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Mid-infrared semiconductor laser based trace gas analyzers: advances, applications & future outlook

S. So [1a], R. Lewicki[1,2],
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RICE

sentinel photonics

OUTLINE

Laser Optics 2014

Philadelphia, PA

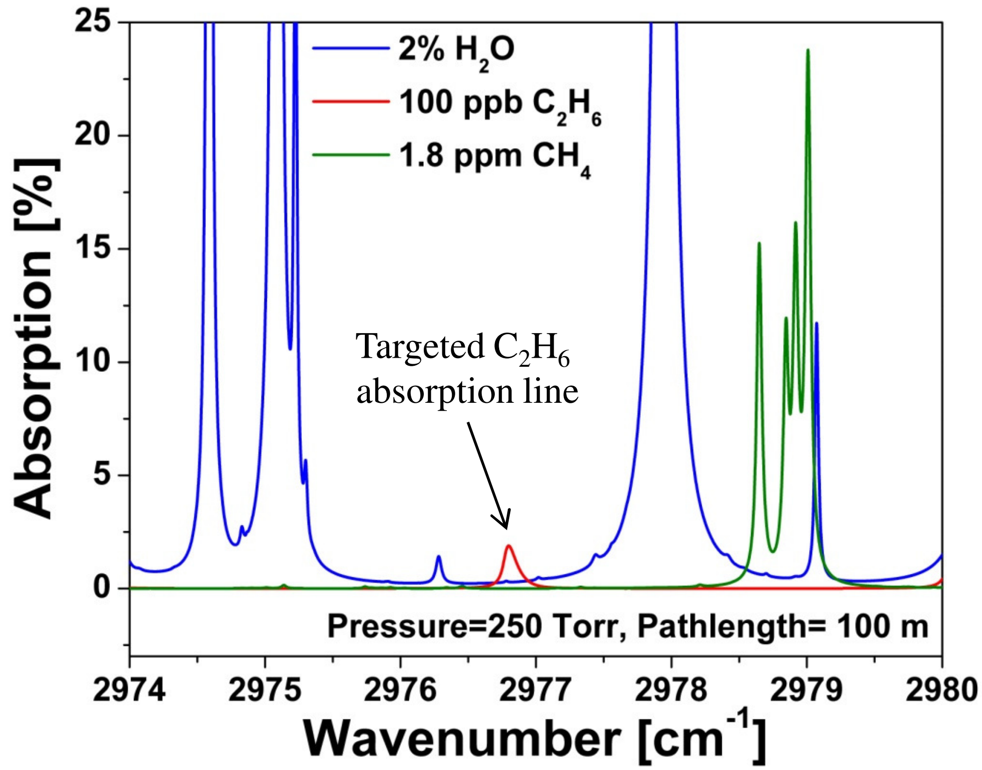
September, 8-10, 2014

- New Laser Based Trace Gas Sensor Technology
  - Novel Multipass Absorption Cell & Electronics
  - Quartz Enhanced Photoacoustic Spectroscopy
- Examples of Mid-Infrared Sensor Architectures
  - C_2H_6, NO, CO and CH_4
  - Future Directions of Laser Based Gas Sensor Technology and Conclusions

Research support by NSF ERC MIRTHE, NSF-ANR NexCILAS, the Robert Welch Foundation, and Sentinel Photonics Inc. via an EPA and NSF SBIR sub-award is acknowledged

Motivation for Mid-infrared C_2H_6 Detection

- Atmospheric chemistry and climate
  - Fossil fuel and biofuel consumption,
  - biomass burning,
  - vegetation/soil,
  - natural gas loss
- Oil and gas prospecting
- Application in medical breath analysis (a non-invasive method to identify and monitor different diseases):
  - asthma,
  - schizophrenia,
  - Lung cancer,
  - vitamin E deficiency.



HITRAN absorption spectra of C_2H_6, CH_4, and H_2O

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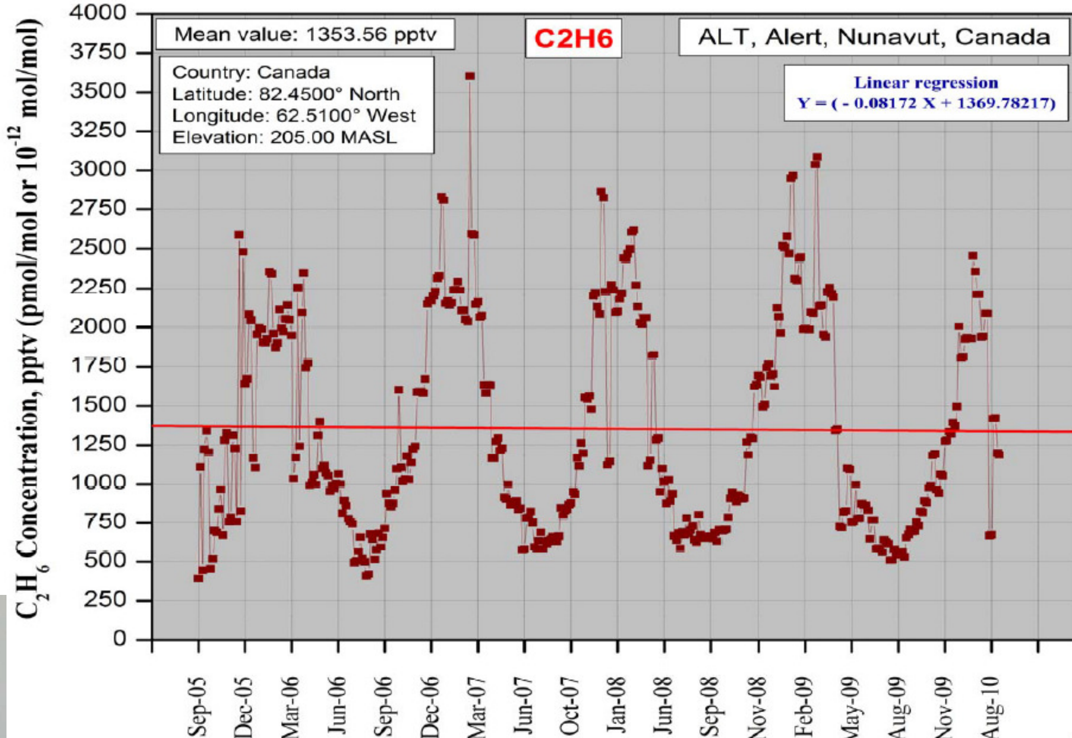
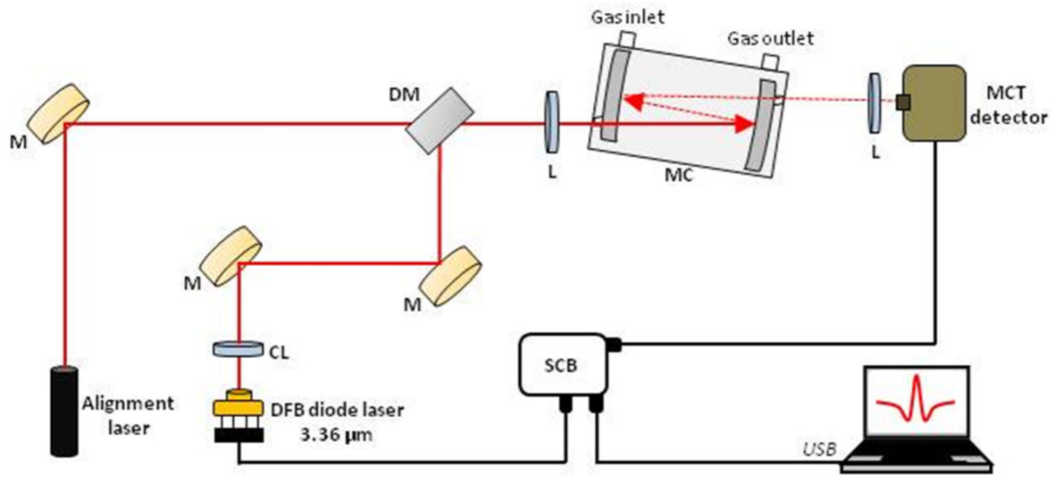
NOAA Monitoring & Sampling Location: Alert, Nunavut, Canada

ALT, Ethane Concentration Measurements

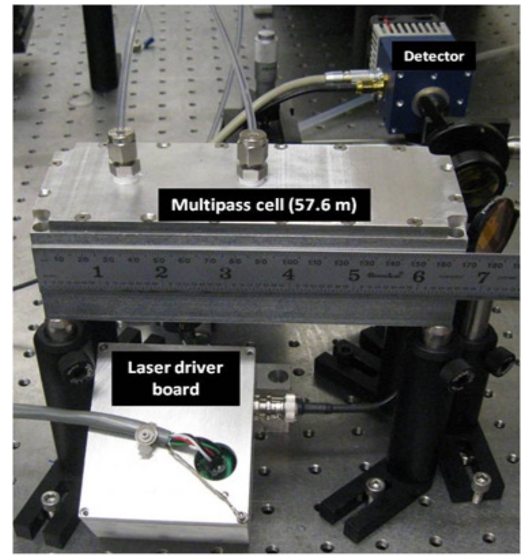


General View on the Facility
Latitude: 82.4508º North
Longitude: 62.5056º West
Elevation: 200.00 m

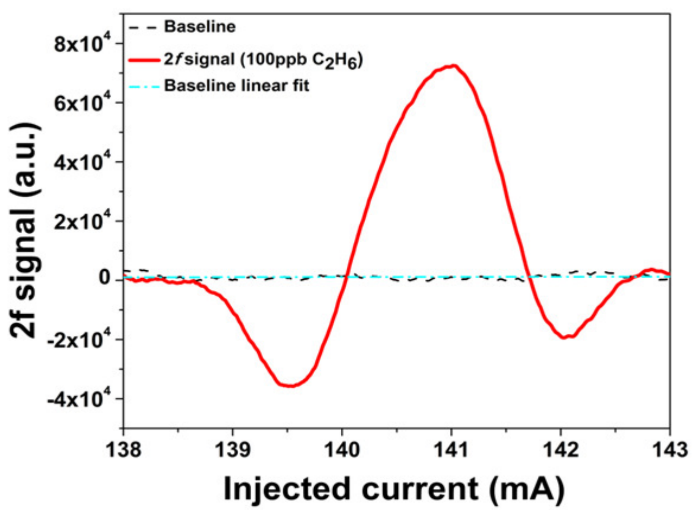
C_2H_6 Detection with a 3.36 µm CW DFB LD using a Novel Compact Multipass Absorption Cell and Control Electronics



Schematic of a C_2H_6 gas sensor using a Nanoplus 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.

