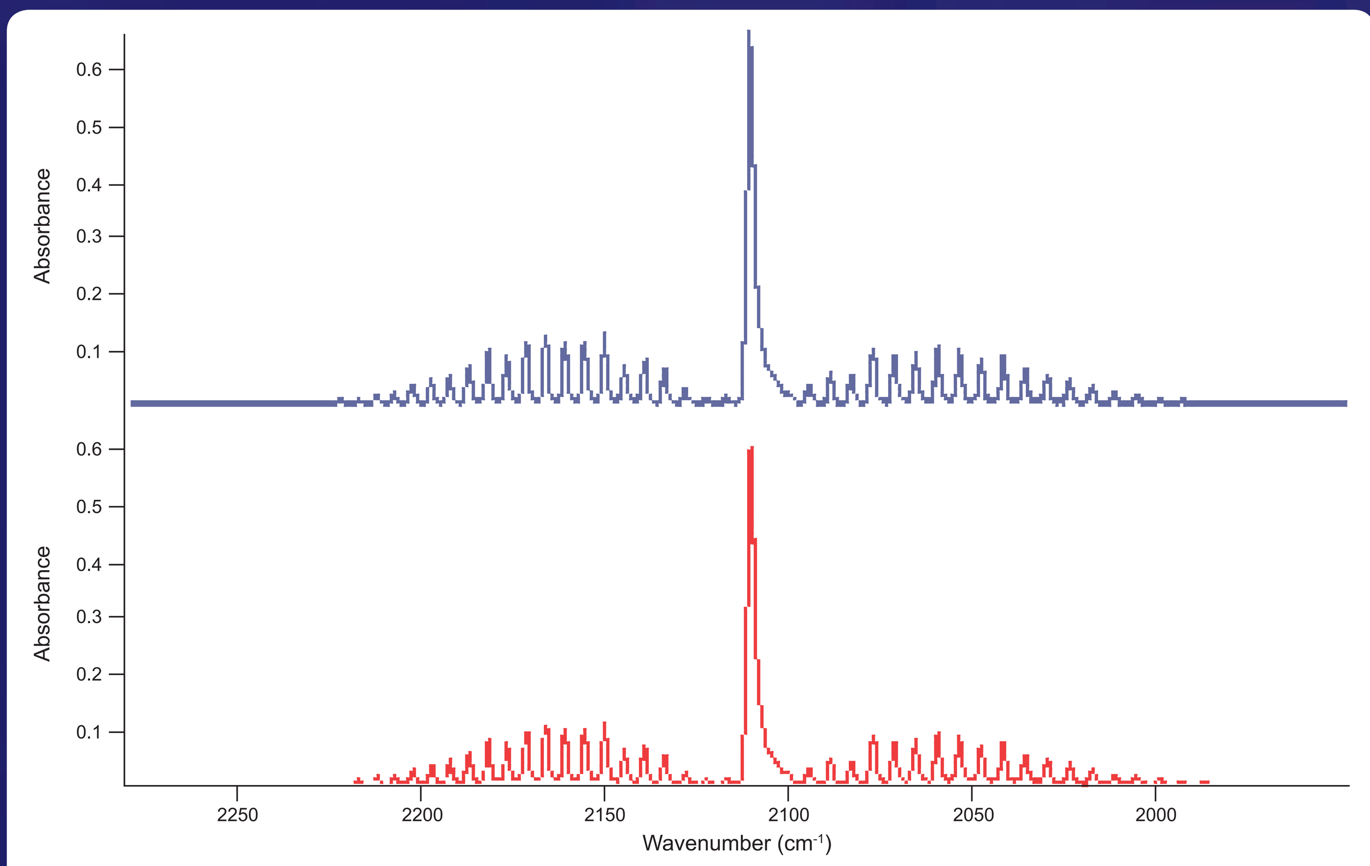
One of the most reliable features of optical spectroscopy is that once a calibration is made at a known/controlled temperature and path length, that calibration is forever valid (notwithstanding any defects in the instrument). Because FTIR spectroscopy is an optical technique, it obeys the Beer-Lambert law which relates the absorption of light to the properties of the material and distance through which the light is travelling. This law means that aside from running an occasional performance or quality control check, today's calibration will be accurate ten years from now, and it can be used indefinitely.

