perspective

lectures

The state-of-the-art and grand Challenges of Mid-infrared Technology and Applications

RICE

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OUTLINE

US-UK Workshop in Mid-IR to THz Technology and Applications

Royal Society of Edinburgh, UK

Feb. 18-19, 2013

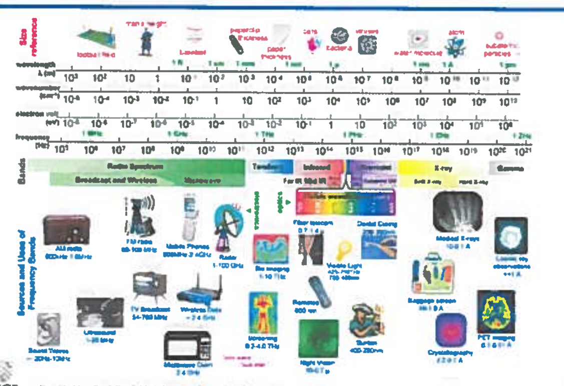
- New Laser Based Trace Gas Sensor Technology
  - Novel Multipass Absorption Cell & Electronics
  - Quartz Enhanced Photoacoustic Spectroscopy
- Seven Examples of Mid-Infrared Sensor Architectures
  - C_2H_6, NH_3, NO, CO, SO_2, CH_4 & N_2O
  - Future Directions of Laser Based Gas Sensor Technology and Conclusions

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

(1)

international team

Mid-IR and THz Spectroscopic Phenomena



Provided by: Prof. Daniel Mittleman, Rice University, Houston, TX

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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, 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**
  - Breath Analysis
- **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, 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)

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Other Spectroscopic Methods

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

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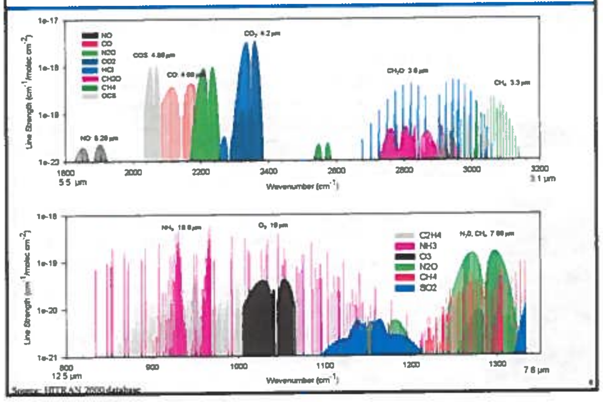
HITRAN Simulated Mid-Infrared Molecular Absorption Spectra



Source: HITRAN 2000 database

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

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
  - ~100cm^{-1} using current and temperature control for QCL DFB Array
  - ~ 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
  - > 1W, TEC CW DFB @ 4.6 μm
  - > 600 mW (CW FP) @ RT, wall plug efficiency of ~17 % at 4.6 μm;

4 mm

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 TEM_{00} 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 (temperature, pressure, etc…)

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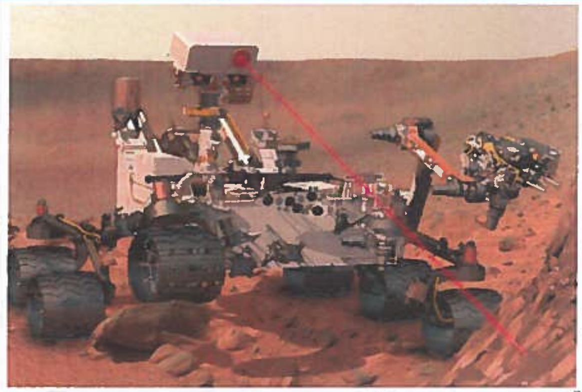
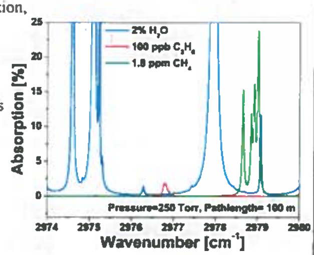
"Curiosity" landed on Mars on August 6, 2012



