

The state-of-the-art and grand challenges of mid-infrared technology and applications



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www.ece.rice.edu/lasersci

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- New Laser Based Trace Gas Sensor Technology
 - Novel Multipass Absorption Cell & Electronics
 - Quartz Enhanced Photoacoustic Spectroscopy
- Examples of Mid-Infrared Sensor Architectures
 - C₂H₆, NH₃, NO, CO, SO₂, CH₄ & N₂O
 - Future Directions of Laser Based Gas Sensor Technology and Conclusions

Research support by NSF ERC MIRTHE, NSF-ANR NexCILAS, the Robert Welch Foundation, Scinovation, Inc. and Sentinel Photonics Inc. via an EPA Phase 1 SBIR sub-award is acknowledged

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 (e.g. isotopic signatures)
 - 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 Medical Diagnostics and the Life Sciences
- Technologies for Law Enforcement, Defense and Security
- Fundamental Science and Photochemistry



Laser based Trace Gas Sensing Techniques

Optimum Molecular Absorbing Transition

- Overtone or Combination Bands (NIR)
- Fundamental Absorption Bands (MID-IR)

Long Optical Pathlength

- Multipass Absorption Cell (White, Herriot (Aerodyne),
 Chernin and Sentinel Photonics, Inc)
- Cavity Enhanced and Cavity Ringdown Spectroscopy
- Open Path Monitoring (with retro-reflector): Standoff and Remote Detection
- Fiberoptic Evanescent Wave Spectroscopy

• Spectroscopic Detection Schemes

- Frequency or Wavelength Modulation
- Balanced Detection
- Zero-air Subtraction
- Photoacoustic & Quartz Enhanced Photoacoustic
 Spectroscopy (QEPAS)



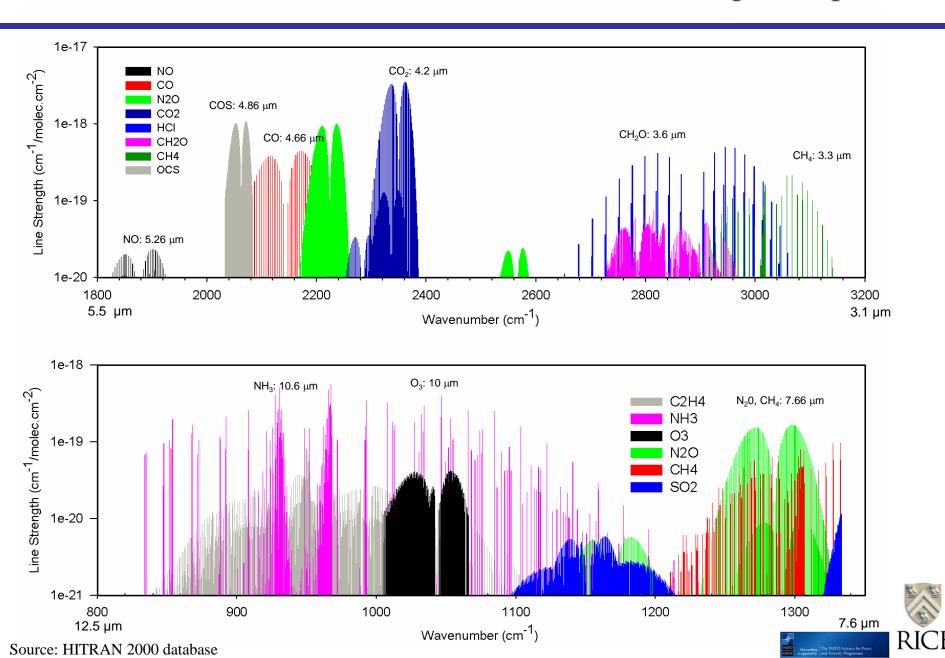
Other Spectroscopic Trace Gas Sensing Techniques

- Faraday Rotation Spectroscopy (limited to paramagnetic chemical species)
- Noise Immune Cavity Enhanced-Optical Heterodyne Molecular Spectroscopy (NICE-OHMS)
- Frequency Comb Spectroscopy
- Laser Induced Breakdown Spectroscopy (LIBS)





HITRAN Simulated Mid-Infrared Molecular Absorption Spectra



Mid-IR Source Requirements for Laser Spectroscopy

REQUIREMENTS	IR LASER SOURCE		
Sensitivity (% to ppt)	Optimum Wavelength, Power		
Selectivity (Spectral Resolution)	Stable Single Mode Operation and Narrow Linewidth		
Multi-gas Components, Multiple Absorption Lines and Broadband Absorbers	Mode Hop-free Wavelength Tunability		
Directionality or Cavity Mode Matching	Beam Quality		
Rapid Data Acquisition	Fast Time Response		
Room Temperature Operation	High wall plug efficiency, no cryogenics or cooling water		
Field deployable in harsh environments	Compact, Robust, Packaging, Low Noise		

Key Characteristics of Mid-IR QCL& ICL Sources – Feb 2013

Band – structure engineered devices

Emission wavelength is determined by layer thickness – MBE or MOCVD; Type I QCLs operate in the 3 to 24 µm spectral region; Type II and GaSb based ICLs can cover the 3 to 6 µm spectral range.

- Compact, reliable, stable, long lifetime, and commercial availability
- Fabry-Perot (FP), single mode (DFB) and multi-wavelength devices

Wide spectral tuning ranges in the mid-IR

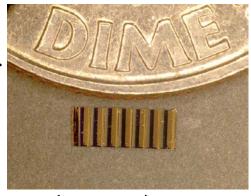
- 1.5 cm⁻¹ using injection current control for DFB devices
- 10-20 cm⁻¹ using temperature control for DFB devices
- ~ 525 cm⁻¹ (22% of c.w.) using an external grating element and FP chips with heterogeneous cascade active region design; also QCL DFB Array

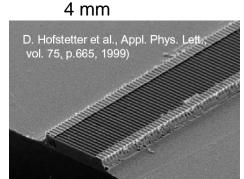
Narrow spectral linewidths

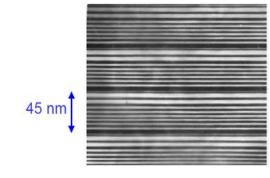
- CW: 0.1 3 MHz & <10kHz with frequency stabilization (0.0004 cm⁻¹) Pulsed: ~ 300 MHz

High pulsed and CW powers of QCLs at TEC/RT **temperatures**

- Room temperature pulsed power of > 30 W with 27% wall plug efficiency and CW powers of ~ 5 W with 21% wall plug efficiency
- > 1 W, TEC CW DFB @ 4.6 μm
- > 600 mW (CW FP) @ RT; wall plug efficiency of ~17 % at 4.6 µm;