This feature of the FTIR spectrometer can be utilized overseas where calibration standards may be expensive and or difficult to acquire. For example, an instrument to be used in the manufacture and packaging of 1.00 vol % germane in hydrogen where accurate standards of germane are unavailable in the destination country could use a calibration made on the instrument before it is shipped overseas which because of the Beer-Lambert law, will be valid on its arrival allowing it to provide the control for precision packaging required.



Calibration (right) and spectra (below) of 1% germane analyzed on a Thermo iS5 FTIR spectrometer

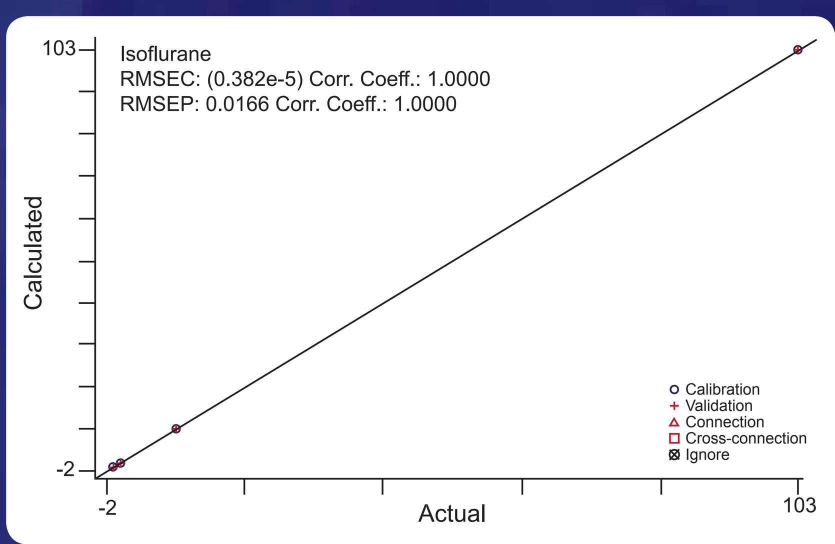
This feature of the FTIR spectrometer also allows us to extrapolate calibrations at higher concentrations by simply using a longer path cell. In the example above, calibrations for 10% and 20% concentrations which would ordinarily be difficult and expensive to acquire are easily created using a 10 cm path length cell. In addition, the new calibrations can also be made remotely in the same way as the original calibration. The tools required to do this are an instrument similar to the remote instrument (in this case, a Thermo Avatar 360), 1 cm and 10 cm path cells, and 1% and 2% germane standards. Both standards when analyzed on the Avatar 360 using a 1 cm short path cell result in a spectral shape and absorbance value identical to

