



Recent Developments of Diode and Quantum Cascade Laser based Trace Gas Sensor Technology: Opportunities and Challenges

F.K. Tittel, A.A. Kosterev, Yu. Bakhirkin, S. So, G. Wysocki & R.F.Curl

Rice Quantum Institute, Rice University, Houston, TX

<http://ece.rice.edu/lasersci/>

OUTLINE

Coherent
Palo Alto, CA
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- Motivation: Wide Range of Chemical Sensing
- Fundamentals of Laser Absorption Spectroscopy
- Selected Applications of Trace Gas Detection
 - Quartz Enhanced Laser-PAS of NH₃, CO₂, H₂CO and N₂O
 - OA-ICOS CO and NO based Sensor Technology
- Conclusions and Outlook

Motivation: Wide Range of Gas Sensing Applications

- **Urban and Industrial Emission Measurements**
 - Industrial Plants
 - Combustion Sources and Processes (eg. early fire sensing)
 - Automobile and Aircraft Emissions
- **Exploration**
 - Agriculture and Animal Facilities
- **Environmental Monitoring**
 - Atmospheric Chemistry (eg ecosystems and airborne)
 - Volcanic Emissions
- **Chemical Analysis and Industrial Process Control**
 - Chemical, Pharmaceutical, Food & Semiconductor Industry
 - Toxic Industrial Chemical Detection
- **Spacecraft and Planetary Surface Monitoring**
 - Crew Health Maintenance & Human Life Support Program
- **Medical Diagnostics** (eg. breath analysis)
- **Fundamental Science and Photochemistry**

Potential Sensor Applications in the Oil & Gas Industry

- **Industrial and Urban Emission Measurements**
 - Industrial Plants (eg. gas turbines in refineries)
 - Combustion Sources and Processes (eg. early fire sensing)
 - Plant Safety (Fence line perimeter)
 - Automobile and Aircraft Emissions
- **Environmental Monitoring**
 - Atmospheric Chemistry (e.g. ecosystems and airborne)
- **Chemical Analysis and Industrial Process Control**
 - Toxic Industrial Chemical Detection
 - Smokestack emissions
- **Meeting Energy Demanded Technology**
 - Gas to Liquid Technology
 - Exploration (down well monitoring, gas hydrates)
- **Advanced Engine Systems**
 - Advanced Fuels

Trace Gas Monitoring in a Petrochemical Plant



Worldwide Megadirty Mega Cities

	Population, m	Sulphur	Particulate	Lead	Carbon	Nitrogen	Ozone
	1990, ext.	2000, proj.	dioxide	matter	monoxide	dioxide	
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