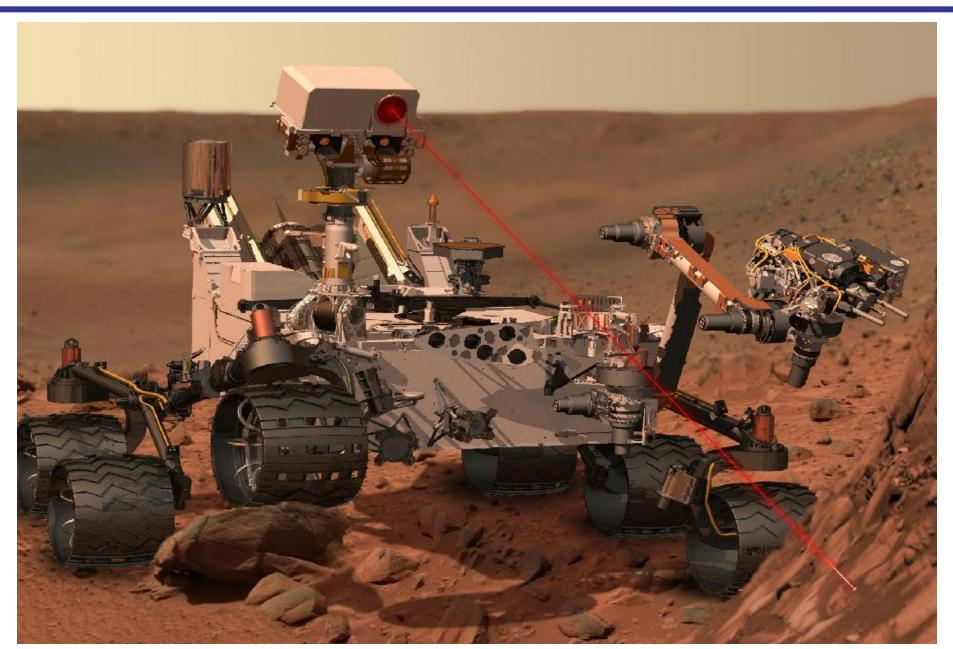
Recent Improvements and New Capabilities of QCLs and ICLs

- Optimum wavelength (> 3 to < 20 µm) and power (>10 mw to < 1 W) at room temperature (> 15 °C and < 30 °C) with state-of-the-art fabrication/processing methods based on MBE and MOCVD, good wall plug efficiency and lifetime (> 20,000 hours) for detection sensitivities from % to pptv with low electrical power budget
- Stable single TEM₀₀ transverse and axial mode, CW and pulsed operation of mid-infrared laser sources (narrow linewidth of ~ 300 MHz to < 10kHz)
- Mode hop-free ultra-broad wavelength tunability for detection of broad band absorbers and multiple absorption lines based on external cavity or mid-infrared semiconductor arrays
- Good beam quality for directionality_and/or cavity mode matching. Implementation of innovative collimation concepts.
- Rapid data acquisition based on **fast time response**.
- Compact, robust, readily commercially available and affordable in order to be field deployable in harsh operating environments (low and high temperature, pressure, etc...)



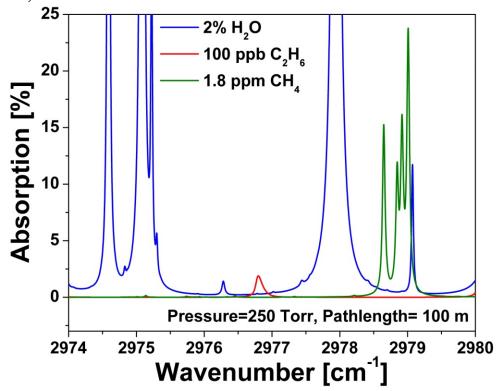


"Curiosity" landed on Mars on August 6, 2012



Motivation for Mid-infrared C₂H₆ Detection

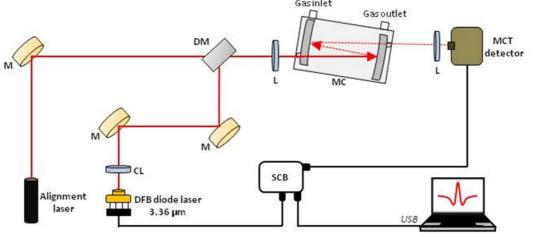
- Atmospheric chemistry and climate
 - Fossil fuel and biofuel consumption,
 - Biomass burning,
 - Vegetation/soil,
 - Natural gas loss
- Oil and gas prospecting
- Application in medical breath analysis (a non-invasive method to identify and monitor different diseases):
 - Asthma,
 - Schizophremia,
 - Lung cancer,
 - Vitamin E deficiency.



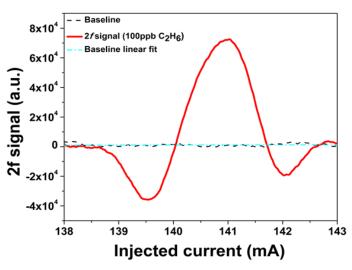
HITRAN absorption spectra of C_2H_6 , CH_4 , and H_2O



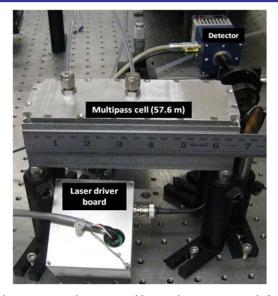
C₂H₆ Detection with a 3.36 μm DFB LD using a Novel Compact Multipass Absorption Cell and Control Electronics



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



2f WMS signal for a C_2H_6 line at 2976.8 cm⁻¹ at a pressure of 200 Torr



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

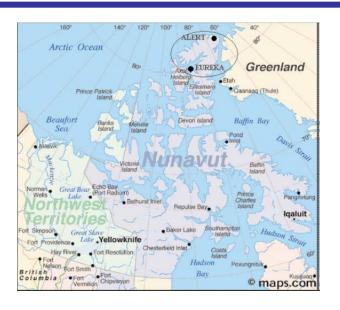


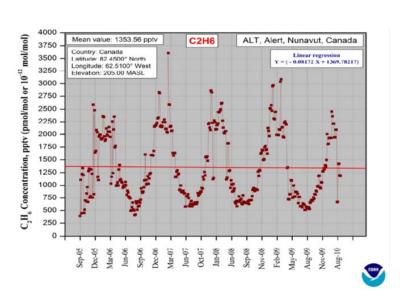
Minimum detectable C_2H_6 concentration is:

~ 130 pptv (1σ; 1 s time resolution)

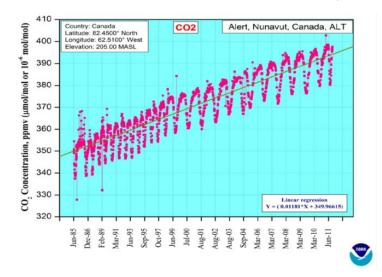
MC dimensions: **17** x **6.5** x **5.5** (cm) Distance between the MGC mirrors: 12.5 cm

