Advanced Infrared Semiconductor Laser based Chemical Sensing Technologies: Opportunities and Challenges

F.K. Tittel, Y. Bakhirkin, R.F. Curl, A.A. Kosterev, R. Lewicki, S. So and G. Wysocki

Rice Quantum Institute, Rice University, Houston, TX, USA
http://ece.rice.edu/lasersci/

OUTLINE

Zhejiang University
Hangzhou, China
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- Motivation: Wide Range of Chemical Sensing
- Fundamentals of Laser Absorption Spectroscopy
- New Laser Sources and Sensing Technologies
- Selected Applications of Trace Gas Detection
  - Quartz Enhanced L-PAS (NH_3, Freon 125, Acetone and TATP)
  - Nitric Oxide Detection (Faraday Rotation Spectroscopy, Remote Sensing & Cavity Enhanced Spectroscopy)
- Future Directions and Conclusions

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Wide Range of Trace Gas Sensing Applications

- **Urban and Industrial Emission Measurements**
  - Industrial Plants
  - Combustion Sources and Processes (e.g. fire detection)
  - Automobile, Truck, Aircraft and Marine Emissions
- **Rural Emission Measurements**
  - Agriculture & Forestry, Livestock
- **Environmental Monitoring**
  - Atmospheric Chemistry
  - Volcanic Emissions
- **Chemical Analysis and Industrial Process Control**
  - Petrochemical, Semiconductor, Nuclear Safeguards, Pharmaceutical, Metals Processing, Food & Beverage Industries
- **Spacecraft and Planetary Surface Monitoring**
  - Crew Health Maintenance & Life Support
- **Applications in Health and Life Sciences**
- **Technologies for Law Enforcement and National Security**
- **Fundamental Science and Photochemistry**

Air Composition

Main Components		Trace Components	
Nitrogen	78%	Methane	1.7 ppm
Oxygen	21%	CO	0.4 ppm
Water	0.8%	N_2O	0.3 ppm
CO_2	0.03 %	O_3	0.03 ppm
		H_2CO	0.001 ppm
		…	

Worldwide Megadirty Megacities

	Population, m 1990. est.	2000. proj.	Sulphur dioxide	Particulate matter	Lead	Carbon monoxide	Nitrogen dioxide	Ozone
Bangkok	7.16	10.26	○	●	◐	○	◐	○
Beijing	9.74	11.47	●	●	○	-	○	◐
Bombay	11.13	15.43	○	●	○	○	○	-
Buenos Aires	11.58	13.05	-	◐	○	-	-	-
Cairo	9.08	11.77	-	●	●	◐	-	-
Calcutta	11.83	15.94	○	●	○	-	○	-
Delhi	8.62	12.77	○	●	○	○	○	-
Jakarta	9.42	13.23	○	●	○	○	○	○
Karachi	7.67	11.57	○	●	●	-	-	-
London	10.57	10.79	○	○	◐	◐	○	○
Los Angeles	10.47	10.91	○	◐	○	◐	◐	●
Manila	8.40	11.48	○	●	◐	-	-	-
Mexico City	19.37	24.44	●	●	●	●	◐	●
Moscow	9.39	10.11	-	◐	○	○	○	-
New York	15.65	16.10	○	○	○	○	○	◐
Rio de Janeiro	11.12	13.00	○	◐	○	○	-	-
Sao Paolo	18.42	23.60	○	●	○	○	○	●
Seoul	11.33	12.97	●	●	○	○	○	◐
Shanghai	13.30	14.69	◐	●	-	-	-	-
Tokyo	20.52	21.32	○	○	-	○	○	●

Source: United Nations ● High pollution ◐ Moderate to heavy pollution ○ Low pollution - No data available

Megacity Air Pollution: Houston, TX

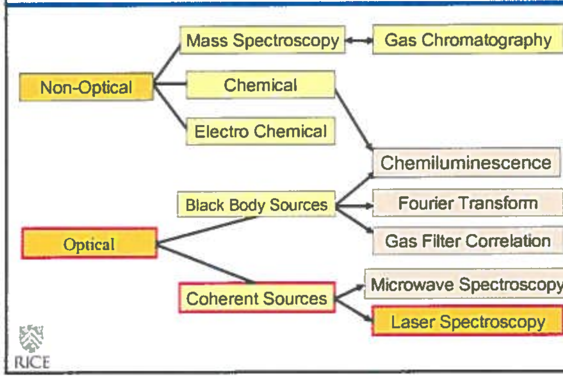
Monitoring Methane in Rice - Based Agroecosystems

International Space Station

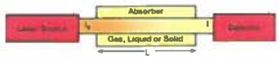
Mars NASA Pathfinder Climate Monitoring

Atmospheric Chemistry of Volcanic Plumes

Existing Methods for Trace Gas Detection



Fundamentals of Laser Absorption Spectroscopy

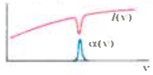


Beer-Lambert's Law of Linear Absorption

$$I(\nu) = I_0 \, e^{-\alpha(\nu) P_a L}$$

$\alpha(\nu)$ - absorption coefficient [cm^{-1} atm^{-1}]; L - path length [cm]

ν - frequency [cm^{-1}]; P_a - partial pressure [atm]



$$\alpha(\nu) = C \cdot S(T) \cdot g(\nu - \nu_0)$$

C - total number of molecules of absorbing gas/atm/cm^3 [molecule cm^{-3} atm^{-1}]

