NEAR-IR DIODE LASER-BASED SENSOR
FOR PPB-LEVEL WATER VAPOR IN INDUSTRIAL GASES

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Near-IR diode laser-based sensor for ppb-level water vapor in industrial gases

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ABSTRACT

The verification of low water vapor impurity levels in semiconductor manufacturing feed gas supplies is becoming critically important for the development of advanced electronic devices. Ammonia is one of the important precursor gases for electronic manufacturing. In this paper we present data from a water vapor absorption spectroscopy sensor designed to continuously measure ppb water impurities in pure ammonia gas with a 1 Hz bandwidth. The sensor is built using a near-IR diode laser, commercial fiber optic components, room-temperature InGaAs photodiodes, an ultra-sensitive balanced ratiometric detection circuit, and a modified commercially available multipass cell. We present water vapor collisional broadening data by ammonia used to determine the optimal operating pressure for maximum system sensitivity. The commercial multipass cell was modified for ease of alignment, a nearly continuously variable pathlength, and to minimize the atmospheric air pathlength outside of the cell. The computer controlled sensor is applicable to making water impurity measurements in a number of additional commercially important gases such as hydrogen chloride, hydrogen fluoroide, hydrogen bromide, silane, etc. The sensor is also applicable to moisture measurements in natural gas, and manufacturing dryer applications such as those found in the plastics industry or the pharmaceutical industry where in-line process control is critical.

Keywords: diode laser, absorption spectroscopy, water vapor, multipass cell, semiconductor gases

1. INTRODUCTION

Accurate water concentration measurements are becoming more important in a number of industries as technological advances begin to push the limits of advanced material applications, manufacturing techniques, and structure designs. Water is often deleterious to a manufacturing process limiting material yields or important chemical processes. These limitations are particularly important in the semiconductor manufacturing community. Many manufacturers are now requiring that gas suppliers certify the water levels within their commercial gas product deliveries. Those suppliers who are able to deliver these certified commercial gas supplies will enjoy a competitive advantage over their rivals and gain additional market share. We believe the instrument described below is an important step in the development of this certification process. For inert gas species such as nitrogen, helium and argon, there are a number of potential measurement techniques that may be used to measure water concentrations down to the parts-per-billion (ppb) level. These techniques include chilled-mirror hygrometers, quartz crystal microbalances (QCM), capacitance hygrometers, atmospheric pressure ionization mass spectroscopy (API-MS) and surface acoustic wave (SAW) micro sensors. Nearly all of these techniques are incompatible with moisture measurements in environments that contain reacting species such as ammonia, HCl and HF. Optical sensors based upon tunable diode laser (TDL) absorption spectroscopy can fill this gap.

In years past TDL sensors were based upon the used of multi-mode lead salt diode lasers in the mid- and far-IR spectral regions. These lasers accessed the strong fundamental absorption bands of water vapor allowing sensitive detection, but at a high cost in the complexity of the instrument. The lasers were coupled to large monochromators to isolate individual lasing modes. Further complicating the instruments was the need for liquid nitrogen cooling and the use of cooled detectors. As the development of near-IR diode laser sources matured and were adopted by the telecommunications industry it become possible to utilize these same sources and InGaAs detectors for near-IR overtone absorption spectroscopy. The near-IR diode lasers were single mode devices and allowed the use of fiber optic coupling and transmission technology to multiplex absorption
measurements at multiple locations and using multiple laser sources. The sensor we describe below is based upon this technology.

There are several commercially available, currently-off-the-shelf (COTS) technologies which we have taken advantage of in the development of this sensor. These technologies include telecommunications grade NIR distributed feedback (DFB) diode lasers, single mode fiber optic components including splitters, collimators and patch cords, a balanced ratiometric detector (BRD) circuit, and a long pathlength multi-pass Herriott cell. The sensor architecture, calibration and resulting sensitivity is discussed below. We note that although water is probably one of the most important species to measure it is also one of the most difficult to measure and control. It is a very “sticky” gas tending to adhere to all surfaces and it is slow to be vacuum pumped away. This fact also contributes to the importance of its measurement, especially for the semiconductor manufacturing community.