NOAA Monitoring and Sampling Location: Alert (ALT), Nunavut, Canada





General view on Alert NOAA Facility



Ethane Concentration Measurements at ALT

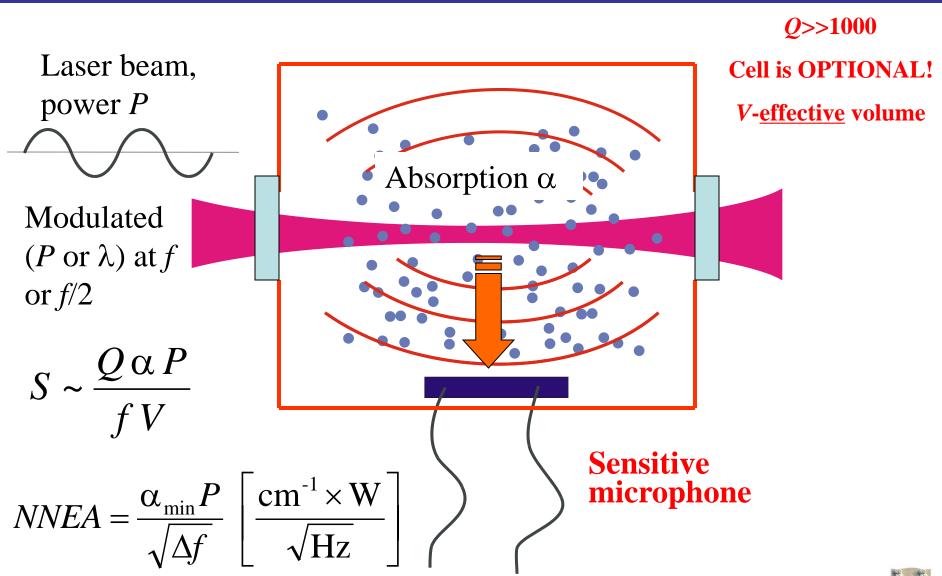
Motivation for NH₃ Detection

- Detection of ammonium-nitrate explosives
- Spacecraft related gas monitoring
- Monitoring NH₃ concentrations in the exhaust stream of NO_x removal systems based on selective catalytic reduction techniques
- Semiconductor process monitoring & control
- Monitoring of industrial refrigeration facilities
- Pollutant gas monitoring
- Atmospheric chemistry
- Medical diagnostics (kidney & liver diseases)





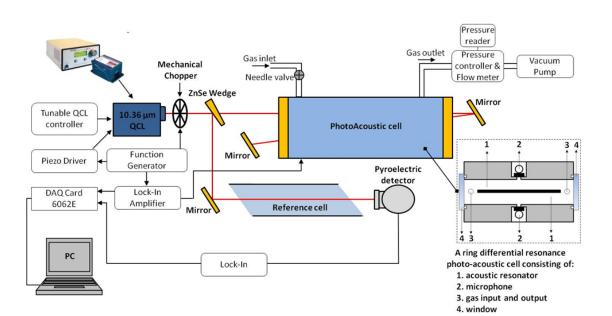
Conventional PAS

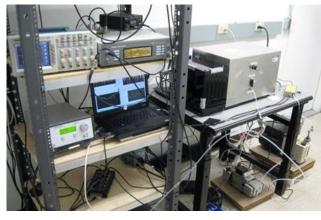






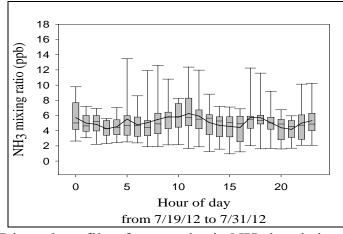
Atmospheric NH₃ Measurements using a CW EC-QCL PAS Sensor



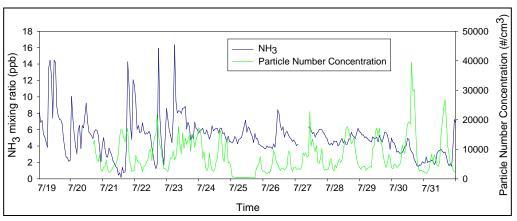


NH₃ sensor deployed at the UH Moody Tower rooftop monitoring site.

Schematic of a Daylight Solutions 10.36 µm CW TEC EC-QCL based PAS NH₃ Sensor.

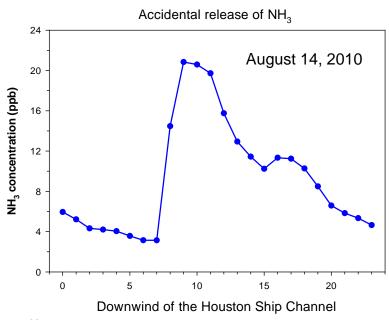


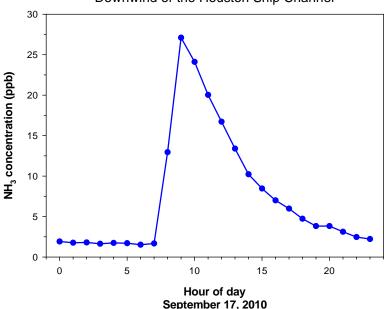
Diurnal profile of atmospheric NH₃ levels in Houston, TX.



Comparison between NH₃ and particle number concentration time series from July 19 to July 31 2012.

NH₃ Detection due to a Fire resulting from a two Truck Collision





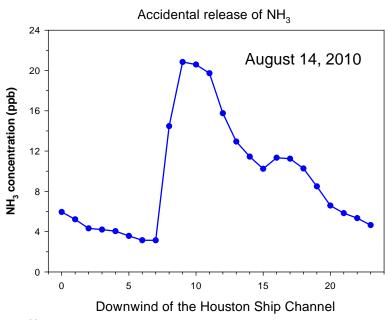


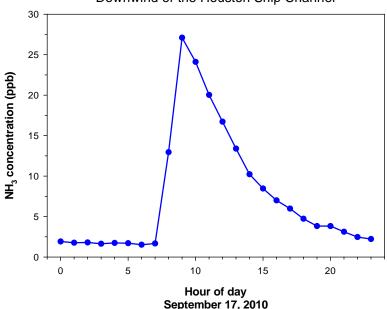
A chemical incident occurred at ~ 6 a.m. after two trucks collided on I-59. Both trucks caught fire. [www.chron.com]



Estimated hourly NH₃ emission from the Houston Ship Channel area is about 0.25 ton. Mellqvist et al., (2007) Final Report, HARC Project H-53.

NH₃ Detection due to a Fire resulting from a two Truck Collision





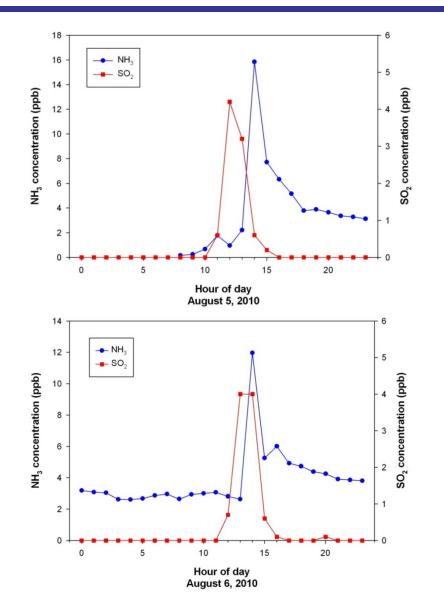


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Sporadic increased NH₃ Concentration Levels related to Emissions by the Parish Electric Power Plant, TX





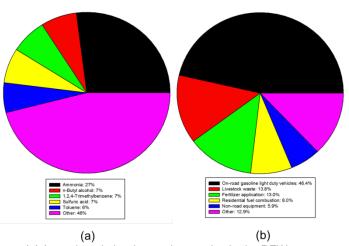
The Parish electric power plant, is located near the Brazos River in Fort Bend County, Texas (~27 miles SW of downtown Houston)



Atmospheric NH₃ Measurements and Implications for PM Formation near Fort Worth

- Four specific aims are being pursued:
 - Investigation of the NH₃ dynamics (e.g., temporal variations);
 - Identification of NH₃ sources using auxiliary data for CO and NO_x;
 - Performing NH₃ source apportionment using the EPA Positive Matrix Factorization 3.0 model including VOC_s data;
 - Evaluation of NH₃ effects on local and regional air quality with respect to PM formation using aerosol/PM data collected by an Aerodyne High Resolution Timeof-Flight Mass Spectrometer, a Scanning Electrical Mobility Spectrometer, and a Mist Chamber-Ion Chromatography system.