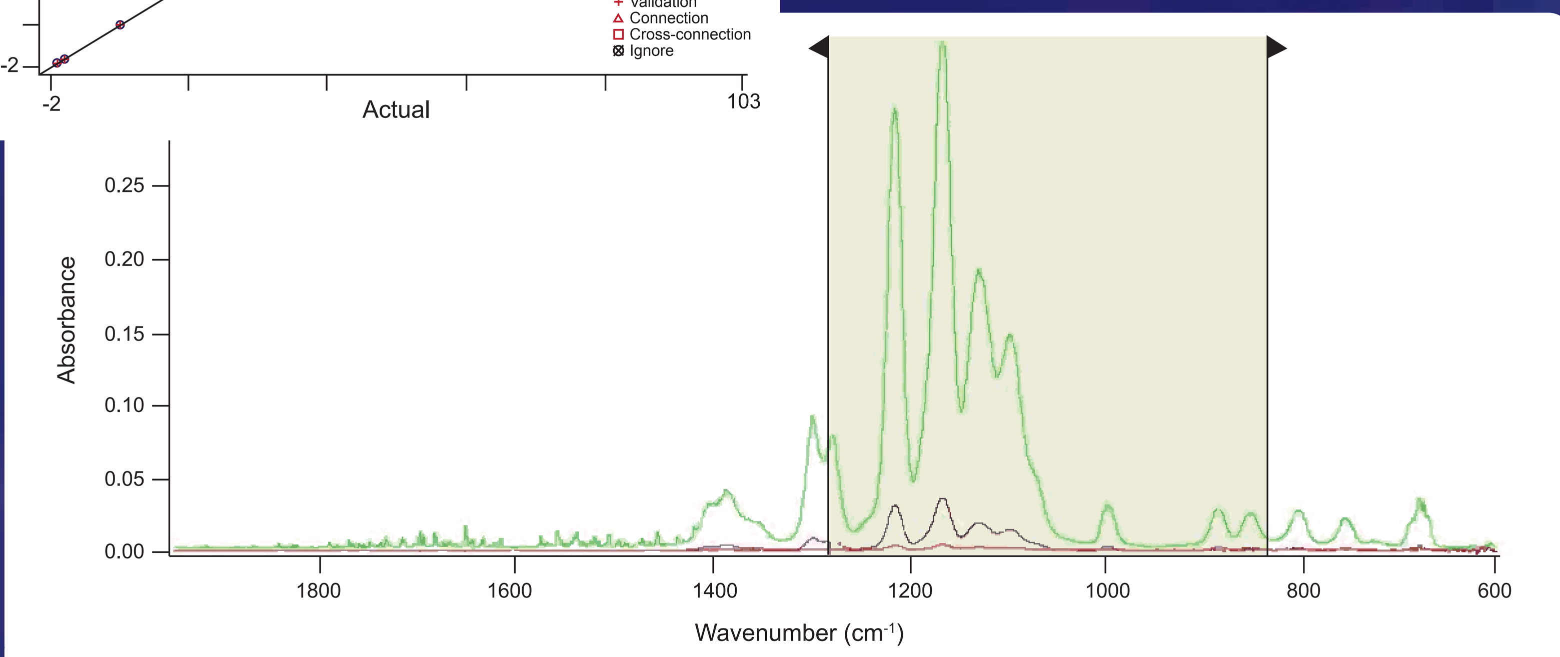


Spectra of 1% germane calibrations using a 1 cm path length cell on a Thermo iS5 FTIR spectrometer (top) and a Thermo Avatar 360 FTIR spectrometer (bottom) that of the remote instrument (Thermo iS5).

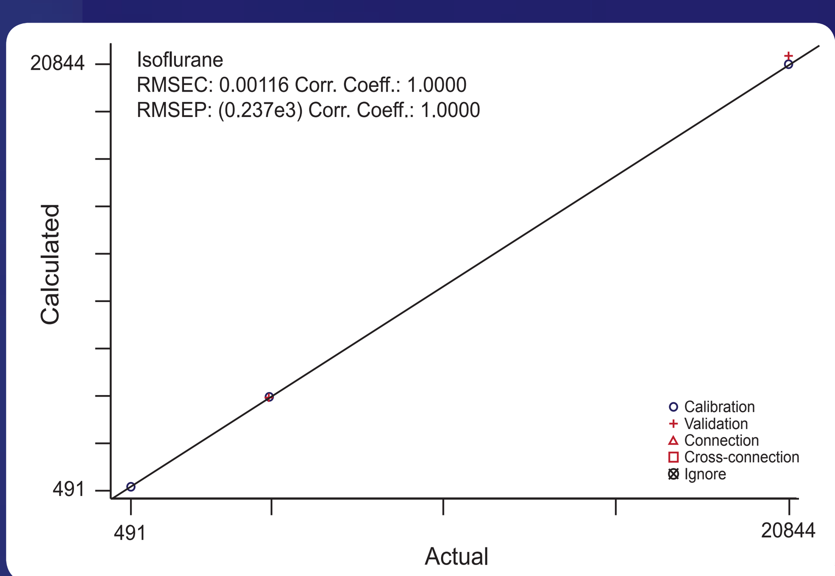
Therefore, the 1% and 2% standards on a longer 10 cm path cell become 10% and 20% virtual calibrations on the 1 cm path cell. Using path cells of varying lengths (1 cm, 10 cm, 50 cm, 2 m, and 10 m) to make calibrations is a robust technique when limited by the availability of calibration gasses.