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,
  - schizophremia,
  - Lung cancer,
  - lung cancer,
  - vitamin E deficiency



HITRAN absorption spectra of C_2H_6, CH_4, and H_2O

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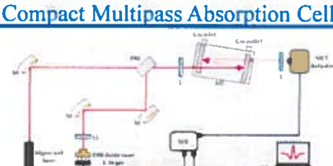
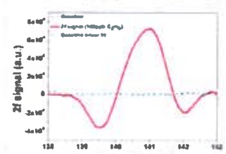
C_2H_6 Detection with a 3.36 µm DFB LD using a Novel Compact Multipass Absorption Cell and Control Electronics



Schematic of a C_2H_6 gas sensor using a Nanoplus 3.36 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

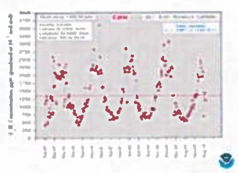
Minimum detectable C_2H_6 concentration is: ~ 130 pptv (1σ; 1 s time resolution)

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

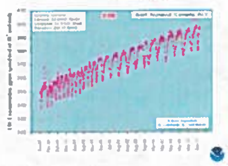
3.36 µm – CW DFB – GaSb based laser diode
2169.2 cm^{-1} @ 9.5°C opt: 2986.7 cm^{-1}
> 10mW
reference cell?

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

General view on Alert NOAA Facility



Ethane Concentration Measurements at ALT



CO_2 Concentration Measurements at ALT

Motivation for NH_3 Detection

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

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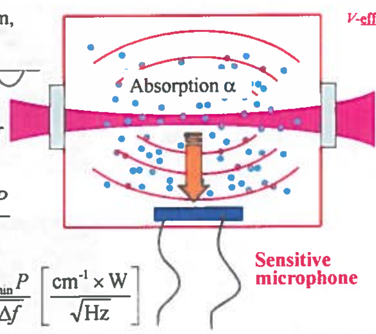
Conventional Photoacoustic Spectroscopy (PAS)

Cell is OPTIONAL!

V-effective volume

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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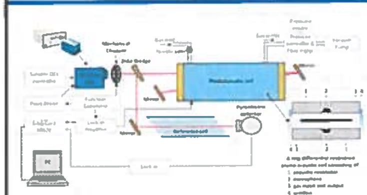
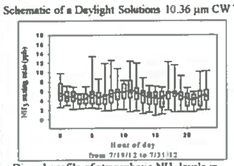
Atmospheric NH_3 Measurements using a CW EC-QCL PAS Sensor

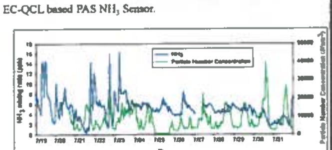


Schematic of a Daylight Solutions 10.36 μm CW TEC EC-QCL based PAS NH_3 Sensor.

NH_3 sensor deployed at the UH Moody Tower rooftop monitoring site.



Diurnal profile of atmospheric NH_3 levels in Houston, TX.



Comparison between NH_3 and particle number concentration time series from July 19 to July 31 2012.

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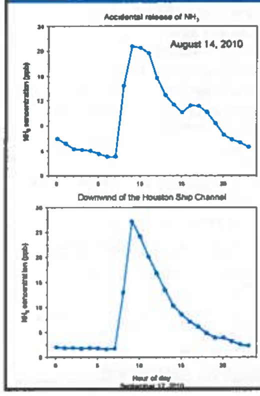
unexpected benefits of remote detection.

NH_3 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 NH_3 emission from the Houston Ship Channel area is about 0.25 ton. Mellqvist et al., (2007) Final Report, HARC Project H-53.