2. DIODE LASER-BASED ABSORPTION SPECTROSCOPY

The automated water vapor sensor discussed in this publication is based on the quantitative absorption of a near-IR diode laser beam propagating through a gas sample. The transmission, $T$, is given by the Beer-Lambert relation:

$$T = \frac{I}{I_o} = \exp \left[ -S(T)g(v - v_o)N\ell \right]$$

where $I_o$ is the transmitted intensity at frequency $v$ after propagation through a pathlength $\ell$ (cm), $I_o$ is the initial intensity, $S(T)$ is the linestrength of the transition centered at $v_o$ (cm$^{-1}$/molecule/cm$^2$), $g(v - v_o)$ is the lineshape function (cm) at pressure, $P$, and $N$ is the number density of the absorbing species (cm$^{-3}$). The peak absorption coefficient, $\alpha(v_o, T)$ is given by:

$$\alpha(v_o, T) = S(T)g(v - v_o)N$$

The integrated absorption coefficient is:

$$\alpha(T) = S(T)N$$

since by definition:

$$\int_{-\infty}^{\infty} g(v - v_o) \, dv = 1$$

The lineshape function, $g$, depends on the gas temperature and pressure and the absorption linestrength depends upon the gas temperature. The water concentration, [H$_2$O], in molecules cm$^{-3}$ is given by:

$$N = -\int \frac{\ln \left[ \frac{I(v)}{I_o(v)} \right]}{S(T)\ell} \, dv$$
where \( dv \) is the laser scan rate per data point across the measured absorption feature. By scanning the laser frequency across the entire absorption lineshape, the pressure dependency of the lineshape function is removed from the measurement. Scanning fully resolved absorption lineshapes also reduces the effect of broadband absorbers in the background gas and non-resonant scattering from aerosols. We have chosen to probe the 1.3925 \( \mu \text{m} \) water vapor absorption feature which arises from the \( 3,03 - 2,02 \) rotational line within the \( v_1 + v_2 \) vibrational band. The linestrength for this absorption feature has a 25% temperature dependence over the temperature range of interest (273 to 375 K).\(^1\)

To measure ppb water vapor concentrations in the industrially important gases of interest, it is typically necessary to be able to make absorption measurements of 1 part in \( 10^4 \) or smaller. To achieve these measurement sensitivities, we utilize a dual beam instrument architecture which takes advantage of a novel noise canceling electronic circuit fragment which was developed at IBM.\(^4,5\) The important aspects of the circuit which we have called the Balanced Ratiometric Detector circuit are shown in Figure 1. In the experimental setup we arrange the laser intensity detected using a reference photodiode to be greater than that detected by the signal photodiode (typically a factor of 2 or higher). The circuit cancels the amplitude noise associated with the diode laser light source by splitting the reference photocurrent across a matched bipolar transistor pair, \( Q_1 \) and \( Q_2 \). The signal diode photocurrent passes directly through an additional transistor, \( Q_3 \). The signal photocurrent and the split reference currents are summed at the junction \( A_1 \). A feedback loop is set up to null the current flow at \( A_1 \) by adjusting the split ratio of the reference photocurrent. The reference current split ratio is adjusted by changing the base emitter voltage of the reference bipolar transistor.

This process results in an electronic photocurrent balance which can reduce the common mode laser noise on the circuit log output signal by more than 50 dB. This allows the BRD to provide nearly shot-noise limited measurements for the detection of weak water vapor absorption spectra.\(^4,6\) Equation (6) shows the log ratio output signal dependence on the reference and signal beam photocurrents.

\[
V_1 = -G \ln \left( \frac{I_{\text{ref}}}{I_{\text{sig}}} - 1 \right)
\]

\( G \) is the gain of the integrating amplifier and \( I_{\text{ref}} \) and \( I_{\text{sig}} \) are the photodiode currents. By combining Equation (6) with Beer’s Law and the water vapor absorption linestrength, the absorption pathlength and the laser tuning characteristics we can directly determine the water vapor number density from the measured absorption lineshape. Thus, the water vapor monitor described below is not a transducer type of sensor, but determines the water vapor number density from known spectroscopic parameters and fixed, measured laser characteristics. Because the sensor scans across the entire water vapor absorption lineshape the technique is immune from total reductions in the laser output power due to minor misalignments of the diode laser or broadband attenuation due to changes in the absorption cell window transmission.