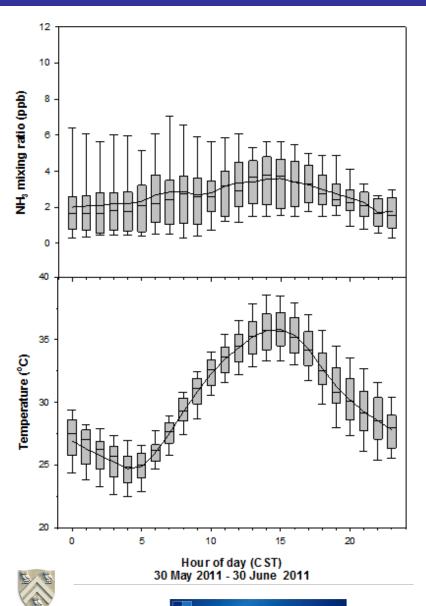


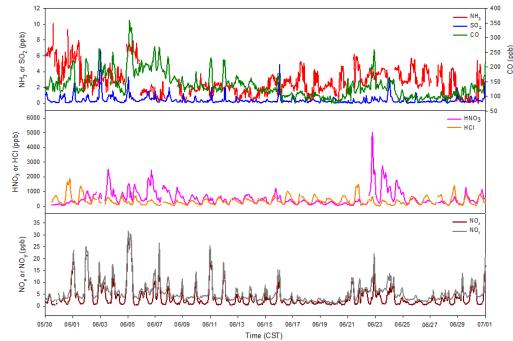
(a) Annual total air releases by species in the DFW area (U.S. EPA TRI, 2010); (b) Annual NH₃ emissions by source categories in Tarrant County, Texas (U.S. EPA NEI, 2008)





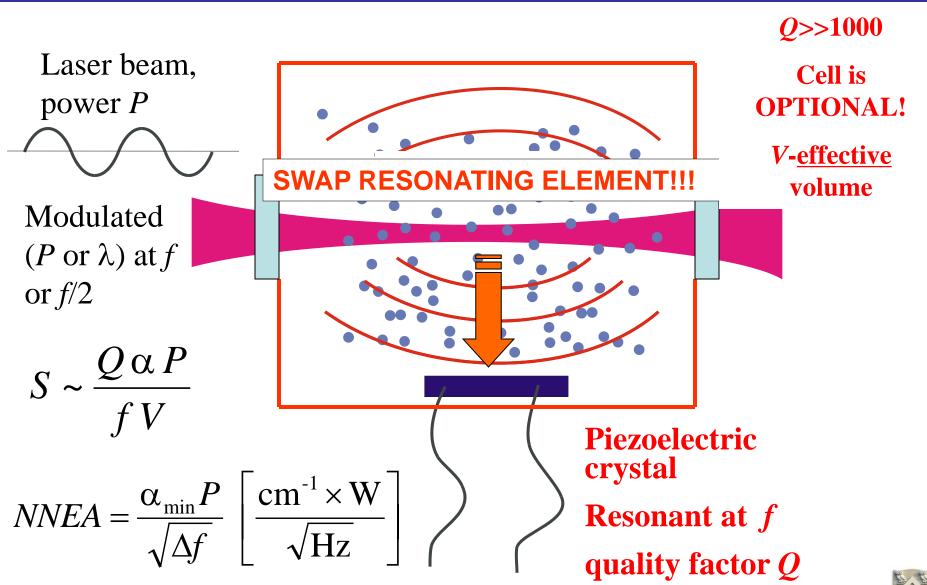
NH₃ vs. Temperature data & Time series of NH₃, SO₂, CO





- Emission events from specified point sources (i.e., industrial facilities)
- Estimated NH₃ emissions from cows (1.3 tons/day)
- Estimated NH₃ emissions from soil and vegetation (0.15 tons/day)
- EPA PMF (biogenic: 74.1%; light duty vehicles: 12.1%; natural gas/industry: 9.4%; and heavy duty vehicles: 4.4%)
- Livestock might account for approximately 66.4% of total NH₃ emissions
- Increased contribution from industry (\rightarrow 18.9%)

From Conventional PAS to QEPAS

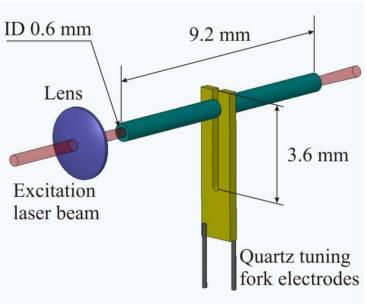






Quartz Tuning Fork as a Resonant Microphone for QEPAS





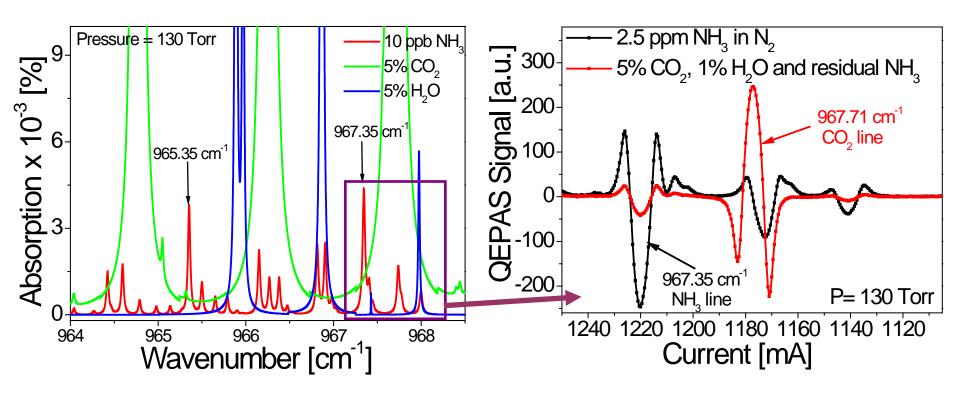
Unique properties

- 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 (~10⁶) linear from thermal noise to breakdown deformation
 - 300 K noise: $x \sim 10^{-11}$ cm & breakdown: $x \sim 10^{-2}$ cm
- Wide temperature range: from 1.6K to ~700K
- Low cost (< 1 zl)
- Resonant frequency;: $\sim 32.8 \text{ KHz} (2 \times 10^{15})$

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 $(\sim \lambda/4 < l < \lambda/2 \text{ for sound at } 32.8 \text{ kHz})$
- SNR of QTF with mR tubes: ×30 (depending on gas composition and pressure)