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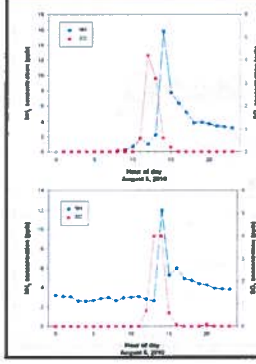
Sporadic increased NH_3 Concentration Levels related to NO_x Emission Reduction by the Parish Electric Power Plant, TX



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

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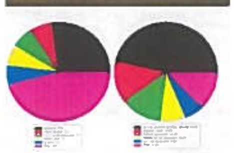
Atmospheric NH_3 measurements and implications for PM formation near Fort Worth

Four specific aims are being pursued:

- Investigation of the NH_3 dynamics (e.g., temporal variation);
- Identification of NH_3 sources using auxiliary data for CO and NO_x;
- Performing NH_3 source apportionment using the EPA Positive Matrix Factorization 3.0 model including VOC, data;
- Evaluation of NH_3 effects on local and regional air quality with respect to PM formation using aerosol/PM data collected by an Aerodyne High Resolution Time-of-Flight Mass Spectrometer, a Scanning Electrical Mobility Spectrometer, and a Mist Chamber-Ion Chromatography system.



(a) (b)

(a) Annual total air releases by species in the DFW area (U.S. EPA TRI 2012); (b) Annual NH_3 emissions by source categories in Tarrant County, Texas (U.S. EPA NEI 2008)

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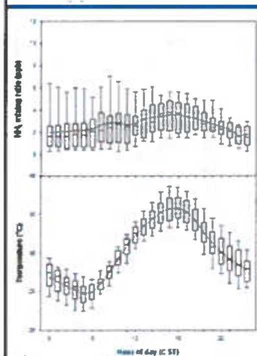
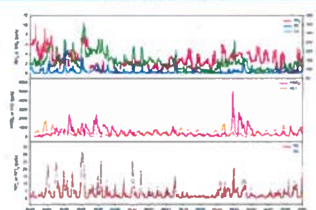
NH_3 vs. Temperature data & Time series of NH_3, SO_2, CO





- Emission events from specified point sources (i.e., industrial facilities)
- Estimated NH_3 emissions from cows (1.3 tons/day)
- Estimated NH_3 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 NH_3 emissions
- Increased contribution from industry (→ 18.9%)

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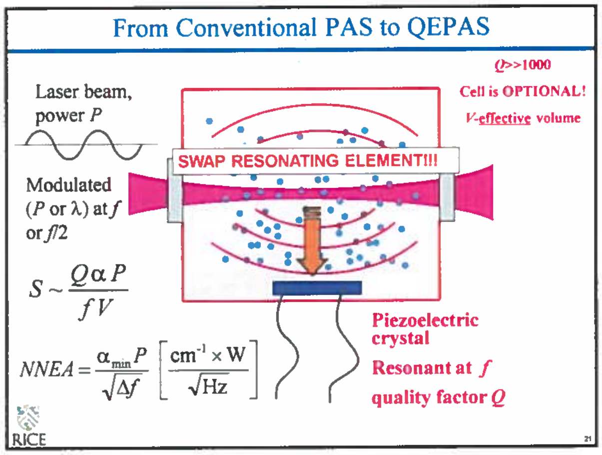

