CW DFB RT diode laser based sensor for trace-gas detection of ethane using novel compact multipass gas absorption cell

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ABSTRACT

The development of a continuous wave (CW), thermoelectrically cooled (TEC), distributed feedback (DFB) laser diode based spectroscopic trace-gas sensor for ultra sensitive and selective ethane (C_2H_6) concentration measurements is reported. The sensor platform used tunable laser diode absorption spectroscopy (TDLAS) and wavelength modulation spectroscopy (WMS) as the detection technique. TDLAS was performed with an ultra-compact 57.6 m effective optical path length innovative spherical multipass cell capable of 459 passes between two mirrors separated by 12.5 cm. For an interference free C_2H_6 absorption line located at 2976.8 cm^{-1} a 1σ minimum detection limit of 130 pptv with a 1 second lock-in amplifier time constant was achieved.

Keywords: ethane trace-gas detection, CW TEC DFB GaSb based laser diode, novel compact multipass gas absorption cell, integrated electronic control and data acquisition module, tunable diode laser absorption spectroscopy

1. Introduction

In this work we focused on ethane detection and monitoring using tunable laser diode absorption spectroscopy (TDLAS) and wavelength modulation spectroscopy (WMS) as the detection technique. Ethane is one of the most abundant hydrocarbons in the atmosphere that strongly affect both atmospheric chemistry and the climate [1-2]. The major sources of ethane in the atmosphere are related to fossil fuel and biofuel consumption, biomass burning process, vegetation and soil leaks. Of particular interest is oil and gas prospecting since naturally occurring ethane seepages are indicative of hydrocarbon reservoirs [2]. Furthermore, ultra-sensitive detection has found application in medical breath analysis. Monitoring elevated levels of ethane in exhaled human breath can be used as a non-invasive method to identify and monitor different diseases such as asthma, schizophrenia, and lung cancer [3].

A TDLAS trace-gas sensor based on a 3.36 µm continuous wave (CW) thermoelectrically cooled (TEC), distributed feedback (DFB) laser diode from nanoplus GmbH was developed [4-6]. An optimum interference free C_2H_6 absorption line located at 2976.8 cm^{-1} was selected as the optimum target wavelength. TDLS was performed with an ultra-compact multipass gas absorption cell with an effective optical path length of 57.6 m as a result of 459 passes between two spherical mirrors separated by 12.5 cm. In addition a compact state of the art surface mounted electronic control board and data acquisition

Quantum Sensing and Nanophotonic Devices X, edited by Manijeh Razeghi, Eric Tournié, Gail J. Brown,
Proc. of SPIE Vol. 8631, 86312G · © 2013 SPIE · CCC code: 0277-786X/13/$18 · doi: 10.1117/12.2004528

module was used for the first time in order to replace bulky and costly laboratory instrumentation. A noise equivalent concentration (NEC) of 130 pptv (1σ) in dry nitrogen for a 1 second lock-in time constant was achieved at a pressure of 200 Torr, due to low electrical and optical noise, and the high sensitivity of the TEC Mercury-Cadmium-Telluride (MCT) detector (VIGO PVI-4TE-4).

2. Optical sensor architecture

The C_2H_6 optical sensor, depicted in Fig. 1, uses a 3.36 µm CW TEC GaInAsSb/AlGaInAsSb DFB laser diode as a spectroscopic source to target the optimum C_2H_6 absorption line located at 2976.8 cm-1. The DFB laser diode is packaged in a TO5 can. Its output beam is collimated by a Black Diamond™ aspheric lens, CL, (Thorlabs, model C036TME-E) and focused by a second lens, L, by using a 200 mm focal length plano-convex CaF2 lens (Thorlabs LA5714-E) into the input of a novel 57.6 m optical pathlength spherical multi-pass gas absorption cell, MC, (Sentinel Photonics).