Optimum NH₃ Line Selection for a 10.34 µm CW TEC DFB QCL

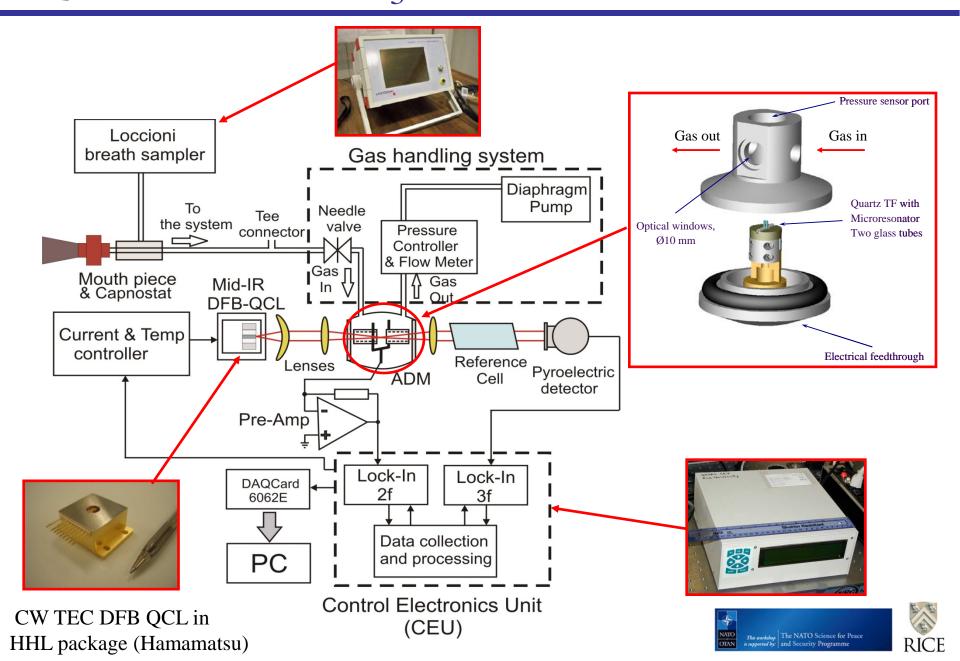


Simulated HITRAN high resolution spectra @ 130 Torr indicating two NH₃ absorption lines of interest

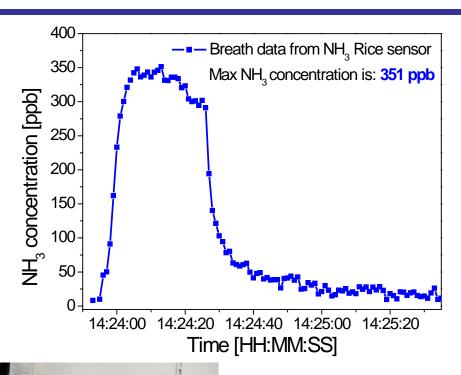
No overlap between NH_3 and CO_2 absorption lines was observed for the selected **967.35 cm⁻¹** NH_3 absorption line in the v_2 R band.

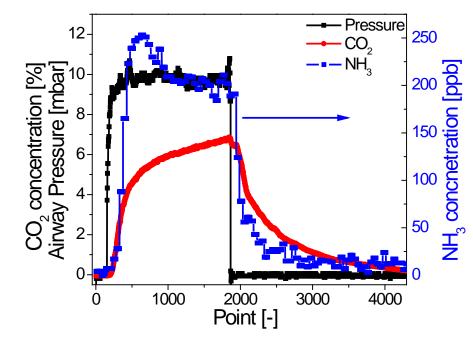


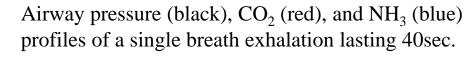
QEPAS based NH₃ Gas Sensor Architecture



Real-time exhaled human NH₃ Breath Measurements







Successful testing of a 2nd generation breath ammonia monitor installed in a clinical environment.(Johns Hopkins, Baltimore, MD and St. Luke's Hospital, Bethlehem, PA)



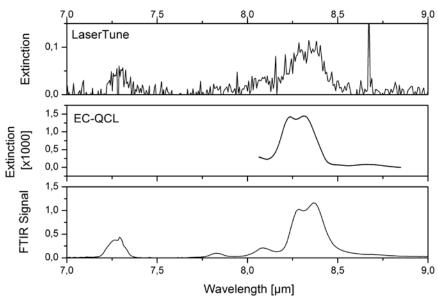




Minimum detectable concentration of NH₃ is:

~ 6 ppbv at 967.35 cm⁻¹ (1σ ; 1 s time resolution)