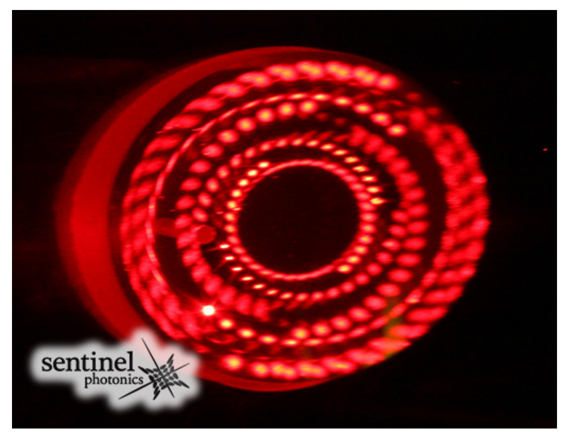


Innovative long path, small volume multipass gas cell: 57.6m with 459 passes



2f WMS signal for a C_2H_6 line at 2976.8 cm^{-1} at a pressure of 200 Torr

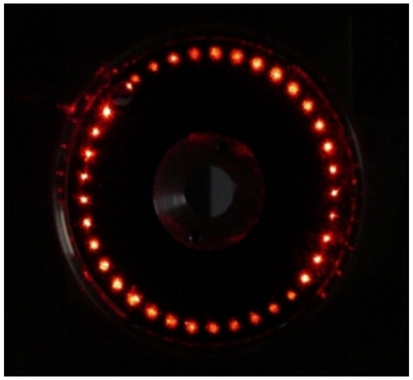
Minimum detectable C_2H_6 concentration is:
~ 740 pptv (1σ; 1 s time resolution)



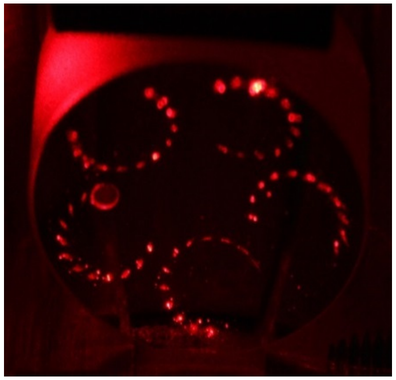
MPC dimensions: **17** x **6.5** x **5.5** (cm)
Distance between the MPC mirrors: 12.5 cm

MULTIPASS CELL TECHNOLOGY

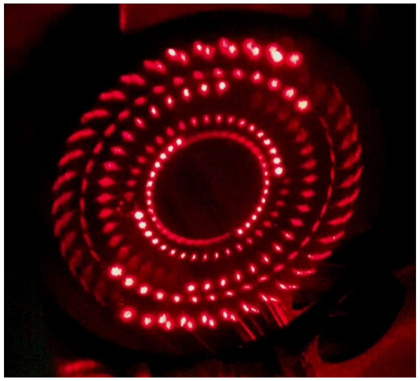
- High pathlength/volume ratio
- Simple spherical mirrors
- Utilize entire mirror surface - embrace optical aberration
- Flexible design – two opposing mirrors
- Typ. 10% throughput for 459 passes



Herriott Cell Pattern



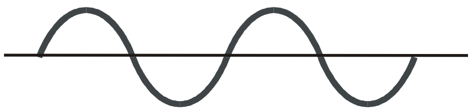
Sentinel 3.7 m cell



Sentinel 57 m cell

From Conventional PAS to QEPAS

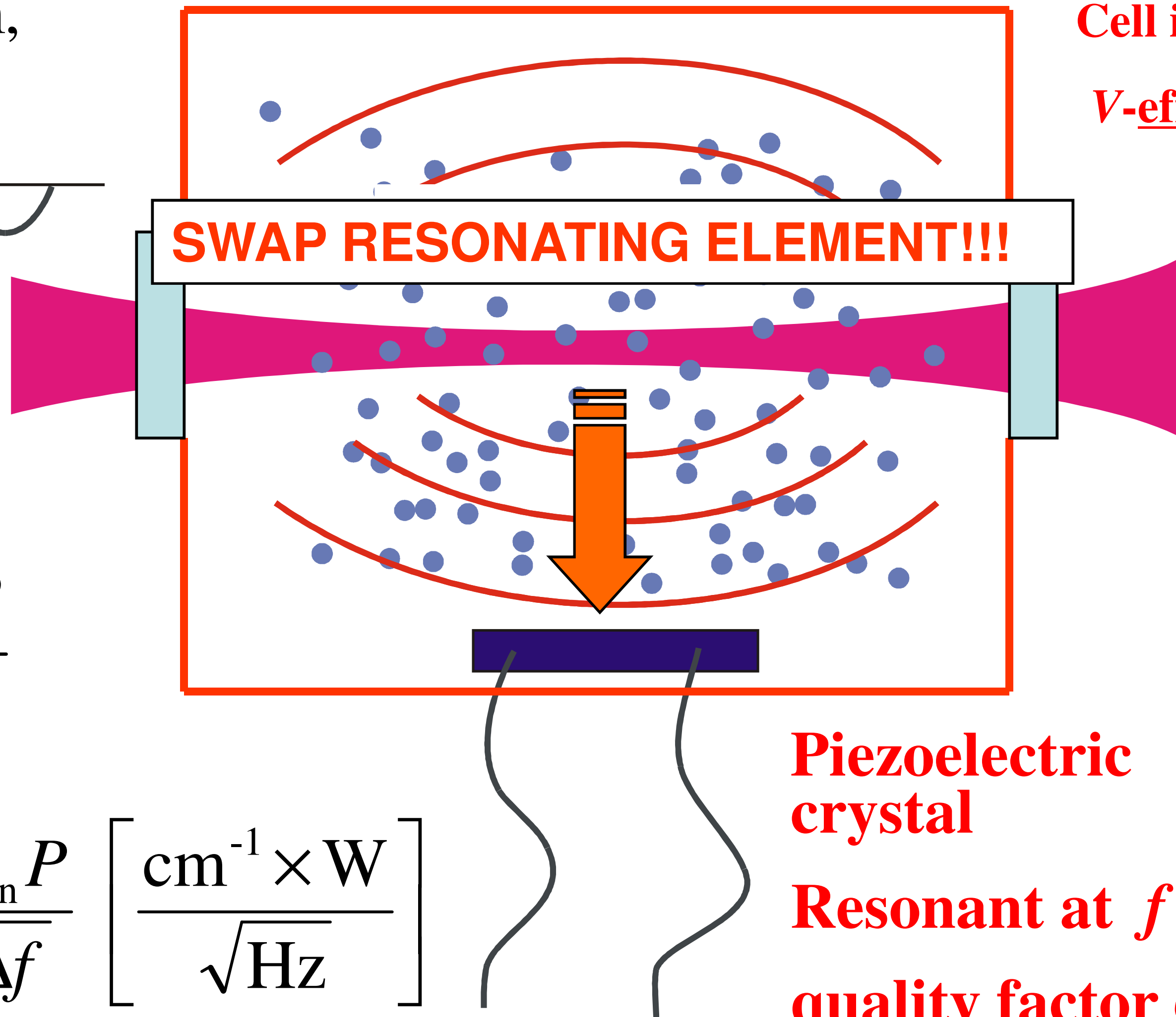
Laser beam, power P



Modulated (P or λ) at f or $f/2$

$$S \sim \frac{Q\,\alpha\,P}{f\,V}$$

$$NNEA = \frac{\alpha_{min} P}{\sqrt{\Delta f}} \left[\frac{\text{cm}^{-1} \times \text{W}}{\sqrt{\text{Hz}}} \right]$$



***Q*>>1000**

Cell is OPTIONAL!

