Trace moisture detection at high pressures incorporating a compact GaAs-based VCSEL array

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KEYWORDS

GAS ANALYZERS, TUNABLE LASERS, VCSEL, LPG, MOISTURE, PROCESS CONTROL

ABSTRACT

In the LPG industry, there is a need for moisture monitoring to prevent the formation of hydrates, which create ice-like blockages in critical infrastructure. LPG is usually processed and transported at 7 bara, a high-pressure environment that is challenging for typical optical moisture detectors. This presentation discusses a vertical-cavity surface-emitting laser(VCSEL)-based moisture detector operating at 7 bara. The system is based on tunable diode laser absorption spectroscopy (TDLAS) and uses a single-mode 2×2 VCSEL array with fast, reproducible tuning capabilities to implement wavelength modulation spectroscopy and achieve a high signal-to-noise ratio. The sensor is a real-time, online, maintenance-free gas monitor with a 7.8 m optical path realized using a Herriot cell. The VCSEL is mounted on a thermoelectric cooler (TEC) and packaged in a hermetically sealed transistor outline(TO)-can, enabling stable performance in harsh ambient conditions. Ultra-low concentrations (3 ppb) of H_2O have been measured using TDLAS in the mid-IR at low pressure. VCSELs are significantly less complex and less expensive components that enable compact, low-cost systems such as oxygen analyzers. Previously, VCSEL-based H_2O sensors achieved limit of detection (LOD) of only 39 ppm. Since then, not enough research has been done to use VCSELs in trace moisture sensors. In this presentation a system with LOD of 0.2 ppm is presented. The concepts to increase sensor functionality by placing multiple VCSELs in one TO-package for multiwavelength real-time sensing are presented, which can be extended to detect gases other than H_2O.

INTRODUCTION

In the modern industrial landscape, the transition toward Industry 4.0 and high-efficiency manufacturing requires a shift from reactive to proactive process monitoring. While industrial gas monitoring has historically been viewed through the lens of emergency mitigation, its primary value now lies in operational cost reduction and asset longevity. Precise gas analysis allows for the real-time adjustment of feedstocks and the prevention of catastrophic equipment degradation.

Trace moisture (H_2O) is one of the most significant analytical nuisances in industry. Unlike inert contaminants, water is highly reactive and polar, leading to complex interactions within process pipelines. In hydrocarbon processing, the presence of water vapor at parts-per-million (ppm) levels can lead to the formation of solid clathrates, which induce mechanical blockages in heat exchangers, valves, orifices, and narrow-diameter piping. Such blockages can cause catastrophic mechanical failure or emergency shutdowns.

Detection of low moisture concentrations in complex hydrocarbon matrices (with a limit of detection of 3 ppb) using an interband cascade laser (ICL) emiting at around 2.7 µm, has been previously reported[1]. However, in order to suppress cross-interference from hydrocarbon backgrounds, low sample pressures were needed. In this presentation a detection scheme is realized in the 940 nm range that is free from significant hydrocarbon interferences. Thus, there is no need for complex sample handling to maintain low pressure in the sample cell. Moreover, VCSELs can be tuned over a wider frequency range than edge-emitting lasers with distributed feedback (DFB) gratings. In addition, VCSELs are both much less complex and less expensive components that enable compact, low-cost systems such as oxygen analyzers. Previously, VCSEL-based H_2O sensors could only achieve an accuracy of 39 ppm [2]. Here, a record sensitivity for VCSEL-based sensors with a limit of detection of 0.2 ppm is presented.

The strategic imperative for monitoring H_2O in liquefied petroleum gas (LPG) is fundamentally driven by the need to prevent the formation of solid hydrocarbon hydrates. While pure LPG is non-corrosive, the presence of even trace moisture enables the formation of corrosive agents. Additionally, LPG often contains trace amounts of CO2 or H2S. In the presence of liquid water, these form carbonic acid or sulfuric acid. Trace moisture monitoring is also crucial for the LPG industry to comply with international standards such as ASTM D2713 and ISO 6251.

GAS ANALYZER SETUP

Trace moisture detection has been implemented using the GasEye™ Extractive Laser Gas Analyzer. The system is a versatile gas analysis tool for industrial process

applications, adapted here for demanding high-pressure (7 bara) water vapor monitoring.

Gas detection is based on a laser spectrometer setup that uses molecular absorption spectroscopy. A semiconductor VCSEL emits a beam that passes through the cell and impinges on a detector on the receiver side. When water vapor is present, the light intensity changes due to absorption, and this change is used to determine the concentration. The laser continuously scans a single absorption line with very high spectral resolution. The measurement is highly selective due to the narrowband nature of the laser light.

The overall system schematic is shown in Figure 1. The main building blocks consist of the power supply, the electronics module, and the gas-sensing setup. The gas detection scheme includes a laser module, a multipass cell, and a detector module. To exclude the ambient moisture from the measurement the enclosure can be purged with dry air.