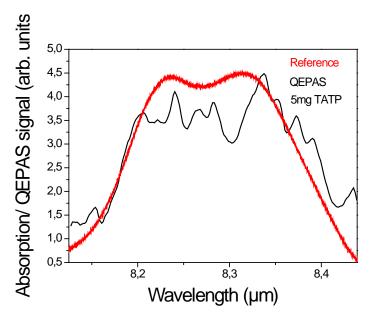
QEPAS Based TATP Detection

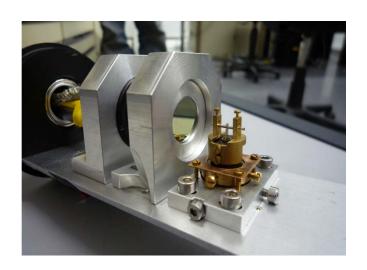


Multi pass 105 cm Gas phase from TATP stored in ionic liquid

Single pass 10 cm Gas phase from TATP powder

Single pass 10 cm
Gas phase from
TATP powder













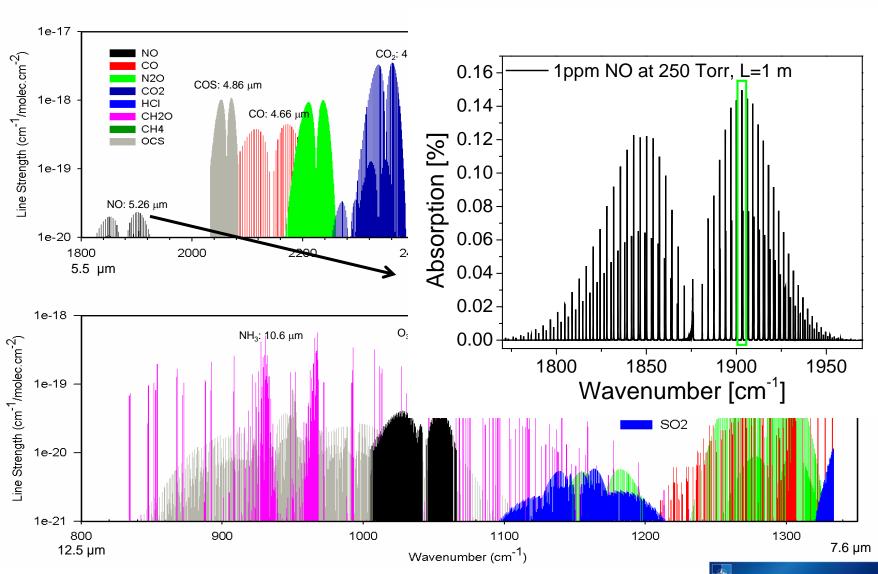


Motivation for Nitric Oxide Detection

- 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

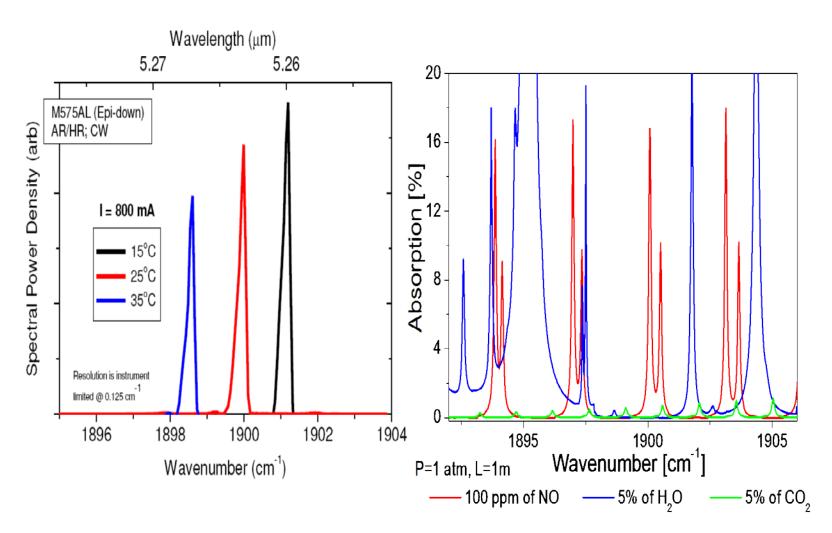


Source: HITRAN 2000 database



This workshop The NATO Science for Peace and Security Programme

Emission spectra of a 1900cm⁻¹ TEC CW DFB QCL and HITRAN Simulated spectra

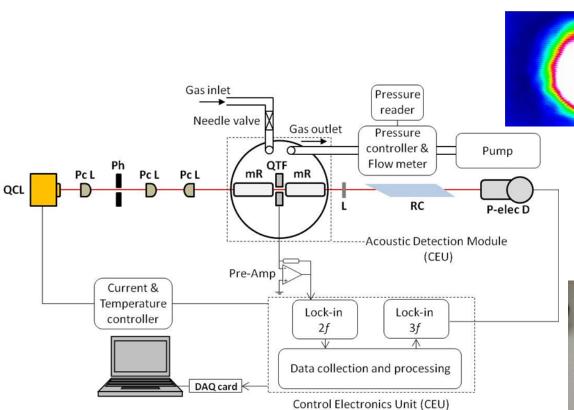






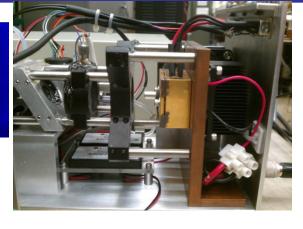


QCL based WMS QEPAS NO Gas Sensor Platform



Schematic of a QCL QEPAS based Gas Sensor. M – mirror, PcL – plano-convez lens, Ph – pinhole, QTF – quartz tuning fork, mR – acoustic micro-resonator, RC – reference cell.



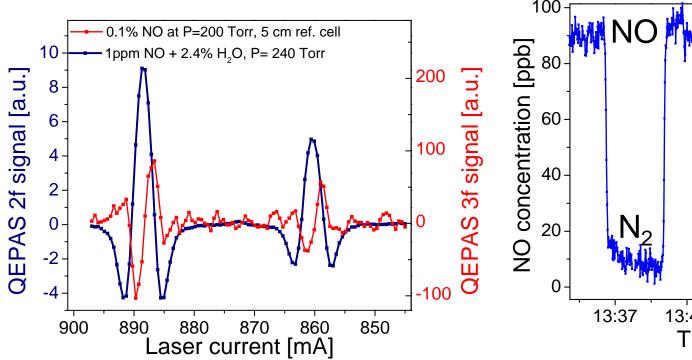


CW HHL TEC DFB-QCL package and IR camera image of the laser beam at 630 mA and 20.5 deg C through tubes after ADM



Compact Prototype NO Sensor (September 2012)

Performance of 2012 CW DFB-QCL based WMS QEPAS NO Sensor Platform



95 ppb NO reference cylinder 13:40 13:43 13:46 13:49 Time [HH:MM]

2f QEPAS signal (navy) and reference 3f signal (red) when laser was tuned across 1900.08 cm⁻¹ line.

2f QEPAS signal amplitude for 95 ppb NO when laser was locked to the **1900.08 cm⁻¹** line.

Minimum detectable NO concentration is:

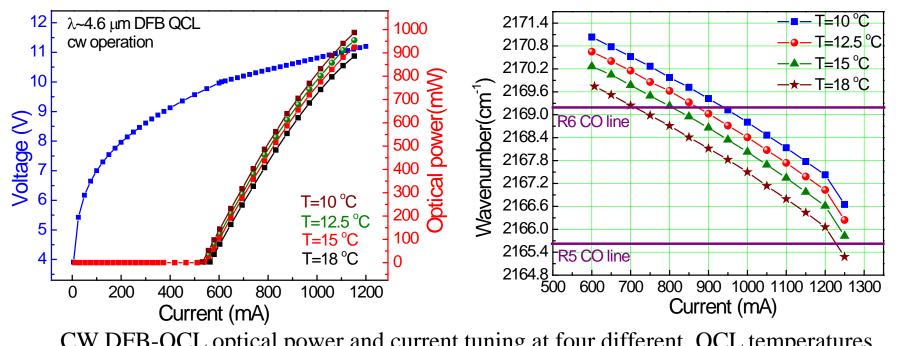
~ 3 ppbv (1σ ; 1 s time resolution)



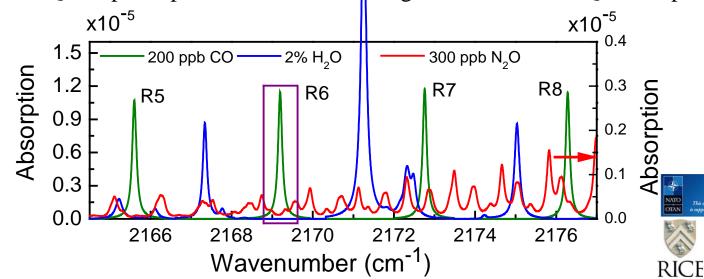
Motivation for Carbon Monoxide Detection

- 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. CH₄).
- Public Health
 - Extremely dangerous to human life even at a low concentrations. Therefore CO must be carefully monitored at low concentration levels.
- 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 4.6 µm high power CW TEC DFB QCL

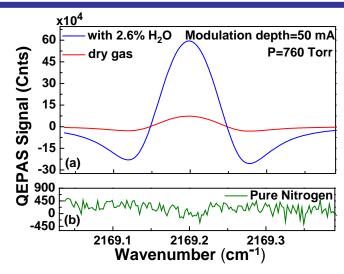


CW DFB-QCL optical power and current tuning at four different QCL temperatures.