### 3. SENSOR DESIGN

There are two basic spectroscopic parameters which govern the design of any absorption spectrometer when attempting to achieve a minimum number density detection sensitivity. The first is the absorption linestrength, \( S(T) \), at the anticipated measurement temperature. The reported linestrength value for the 1.39253 \( \mu \text{m} \) absorption feature in the HITRAN database...
is $1.80 \times 10^{-20}$ cm/molecule. We have undertaken an independent measurement of the linestrength using a diode laser-based instrument and measured the linestrength to be $1.24 \times 10^{-20}$ cm/molecule. The second important parameter is the collisional broadening parameter, $\gamma$, for the bath gas of interest. The collisional broadening parameter is important because it determines the spectral width of the absorption lineshape along with the gas temperature. In the low pressure limit the absorption lineshape is characterized by a Gaussian distribution function with the temperature determining the Doppler width of the absorption function. Doppler lineshapes tend to be quite narrow with highly peaked absorbance values. As the pressure of the sample gas rises collisional broadening mechanisms begin to change the spectral width of the absorption lineshape, adding Lorentzian character. In the limit of low temperature and finite pressure the lineshape would be primarily Lorentzian. Lorentzian lineshapes tend to be broad, with depressed peak absorbance values. In typical sensor operation the lineshape falls somewhere between Gaussian and Lorentzian, and is thus described by a Voigt lineshape which is a convolution of Gaussian and Lorentzian lineshape functions.

The Voigt profile is a convolution of the Doppler (or thermal) broadening profile and the collisional broadening profile. It may be described by two parameters: the Voigt $a$-parameter and the gas temperature. The Voigt $a$-parameter is a measure of the collisional width of the convolution profile normalized by the Doppler width and is defined as

$$a = \sqrt{n} \left( \frac{\Delta \omega_c}{\Delta \omega_D} \right)$$  \hspace{1cm} (7)

where $\Delta \omega_c$ is the collision width, and $\Delta \omega_D$ is the Doppler width. The Doppler width is calculated using the formula

$$\Delta \omega_D = 2\omega_0 \left( 2kT / m \omega_0 c^2 \right)^{1/2}$$  \hspace{1cm} (8)

where $\omega_0$ is the linecenter frequency, $k$ is Boltzmann’s constant, $m$ is the mass of a H$_2$O molecule, and $c$ is the velocity of light.

The total collision width contains contributions from all of the available collision partners in the measurement volume. Thus we use the formula

$$\Delta \omega_c = \sum_i 2\gamma_i P_i$$  \hspace{1cm} (9)

where $\gamma_i$ is the collisional broadening coefficient for species $i$ at partial pressure $P_i$. The collisional broadening coefficient is also temperature dependent, as described by

$$2\gamma = 2\gamma_0 (T / T_{ref})^n$$  \hspace{1cm} (10)

where $\gamma_0$ is the value at the reference temperature, $T_{ref}$. In this investigation we are making measurements at room-temperature.

With these considerations in mind, the first task in designing the water monitor for measuring moisture in ammonia was to measure the collisional broadening parameter for ammonia on the 1.3925 $\mu$m water vapor absorption feature. This was accomplished using a dual beam direct absorption technique which simply ratioed the output from two transimpedence amplifiers, signal and reference, to measure the water vapor absorption lineshape as a function of ammonia bath gas pressure. After recording the lineshapes on an absolute laser frequency scale, we fit each of the lineshapes to a Voigt absorption profile. The resulting fits provide Voigt $a$-parameters which were converted to collisional broadening widths using the known gas pressure and temperature. A plot of the collisional widths (full width at half maximum, FWHM) is shown in Figure 2. The
data shows excellent linearity, with the slope of the line providing the value of \(2\gamma\), or twice the collisional broadening parameter, 23.77 MHz/Torr.

The collisional broadening parameter was used in conjunction with the sample gas temperature (300 K) to calculate a peak water vapor absorbance value as a function of total pressure. This was accomplished by inputting the temperature, the Voigt \(a\)-parameter and the center absorption line frequency into a computer Voigt simulation routine. This provided a lineshape function value at the peak of the absorption feature which was used in conjunction with the known value of the absorption linestrength, \(S(T)\). Figure 3 shows the plot of the calculated peak absorption as a function of total ammonia sample gas pressure for a sample gas containing 50 ppb of water vapor. The plot shows that the peak absorbance remains nearly constant (dropping 4%) from pressures of 1 atmosphere (760 Torr) down to approximately 100 Torr, despite the factor of 7.6 loss in total water vapor number density (the peak absorption simulations were performed at a constant water vapor/ammonia vapor...
mole fraction). From 100 to 50 Torr there is a 53% loss in the peak absorbance. This analysis suggested that we should operate the water vapor sensor between 100 and 760 Torr to obtain a peak absorbance value of $1.53 \times 10^{-4}$ using a 100 m pathlength.