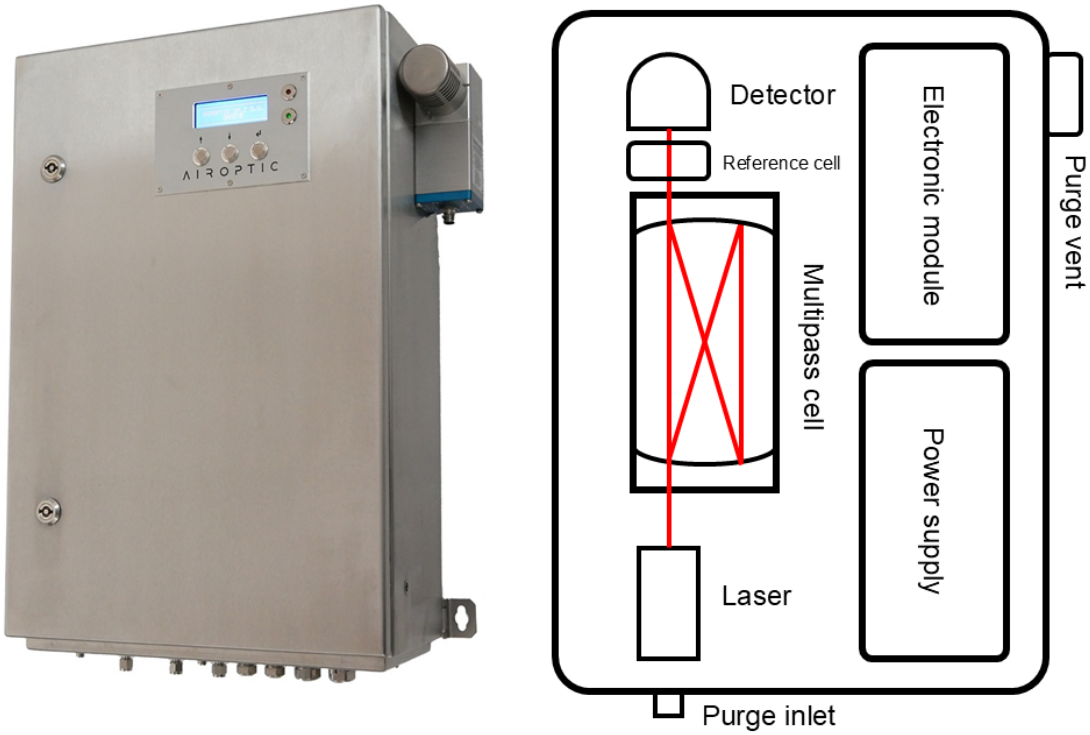

FIGURE 1. Laser Gas Analyzer enclosure and internal set-up scheme.

The light source used for H_2O detection is a compact, high-modulation-rate gallium arsenide (GaAs)–based VCSEL [3], which offers key advantages for high-precision spectroscopy. The use of GaAs quantum wells with specifically tailored, few-nanometer-thick layers enables emission near 940 nm. VCSELs compared to edge-emitting lasers have much shorter internal cavities, this structural difference

allows the VCSELs to achieve an extremely narrow linewidth and high side-mode suppression, both of which are critical for resolving specific molecular absorption lines.

VCSELs typically exhibit a strong tunability coefficient of ~0.5 nm/mA, making them well suited to sensing broad spectral features such as pressure-broadened absorption lines. Unlike traditional edge-emitting lasers, a VCSEL emits light perpendicularly from the wafer surface, enabling robust and simplified placement of the laser chip within standard packages. Furthermore, VCSELs exhibit high thermal stability and low threshold currents, allowing the analyzer to perform rapid wavelength tuning with minimal power consumption.

VCSELs are significantly more cost-effective to produce than traditional DFB and ICL lasers (~1/10 the cost) because they can be tested at the wafer level and manufactured in high volumes. By integrating these more affordable components, the overall cost of the gas analyzer is reduced, making high-end spectroscopy accessible to a wider range of industrial sites. This lower entry barrier allows facilities to deploy more sensors across their infrastructure, improving plant safety through comprehensive monitoring and enabling finer process optimization that was previously cost-prohibitive. This can enable for instantaneous and pinpoint leak detection across many industries. In the LPG industry it can help to monitor precise gas composition across the facility to prevent boiling liquid expanding vapor explosion.