***V*-effective volume**

Piezoelectric crystal

Resonant at *f*

quality factor *Q*

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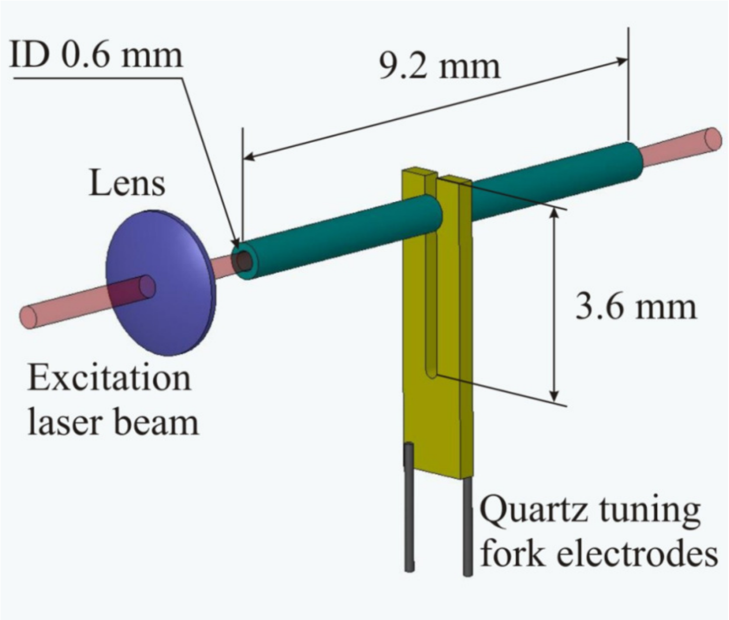
Quartz Tuning Fork as a Resonant Microphone for QEPAS



Unique properties

- Extremely low internal losses:
  - Q~10 000 at 1 atm
  - Q~100 000 in vacuum
- Acoustic quadrupole geometry
  - Low sensitivity to external sound
- Large dynamic range (~10^6) – linear from thermal noise to breakdown deformation
  - 300K noise: x~10^{-11} cm
  - Breakdown: x~10^{-2} cm
- Wide temperature range: from 1.6K to ~700K

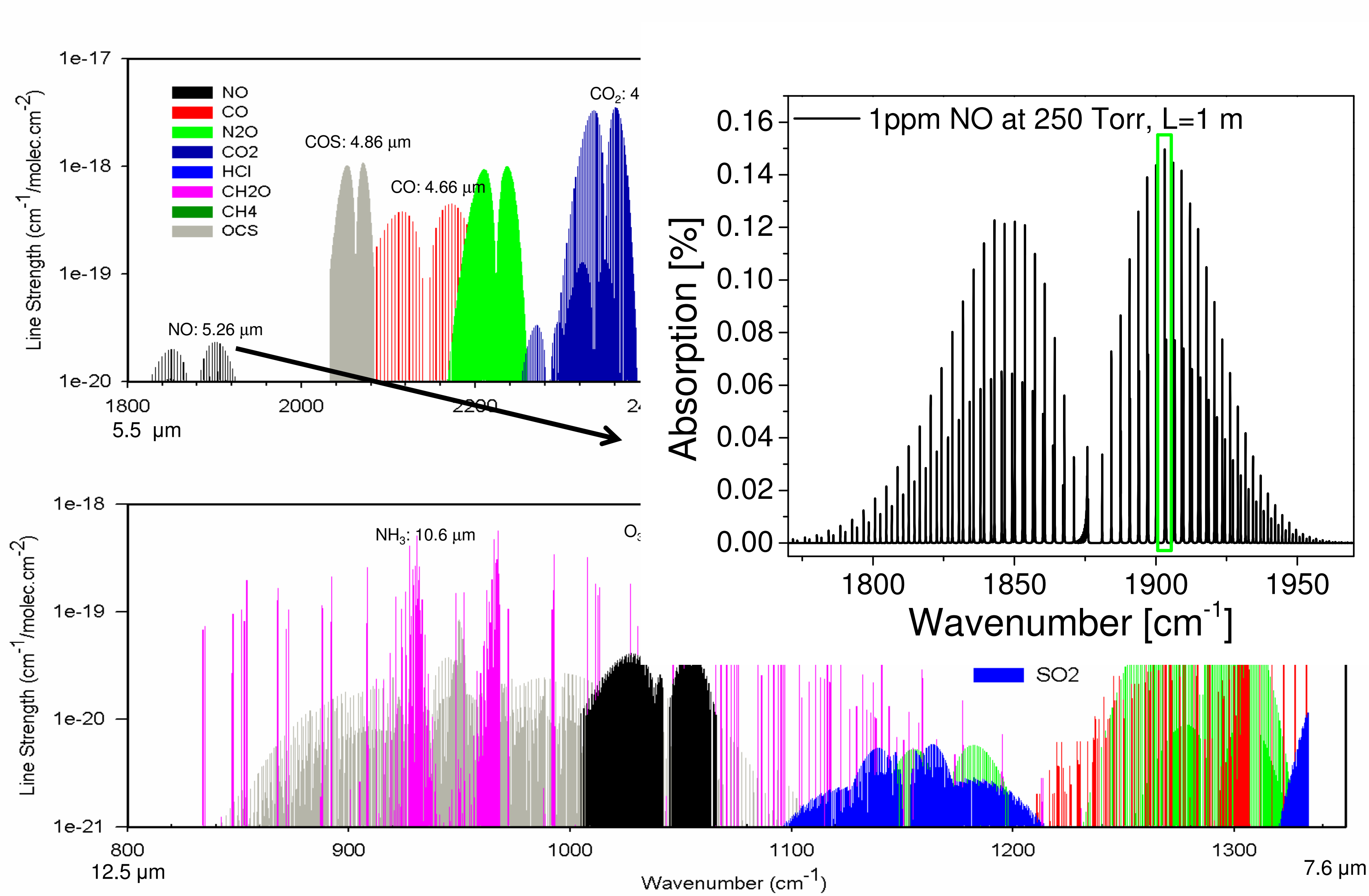
Acoustic Micro-resonator (mR) tubes

- Optimum inner diameter:0.6 mm; mR-QTF gap is 25-50 μm
- Optimum mR tubes must be ~ 4.4 mm long (~λ/4<l<λ/2 for sound at 32.8 kHz)
- SNR of QTF with mR tubes: ×30 (depending on gas composition and pressure)

Motivation for Nitric Oxide Detection

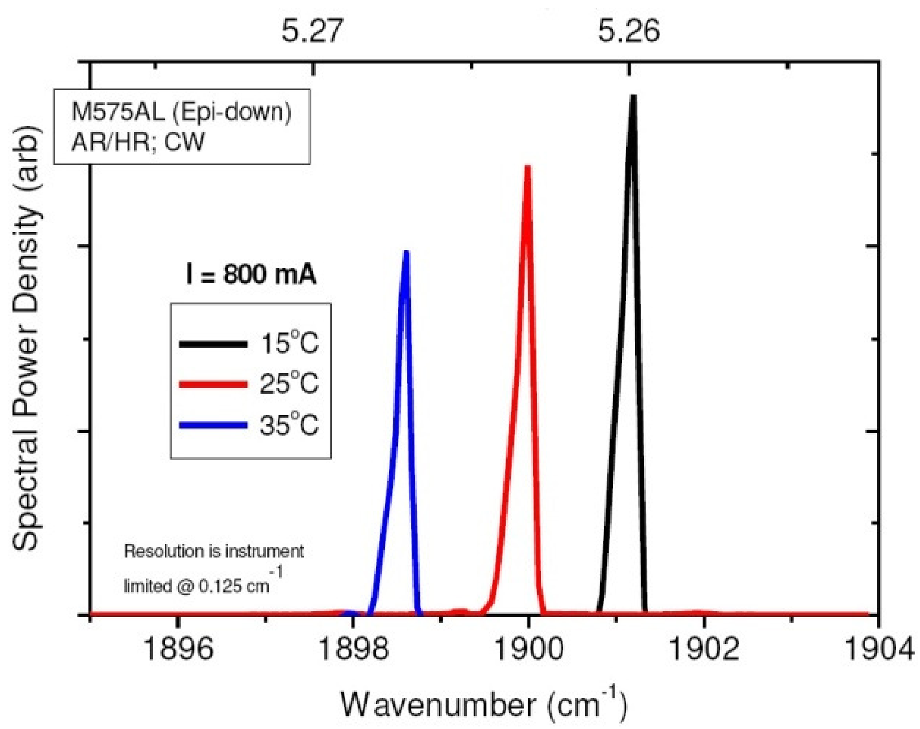
- Atmospheric Chemistry
- Environmental pollutant gas monitoring
  - NO_x monitoring from automobile exhaust and power plant emissions
  - Precursor of smog and acid rain
- Industrial process control
  - Formation of oxynitride gates in CMOS Devices
- NO in medicine and biology
  - Important signaling molecule in physiological processes in humans and mammals (1998 Nobel Prize in Physiology/Medicine)
  - Treatment of asthma, COPD, acute lung rejection
- Photofragmentation of nitro-based explosives