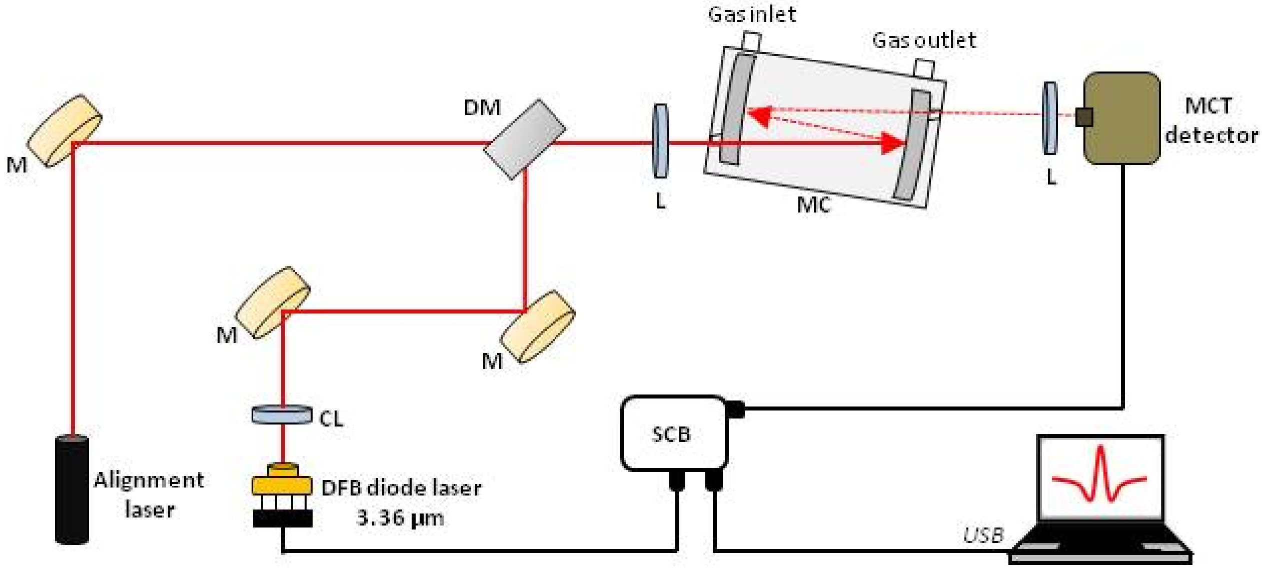

Figure 1: Schematic of a C_2H_6 gas sensor using a 3.36 µm DFB laser diode as an excitation source. M – mirror, CL – collimating lens, DM – dichroic mirror, MC – multipass cell, L – lens, SCB – sensor control board.

The DFB laser diode light intensity and voltage as function of injected current (LIV) curve, experimentally determined, are presented in figure 2(a), and its temperature and current tuning are shown in figure 2(b). Experimentally, and based on figure 2(b), the DFB laser diode current and temperature tuning coefficients are -0.022 cm^{-1} /mA and -0.26 cm^{-1} /ºC, respectively. The novel ultra-compact multipass cell is formed by 2 dielectric-coated spherical glass substrates. The reflectivity provided by these substrates exceeds 99.5%. Based on the spherical Herriott cell concept with two coaxial spherical mirrors, and with a distance of 13 cm between the mirrors providing a minimal spot overlap to minimize etalon fringe effects, 459 laser beam passes are created inside the multipass cell, giving an effective optical path length of 57.6 m. The spot pattern is shown in Fig. 3.

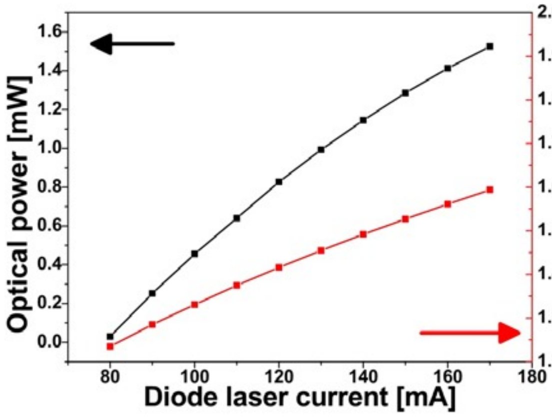

Figure 2(a): Light intensity vs. current and voltage (LIV) curve for the 3.36 µm CW TEC DFB laser diode operating at 10°C.