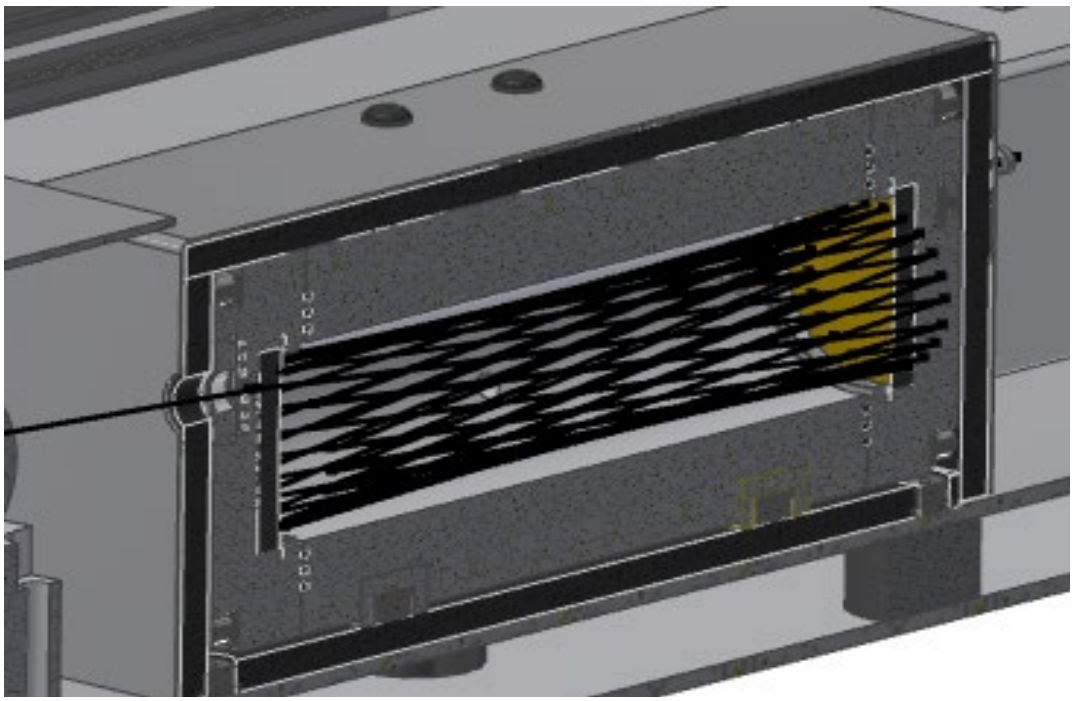

FIGURE 2. Cross section of Herriot cell design with the laser beam propagation.

The multipass cell used in the analyzer utilizes a Herriott cell configuration (Figure 2). The optical assembly is designed to maximize the interaction between the laser light

and the gas sample within a small volume, resulting in a 7.8 m optical path length and a volume of 0.35 L. By reflecting the laser beam multiple times between two spherical mirrors, the cell effectively increases the interaction length between the light and the gas molecules, which greatly enhances the signal-to-noise ratio necessary for detecting moisture at ppm levels. The Herriot cell design is inherently more stable than other multipass configurations, as it is less susceptible to mechanical vibrations and thermal shifts common in industrial settings. This is due to the high tolerance for the accuracy of the laser beam alignment inside of the cell. The small internal volume ensures a fast response time due to rapid gas exchange. Additionally, the multipass cell can be equipped with both temperature and pressure stabilization if further optimization of measurement conditions is needed. Here only temperature stabilization was used.

To realize the detection scheme, a planar diffused silicon photodiode was used. The detector exhibits high sensitivity and a spectral response ranging from 350 nm to 1100 nm. The diode has high shunt resistance, low noise, and long-term stability, making it well suited for industrial applications.

The instrument utilizes an internal reference gas cell for real-time verification of calibration status. Internal reference gas is used for closed loop control of the zero and span drift. Thus, long-term stability and accuracy can be assured.

RESULTS

Measuring ppm-levels of water vapor requires a strictly dry environment inside the analyzer enclosure to ensure that the detected moisture originates solely from the process gas. To maintain these conditions, the system is continuously purged with dry air (dew point < -40 °C). However, achieving a stable baseline is time-intensive due to the polar nature of water molecules. The molecules tend to adsorb onto the internal surfaces of the enclosure, electronics, and mechanical parts. During purging, the adsorbed molecules begin to desorb and influence the readings. To achieve a sufficiently dry background environment, at least one hour of purging is required before starting measurements.

The results below show the system performance for trace moisture sensing. For these tests, the analyzer was supplied with a gas mixture at around 1 bara, and the measurement cell was heated to a constant 50 °C. Keeping the temperature stable is important because it reduces thermal drifting of the water vapor absorption lines and prevents water from condensing inside the cell.

Calibration of the device was performed using a gas standard generator equipped with a precision trace moisture source. This setup uses stable molecular permeation of

water vapor into a controlled flow of dilution gas, creating a known, highly accurate ppm-level mixture. While these gas mixtures enabled precise calibration of the analyzer, the transition between different concentration levels required significant stabilization time (~20 minutes).

Validation of the results was carried out by measuring at increasing moisture concentrations. The steps included 0.0, 25.0, 50.0, 74.9, and 100.9 ppm water vapor, and were limited by the accuracy of the dilution gas flow (0.01 standard liters per minute). Dry air was used as a dilution gas in the presented validation.

As seen in Figure 3, the system exhibits an excellent response to the gas concentration being measured (the true moisture content is indicated by vertical lines with labels on the right side of the figure). Small reading fluctuations correspond to real fluctuations in moisture content rather than analyzer performance, as evidenced by the flat zero-sample readings. The results also verify the system's repeatability, as shown by the repeated measurements at 74.9 ppm moisture.