S - molecular line intensity [cm/molecule^{-1}]

$g(\nu - \nu_0)$ - normalized spectral lineshape function [cm], (Gaussian, Lorentzian, Voigt)

Key Requirements: Sensitivity, specificity, rapid data acquisition and multi-species detection

Optimum Molecular Absorbing Transition
- Overtone or Combination Bands (NIR)
- Fundamental Absorption Bands (MID-IR)

Long Optical Pathlengths
- Multipass Absorption Cell
- Cavity Enhanced, Cavity Ringdown & Intracavity Spectroscopy
- Open Path Monitoring (with retro-reflector)

Spectroscopic Detection Schemes
- Wavelength & Frequency Modulation
- Balanced Detection
- Zero-air Subtraction
- Photoacoustic Spectroscopy

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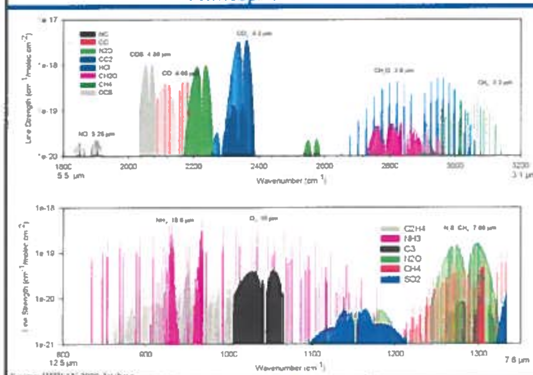
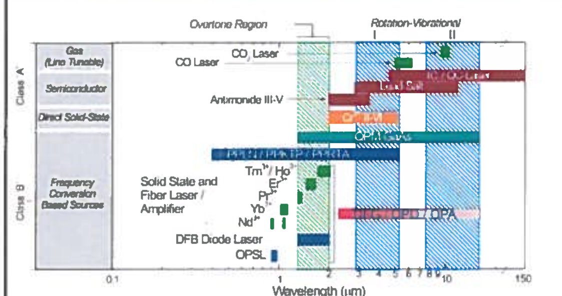
Mid-IR Source Requirements for Laser Spectroscopy

REQUIREMENTS	IR LASER SOURCE
Sensitivity (% to ppt)	Wavelength, Power
Selectivity (Spectral Resolution)	Single Mode Operation and Narrow Linewidth
Multi-gas Components, Multiple Absorption Lines and Broadband Absorbers	Tunable Wavelength
Directionality or Cavity Mode Matching	Beam Quality
Rapid Data Acquisition	Fast Time Response
Room Temperature Operation	No Consumables
Field deployable	Compact & Robust

Molecular Absorption Spectra within the two Mid-IR Atmospheric Windows



IR Laser Sources and Wavelength Coverage



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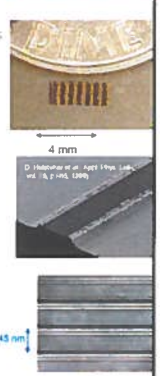
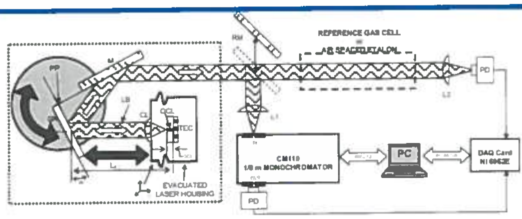
Key Characteristics of mid-IR QCLs and ICL Sources-2008

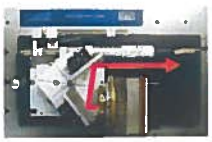
- **Band – structure engineered devices**
  (emission wavelength is determined by layer thickness – MBE or MOCVD); mid-infrared QCLs operate from 3 to 24 µm
- Compact, reliable, stable, long lifetime, and commercial availability
- Fabry-Perot (FP), single mode (DFB) and multi-wavelength
- **Spectral tuning range in the mid-IR**
  (4-24 µm for QCLs and 3-5 µm for ICLs)
  - 1.5 cm^{-1} using injection current control
  - 10-20 cm^{-1} using temperature control
  - ~ 300 cm^{-1} using an external grating element and heterogeneous cascade active region design; also QC laser array (Harvard)
- **Narrow spectral linewidth**
  cw: 0.1 - 3 MHz & <10Khz with frequency stabilization (0.0004 cm^{-1}); pulsed: ~ 300 MHz (chirp from heating)
- **High pulsed and cw powers of QCLs at TEC/RT temperatures**
  - Pulsed and CW powers of ~ 1.5 W; high temperature operation ~300 K
  - Average power levels: 1-600 mW (current wall plug η~ 9 %)
  - ~ 50 mW, TEC CW DFB @ 5 and 10 µm (DLS, Alpes, Princeton)
  - ~ 300 mW @ 8.3 µm (Harvard, Hamamatsu, Alpes, Northwestern)
  - > 600 mW (CW FP) @ RT & a wall plug efficiency of ~9 %, Adtech Optics, Harvard, Pranalytica
  - >150 mW (CW DFB) at 298 K (Northwestern)



Widely Tunable, CW, TEC Quantum Cascade Lasers

Tunable external cavity QCL based spectrometer





- Fine wavelength tuning
  - PZT controlled EC-length
  - PZT controlled grating angle
  - QCL current control
- Motorized coarse grating angle tuning
- Vacuum tight QCL enclosure with build-in 3D lens positioner (TEC laser cooling + optional chilled water cooling)