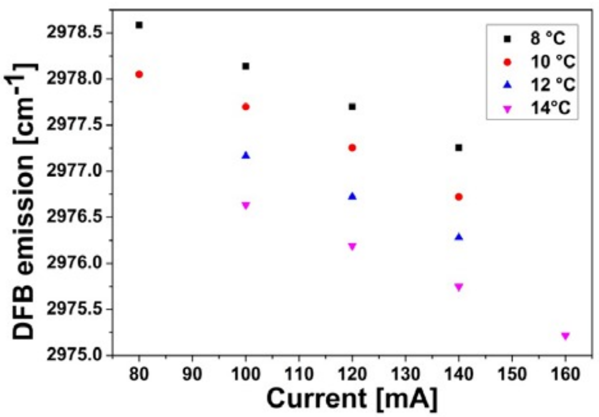

Figure 2(b): Temperature and current tuning characteristics of CW TEC DFB laser diode.

Figure 3: The novel ultra-compact multipass cell spot pattern.

The output DFB laser diode beam is focused by a 75 mm focal length plano-convex CaF2 lens, L, (Thorlabs LA5042-E) onto a TEC Mercury-Cadmium-Telluride (MCT) detector (VIGO PVI-4TE-4). Ultra-compact electronics provided by Sentinel Photonics, designated as the sensor control board in Fig. 1 (SCB) was used in order to provide the diode laser current and temperature control. The TEC of the MCT detector is also connected to the SCB for signal data acquisition. A photo of the SCB, the multipass cell, and the MCT detector are depicted in Fig. 4. The entire optical sensor alignment was realized by adding a visible semiconductor laser diode beam (λ = 630 nm). This beam was combined with the mid-IR beam by using a dichroic mirror (ISO Optics, model BSP-DI-25-3). The processing unit used for the sensor control board (SCB) is based on a TI MSP430. The laser diode temperature and TEC are controlled by thermistor sensing and the power output, respectively. In addition, the SCB provides laser diode current drive and modulation. The SCB board dimensions are 70mm × 50mm × 10mm. During wavelength modulation performance, the data is sampled via an embedded analog-to-digital converter synchronously and characteristic absorption spectra are produced by a digital lock-in amplifier algorithm. The SCP can also synchronously apply a continuous saw-tooth current ramping at 8 Hz which is the 32-bit lock-in amplifier signal. Therefore, the total control and acquisition systems power consumption to generate wavelength modulated ramp spectra is less than 0.4W.

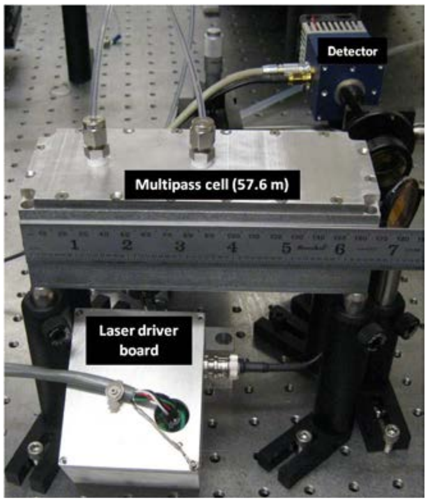

Figure 4: Photo of the novel spherical compact multipass gas absorption cell, the laser driver module and the MCT detector.

3. C_2H_6 sensor tests

The targeted C_2H_6 absorption line located at 2976.8 cm^{-1} is detected by setting the laser diode temperature and injected current to 9.5 °C and 136.7 mA, respectively. Wavelength scanning across the C_2H_6 absorption line is performed with 8 Hz of continuous saw-tooth current ramping provided by the SCB. In addition, a sinusoidal modulation of 16 KHz was superimposed on the ramping signal with amplitude of 15.6 mA. These parameters are required for the 2f WMS detection technique. A vacuum pump was connected to the multipass cell in order to operate at a cell pressure of 200 Torr. The absorption spectrum for a mixture consisting of 100 ppbv C_2H_6, 1.8 ppmv CH4 and 2% H2O for an interaction length of 57.6 m at a temperature of 23 °C and at pressure of 200 Torr was computed using Hitran [7] (Fig. 5).