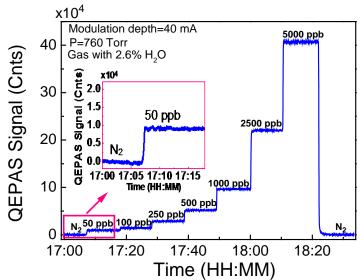


CQD

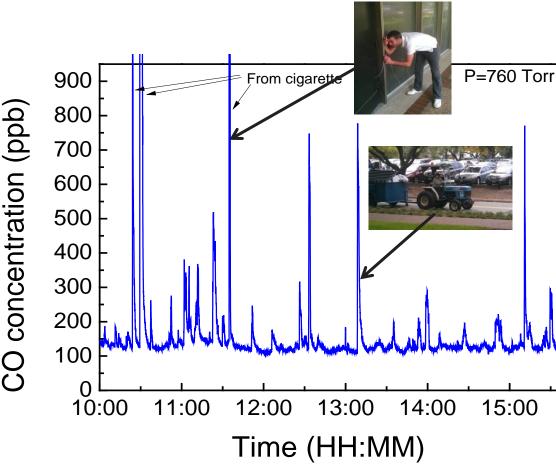
CW DFB-QCL based CO QEPAS Sensor Results



2f QEPAS signal for dry (red) and moisturized (blue) 5 ppm CO:N₂ mixture near **2169.2** cm⁻¹.



Dilution of a 5 ppm CO reference gas mixture when the CW DFB-OCL 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)



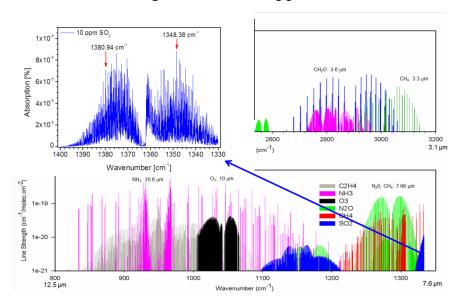


CW DFB-QCL based SO₂ QEPAS Results

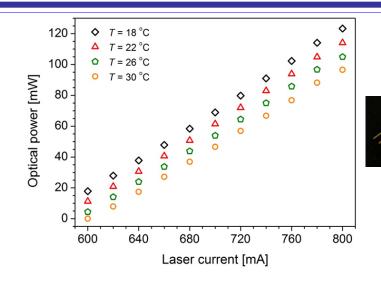
Motivation for Sulfur Dioxide Detection

- Prominent air pollutant
- Emitted from coal fired power plants (~73%) and other industrial facilities (~20%)
- In atmosphere SO₂ converts to sulfuric acid

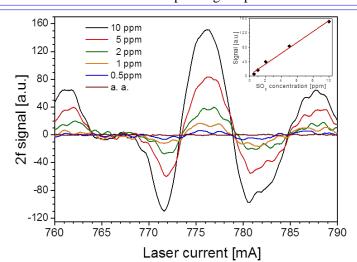
 → primary contributors to acid rain
- SO₂ reacts to form sulfate aerosols
- Primary SO₂ exposure for 1 hour is 75 ppb
- SO₂ exposure affects lungs and causes breathing difficulties
- •Currently, reported annual average atmospheric SO_2 concentrations range from ~ 1 6 ppb



Molecular Absorption Spectra within two Mid-IR Atmospheric Windows



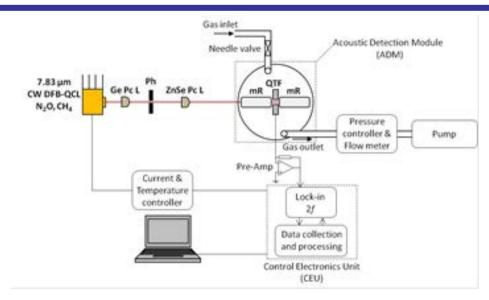
7.24 µm CW DFB-QCL optical power and current tuning at three different operating temperatures.



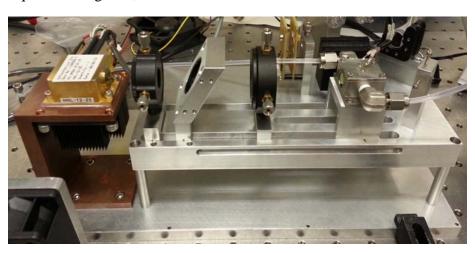
2f WMS QEPAS signals for different SO₂ concentrations when laser was tuned across 1380.9 cm⁻¹ line.

Minimum detectable SO_2 concentration is: ~ 100 ppby (1 σ ; 1 s time resolution)

CW TEC DFB QCL based QEPAS CH₄ & N₂O Gas Sensor



Ge Pc L and ZnSe Pc L – plano-convex lenses, Ph – pinhole, QTF – quartz tuning fork, mR – acoustic micro-resonator

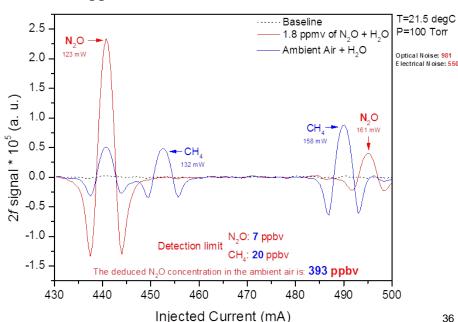


Motivation for CH₄ Detection

- Prominent greenhouse gas
- Leakage from Natural Gas Systems
- Animal Husbandry
- Fossil Fuel Production

Motivation for N₂O Detection

- Major greenhouse gas and air pollutant
- Agriculture
- Fossil Fuel Combustion
- Wastewater Management
- Industrial processes
- Medical Applications



QCL based QEPAS Performance for 10 Trace Gas Species (February 2013)

Molecule (carrier gas)	Frequency cm ⁻¹	Pressure Torr	NNEA cm ⁻¹ W/Hz ^{1/2}	QCL Power mW	NEC (τ=1s) ppbV
CH ₂ O (N ₂ :75% RH)*	2804.90	75	8.7×10 ⁻⁹	7.2	120
CO (N ₂ + 2.2% H ₂ O)*	2176.28	100	1.57×10 ⁻⁸	71	2
CO (propylene)	2196.66	50	7.4×10 ⁻⁸	6.5	140
N ₂ O (air+5%SF ₆)	2195.63	50	1.5×10 ⁻⁸	19	7
N ₂ O (N ₂ +2.37%H ₂ O)	2201.75	200	2.9×10 ⁻⁸	70	2.5
C ₂ H ₅ OH (N ₂)**	1934.2	770	2.2×10 ⁻⁷	10	$9x10^{4}$
NO (N ₂ +H ₂ O)	1900.07	250	7.5×10 ⁻⁹	100	3.6
SO ₂ (N ₂ +2.4%H ₂ O)	1380.94	100	2.0×10 ⁻⁸	40	100
N ₂ O (air)	1275.49	230	5.3×10 ⁻⁸	100	30
CH ₄ (air)	1275.39	230	1.7×10 ⁻⁷	100	118
C ₂ HF ₅ (N ₂)***	1208.62	770	7.8×10 ⁻⁹	6.6	9
NH ₃ (N ₂)*	1046.39	110	1.6×10 ⁻⁸	20	6
SF ₆ ***	943.73	75	2.7×10 ⁻¹⁰	40	5×10 ⁻²

^{* -} Improved microresonator

NNEA – normalized noise equivalent absorption coefficient.