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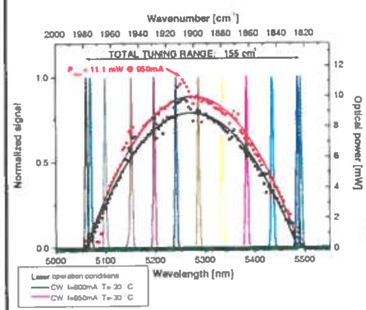
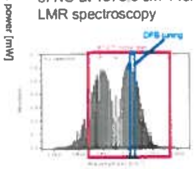
Wide Wavelength Tuning of a 5.3μm EC-QCL



- Coarse wavelength tuning of **155 cm⁻¹** is performed by varying diffraction grating angle
- Power output is ~ **11mW**
- Access to Q(3/2) transition of NO at 1875.8 cm-1 for LMR spectroscopy



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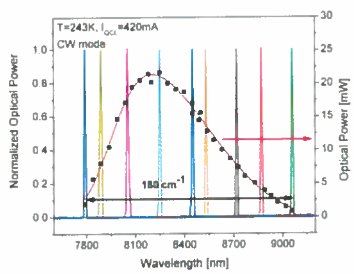
Performance of 8.4 μm cw EC-QCL Spectroscopic Source



Tunability **182 cm⁻¹** @8.4 μm;(1100 to 1280 cm⁻¹); λ_c=15%

AR coating: $R_{AR} \approx$ **2 x 10⁻⁴**

$P_{EC\text{-}opt}$: ~ **50 mW @ -30 C, ; also 103 mW @ 8.3 μm & -25 C; ~700mA; 2007 QCL technology**

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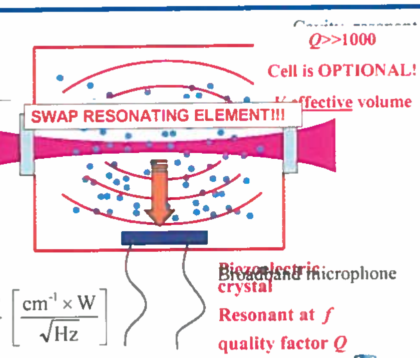
Quartz Enhanced Photoacoustic Spectroscopy

From conventional PAS to QEPAS

Laser beam, power P

Modulated (P or λ) at f or $f/2$



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

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

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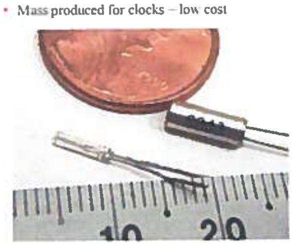
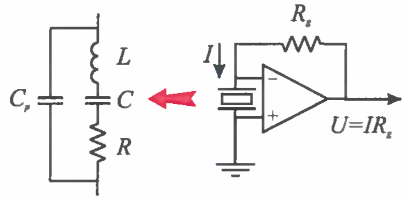
Quartz Tuning Fork (TF) as a Resonant Microphone

- Resonant frequency f=32.8 kHz
- Intrinsically high Q factor: Q_{vacuum} ~ 125 000, Q_{air} ~10 000 at ambient conditions,
- Piezoelectric: requires no transducer
- Miniature size
- Mass produced for clocks – low cost



QEPAS Signal Detection



Amplifier
- Fast
- Low noise
- High impedance
- Low 1/f noise

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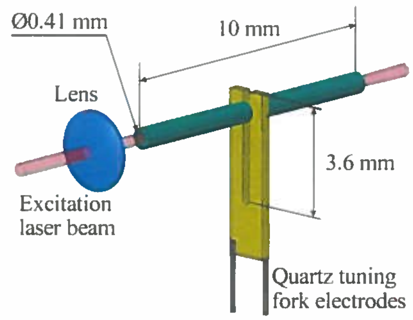
Absorption Detection Module for QEPAS based Gas Sensor



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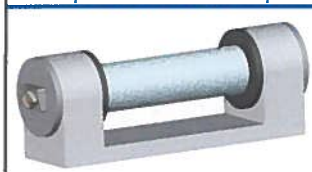
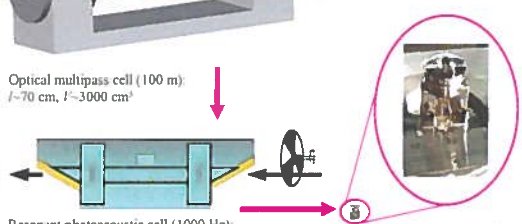
Comparative Size of Absorption Detection Modules (ADM)



- Resonant frequency f = 32.8 kHz
- Intrinsically high Q factor: Q_{vacuum} ~ 125 000, Q_{air} ~10 000 for ambient conditions
- Piezoelectric: requires no transducer
- Miniature size
- Mass produced for watches & clocks – low cost

Optical multipass cell (100 m): l~70 cm, V~3000 cm^3



Resonant photoacoustic cell (1000 Hz): l~60 cm, V~50 cm^3

QEPAS spectrophone: l~1 cm, V~0.05 cm^3

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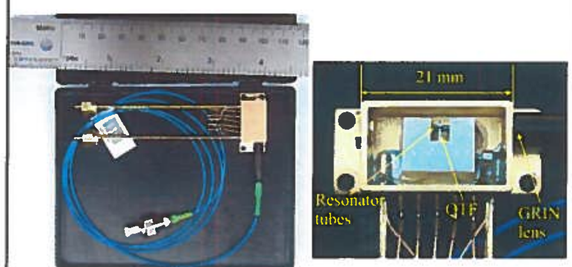
Alignment-free QEPAS Absorption Detection Module