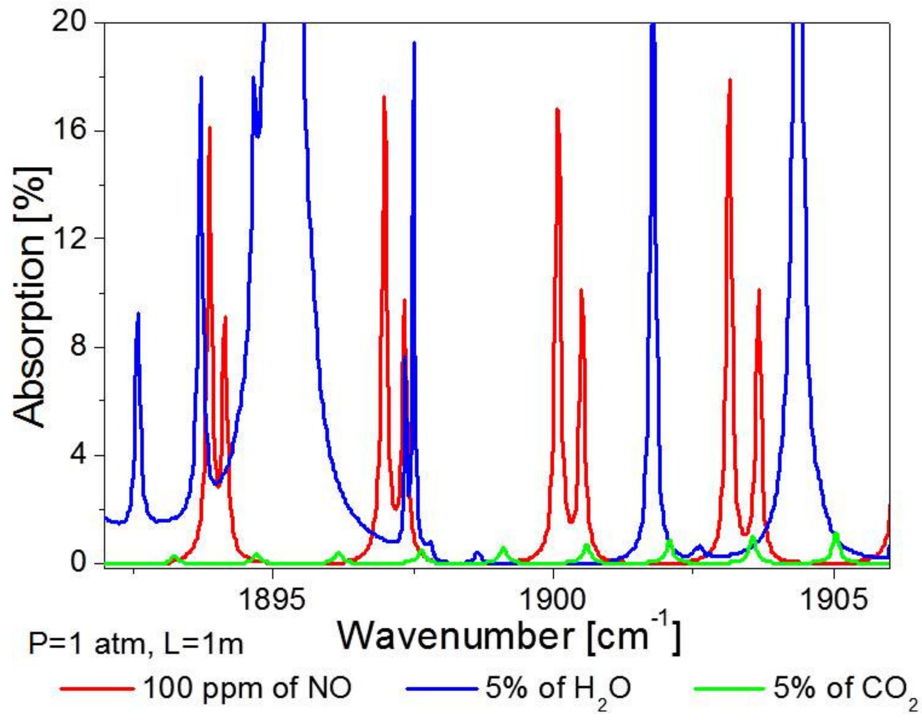
Molecular Absorption Spectra within two Mid-IR Atmospheric Windows and NO absorption @ 5.26µm



Source: HITRAN 2000 database

Emission spectra of a 1900cm^{-1} TEC CW DFB QCL and HITRAN Simulated spectra





Output power: 117 mW @ 25 C
Thorlabs/Maxion

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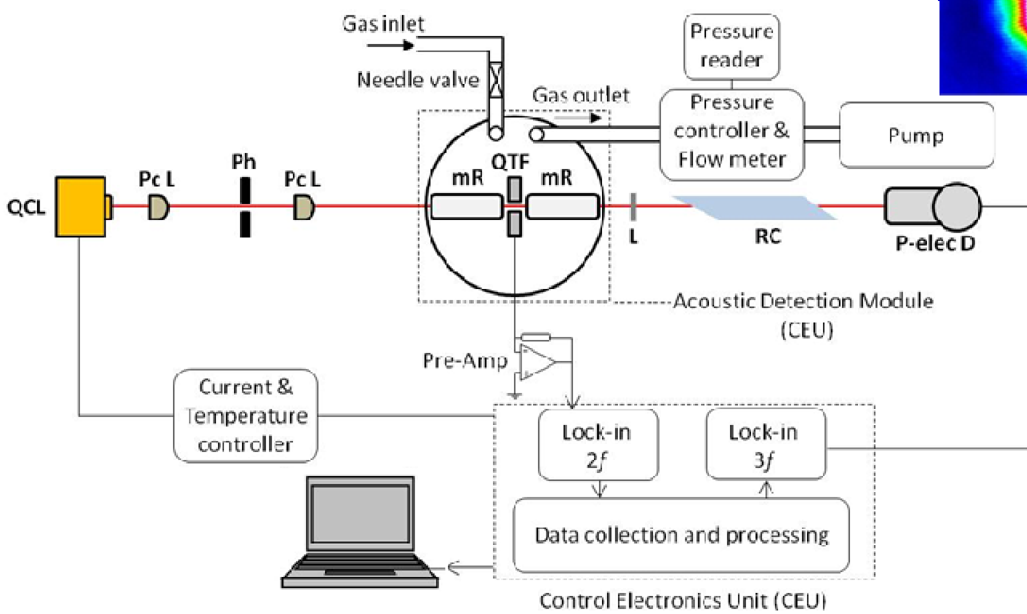
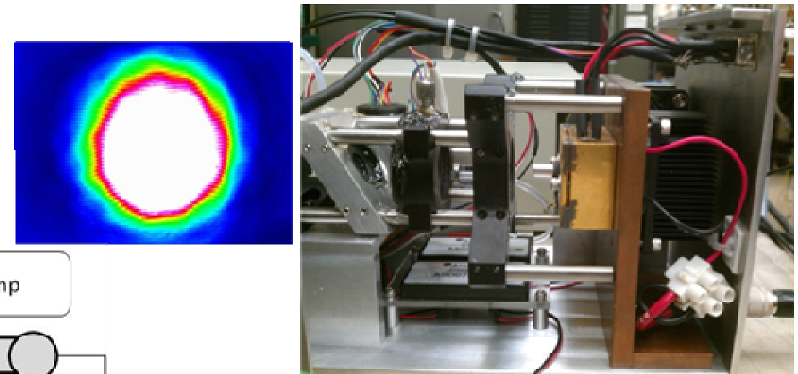
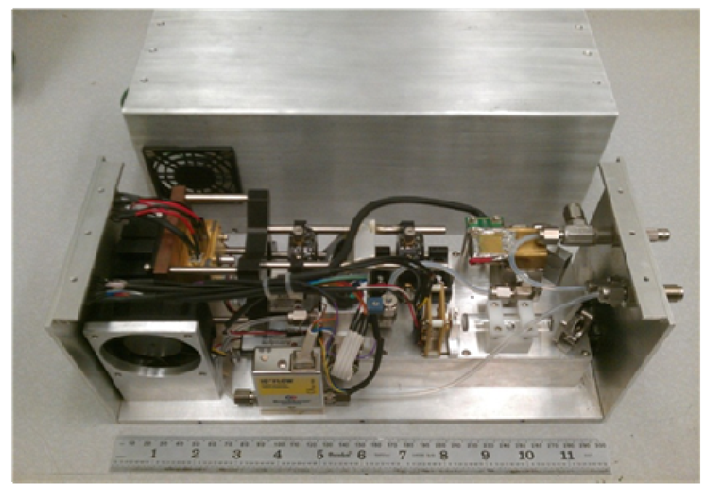
CW TEC DFB QCL based QEPAS NO Gas Sensor



Schematic of a DFB-QCL based Gas Sensor.
PcL – plano-convex lens, Ph – pinhole,
QTF – quartz tuning fork, mR – microresonator,
RC- reference cell, P-elec D – pyro electric detector



CW HHL TEC DFB-QCL package and IR camera image of the laser beam at 630 mA and 20.5 deg C through tubes after ADM



Compact Prototype NO Sensor (September 2012)

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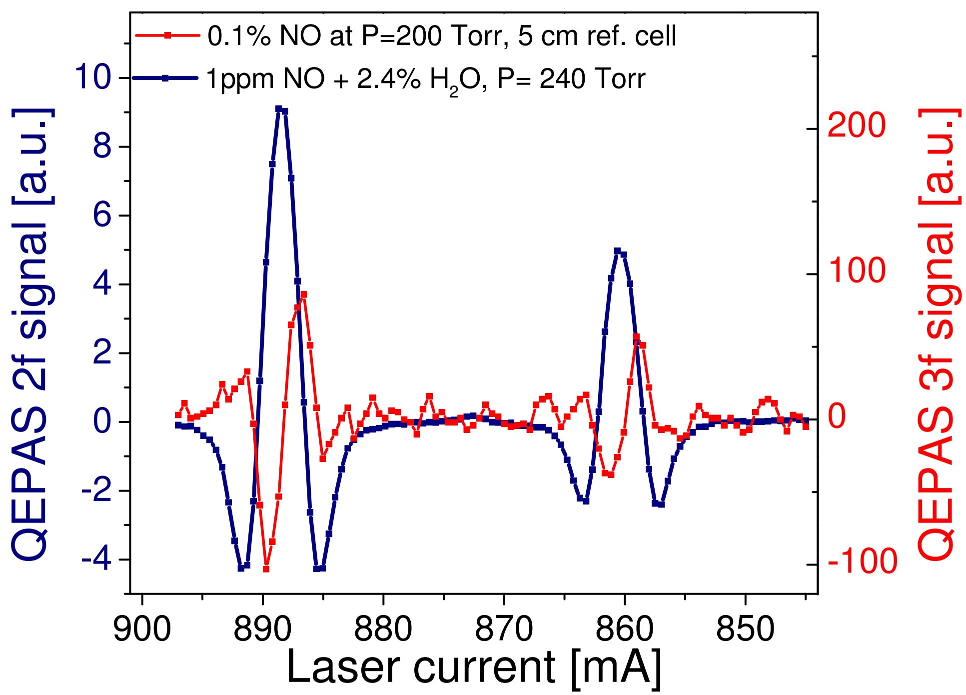
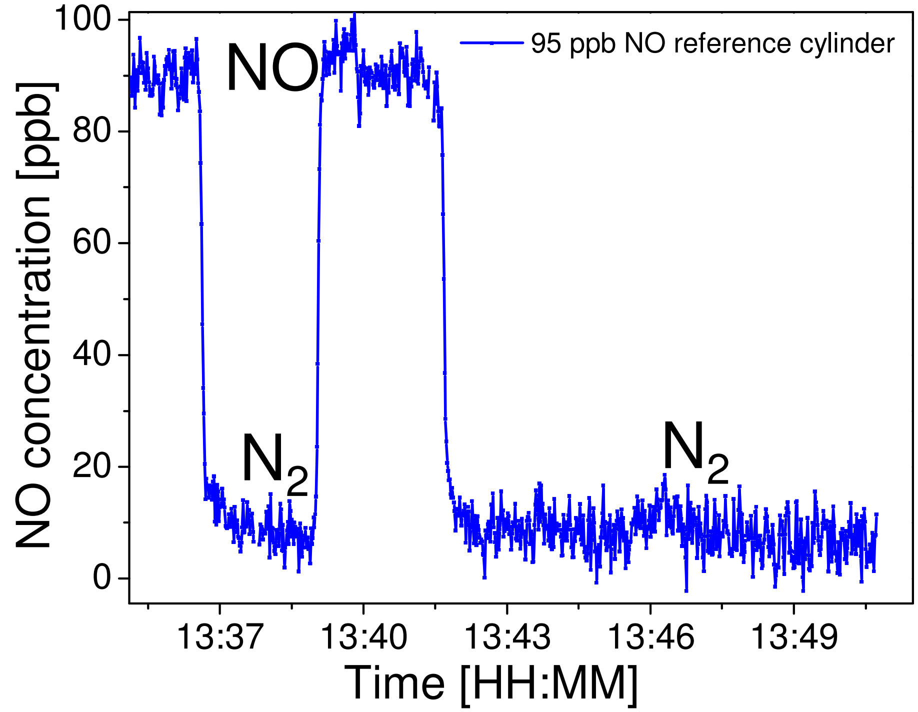
Performance of CW DFB-QCL based WMS QEPAS NO Sensor Platform