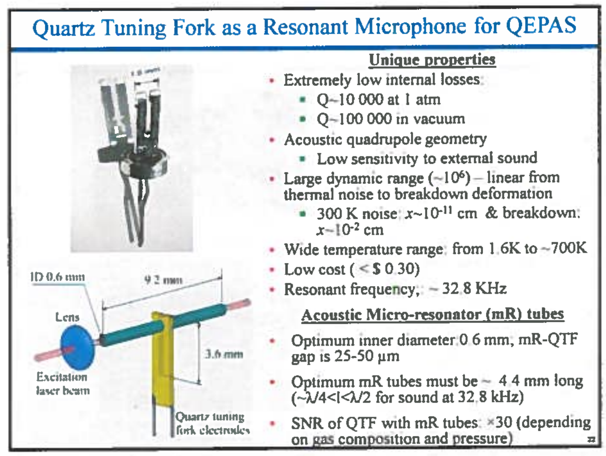

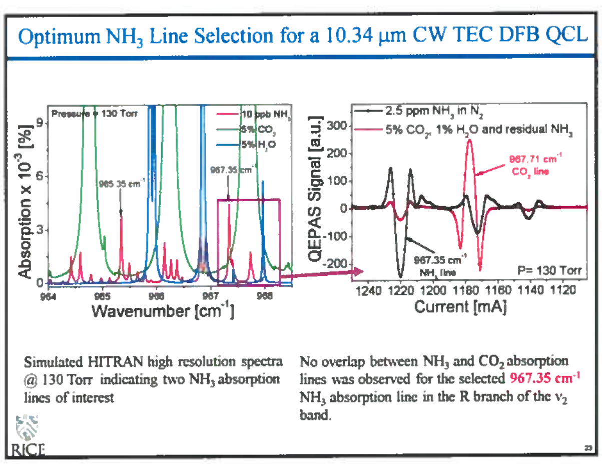

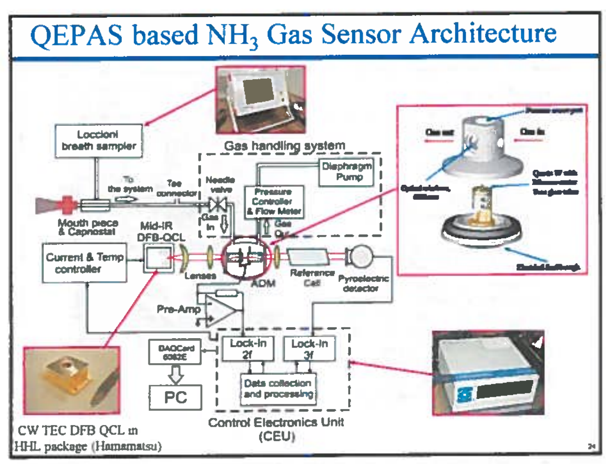

autonomous
skype repair.

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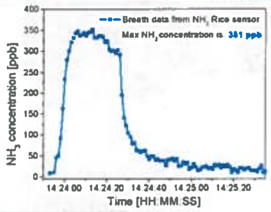
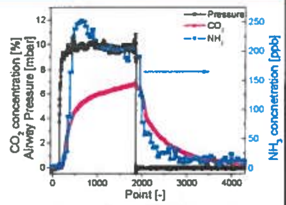
Real-time exhaled human NH_3 Breath Measurements





Airway pressure (black), CO_2 (red), and NH_3 (blue) profiles of a single breath exhalation lasting 40sec.

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 NH_3 is: ~ 6 ppbv at 967.35 cm^{-1} (1σ; 1 s time resolution)

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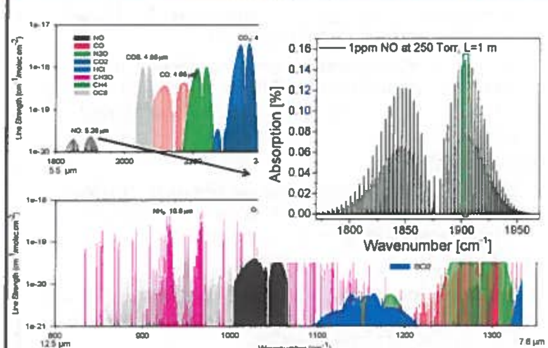
Journal of breath analysis

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

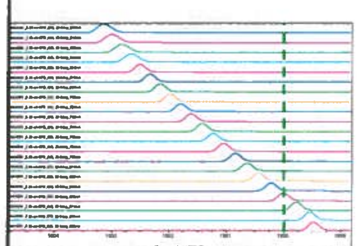
Molecular Absorption Spectra within two Mid-IR Atmospheric Windows and NO absorption @ 5.26µm



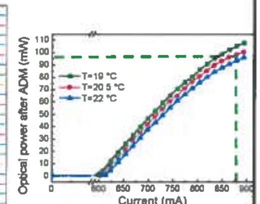
Source: HITRAN 2000 database

marion (Thorlabs)

Performance of a 5.26 µm CW HHL TEC DFB-QCL



Single frequency QCL radiation recorded with FTIR for different laser current values at a QCL temperature of 20.5°C.