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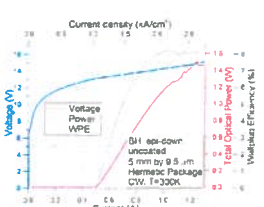
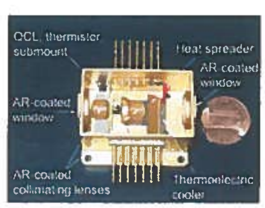
High Performance 4.6 μm CW, RT QC Laser - 2008





A. Lyakh et al., Pranalytica, Harvard, Adtech, Applied Physics Letters 92, 111110, 2008

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Merits of QE Laser-PAS based Trace Gas Detection

- High sensitivity (ppm to ppb gas concentration levels) and excellent dynamic range
- Immune to ambient and flow acoustic noise, laser noise and etalon effects
- Significant reduction of sample volume (< 1 mm^3)
- Applicable over a wide range of pressures
- Temperature, pressure and humidity insensitive
- Ultra-compact, rugged and low cost (compared to other laser based sensor architectures)

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Trace Gas Sensing Examples

Motivation for NH_3 Detection

- Monitoring of gas separation processes
- Detection of ammonium-nitrate explosives
- Spacecraft related gas monitoring
- Monitoring NH_3 concentrations in the exhaust stream of NO_x removal systems based on selective catalytic reduction (SCR) techniques
- Semiconductor process monitoring & control
- Monitoring of industrial refrigeration facilities
- Pollutant gas monitoring
- Atmospheric chemistry
- Medical diagnostics (kidney & liver diseases)

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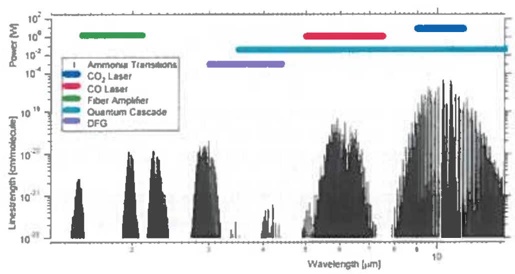
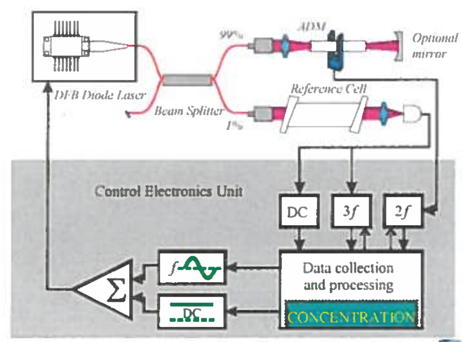
Infrared NH_3 Absorption Spectra



M. Webber et al. 2003, Pranalytica

QEPAS based Gas Sensor Architecture



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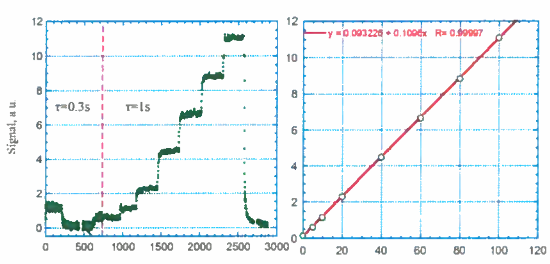
Calibration and Linearity of a 1.53 µm QEPAS based NH_3 Sensor

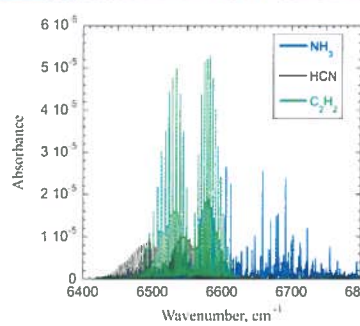


Noise-equivalent concentration (NEC) for t=1s time constant is 0.06 ppm for 60mW excitation power at 6528.76 cm^{-1}

90 last points of each step averaged

Noise-equivalent absorption (NEA) coefficient $k=3.1\times10^{-9}$ $cm^{-1}W/Hz^{1/2}$

Infrared NH_3, HCN and C_2H_2 Absorption Spectra



1atm,1ppmv,1m (base 10)

PNNL FTIR Database

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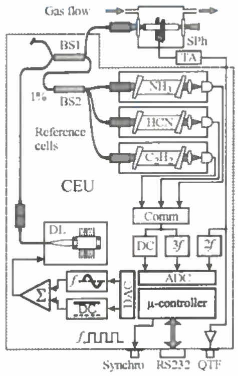
QEPAS based Multi-Gas Sensor Architecture



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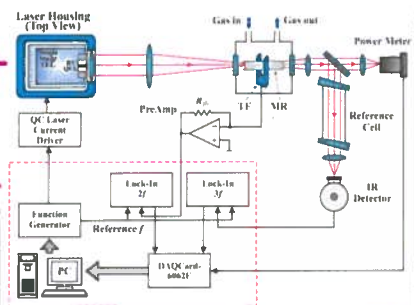
Biomarkers Present in Exhaled Human Breath