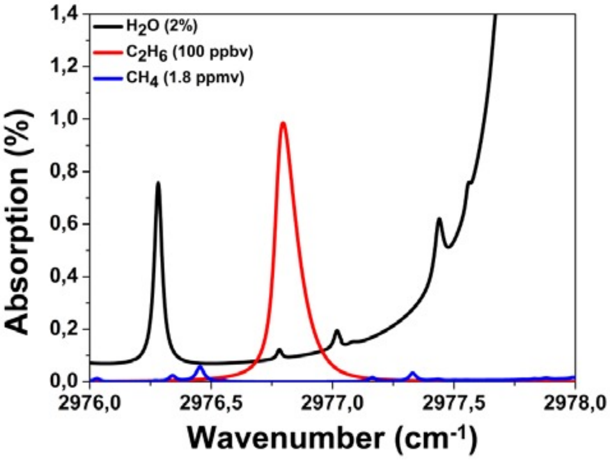

Figure 5: Simulated HITRAN spectra of C_2H_6 (100 ppbv), CH_4 (1.8 ppmv) and H_2O (2%) (T=23 °C; p=200 Torr, L=57.6 m).

The ethane line located at 2976.8 cm^{-1} is sufficiently isolated and free from interferences from other gases present in the ambient air such as CO2, CH4 and H2O at the pressure of 200 Torr asis evident in Fig. 5. This rotational-vibrational transition was therefore chosen for this study. The mutlipass cell was filled with a calibrated mixture of 100 ppbv C_2H_6 in nitrogen (N2). The 2f signal of 100 ppbv C_2H_6 and the baseline which is determined after filling the multipass cell with pure N2 at a pressure of 200 Torr are presented in Fig. 6.

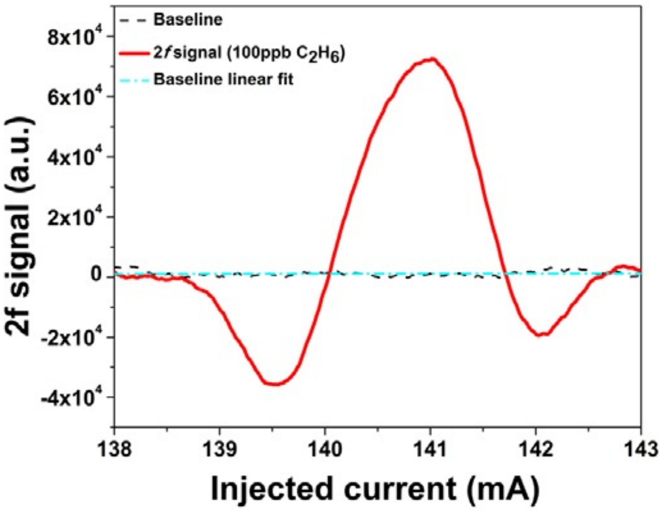

Figure 6: 2*f* WMS signal for a C_2H_6 absorption line located at 2976.8 cm^{-1} (p=200 Torr).

A low noise C_2H_6 sensor baseline was achieved by using a single driver module for laser diode and data acquisition. A signal-to-noise ratio (SNR) of 770 was obtained, yielding a NEC of 130 pptv (1σ) for 1 s sample averaging time.
To assess the performance of the TDLAS-WMS sensor for the detection of C_2H_6, we studied its linearity by plotting the 2f amplitude signal as measured by e lock-in detection as a function of the calibrated concentration of ethane in the multipass gas cell (at 5, 10, 15, 25, 50 and 100 ppbv concentration levels). Gas dilution was realized by using a commercial gas mixer (Gas Dilution System -series 4040} from Environics. Figure 7 depicts the response of the C_2H_6 sensor versus concentration fitted with a linear slope.

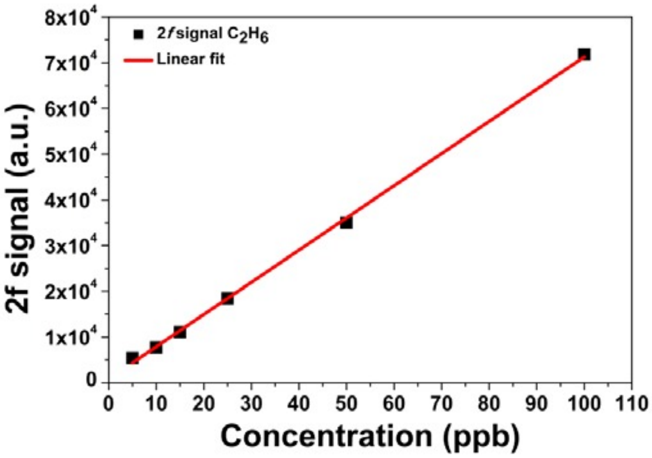

Figure 7: 2*f* WMS amplitude signal as function of the C_2H_6 concentration.