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

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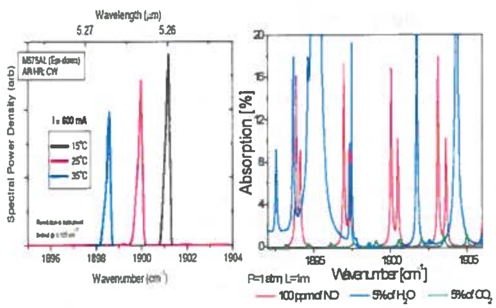
Emission spectra of a 1900cm^{-1} TEC CW DFB QCL and HITRAN Simulated spectra



Output power: 117 mW @ 25 C

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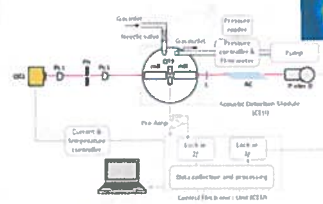
CW TEC DFB QCL based QEPAS NO Gas Sensor



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 650 mA and 20 deg C through tubes after ADM

Compact Prototype NO Sensor
(September 2012)

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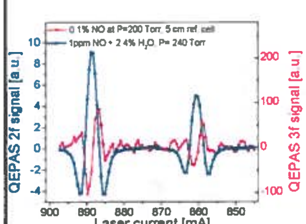
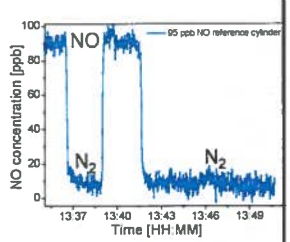
Performance of CW DFB-QCL based WMS QEPAS NO Sensor Platform



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



2f 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).
- 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.