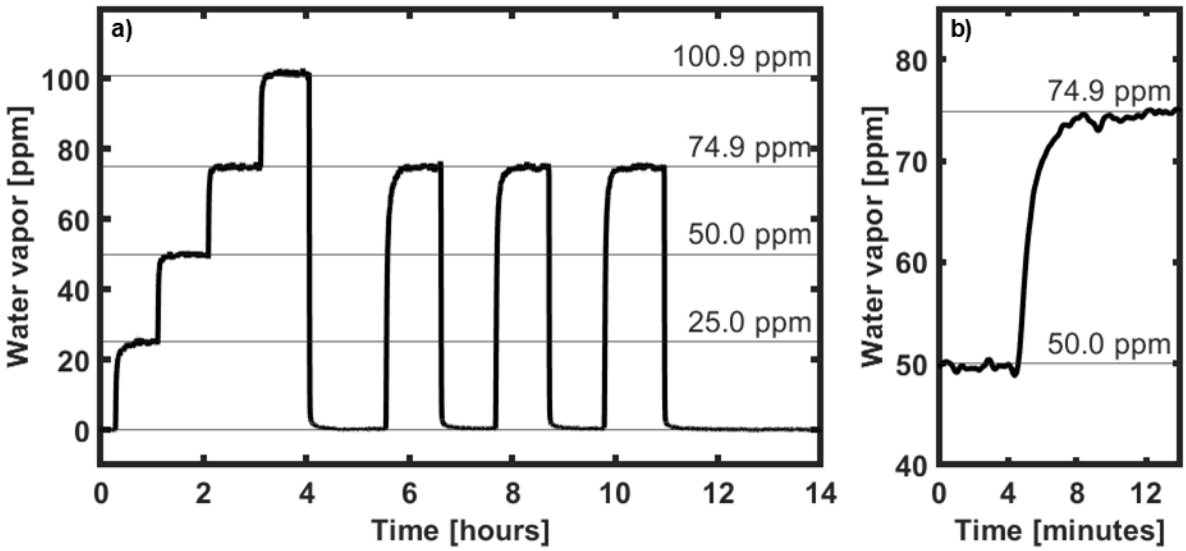

FIGURE 3. a) Water vapor at different concentrations and repeatability measurements, b) zoomed in view.

To determine the limit of detection, the system enclosure and measurement cell were purged with dry instrument air for an extended period until a zero reading was reached. Figure 4 displays the analyzer's readings during this zero-sample test. Based on the data and the observed noise levels, the limit of detection was calculated to be 0.2 ppm.

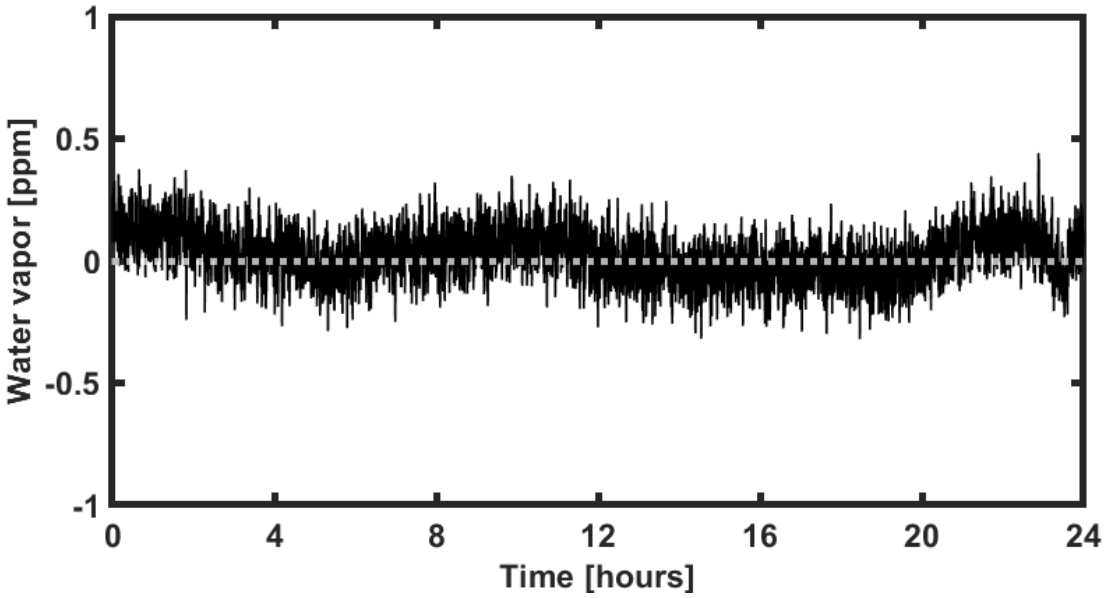

FIGURE 4. Zero sample measurement.