More than 400 different molecules in breath; many with well defined biochemical pathways

compound	concentration	physiological basis
Acetaldehyde	ppb	ethanol metabolism
Acetone	ppm	decarboxylation of acetoacetate
Ammonia	ppb	protein metabolism
Carbon dioxide	%	product of respiration
Carbon disulfide	ppb	gut bacteria
Carbon monoxide	ppm	production catalyzed by *heme oxygenase*
Carbonyl sulfide	ppb	gut bacteria
Ethane	ppb	lipid peroxidation
Ethanol	ppb	gut bacteria
Ethylene	ppb	lipid peroxidation
Hydrocarbons	ppb	lipid peroxidation/metabolism
Hydrogen	ppm	gut bacteria
Isoprene	ppb	cholesterol biosynthesis
Methane	ppm	gut bacteria
Methanethiol	ppb	methionine metabolism
Methanol	ppb	metabolism of fruit
Methylamine	ppb	protein metabolism
Nitric oxide	ppb	production catalyzed by *nitric oxide synthase*
Oxygen	%	required for normal respiration
Pentane	ppb	lipid peroxidation
Water	%	product of respiration

Terence Risby, Johns Hopkins University

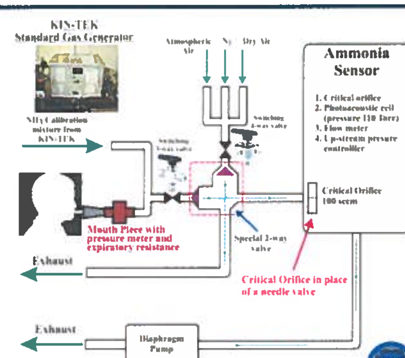
Mid-IR QEPAS based NH_3 Gas Sensor Architecture



QEPAS Driver

Noise–equivalent concentration (NEC) for t=1s time constant is 0.006 ppm for 20mW excitation power at 1046.4 cm-1 (110 Torr)

Breath Collection Interface to Trace Gas Sensor



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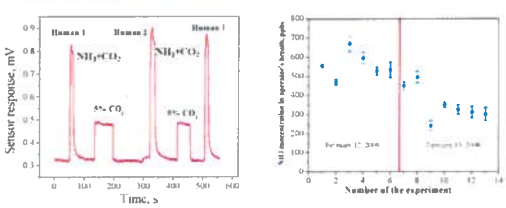
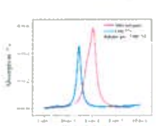
Real Time Exhaled NH_3 Sensor Response from a Healthy Volunteer





For the available QCL the presence of CO_2 in breath (~ 4% - 4.5%) contributes to the sensor signal. This fact has to be taken into account in the quantification of the exhaled ammonia concentration.

NH_3 Measurements at an Oklahoma State University Research Feedyard

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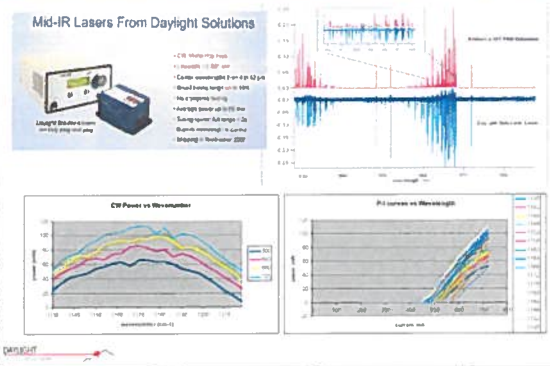
Ekips

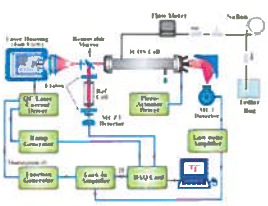
Commercial widely tunable cw EC-QCL



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 (TNT)

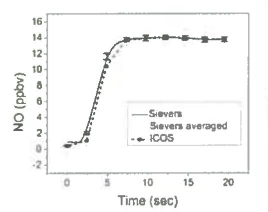
Laser-based ICOS Nitric Oxide Sensor



NO:N2 mixture @ 100 Torr
Effective L= 700 m
A 1σ deviation of the amplitude corresponds to a 0.7 ppb detection limit (1 sec.)

Online NO concentration measurements at 3l/min exhalation.

Intercomparison of ICOS and commercial chemiluminescence sensor (Sievers – solid line)



M.R. McCurdy, Y. Bakhirkin, G. Wysocki, F.K. Tittel, "Performance of an exhaled nitric oxide and carbon dioxide sensor using quantum cascade laser based integrated cavity output spectroscopy", to appear in Journal of Biomedical Optics 2007

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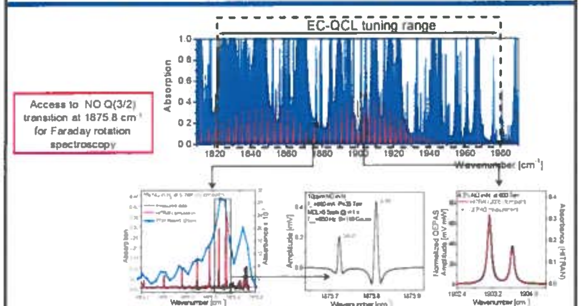
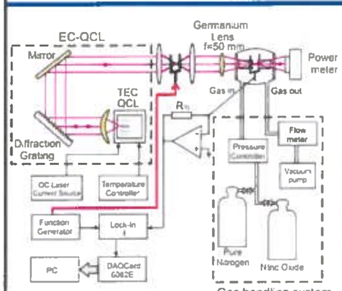
High resolution spectroscopy with a 5.3μm EC-QCL