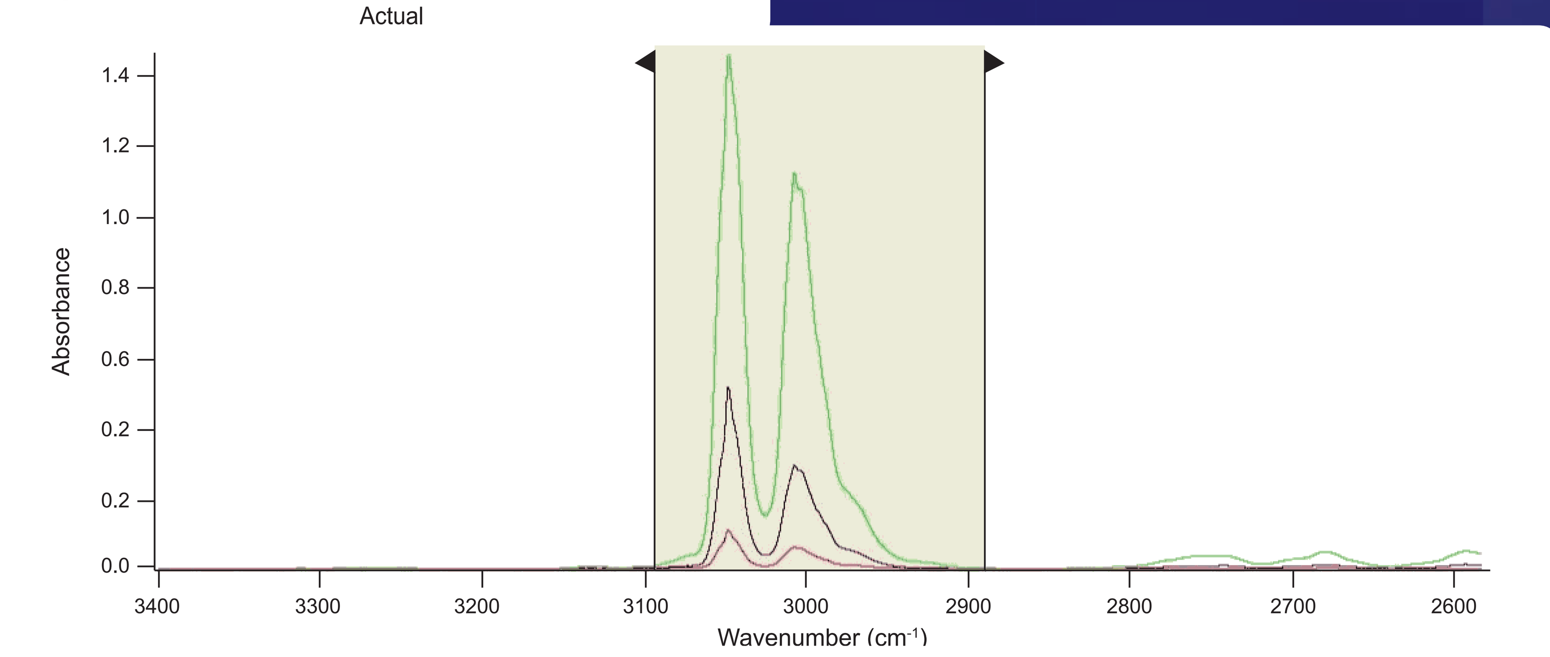
A broad range of calibrations can be made by using different wavelength regions. Low and high level calibrations of isoflurane, an anesthetic, demonstrate this range (below).