In Figure 5, the Allan deviation is presented. From this statistic, it can be determined that a possible limit of detection of 0.066 ppm can be achieved for an averaging time of 2000 s. The relatively long averaging time corresponding to the minimum in the Allan deviation indicates that the system is very stable and essentially free from drift over long periods.

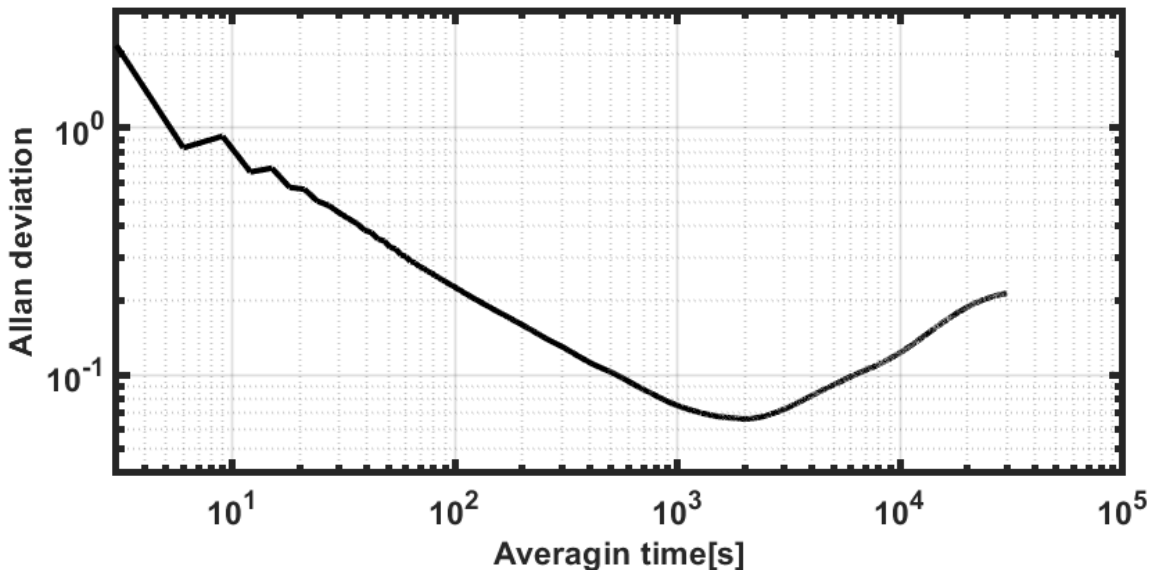

FIGURE 5. Allan deviation vs averaging time.

FURTHER CONCEPTS

HIGH PRESSURE H_2O DETECTION IN LPG

Thanks to the excellent tuning capabilities of VCSELs, it is possible to scan over a wide wavelength range. This enables the simultaneous scanning of multiple spectrally separated features or pressure-broadened absorption lines.

In this section, preliminary results are presented for high-pressure trace moisture detection in an LPG mixture. The system was prepared and calibrated in a similar manner to that described in the previous sections.

In air, water molecules primarily collide with nitrogen and oxygen, which are small, non-polar molecules. In contrast, LPG consists of heavier hydrocarbons such as propane and butane. These larger, more complex molecules exert stronger intermolecular forces and cause more frequent collisions with the water molecules. As a result, the water absorption line in an LPG mixture becomes broader than the same line in air. The comparison is shown in Figure 6. It can also be seen that the chosen spectral region is free of spectral interferences originating from the LPG mixture. This enables accurate and precise determination of the moisture content.

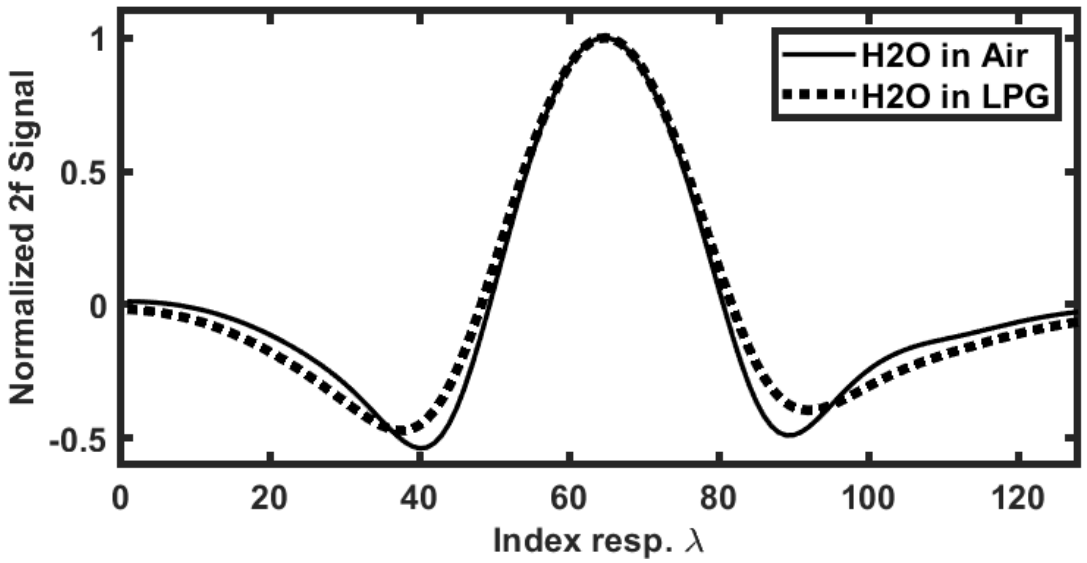

FIGURE 6. Comparison of H_2O absorption line in instrument air and H_2O in LPG.