NEC – noise equivalent concentration for available laser power and $\tau=1s$ time constant, 18 dB/oct filter slope.

For comparison: conventional PAS 2.2 (2.6)×10⁻⁹ cm⁻¹W/ $\sqrt{\text{Hz}}$ (1,800; 10,300 Hz) for NH₃*, (**)

^{** -} Improved microresonator and double optical pass through ADM

^{*** -} With amplitude modulation and metal microresonator

Merits of QEPAS based Trace Gas Detection

- Very small sensing module and sample volume (a few mm³ to ~2cm²)
- Extremely low dissipative losses
- Optical detector is not required
- Wide dynamic range
- Frequency and spatial selectivity of acoustic signals
- Rugged transducer quartz monocrystal; can operate in a wide range of pressures and temperatures
- Immune to environmental acoustic noise, sensitivity is limited by the fundamental thermal TF noise: k_BT energy in the TF symmetric mode
- Absence of low-frequency noise: SNR scales as \sqrt{t} , up to t=3 hours as experimentally verified

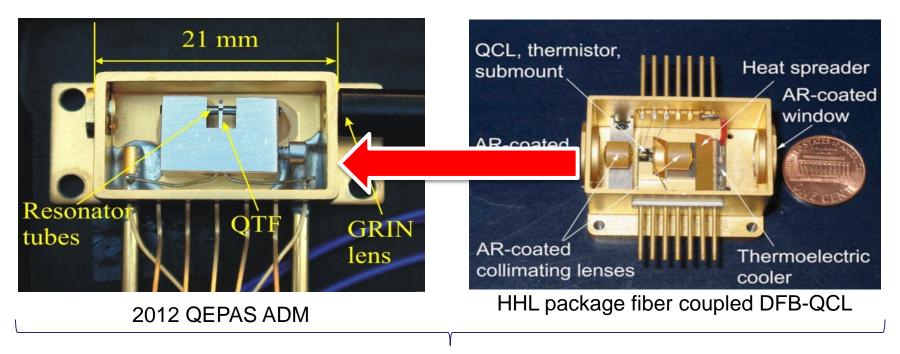
QEPAS: some challenges

- Cost of Spectrophone assembly
- Sensitivity scales with laser power
- Effect of H₂O
- Responsivity depends on the speed of sound and molecular energy transfer processes
- Cross sensitivity issues

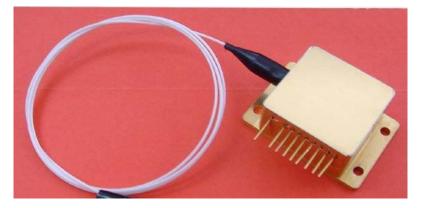
Future Directions and Outlook

- New target analytes such as OCS, CH₂O, nitrous acid (HNO₂), H₂O₂, ethylene (C₂H₄), propane (C₃H₈), and benzene (C₆H₆)
- Ultra-compact, low cost, robust sensors (e.g. C₂H₆, NO, CO...)
- Monitoring of broadband absorbers: acetone (C_3H_6O), acetone peroxide (TATP), UF₆,...
- Low divergence surface emitting quantum cascade lasers
- Finite element modeling for on-axis and off-axis microresonators for QEPAS
- Potential Integration of DFB-QCL or ICL& QEPAS Spectrophone
- Novel optical power build-up cavity designs
- Development of trace gas sensor networks

Potential Integration of a CW DFB- QCL and QEPAS Absorption Detection Module



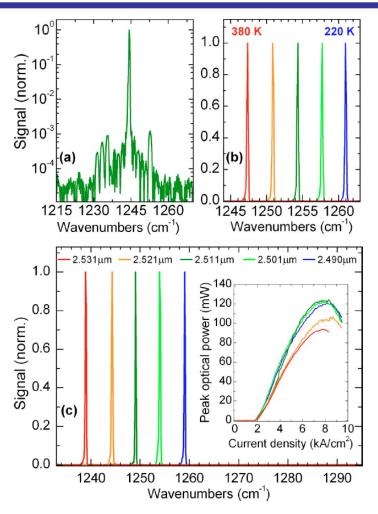
A. Lyakh, et al "1.6 W high wall plug efficiency, continuous-wave room temperature quantum cascade laser emitting at 4.6 μ m", Appl. Phys. Lett. **92**, 111110 (2008)







Ring Resonator-based Surface Emitting QCL



2 experiment simulation 0.8 Device direction y (°) Signal (norm.) 0.2 ب -5 2 Device direction x (°)

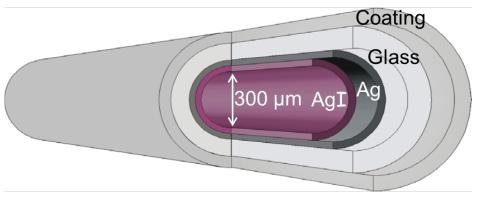
(a) Single-mode spectrum (b) Surface emission spectra recorded at different temperatures. (c) Linear tuning of the resonance. The inset illustrates the corresponding optical power curves.

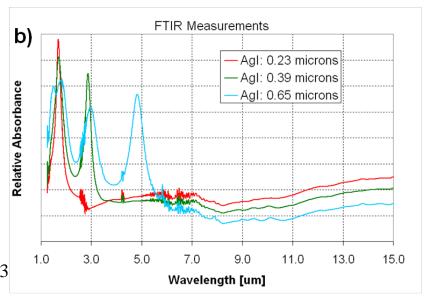
Measured (left half) and simulated (right half) surface emission far-field pattern.



Hollow Core Glass Waveguide

- Hollow Core Glass Waveguides
 - Excellent Infrared transmission out to 20 μm
 - Proven single mode delivery for bore size ~30λ
 - No end reflections
 - High damage threshold
 - Very Robust





For more details: V. Spagnolo, 9:50-10:10 am, Feb 28, 2013 Also to appear in Appl.Phys. B 2013





Summary

- Laser spectroscopy with mid-infrared, TEC, CW, DFB laser diodes, high performance DFB QCLs and tunable EC-QCLs is a promising analytical approach for real time environmental, biomedical and industrial monitoring as well as technology for national security.
- Six mid-infrared from Nanoplus, Daylight Solutions, Maxion Technologies (Thor Labs), Hamamatsu, Northwestern University and Adtech Optics were used recently (2011-2013) in three sensor platforms: TDLAS, PAS and QEPAS
- 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 130 pptv using TDLAS
 - NH₃ at $\sim 10.4 \,\mu\text{m}$ with a detection sensitivity of $\sim 1 \,\text{ppbv}$ (200 sec averaging time);
 - NO at ~5.26µm with a detection limit of 3 ppbv
 - CO at ~ 4.61µm with minimum detection limit of 2 ppbv
 - SO_2 at ~7.24µm with a detection limit of 100 ppbv
 - CH₄ and N₂O at ~7.28 μm concentration are <u>currently in progress</u> with detection limits of 20 and 7 ppbv, respectively.