Low level calibration of isoflurane

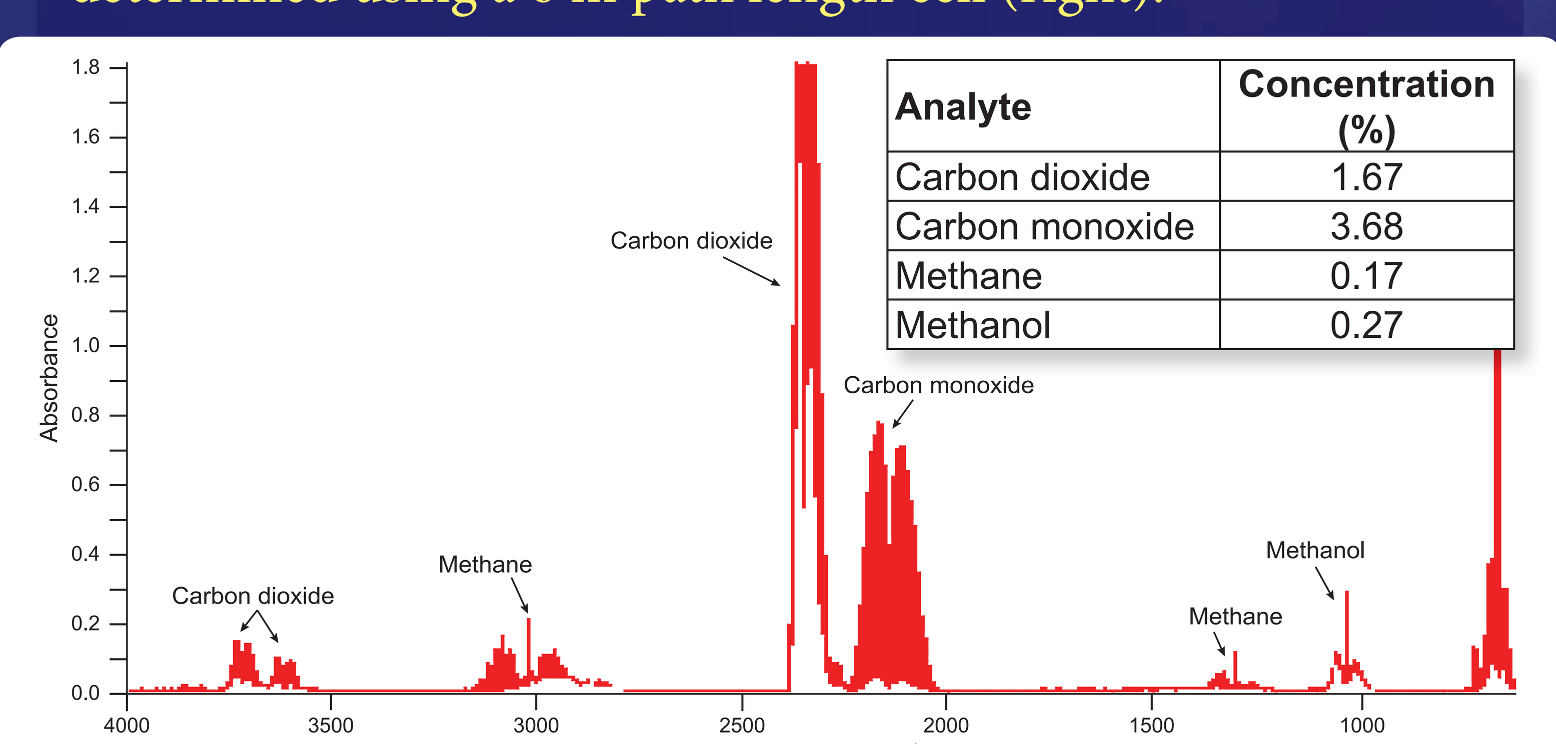




High level calibration of isoflurane

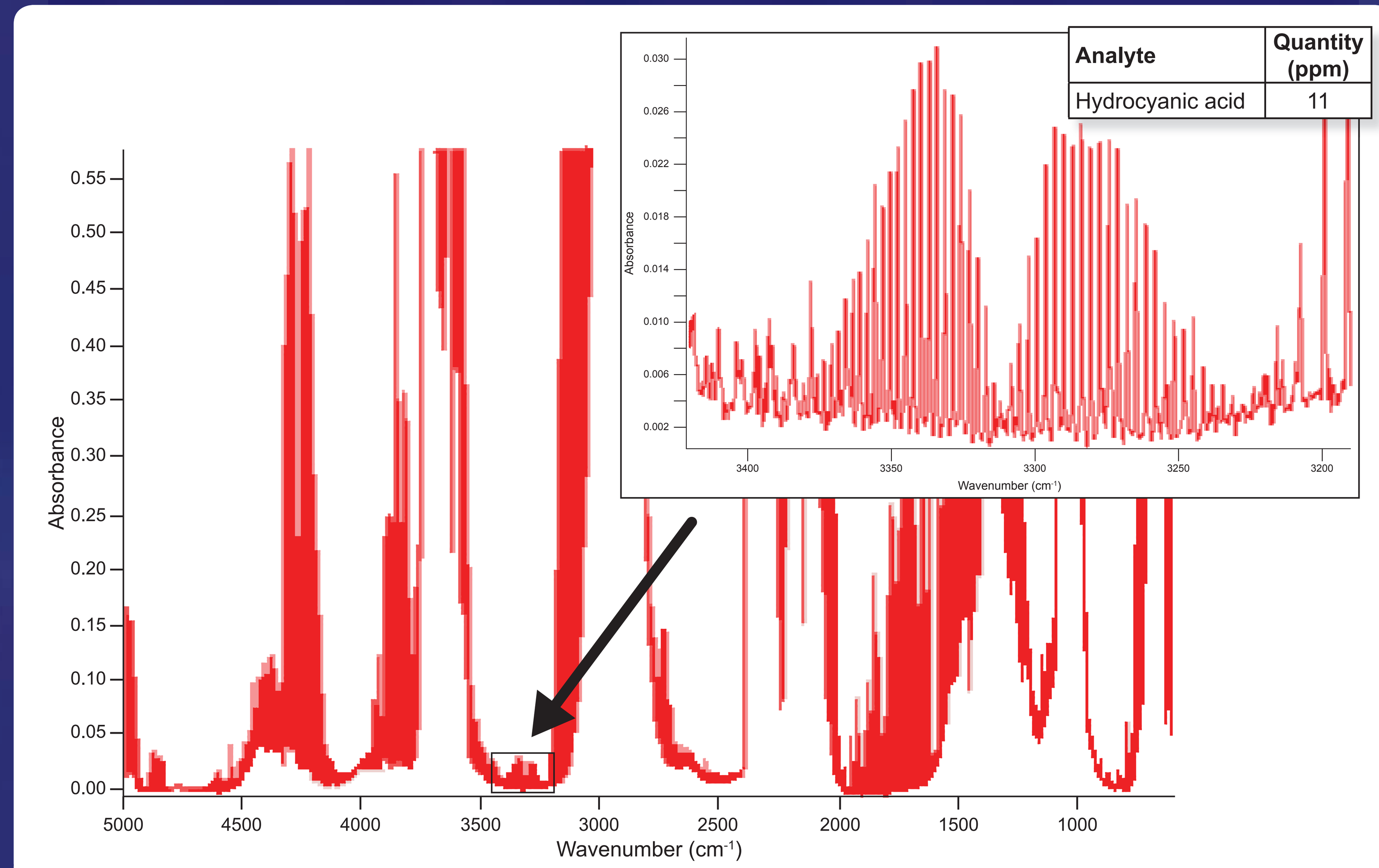
FTIR Spectroscopy vs GC

There is little debate that gas chromatography (GC) is one of the most powerful tools for the analysis of materials amenable to it. However, it can be slow and cumbersome in some cases. A good example is the measurement of methanol, carbon dioxide, carbon monoxide, methane, and hydrocyanic acid (HCN) in a balance stream of hydrogen. To measure the carbon monoxide and carbon dioxide requires two different GCs or a complex valve arrangement, as well as a separate analysis to measure the methanol, and yet another to determine the HCN content. This is more easily accomplished using FTIR spectroscopy and two different path length cells: High level compounds are measured using a 10 cm path length cell (below), and HCN is determined using a 6 m path length cell (right).