2f QEPAS signal (navy) and reference 3*f* signal (red) when DFB-QCL was tuned across **1900.08 cm^{-1}** NO line.



2*f* QEPAS signal amplitude for 95 ppb NO when DFB-QCL was locked to the **1900.08 cm^{-1}** line.

Minimum detectable NO concentration is:
~ 3 ppbv (1σ; 1 s time resolution)

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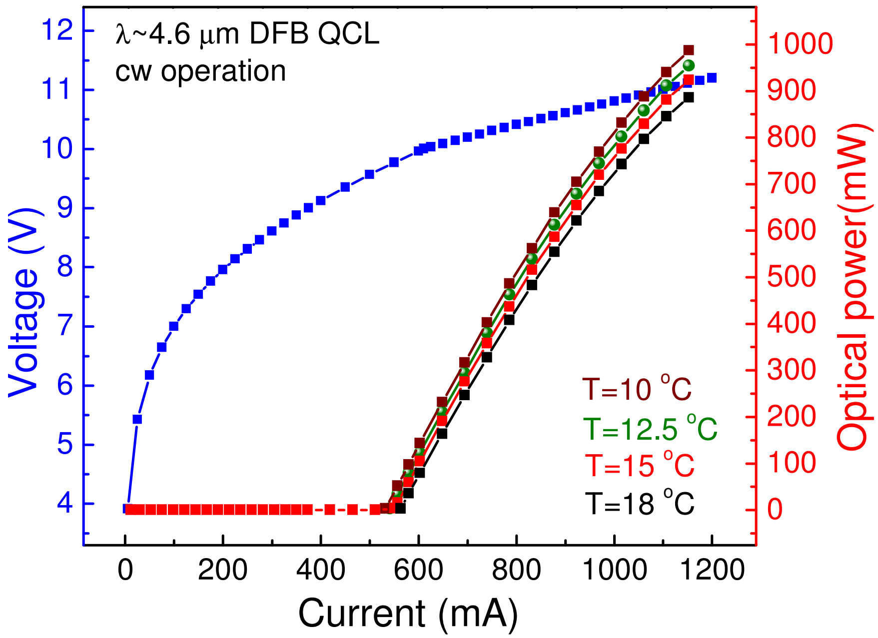
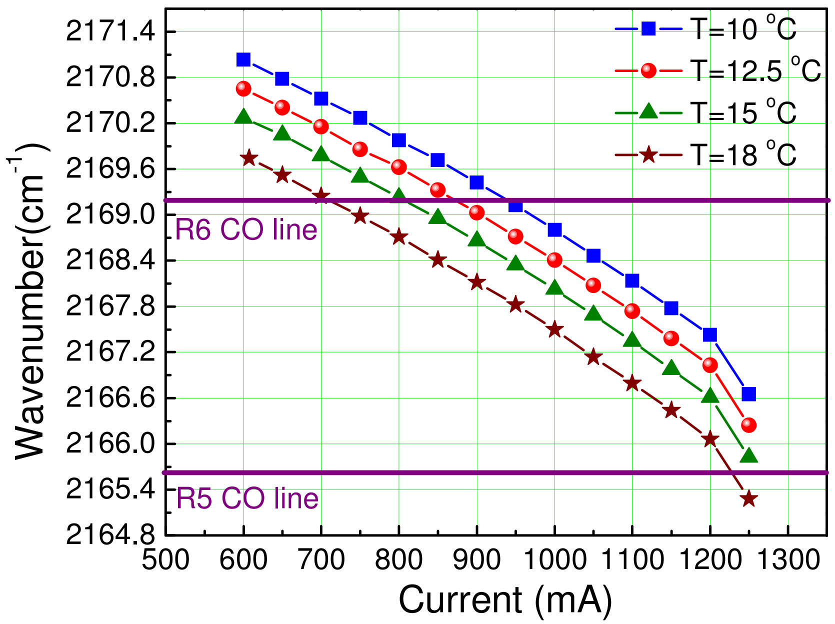
Motivation for Carbon Monoxide Detection

- Atmospheric Chemistry
  - Incomplete combustion of natural gas, fossil fuel and other carbon containing fuels.
  - Impact on atmospheric chemistry through its reaction with hydroxyl (OH) for troposphere ozone formation and changing the level of greenhouse gases (e.g. CH_4).

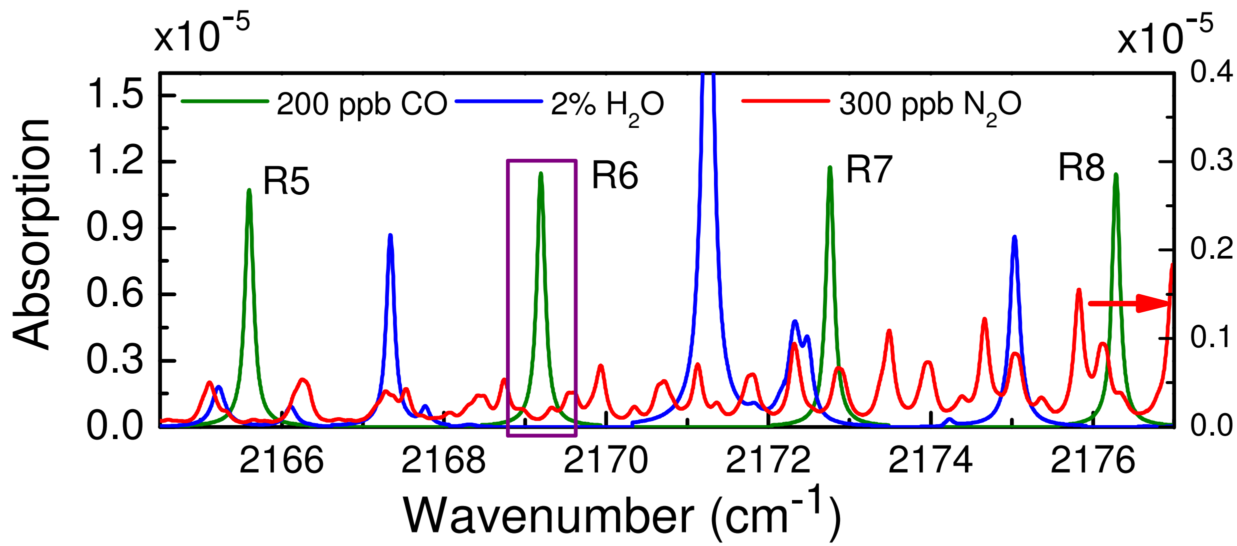
- CO in medicine and biology
  - Hypertension, neurodegenerations, heart failure and inflammation have been linked to abnormality in CO metabolism and function.

Performance of a NWU 4.61 μm high power CW TEC DFB QCL





CW DFB-QCL optical power and current tuning at a four different QCL temperatures.



Estimated max wall-plug efficiency (WPE) is ~ 7% at 1.25A QCL drive-current.