- Mode hop free scan of up to ~2.5 cm⁻¹ with a resolution <0.001cm⁻¹ (30MHz) can be performed anywhere within the tuning range

In collaboration with

QCL based Quartz-Enhanced Photoacoustic Gas Sensor



QEPAS characteristics:

- High sensitivity (ppm to ppb)
- Excellent dynamic range
- Immune to environmental noise
- Ultra-small sample volume (< 1 mm³)
- Sensitivity is limited by the fundamental thermal TF noise
- Compact, rugged and low cost
- Potential for trace gas sensor networks

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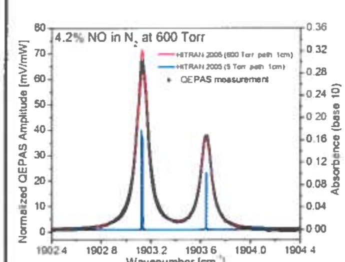
High resolution EC-QCL based QEPAS



External Amplitude Modulation:

- QTF is used as a mechanical chopper at f=~32kHz
- No chirp associated with the laser current modulation
- High resolution mode-hop-free tuning is possible

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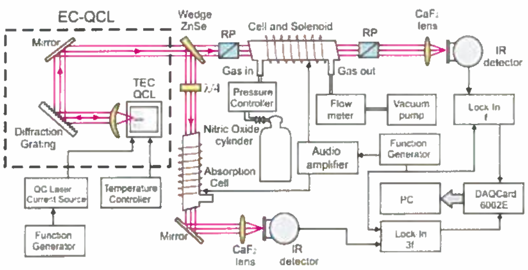
EC-QCL based Magnetic Rotation Spectroscopy



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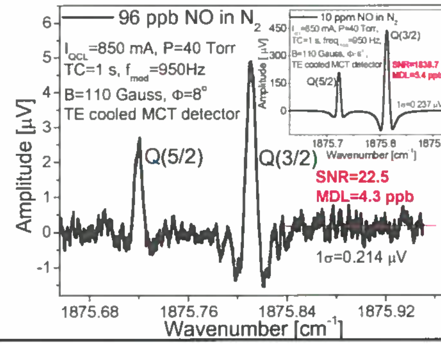
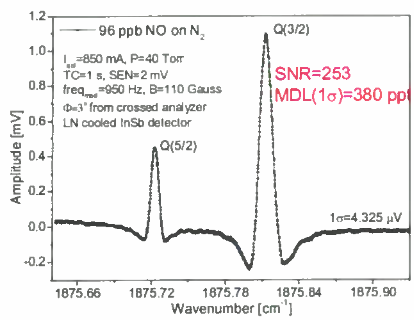
Magnetic rotation spectrum of Q(3/2) and Q(5/2) transitions of nitric oxide



Magnetic Rotation Spectroscopy of Nitric Oxide



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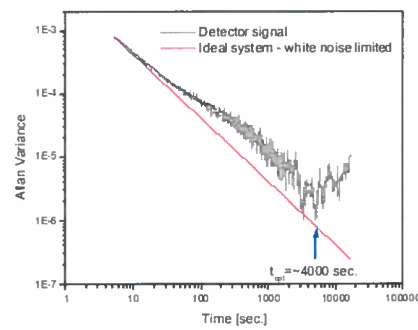
Allan Plot of EC-QCL based MRS NO Sensor



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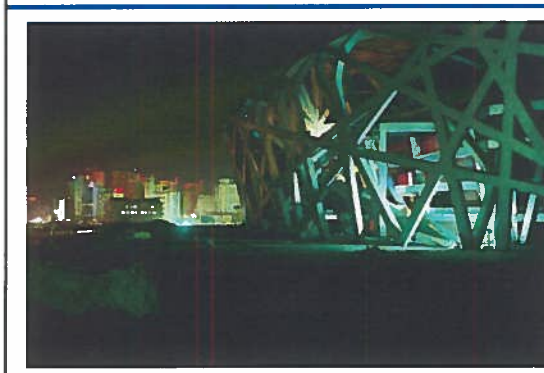
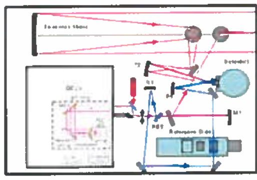
EC-QCLbased MRS NO Sensor for IAP, Beijing

National Stadium, Beijing, July-Sept. 2008

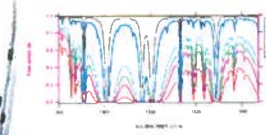


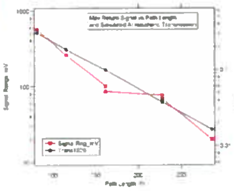
Design of an EC-QCL Based Remote Sensing System



- An upgraded version of a four-laser pulsed QCL system
- The optical set-up, electronics and control software modified for CW-QCL operation
- First tests performed with a DFB CW-QCL operating at ~5.5µm

Outdoor Open Path Measurements (Influence of Atmospheric Transmission)





Monitoring of broadband absorbers

- Freon 125 (C_2HF_5)
  - Refrigerant (leak detection)
  - Safe simulant for toxic chemicals, e.g. chemical warfare agents
- Acetone (CH_3COCH_3)
  - Recognized biomarker for diabetes
- TATP, Acetone Peroxide ($C_6H_{12}O_4$)
  - Highly Explosive