In evaluating the total pressure range in which to operate the sensor, we considered not only the absorbance level for a given water concentration but also the potential for interfering absorbance peaks from the ammonia sample gas. Previous investigations have reported ammonia absorption in the 1.3 to 1.4 µm region. Taking into account our broadening data and the interference potential we targeted a sensor operating pressure of 100 Torr or less. The lower operating pressure provided the potential for spectral discrimination between the water vapor absorption and potential interferences. Assuming a 100 Torr total pressure and a 50 PPB sensitivity, we calculated an absorbance of $\sim 1.53 \times 10^{-4}$ with a total pathlength of 100 m. The use of a fully fiber coupled system and the BRD typically results in limiting absorbance sensitivity of $\sim 1 \times 10^{-5}$. Although these calculations projected a factor of $\sim 10$ additional sensitivity than required, we anticipated the potential need to utilize a weaker absorption line if an ammonia interference was coincident with the 1.3525 µm water vapor absorption. With this in mind, we chose to design the optical pathlength to provide between 75 and 100 m of total absorption pathlength.

2.1 Diode laser module

The diode laser module houses the diode laser, the diode laser controller, the 1 x 2 fiber optic splitter, the BRD, and the DC power supplies for the laser operation. The diode laser operated at 1.3925 µm and had a spectral linewidth of approximately 40 MHZ. The laser tuned at approximately 0.04 cm$^{-1}$ mA$^{-1}$ and was typically scanned over $\sim 0.75$ cm$^{-1}$ to fully capture the low pressure water vapor lineshape. The laser temperature and injection current were controlled by a Melles Griot diode laser controller, model 56DLD403. The optically isolated fiber coupled output from the diode laser was split by a 70/30 single-mode wavelength flattened fused fiber optic splitter. The output from the 30% leg of the fiber splitter was coupled directly to an InGaAs reference photodiode located within the BRD. The 70% leg was connected to the fiber optic launch collimator located within the absorption cell module (described below). After traversing the multipass absorption cell the signal beam was detected by a second 3 mm diameter, windowless InGaAs photodiode.

2.2 Computer module

The automated water vapor sensor was controlled by a 200 MHz Pentium class industrial rack mounted computer. The system was outfitted with a National Instruments AT-MIO-64E-3 data acquisition (DAQ) board and a NI PC-DIO-24 digital I/O (DIO) board. The DAQ board was used to control the laser diode controller and to digitize signals form the BRD, the laser controller and the absorption cell pressure transducer. The DIO board was used to implement software control of the programmable gain amplifiers (PGA) within the BRD. The sensor control software was developed using National Instruments LabView graphical software programming language. The software provided three separate graphical user interface (GUI) screens for sensor operation including a main control and data presentation GUI, an oscilloscope GUI and a digital filter GUI.

2.3 Absorption cell module

The absorption cell module was designed to achieve adjustable absorption path lengths up to 100 m using a modified commercial platform. We purchased a New Focus, Inc. multipass Herriott cell based upon an astigmatic mirror design. The cell was capable of a 100 meter pathlength using a Lissajous’ laser spot pattern on each of the cell mirrors. The commercial cell input and exit for the diode laser beam utilized a single aperture in one of the mirrors and required a substantial atmospheric air pathlength outside of the absorption cell. Due to the complicated alignment procedure and the need for large (8 to 30 cm) external air pathlengths for the laser beam we chose to modify the commercial platform. We replaced the nickel-coated astigmatic cell mirrors with a pair of spherical dichroic mirrors each outfitted with a radial 2 mm diameter through slot from the center of the mirror to the outer edge. This design allowed the collimated diode laser beam to enter one end of the cell, multipass within the cell forming a circular spot pattern and exit the other end of the cell. The external pathlength outside of the cell was less than 1 cm, which was efficiently purged with dry nitrogen.
The 1 m focal length spherical slotted mirrors were mounted within the modified commercial platform separated by 0.5 m. The collimated laser beam entered the cell through a wedged AR-coated window and passed through the mirror slot positioned at the 12 o’clock position. The laser beam was reflected back and forth between the two Herriott cell mirrors before exiting the second mirror with the mirror slot positioned between the 2 and 3 o’clock position. A photograph of the exit mirror and the laser beam spot pattern is shown in Figure 4. Typical absorption pathlengths ranged from 75 to 100 m, with the spot pattern shown in Figure 4 resulting in an 86 m pathlength. After traversing through the exit mirror slot the laser beam passed through a second wedged AR-coated window and was detected by an InGaAs photodiode positioned against the cell exit window. The air gap between the cell window and the windowless photodetector was less than 2 mm.