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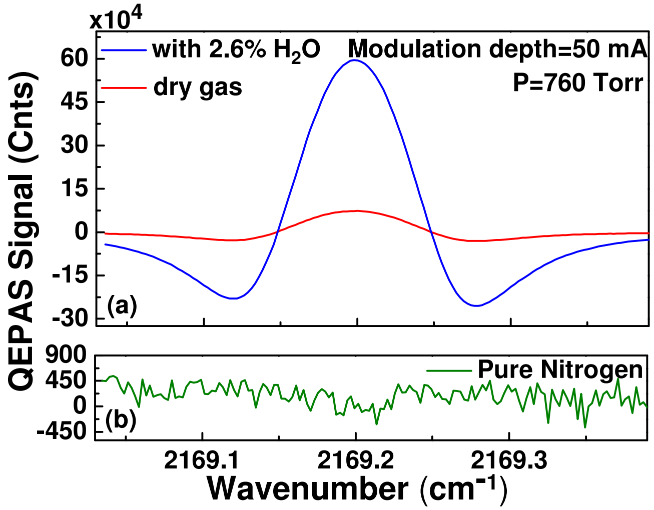
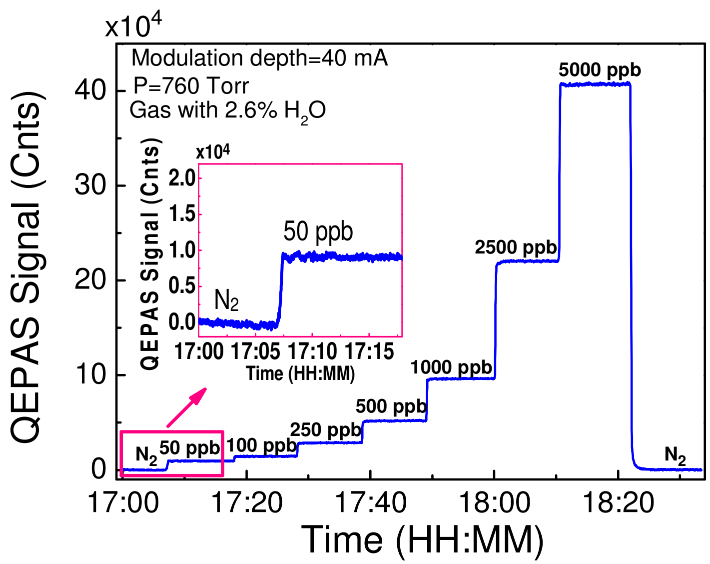
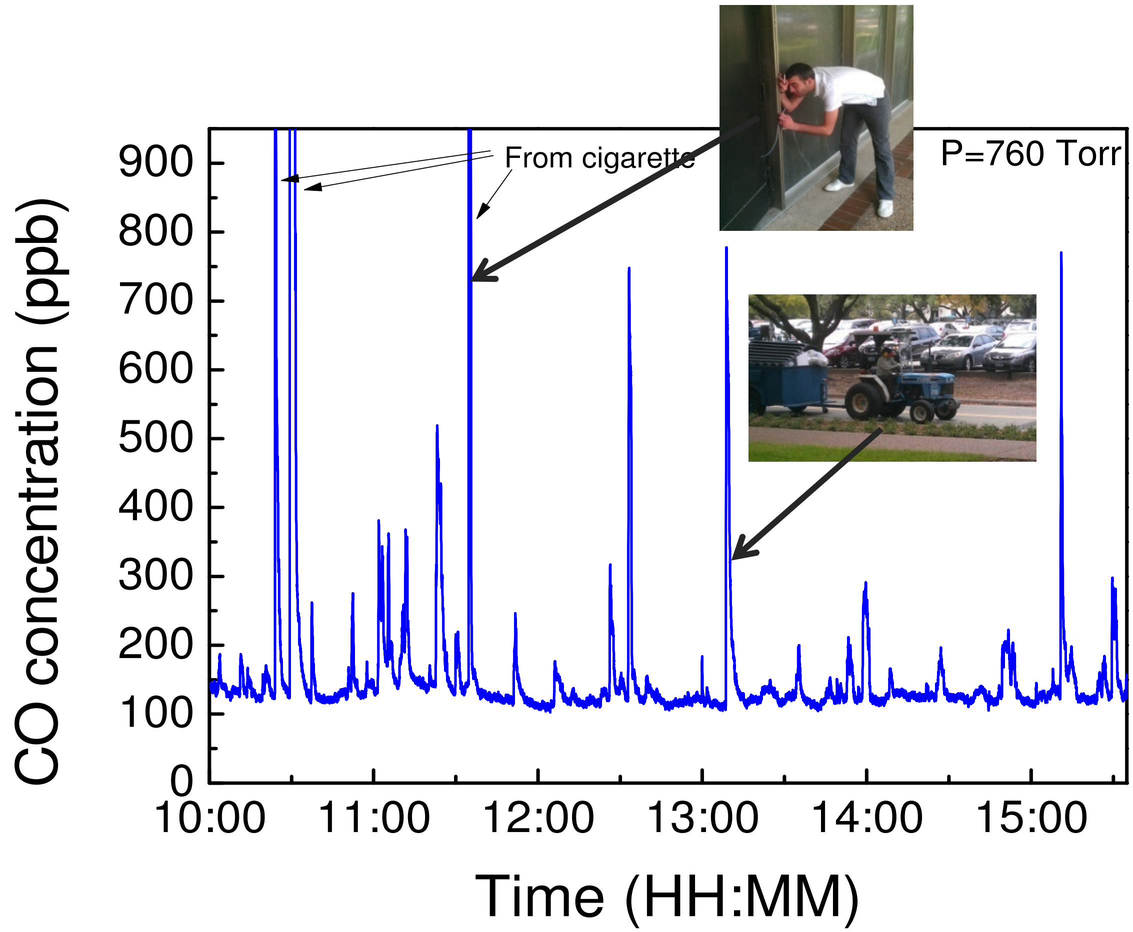
CW DFB-QCL based CO QEPAS Sensor Results



2f QEPAS signal for dry (red) and moisturized (blue) 5 ppm CO:N_2 mixture near **2169.2 cm^{-1}**.



Dilution of a 5 ppm CO reference gas mixture when the CW DFB-QCL is locked to the **2169.2 cm^{-1}** R6 CO line.



Atmospheric CO concentration levels on Rice University campus, Houston, TX

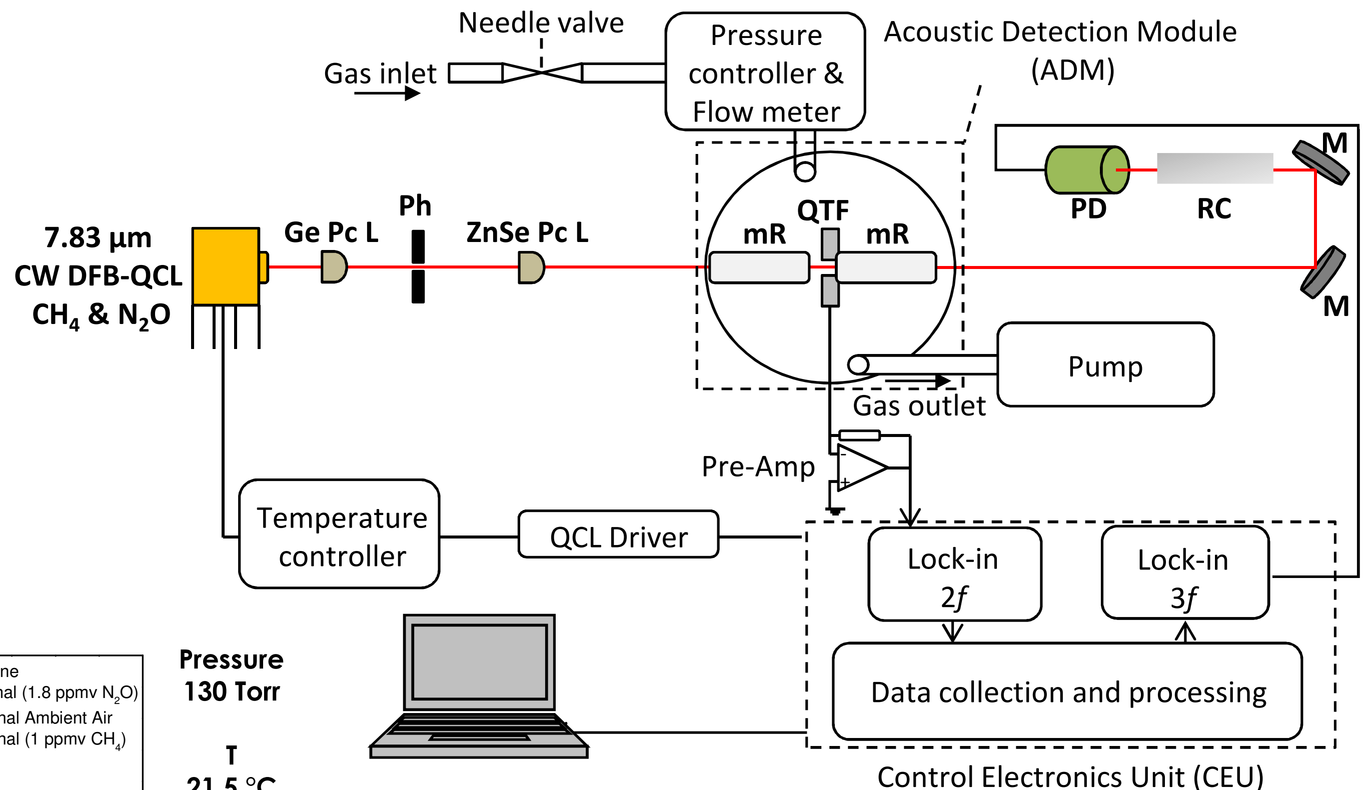
Minimum detectable CO concentration is:

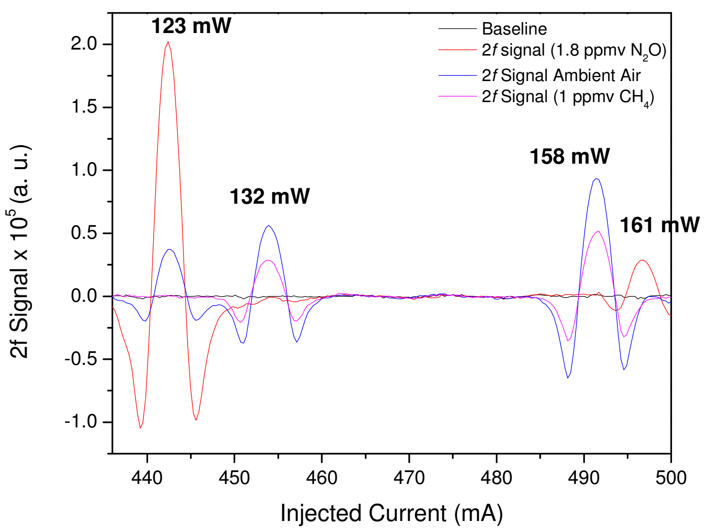
~ 2 ppbv (1σ; 1 s time resolution)

QEPAS based CH_4 and N_2O Gas Sensor

Motivation for CH_4 and N_2O Detection

- Prominent greenhouse gases
- Sources : Wetlands, leakage from natural gas systems, fossil fuel production and agriculture
- Applications: Environmental, medical and aerospace (N_2O)





Pressure 130 Torr

T 21.5 °C

AM *4 mA*

f 32760 Hz

f_{mod} 16380 Hz

Submitted to *The Analyst* Aug. 2013

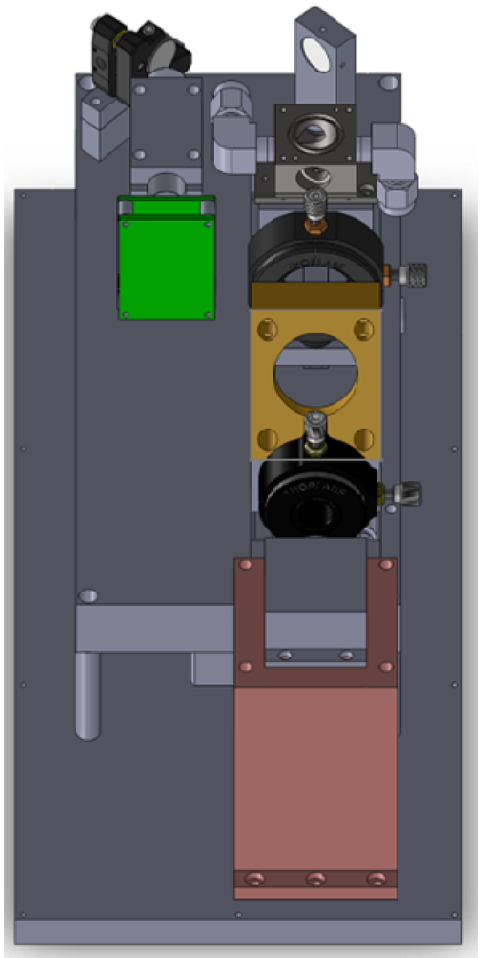
Detection Limit (1σ) with a **1-sec** averaging time

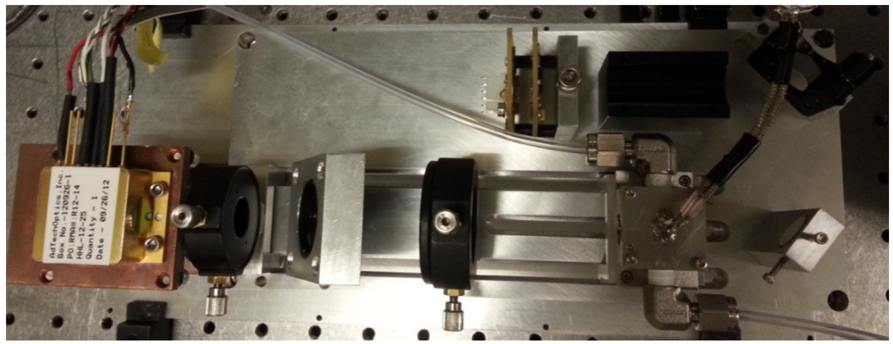
Methane (CH4) (1275.04 cm^{-1}) **13 ppbv**

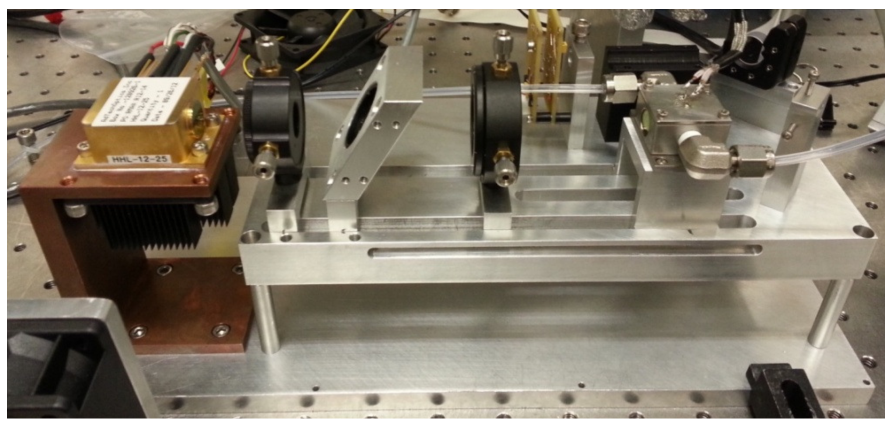
Nitrous Oxide (N_2O) (1275.5 cm^{-1}) **6 ppbv**

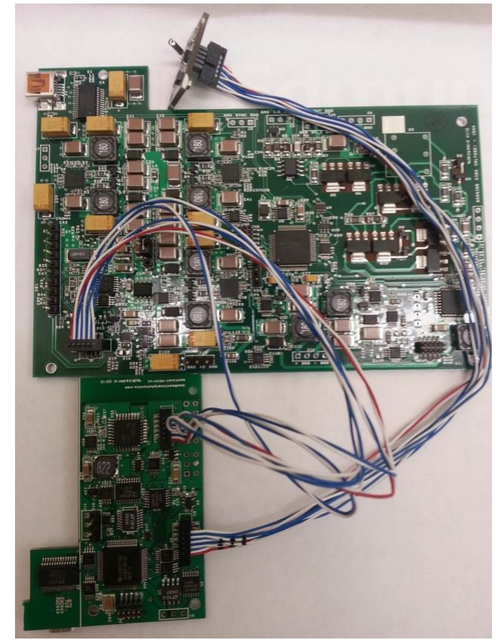
Deduced N_2O concentration in the ambient laboratory air: **331 ppbv**

QEPAS based CH_4 and N_2O Gas Sensor









QEPAS Sensor Control Board

QCL Current and TEC Driver, Performing wavelength modulation, Data acquisition, Applying continuous saw-tooth current ramping at 8 Hz, Testing QTF and low noise pre amplifier

QEPAS Performance for Trace Gas Species (September 2014)

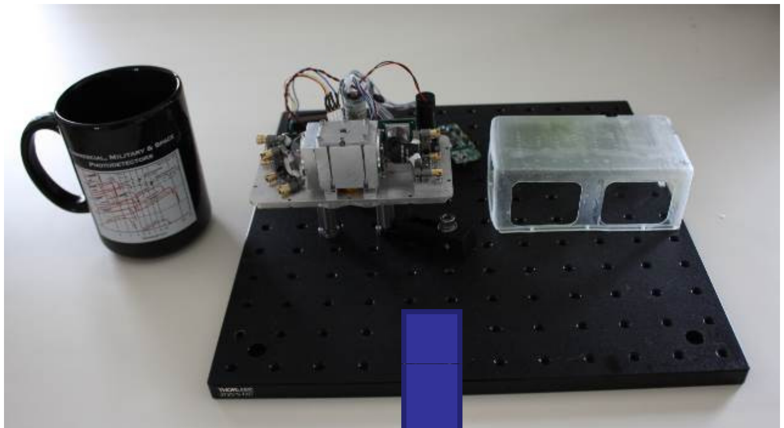
	Molecule (Host)	Frequency, cm^{-1}	Pressure, Torr	NNEA, $cm^{-1}W/Hz^{1/2}$	Power, mW	NEC (τ=1s), ppmv
VIS	**O_3 (air)**	35087.70	700	3.0×10^{-8}	0.8	1.27
	O_2 (N_2)	13099.30	158	4.74×10^{-7}	1228	13
NIR	**C_2H_2 (N_2)***	6523.88	720	4.1×10^{-9}	57	0.03
	NH_3 (N_2)*	6528.76	575	3.1×10^{-9}	60	0.06
	C_2H_4 (N_2)*	6177.07	715	5.4×10^{-9}	15	1.7
	CH_4 (N_2+1.2% H_2O)*	6057.09	760	3.7×10^{-9}	16	0.24
	N_2H_4	6470.00	700	4.1×10^{-9}	16	1
	H_2S (N_2)*	6357.63	780	5.6×10^{-9}	45	5
	HCl (N_2 dry)	5739.26	760	5.2×10^{-8}	15	0.7
	CO_2 (N_2+1.5% H2O) *	4991.26	50	1.4×10^{-8}	4.4	18
Mid-IR	**CH_2O (N_2:75% RH)***	2804.90	75	8.7×10^{-9}	7.2	0.12
	CO (N_2 +2.2% H_2O)	2176.28	100	1.4×10^{-7}	71	0.002
	CO (propylene)	2196.66	50	7.4×10^{-8}	6.5	0.14
	N_2O (air+5%SF_6)	2195.63	50	1.5×10^{-8}	19	0.007
	C_2H_5OH (N_2)**	1934.2	770	2.2×10^{-7}	10	90
	NO (N_2+H_2O)	1900.07	250	7.5×10^{-9}	100	0.003
	C_2HF_5 (N_2)***	1208.62	770	7.8×10^{-9}	6.6	0.009
	NH_3 (N_2)*	1046.39	110	1.6×10^{-8}	20	0.006
	SF_6	948.62	75	2.7×10^{-10}	18	5×10^{-5} (50 ppt)