Analyte	Concentration (%)
Carbon dioxide	1.67
Carbon monoxide	3.68
Methane	0.17
Methanol	0.27

Spectrum of carbon dioxide, carbon monoxide, methane, methanol in a balance stream of hydrogen analyzed by FTIR spectroscopy with a 10 cm path length cell

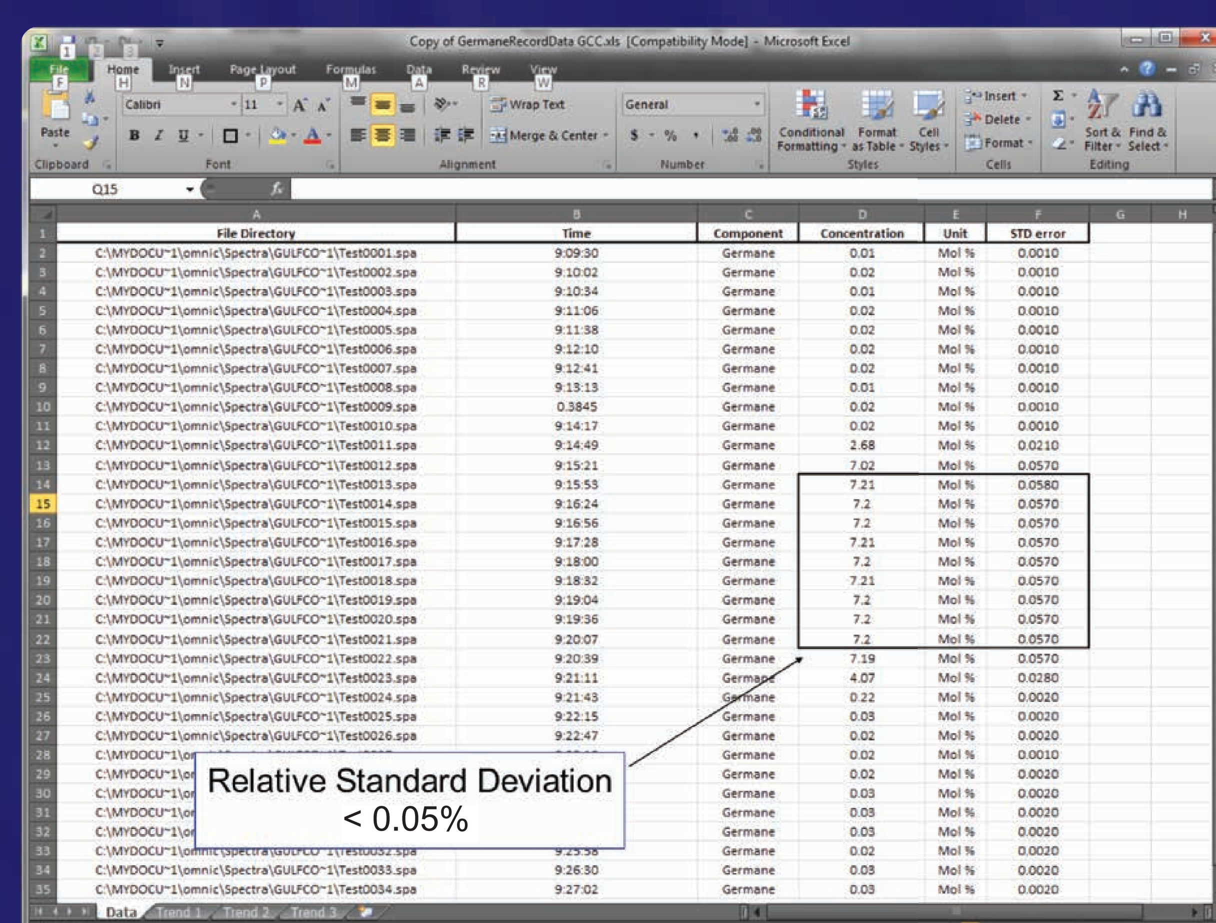


Analyte	Quantity (ppm)
Hydrocyanic acid	11

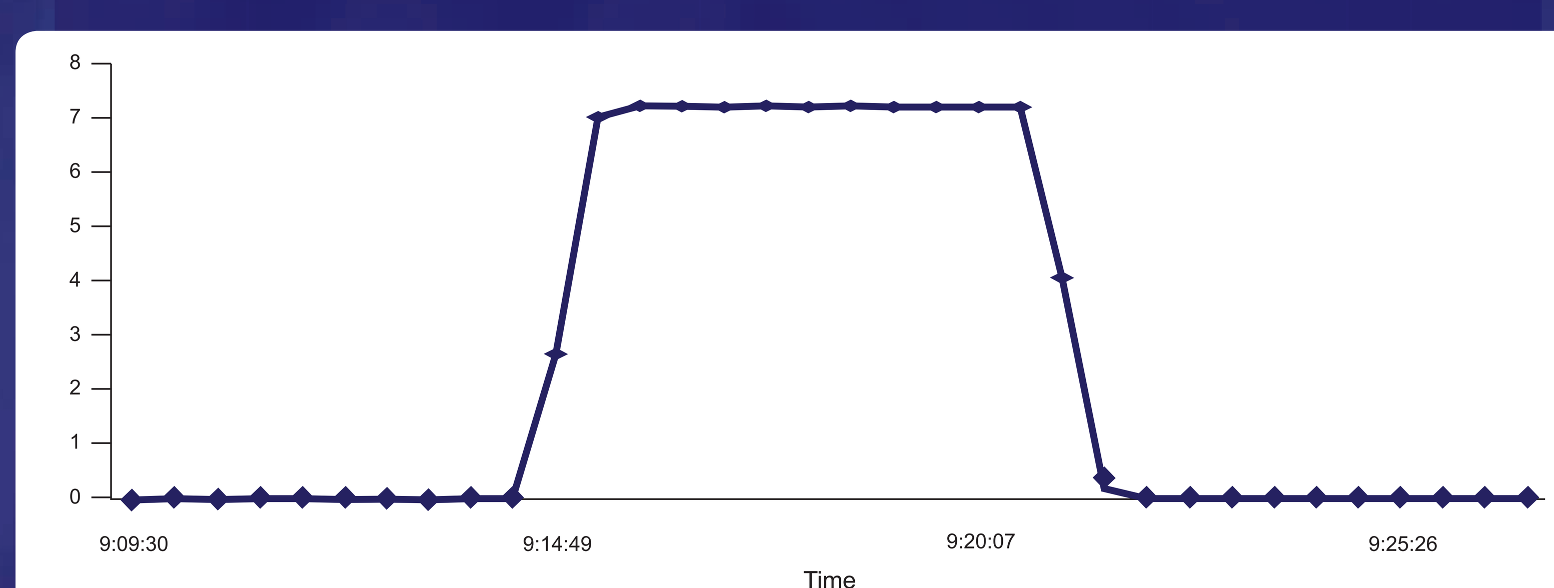
Spectrum of hydrocyanic acid in a balance stream of methane, carbon monoxide, and carbon dioxide analyzed by FTIR spectroscopy with a 6 m path length cell

Analytical speed and precision, whether for laboratory analysis or process control, are always important factors in technique choice. Macros embedded in the control software allow observation of the data quality on a near real time basis, and the results can be immediately observed and written to the computer screen or a spreadsheet file. Each scan can be collected or dumped at the user's discretion. The precision and sample turnover speed on a 10 cm cell at a flow rate of 100 mls/min and the corresponding data are shown below.

In just over a minute, a stable reading is reached. Over approximately a five minute period, nine replicate analyses can be made creating sufficient population for a legitimate statistical evaluation.



Concentration and standard deviation in the analysis of germane in hydrogen



Plot of the above standard deviation over time representing the precision and sample turnover speed in the analysis of germane in hydrogen by FTIR on a 10 cm path length cell at a flow rate of 100 mls/min

Conclusion

Due to its speed, accuracy, stability, and ability to measure many different types of gases with a single instrument and multiple path length cells, FTIR spectroscopy has replaced gas chromatographic methods for many analyses in our laboratory.

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