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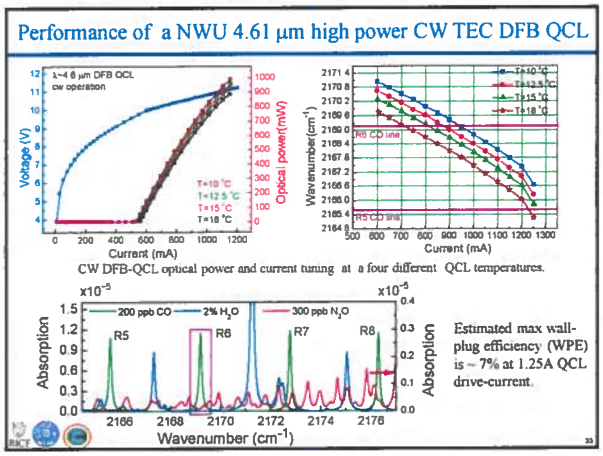
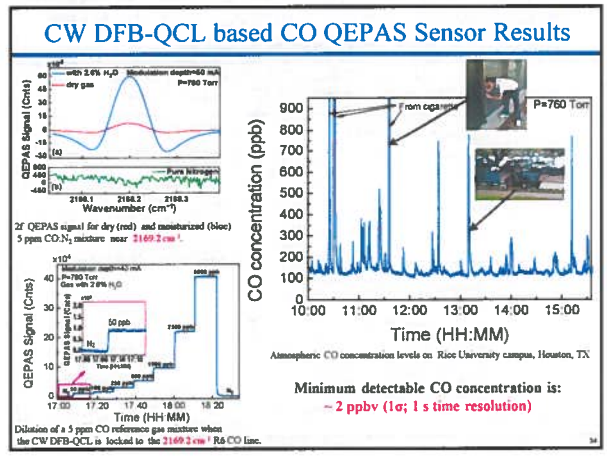
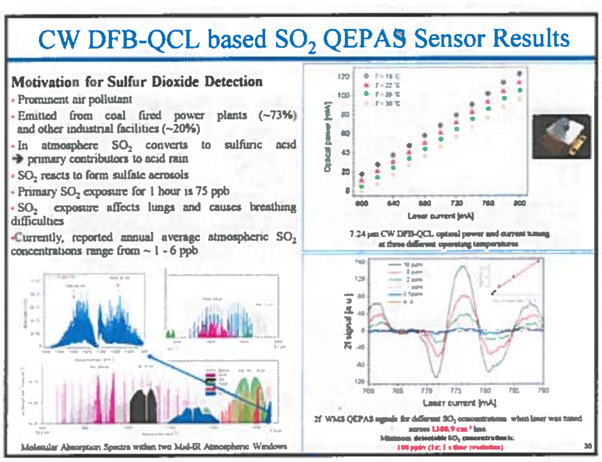
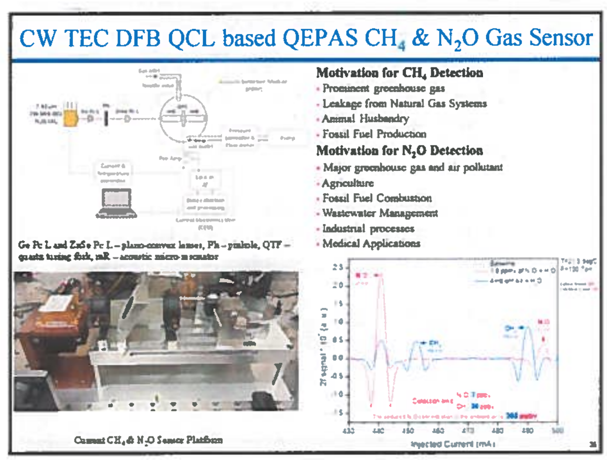

Performance of a NWU 4.61 μm high power CW TEC DFB QCL
λ~4.6 μm DFB QCL
cw operation
Voltage (V)
Optical power(mW)
Current (mA)
T=10 °C
T=12.5 °C
T=15 °C
T=18 °C
Wavenumber(cm-1)
R6 CO line
R5 CO line
Current (mA)
CW DFB-QCL optical power and current tuning at a four different QCL temperatures.
200 ppb CO
2% H2O
300 ppb N2O
R5
R6
R7
R8
Absorption
Wavenumber (cm-1)
Estimated max wall-plug efficiency (WPE) is ~ 7% at 1.25A QCL drive-current.


CW DFB-QCL based CO QEPAS Sensor Results
QEPAS signal (Cnts)
with 2.6% H2O
dry gas
Modulation depth=50 mA
P=760 Torr
Pure Nitrogen
Wavenumber (cm-1)
2f QEPAS signal for dry (red) and moisturized (blue) 5 ppm CO:N2 mixture near 2169.2 cm-1.
QEPAS Signal (Cnts)
P=760 Torr
50 ppb
N2
Time (HH:MM)
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.
CO concentration (ppb)
from cigarette
P=760 Torr
Time (HH:MM)
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 Sensor 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 SO2 concentrations range from ~ 1 - 6 ppb
Optical power (mW)
Laser current (mA)
7.24 μm CW DFB-QCL optical power and current tuning at three different operating temperatures
2f signal (a.u.)
Laser current (mA)
2f WMS QEPAS signals for different SO2 concentrations when laser was tuned across 1380.9 cm-1 line.
Minimum detectable SO2 concentration:
100 ppbv (1σ; 1 s time resolution)
Molecular Absorption Spectra within two Mid-IR Atmospheric Windows