After calibration, the system performance was evaluated again, and zero-sample measurements were conducted to determine the limit of detection. High pressure of 7 bara and the LPG mixture both introduce broadening effects. Because of this, the limit of detection calculated from the measurements in Figure 7 is 1.8 ppm.

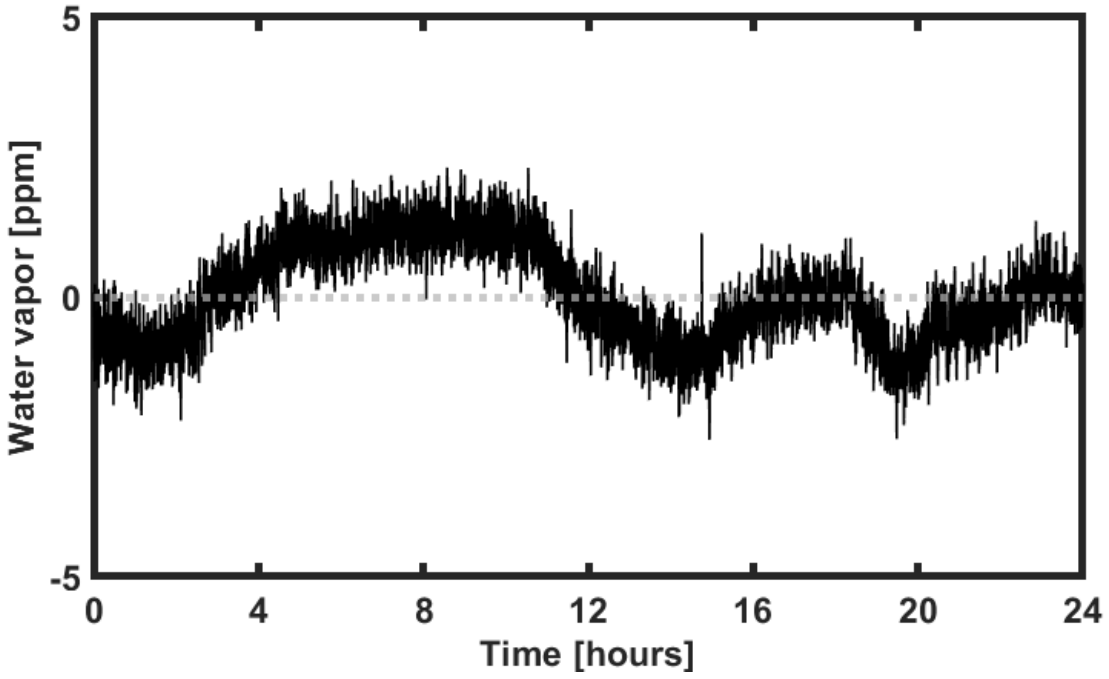

FIGURE 7. Zero sample measurement for a high pressure H_2O in LPG analyzer.

Figure 8 shows an Allan deviation based on measurement conducted at 7 bara. It can be seen that system exhibits excellent long-term stability and can achieve the best limit of detection of 0.23 ppm at averaging time of 14000 s.