The total absorption path length provided by the simplified Herriott cell design could be adjusted by simply rotating the exit mirror slot. In the current system the exit mirror slot could be rotated clockwise providing pathlength of 86 m to only a few meters. No additional adjustments were made to the cell mirrors, with only the cell exit window and the photodetector being repositioned. Additional mirror focal lengths (e.g., 0.5 m) could also be used providing similar spot patterns and functionality. Additional changes to the commercial multipass cell included modified cell end flanges including unsymmetrical wedged window ports. We also replaced the standard glass vacuum sleeve with an electropolished stainless steel vacuum sleeve. This sleeve included gas inlet and pump ports and a pressure tap outfitted with a 100 Torr MKS Baratron pressure transducer. The sleeve was put into place following the optical alignment by removing the entire exit mirror assembly. The sleeve was then positioned into place and the exit mirror was repositioned using the provided alignment pin. The use of the modified commercial platform provided reproducible mirror positioning and allowed the use of the stainless steel tube for operation from vacuum to several atmospheres.

4. SENSOR CALIBRATION AND SENSITIVITY

The response of the sensor could be predicted from known physical constants including the electronic gain within the BRD circuit, the laser tuning characteristics, the temperature dependent water absorption linestrength and the absorption pathlength. Thus the only calibration which was required was the laser tuning rate. This was measured using a Burleigh confocal Fabry-Perot spectrum analyzer, Model SA PLUS, outfitted with a NIR mirror set. The calibration was accomplished by scanning the diode laser current under the exact conditions (temperature and current ramp) which were used for the water vapor absorption
measurements. The resulting interferogram displayed a series of intensity peaks separated by the 2 GHz FSR. The calibrated laser scan rate was 1200 MHz/mA. In addition to the laser calibration we also undertook a series of sensor validation experiments. In these experiments we added neat water vapor to the sealed, evacuated absorption cell. We performed measurements at two different total absorption pathlengths, 64.8 cm, a single pass through the cell, and at 8564.8 cm, multipassing through the cell. The water concentration was measured using a temperature controlled 1 Torr Baratron pressure transducer and a 10 Torr transducer attached to the cell. Under most operating concentrations we observed a rapid equilibrium between the cell walls and the water vapor providing a steady gas pressure. Figure 5 displays a plot of the water vapor concentration measured in the absorption cell using the sensor absorption lineshape data versus the pure water vapor number density as determined by the MKS Baratron measurements. The sensor-based optical measurements were derived from computer spreadsheet simulations of the automated sensor control software calculations. This procedure was undertaken to verify the validity of the sensor analysis algorithm and to verify the previously determined value for the laser scan parameter. The linearity of the plot in Figure 5 was good for over 3½ decades of dynamic range of pure water number density. The line through the data is a line of slope = 1, for the Baratron measurement plotted for both the x and y values. This data was recorded with an absorption pathlength of 64.8 cm. A similar set of data was recorded for the 8564.8 cm pathlength, providing comparable results.