CW TEC DFB QCL based QEPAS CH4 & N2O Gas Sensor
Ge PL and ZnSe PL – plano-convex lenses, Ph – pinhole, QTF – quartz tuning fork, mR – acoustic micro resonator
Current CH4 & N2O Sensor Platform
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
Injected Current (mA)

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QCL based QEPAS Performance for10 Trace Gas Species (February 2013)

Molecule (carrier gas)	Frequency cm^{-1}	Pressure Torr	NNEA cm^{-1}W/Hz^{½}	QCL Power mW	NEC (τ=1s) ppbV
CH_2O (N_2:75% RH)*	2804.90	75	$8.7×10^{-9}$	7.2	120
CO (N_2+ 2.2% H_2O)*	2176.28	100	$1.57×10^{-8}$	71	2
CO (propylene)	2196.66	50	$7.4×10^{-8}$	6.5	140
N_2O (air+5%SF_6)	2195.63	50	$1.5×10^{-8}$	19	7
N_2O (N_2+2.37%H_2O)	2201.75	200	$2.9×10^{-8}$	70	2.5
C_2H_5OH (N_2)**	1934.2	770	$2.2×10^{-7}$	10	$9×10^{4}$
NO (N_2+H_2O)	1900.07	250	$7.5×10^{-9}$	100	3.6
SO_2 (N_2+2.4%H_2O)	1380.94	100	$2.0×10^{-8}$	40	100
N_2O (air)	1275.49	230	$5.3×10^{-8}$	100	30
CH_4 (air)	1275.39	230	$1.7×10^{-7}$	100	118
C_2HF_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
** - 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^{-9}cm^{-1}W/√Hz (1,800; 10,300Hz) 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, *** V. Spagnolo, et al. University and Politecnico of Bari, Italy

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big molecules ✓

Merits of QEPAS based Trace Gas Detection

- Very small sensing module and sample volume (a few mm^3 to ~2cm^2)
- 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 H_2O
- Responsivity depends on the speed of sound and molecular energy transfer processes
- Cross sensitivity issues

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Future Directions and Outlook

- New target analytes such as OCS, CH_2O, nitrous acid (HNO_2), H_2O_2, ethylene (C_2H_4), 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,…
- Low divergence surface emitting quantum cascade lasers
- Finite Element Modeling for on-axis and off-axis micro resonators for QEPAS
- Optical power build-up cavity designs
- Development of trace gas sensor networks

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

2012 QEPAS ADM

HHL package fiber coupled DFB-QCL

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

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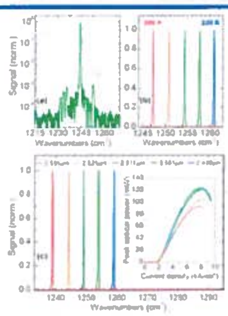
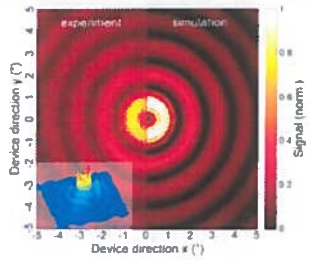
Ring resonator-based surface emitting QCL



(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.

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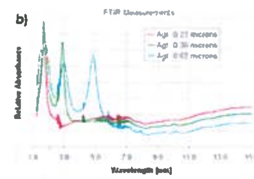
Hollow core waveguide

- 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
  - Bending loss is a primary concern



IFN

OKSI OPTO-KNOWLEDGE SYSTEMS INC.

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George ✓
Noble Barrel
area.

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
  - 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 ~ 4.61µm with minimum detection limit of 2 ppbv
  - SO_2 at ~7.24µm 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.

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Corning.

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1. Thank Prof Reid.
(2) red ~~angle~~ circle, hyperlinked.
QEPAS ~~ten years~~, QCL 25 years, laser 50 yrs.

18 (~~1~~) laser case /#18 (2).

9 color title

older version ✓