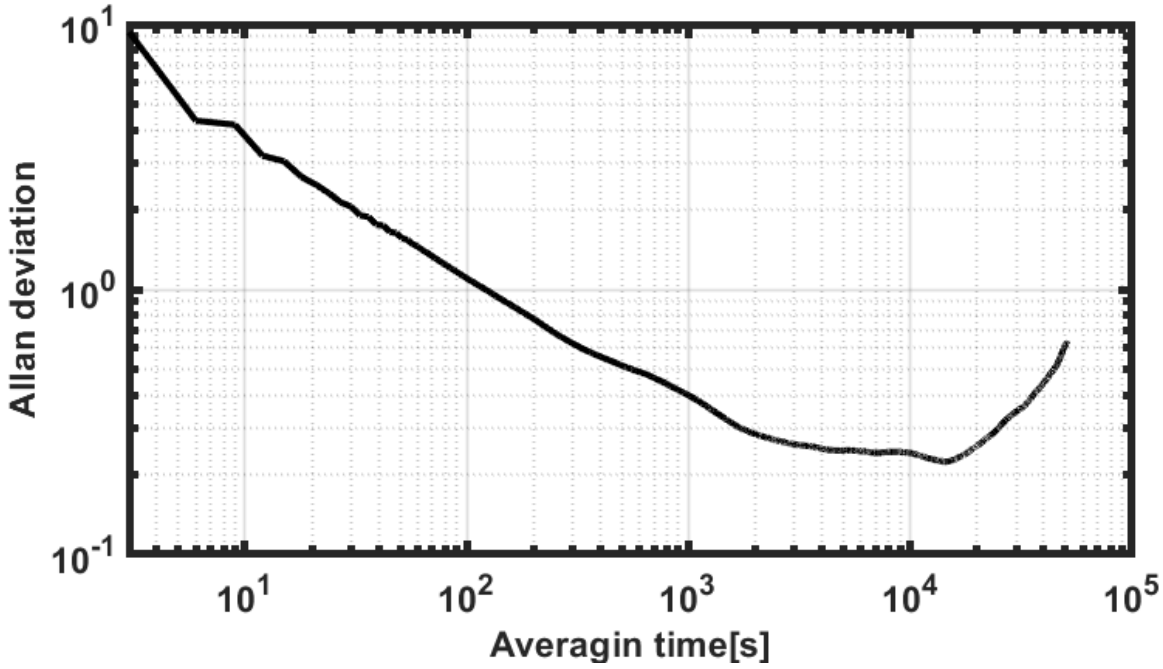

FIGURE 8. Allan deviation vs averaging time for a high pressure H_2O in LPG analyzer.

The results presented here, already show good potential for an industrial application and are soon to be implemented as a standard solution for trace moisture monitoring at high pressure.

2X2 LASER ARRAY

Since VCSELs emit vertically, they are much easier to assemble and integrate into optical setups compared to edge-emitting lasers, which require additional submounts. This advantage makes it possible to create tightly spaced laser arrays.
Here, we present a proof-of-concept 2×2 array assembly utilizing VCSELs with center wavelengths at 937 nm, 938.5 nm, and 940 nm. Because the standard package has a limited number of pin-outs, one dummy laser that was not connected to any pins was included in the array.
All lasers were mounted on a common TEC with a thermistor for temperature stabilization and tuning. Each laser can be individually tuned via the drive current to scan the desired absorption lines. The lasers were arranged in an array with a center-to-center spacing of less than 0.5 mm, as shown in Figure 9.

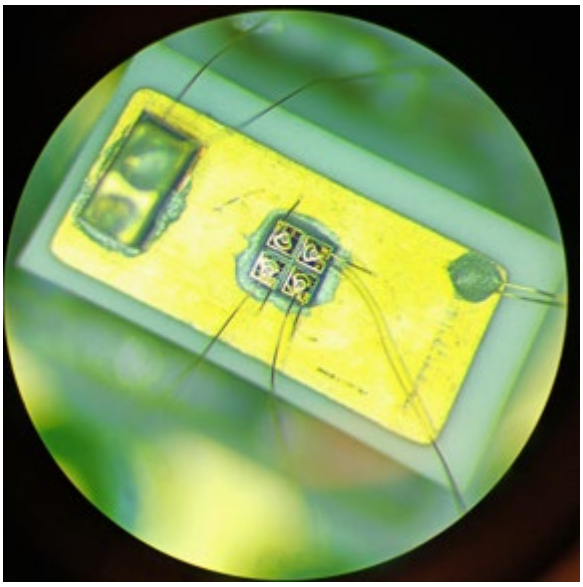

FIGURE 9. Microscope image of the 2×2 laser array.

Thanks to the tight array assembly, it was possible to collimate all lasers with a single lens. The collimated beams propagated along a common axis with only slight lateral displacement from the center. Nevertheless, they can be easily refocused to a single point on the detector. The cross-sections of the collimated beams at a distance of 5 m from the source are shown in Figure 10.