The perfect match between the linear slope and the 2f WMS amplitude signal as function of C_2H_6 concentration confirm the linearity of the TDLAS-WMS C_2H_6 sensor. Also, the long term stability of the TDLAS-WMS C_2H_6 sensor was studied by using Allan variance measurements (Fig. 8). The 100 ppbv C_2H_6 2f signals were detected and averaged in order to determine the Allan plot.

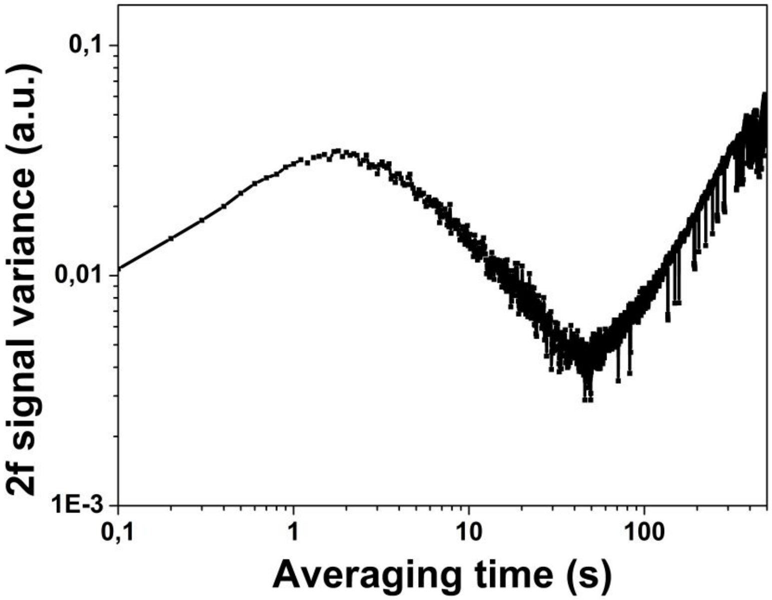

Figure 8: Measured Allan variance for 2*f* signal corresponding to absorption of 100 ppbv of C_2H_6.

The optimum detection sensitivity is obtained by averaging the TDLAS-WMS C_2H_6 signal up to 35 sec. The average of the accumulated 2f spectra yields an increase initially, which is the direct result of filter time constant reduction of short term noise present in the acquired data. By adding a reference channel and/or incorporating line-locking functionality, a further improvement of the TDLAS-WMS C_2H_6 sensor can be obtained.

4. Summary

A TDLAS-WMS sensor based on a 3.36 µm CW TEC DFB laser diode was implemented to detect C_2H_6 of interest in environmental, industrial and medical applications. A novel multipass gas cell and sensor control board was used in order to realize a compact gas sensor. The multlipass gas cell is composed of two dielectric-coated spherical glass substrates separated by 12.5 cm giving an effective optical path-length of 57.6 m. An optimum and interference-free C_2H_6 absorption line located at 2976.8 cm^{-1} was detected, leading to an NEC of 130 pptv (1σ) due to the use of a low noise custom built SCB and a state-of-the-art VIGO MCT detector By implementing line-locking functionality, the stability of the TDLAS-WMS C_2H_6 sensor will be improved and further miniaturization of the sensor architecture an ultra-compact and robust sensitive and selective C_2H_6 sensor can be realized.

Acknowledgements

The Rice University group acknowledges financial support from a National Science Foundation (NSF) grant EEC-0540832 entitled “Mid-Infrared Technologies for Health and the Environment (MIRTHE)”, a NSF-ANR award for international collaboration in chemistry “Next generation of Compact Infrared Laser based Sensor for environmental monitoring (NexCILAS)” and grant C-0586 from the Robert Welch Foundation.

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