The goal of the following experiments was to develop an estimate of the sensor detection sensitivity for water concentration measurements in inert gases such as nitrogen. Following a series of heating and purging cycles of the absorption cell with dry nitrogen the water content of a flowing sample of nitrogen gas was measured. The nitrogen source was gaseous blow-off from a high pressure liquid nitrogen dewar (with additional water being introduced to the flow from outgassing within the stainless steel transfer tubing). Figure 6 displays an absorption lineshape of 370 ppb of water in a total nitrogen pressure of 30 Torr. The measured water number density was $3.5 \times 10^{11}$ cm$^{-3}$. The peak absorption was approximately $1.3 \times 10^{-3}$. The residual fringe amplitude noise in the baseline of the absorption spectrum was likely due to beam steering or turbulence effects of the rapidly flowing nitrogen gas through the absorption cell. Using these fringes as the baseline noise level, and the peak of the absorption lineshape as the signal level, the signal-to-noise ratio equals 29:1. This ratio, combined with the measured water vapor concentration suggests a 13 ppb (S/N=1) detection limit for water vapor. This would correspond to a limiting absorbance value of $4.4 \times 10^{-5}$.

We performed a similar set of experiments using semiconductor grade ammonia as the sample gas of interest. Figure 7 displays a water vapor absorption lineshape recorded using the sensor with an ammonia sample gas. The gas was certified by the supplier to contain <5 ppm of water contamination. The cell pressure was 50 Torr, the signal integration time was 1 s. The water concentration was measured to be 788 ppm. Classifying the residual baseline ripple as noise and the maximum peak height as the signal, the spectrum has a signal-to-noise ratio of 20:1. Based upon this analysis the resulting water
vapor detection sensitivity was 35 ppb. In addition to recording absorption lineshapes to estimate the sensitivity of the sensor, we also recorded temporal plots of water concentration to verify the sensor precision. Figure 8 shows a time plot of the water concentration from an ammonia gas sample. The flowing sample pressure was 54.7 Torr and the average water concentration was 445 ppb. The $1\sigma$ noise level was determined to be 21 ppb. We are currently investigating the reason why we apparently achieved a lower sensitivity limit for water detection in nitrogen as compared to ammonia gas samples. We believe it may be related to the possibility of ammonia spectral interferences within this spectral window. We are also developing potential techniques for subtracting these unwanted absorption signals from our data analysis to achieve higher sensitivity limits with a goal of measurements approaching 1 ppb or below.
Figure 8. Water sensor precision demonstration using a flowing, dry ammonia gas sample.

5. SUMMARY AND CONCLUSIONS

We have developed an industrial water vapor sensor based upon near infrared tunable diode laser absorption spectroscopy at 1.3925 μm absorption spectroscopy. The automated sensor scans the diode laser wavelength across the entire water vapor absorption feature allowing for a robust sensor design which is insensitive to potential reductions in overall laser power or broadband attenuation due to particulate contamination of optical surfaces, or the introduction of broadband absorbers. The water concentration is determined from the absorption pathlength, the fundamental linestrength of the absorption feature, the scan characteristics of the diode laser and the inherent gain built into the BRD circuit. The sensor is calibrated by simply calibrating the laser scanning characteristics using a Fabry-Perot spectrum analyzer. The sensor determines the water concentration from known physical constants. It is not a transducer and thus remains “calibrated” as long as the laser diode maintains its characteristic lasing frequency and tuning rate (typical mean time between failure is greater than 10,000 hours). The sensor utilized a multipass Herriott cell design which minimized the atmospheric air external pathlength while allowing a nearly continuously variable pathlength. The use of spherical mirrors simplified the optical alignment procedure as compared to the the commercially available configuration. One of our goals was to simplify the overall design so the instrument could be fielded without the need for a highly trained technician to implement optical realignment. The use of the modified commercial platform resulted in a robust design which remained optically aligned even after final shipment to a gas manufacturing facility. The water vapor sensor response was verified by measuring neat water concentrations within the multipass absorption cell and comparing the measured values to those provided by a MKS Baratron pressure transducer. Measurements within dry nitrogen gas and ammonia gas samples were also performed to guage the instrument sensitivity. Water content in dry nitrogen was measured to be 370 ppb with signal to noise (S/N=1) projecting a sensitivity of 13 ppb. Water content in semiconductor grade ammonia was measured at 690 ppb and 445 ppb with signal to noise (S/N=1) projecting a sensitivity of 21 and 35 ppb. We are currently investigating the possibility of interfering ammonia absorption features near our spectral window contributing to the “noise” level within the ammonia sample gas measurements. We anticipate that the ultimate sensitivity of the instrument may meet or exceed the 1 ppb level.
6. REFERENCES