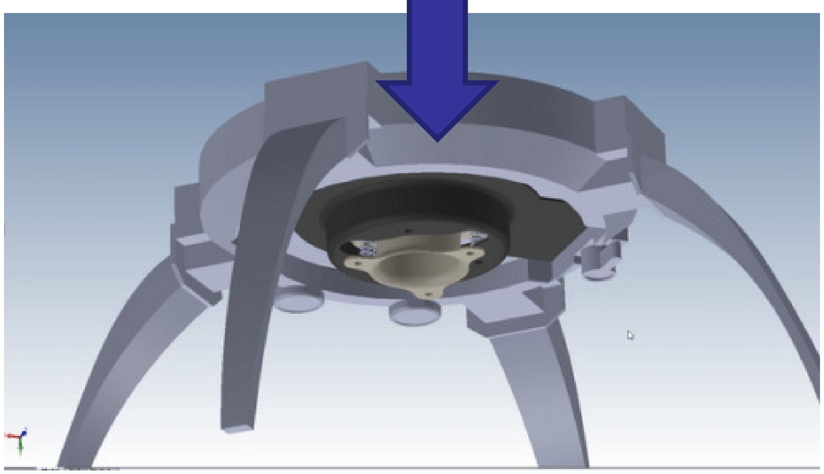
* - Improved microresonator
** - Improved microresonator and double optical pass through ADM
*** - With amplitude modulation and metal microresonator

NNEA – normalized noise equivalent absorption coefficient.
NEC – noise equivalent concentration for available laser power and τ=1s time constant, 18 dB/oct filter slope.

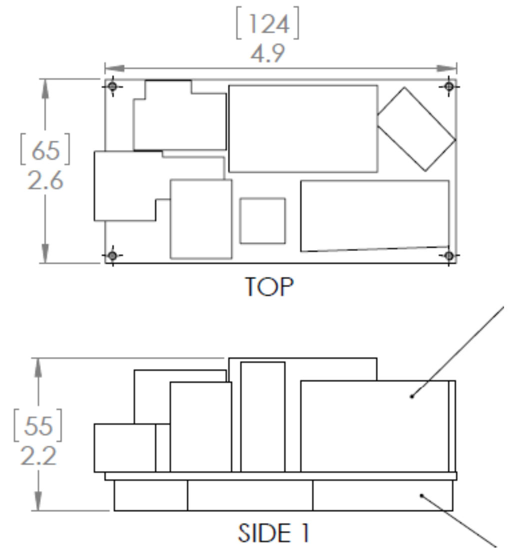
For comparison: conventional PAS 2.2 $\times10^{-9}$ $cm^{-1}W/\sqrt{Hz}$ for NH_3

Mini Methane Sensor for UAVs





Miniaturization



Remote controlled quad-copter UAV
for pipeline sniffing -
payload maximum only 600g!

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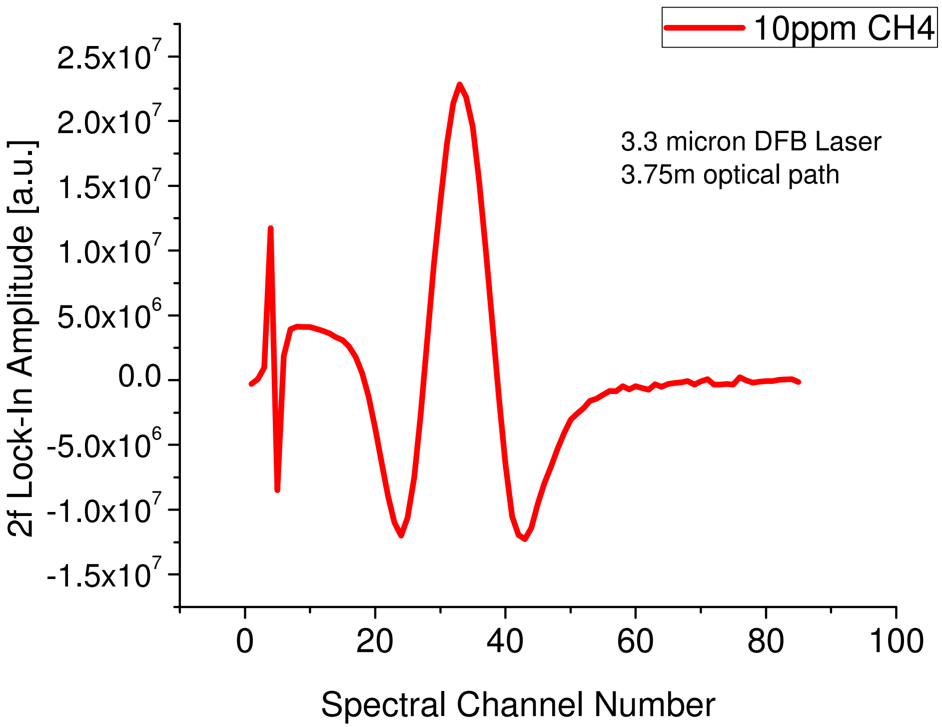
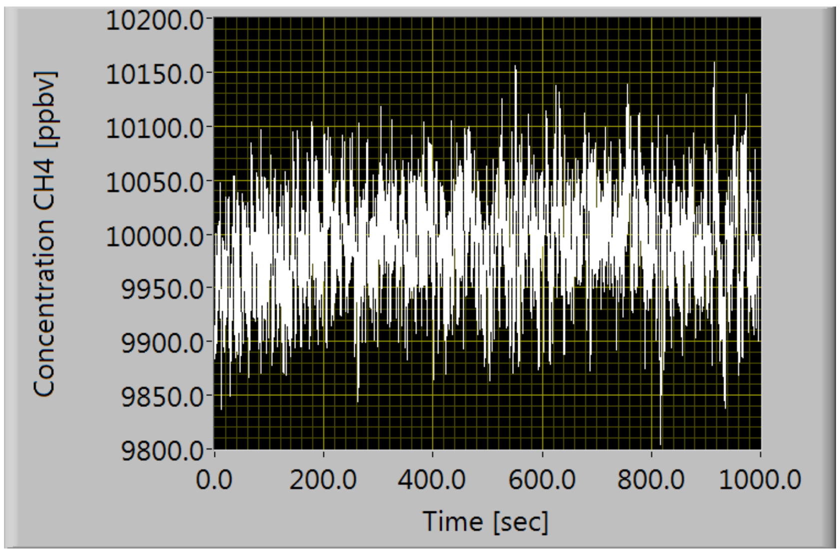
Mini Methane Sensor for UAVs
Sensor Performance





Onboard pressure controller and pump system
Continuous measurement [10Hz] with onboard processing
Direct concentration output – no post-processing necessary

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Future Directions and Outlook

- New target analytes such as carbonyl sulfide (OCS), formaldehyde (CH_2O), nitrous acid (HNO_2), hydrogen peroxide (H_2O_2), ethylene (C_2H_4), ozone (O_3), nitrate (NO_3), propane (C_3H_8), and benzene (C_6H_6)
- Ultra-compact, low cost, robust sensors (e.g. C_2H_6, NO, CO…)
- Monitoring of broadband absorbers: acetone (C_3H_6O), acetone peroxide (TATP), UF_6…
- Optical power build-up cavity designs
- Development of trace gas sensor networks
- QEPAS based detection at THz frequencies

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Future Directions and Outlook

- Development of robust, compact sensitive, mid-infrared trace gas sensor technology based on room temperature, continuous wave, DFB QCL and ICLs for environmental, industrial, biomedical monitoring and security applications
- Seven target trace gas species were detected with a 1 sec sampling time:
  - C_2H_6 at ~ 3.36 μm with a detection sensitivity of 740 pptv using TDLAS
  - NH_3 at ~ 10.4 μm with a detection sensitivity of ~1 ppbv (200 sec averaging time);
  - NO at ~5.26μm with a detection limit of 3 ppbv
  - CO at ~ 4.61 μm with minimum detection limit of 2 ppbv
  - SO_2 at ~7.24 μm with a detection limit of 100 ppbv
  - CH_4 and N_2O at ~7.28 μm currently in progress with detection limits of 20 and 7 ppbv, respectively.
- New target analytes such as CH_2O, H_2O_2, and C_2H_4,
- Monitoring of broadband absorbers such as acetone, C_3H_8, C_6H_6 and UF_6

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