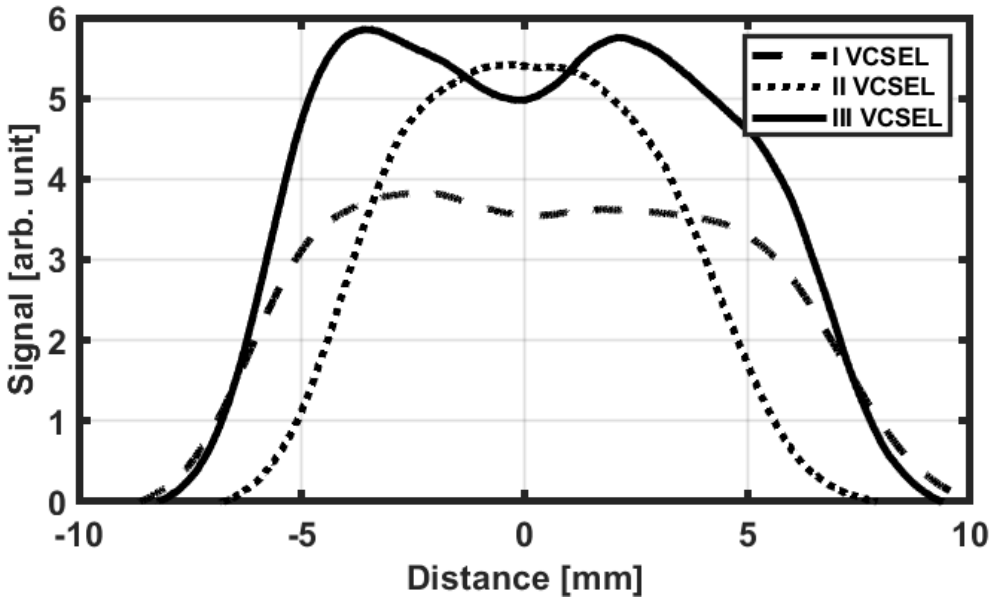

FIGURE 9. Collimated beams cross sections of the 2x2 laser array measured at 5m distance from the source.

The VCSELs used, possess a broad available tuning range and are optimally distributed spectrally with respect to one another. Thanks to this, it was possible to perform simultaneous scanning of multiple H_2O absorption lines, covering a 4 nm-wide spectral range. A comparison of the measured spectra and the HITRAN-based simulation [4] is shown in Figure 11.

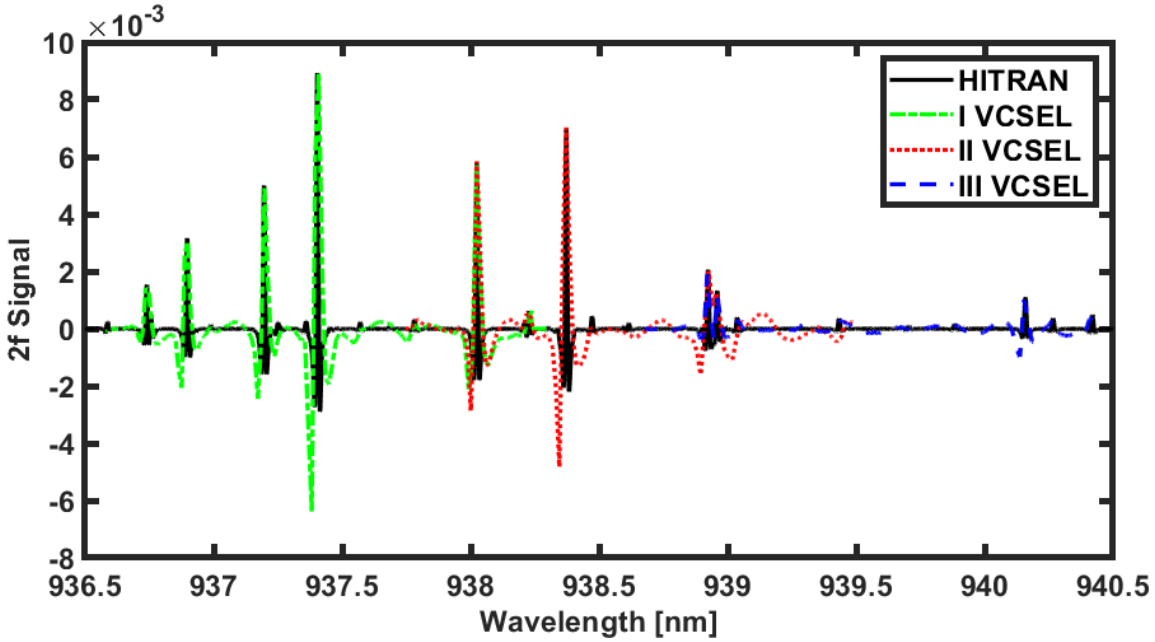

FIGURE 11. Comparison of combined 3 laser measurements with HITRAN-based simulations.

DISCUSSION AND CONCLUSIONS

The results confirm that the presented gas analyzer is highly effective for trace moisture sensing. The temperature-stabilized multipass cell enabled a limit of detection of 0.2 ppm by ensuring a stable spectral baseline and preventing adsorption and condensation of water molecules within the cell. The long stabilization times observed were expected, as the polar nature of water vapor causes it to adsorb onto internal surfaces, requiring thorough purging to reach equilibrium.

Preliminary results for high-pressure H_2O sensing already show strong industrial potential, with a limit of detection of 1.8 ppm. Further improvements remain to be explored to push the device's sensitivity even lower.

The novel 2×2 VCSEL array was collimated with a single lens. The lasers were temperature-stabilized in common and tuned individually by adjusting the current to enable independent gas detection. The results represent a strong proof of concept, and possible applications—especially in multi-gas sensing—will be further investigated.

In conclusion, the integration of a Herriott multipass cell and a GaAs-based VCSEL has produced a robust, high-sensitivity analyzer capable of reliable trace moisture detection. The demonstrated high-pressure moisture detection in LPG mixtures already represents a gap-filling industrial technology. The move toward using a 2×2 VCSEL array offers a clear path for future enhancements: the broad tunability and spectral distribution of the array in a single standard laser package can significantly reduce the size and cost of next-generation multi-gas sensors.

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