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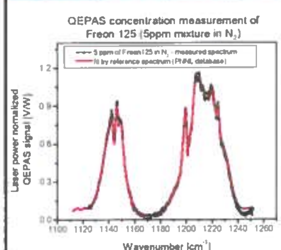
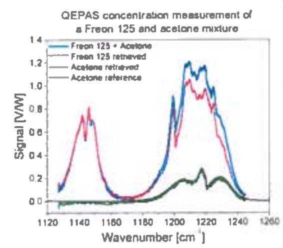
QEPAS based Freon 125 and Acetone concentration measurements with a tunable 8.4 µm CW EC-QCL



- Minimum detection limit (1σ) of **~4.5 ppb** was obtained for Freon 125 with an average laser power of 6.6 mW



- Wide tunability enables excellent molecular selectivity for broad band absorbers

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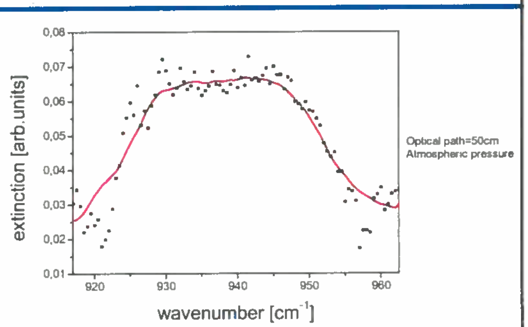
TATP Absorption Spectrum



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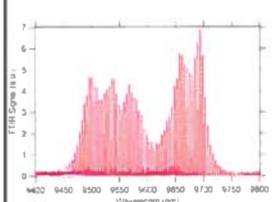
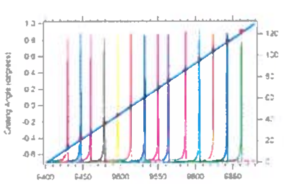
Applications of Efficient Mid-Infrared 9.6µm QCL



FTIR analysis of the broadband output from FP QCL (65mW per facet from uncoated chip)

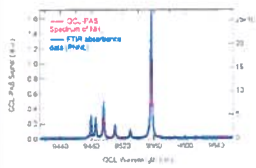


Wavelength Tuning Characteristics and FTIR spectrum

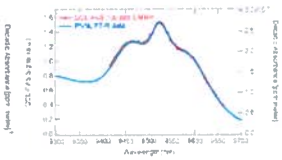
A. Mukherjee et al., Pranalytica & AdTech Optics, Applied Optics 47, 1543-1548, 2008

60

Applications of Efficient Mid-Infrared 9.6μm QCLs



Measured QCL-PAS spectrum of NH_3 comparison with FTIR spectrum of NH_3



Demonstrated QCL-PAS spectrum of 100 ppb DMMP in CDA superimposed on FTIR spectrum

A. Mukherjee et al., Pranalytica, Inc and Adtech, Inc, Applied Optics, 47, 1543-1548, 2008

QEPAS Performance for 12 Trace Gas Species (July '08)

Molecule (Host)	Frequency, cm⁻¹	Pressure, Torr	NNEA, cm⁻¹W/Hz^½	Power, mW	NEC (τ=1s), ppmv
H_2O (N_2)**	7306.75	60	1.9×10[illegible]	9.5	0.09
HCN (air: 50% RH)*	6539.11	60	< 4.3×10[illegible]	50	0.16
C_2H_2 (N_2)*	6523.88	720	4.1×10[illegible]	57	0.03
NH_3 (N_2)*	6528.76	575	3.1×10[illegible]	60	0.06
C_2H_4 (N_2)*	6177.07	715	5.4×10[illegible]	15	1.7
CH_4 (N_2)*	6057.09	950	2.9×10[illegible]	13.7	2.1
CO_2 (breath ~100% RH)	6361.25	150	8.2×10[illegible]	45	40
H_2S (N_2)*	6357.63	780	5.6×10[illegible]	45	0.20
CO_2 (N_2+1.5% H2O)*	4991.26	50	1.4×10[illegible]	4.4	18
CH_2O (N_2:75% RH)*	2804.90	75	8.7×10[illegible]	7.2	0.12
CO (N_2)	2196.66	50	5.3×10[illegible]	13	0.5
CO (propylene)	2196.66	50	7.4×10[illegible]	6.5	0.14
N_2O (air+5%SF_6)	2195.63	50	1.5×10[illegible]	19	0.007
C_2H_5OH (N_2)**	1934.2	770	2.2×10[illegible]	10	90
C_2HF_5 (N_2)***	1208.62	770	7.8×10[illegible]	6.6	0.009
NH_3 (N_2)*	1046.39	110	1.6×10[illegible]	20	0.006

* 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 (2.6)×10[illegible] cm⁻¹W/√Hz (1,800; 10,300 Hz) for NH_3

• M. E. Webber et al, Appl. Opt. 42, 2119-2126 (2003); ** J. S. Pilgrim et al, SAE Intl. ICES 2007-01-3152

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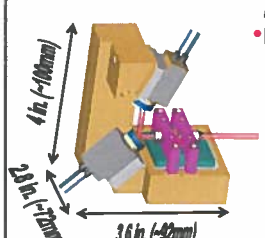
Future of Chemical Trace Gas Sensing

Conceptual Design of Ultra-compact QCL Trace Gas Sensor

Aerodyne, Inc, Barry McManus et al., 2008

New design of fast broadly tunable EC-QCLs (2008)



- New optical configuration *Folded cavity (configuration #1)*
- Fast tuning capabilities:
  - Coarse Broadband Scanning (~55 cm⁻¹ @5μm) ***up to 5 KHz*** *(compared to available technologies <10Hz)*
  - High resolution mode-hop free tuning (~3.2 cm⁻¹ @5μm) ***up to 5 KHz*** *(compared to available technology 100-200 Hz)*

Patent pending, G. Wysocki, F. K. Tittel, 2007

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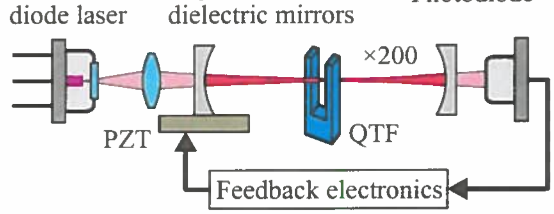
Proposed QEPAS-OPBC Sensor Configuration



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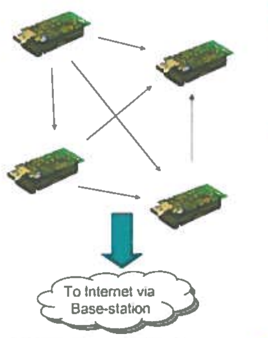
Wireless Sensor Networks for Trace Gas Sensing



- Each point called “mote”
- Advantages?
  - Spatial resolution
  - Measure fluxes
- What is needed?
  - Low power
  - Low cost
  - Ultra miniature
  - Replicable
  - Autonomy

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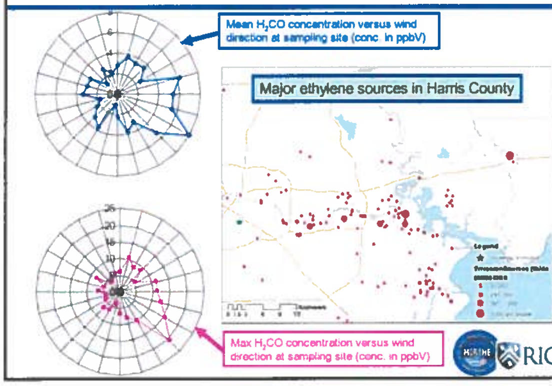
H_2CO Concentration (ppb) Versus Wind Direction



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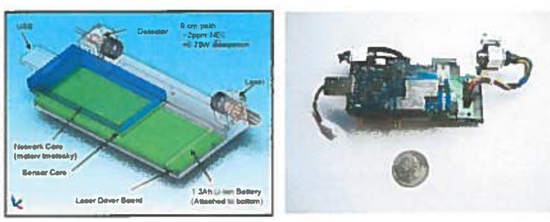
PHOTONS v4.0 - 2.7μm CO2 Direct Absorption Based Sensor



- Small size
- Relatively low cost
- High efficiency switching power supplies
- PWM Peltier cooler driver
- 0 2W control system power consumption
- Detection sensitivity of CO_2 1 ppm with 1sec lock-in time constant
- Over 100x improvement in sensitivity is possible @ 4 2μm

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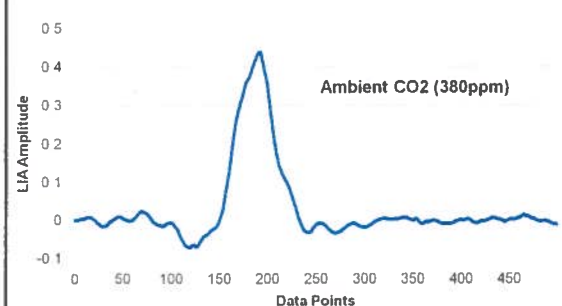
LAS based CO_2 Spectrum at 2.7 μm



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Summary & Future Directions of QCL based Gas Sensor Technology

- **Quantum and Interband Cascade Laser based Trace Gas Sensors**
  - Compact, tunable, and robust
  - High sensitivity (<10^{-4}) and selectivity (3 to 500 MHz)
  - Capable of fast data acquisition and analysis
  - Detected 13 trace gases to date: NH_3, CH_4, N_2O, CO_2, CO, NO, H_2O, COS, C_2H_4, H_2CO, SO_2, C_2H_5OH, C_2HF_5 and several isotopic species of C, O, N and H.
- **New Applications of Trace Gas Detection**
  - Environmental Monitoring (urban quality - H_2CO and, isotopic ratio measurements of CO_2 and CH_4, fire detection and quantification of engine exhausts)
  - Industrial process control and chemical analysis (NO, NH_3, H_2O, and H_2S)
  - Medical & biomedical diagnostics (NO, NH_3, N_2O, H_2CO and CH_3COCH_3)
  - Hand-held sensors and sensor network technologies (CO_2)
- **Future Directions and Collaborations**
  - Improvements of the existing sensing technologies using novel, thermoelectrically cooled, cw, high power, and broadly wavelength tunable mid-IR interband and intersubband quantum cascade lasers
  - New applications enabled by novel broadly wavelength tunable quantum cascade lasers based on heterogeneous EC-QCL (i e sensitive concentration measurements of broadband absorbers, in particular VOCs, HCs and multi-species detection)
  - Development of optically gas sensor networks based on QEPAS and LAS

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