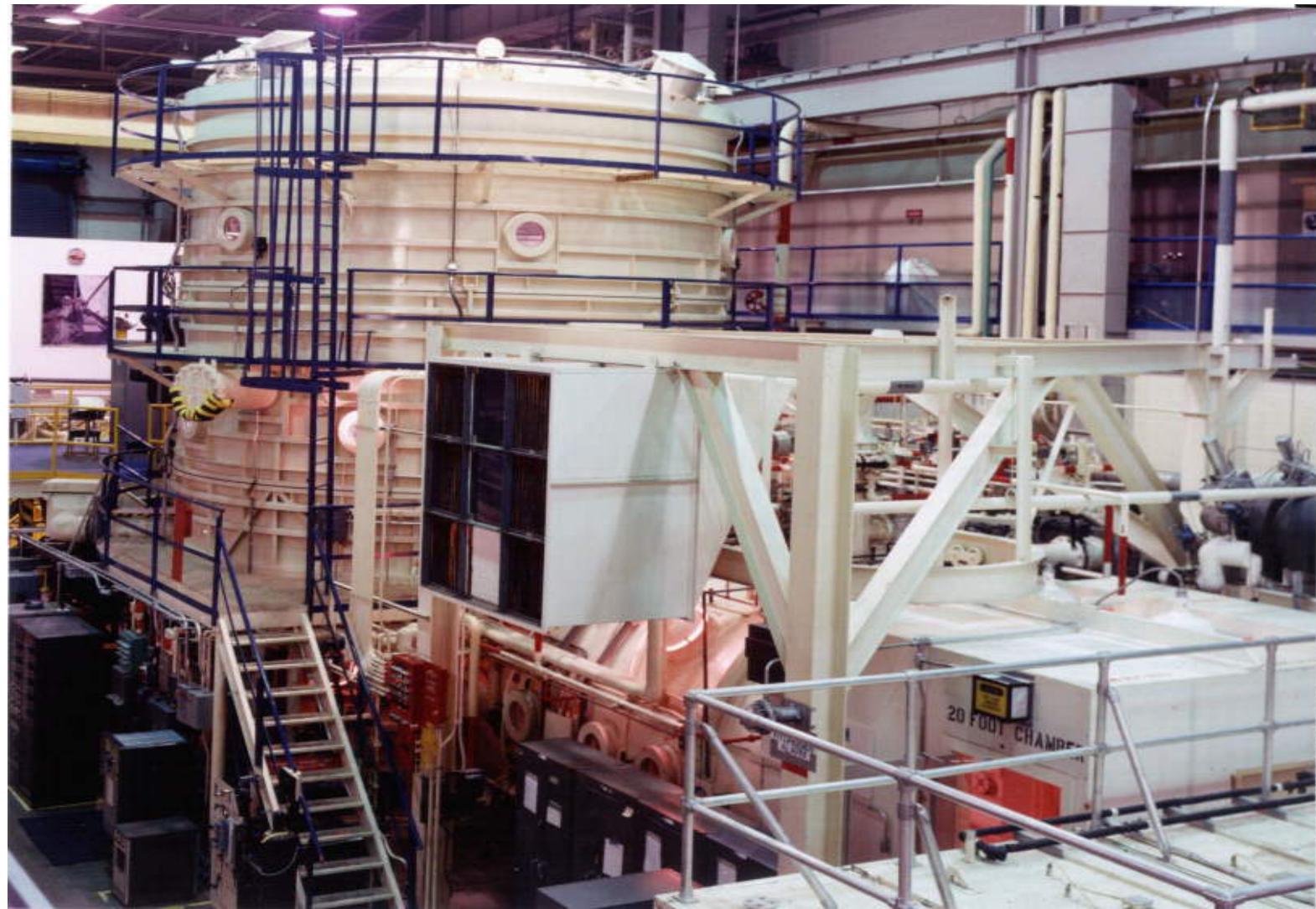


Megacity Air Pollution: Houston, TX



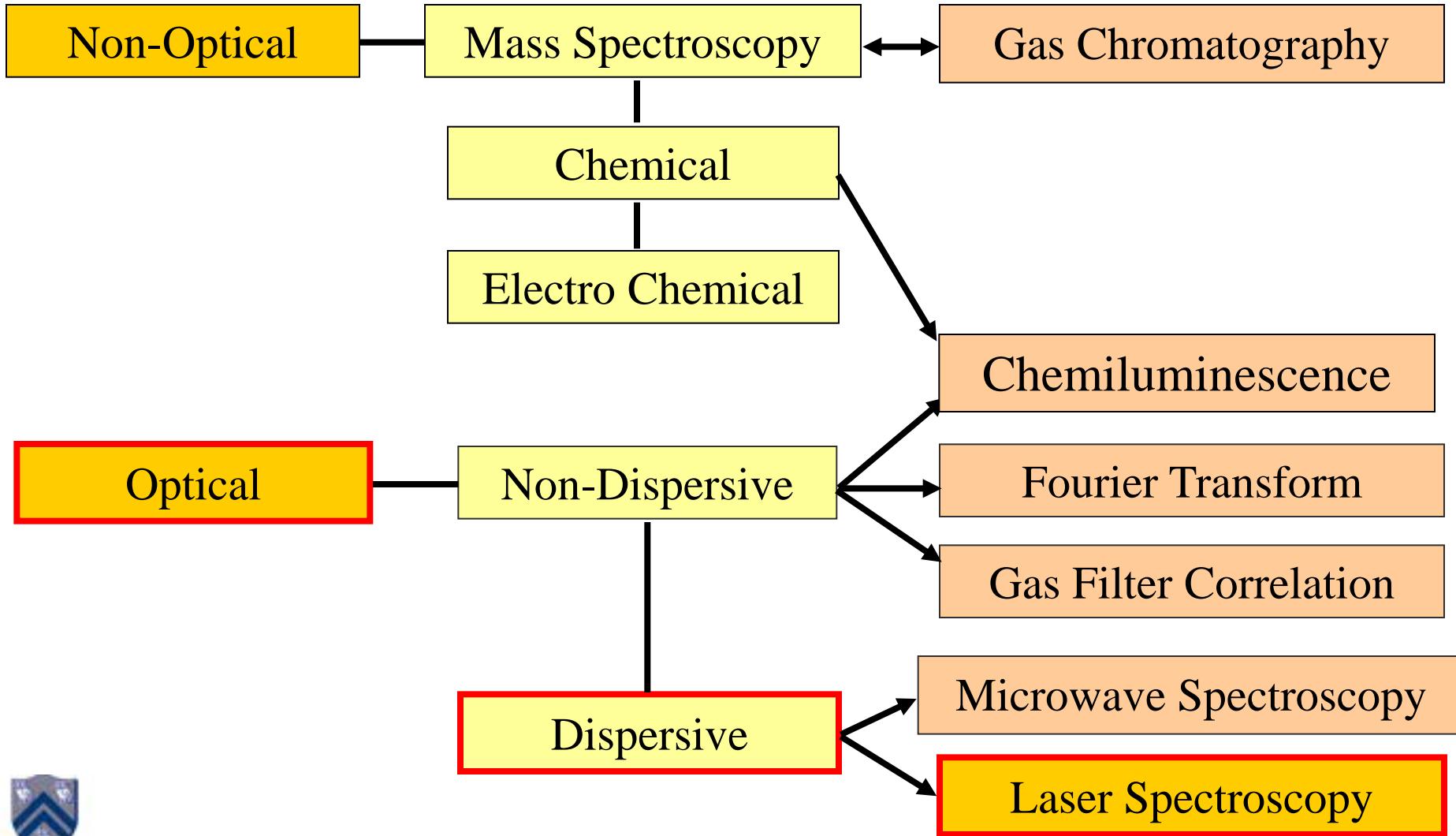
8/21/2000

NASA–JSC Human-Rated Simulation Chamber

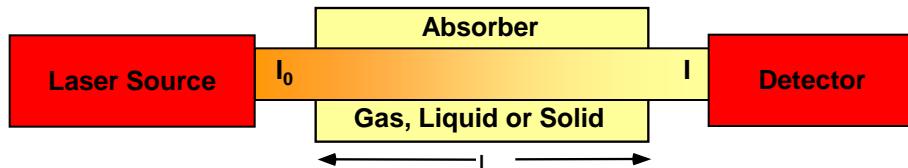


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Existing Methods for Trace Gas Detection



Fundamentals of Laser Absorption Spectroscopy

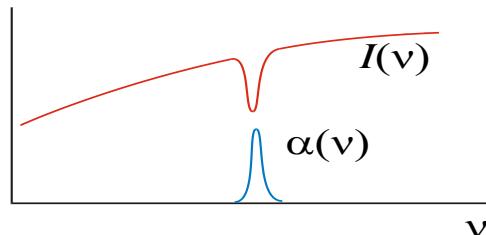


Beer-Lambert's Law of Linear Absorption

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

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

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



$$\alpha(v) = C \cdot S(T) \cdot g(v - v_0)$$

C - total number of molecules of absorbing gas/atm/cm³
[molecule · cm⁻³ · atm⁻¹]

S – molecular line intensity [cm · molecule⁻¹]

$g(v - v_0)$ – normalized spectral lineshape function [cm],
(Gaussian, Lorentzian, Voigt)

Optimum Molecular Absorbing Transition

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

Long Optical Pathlengths

- Multipass Absorption Cell
- Cavity Enhanced and Cavity Ringdown Spectroscopy
- Open Path Monitoring (with retro-reflector)

Spectroscopic Detection Schemes

- Frequency or Wavelength Modulation
- Balanced Detection
- Zero-air Subtraction
- Photoacoustic Spectroscopy

CW IR Source Requirements for Laser Spectroscopy

REQUIREMENTS

- Sensitivity (% to ppt)
- Selectivity
- Multi-gas Components
- Directionality
- Rapid Data Acquisition
- Room Temperature

IR SOURCE

- Power
- Narrow Linewidth
- Tunable Wavelengths
- Beam Quality
- Fast Response
- No Consumables



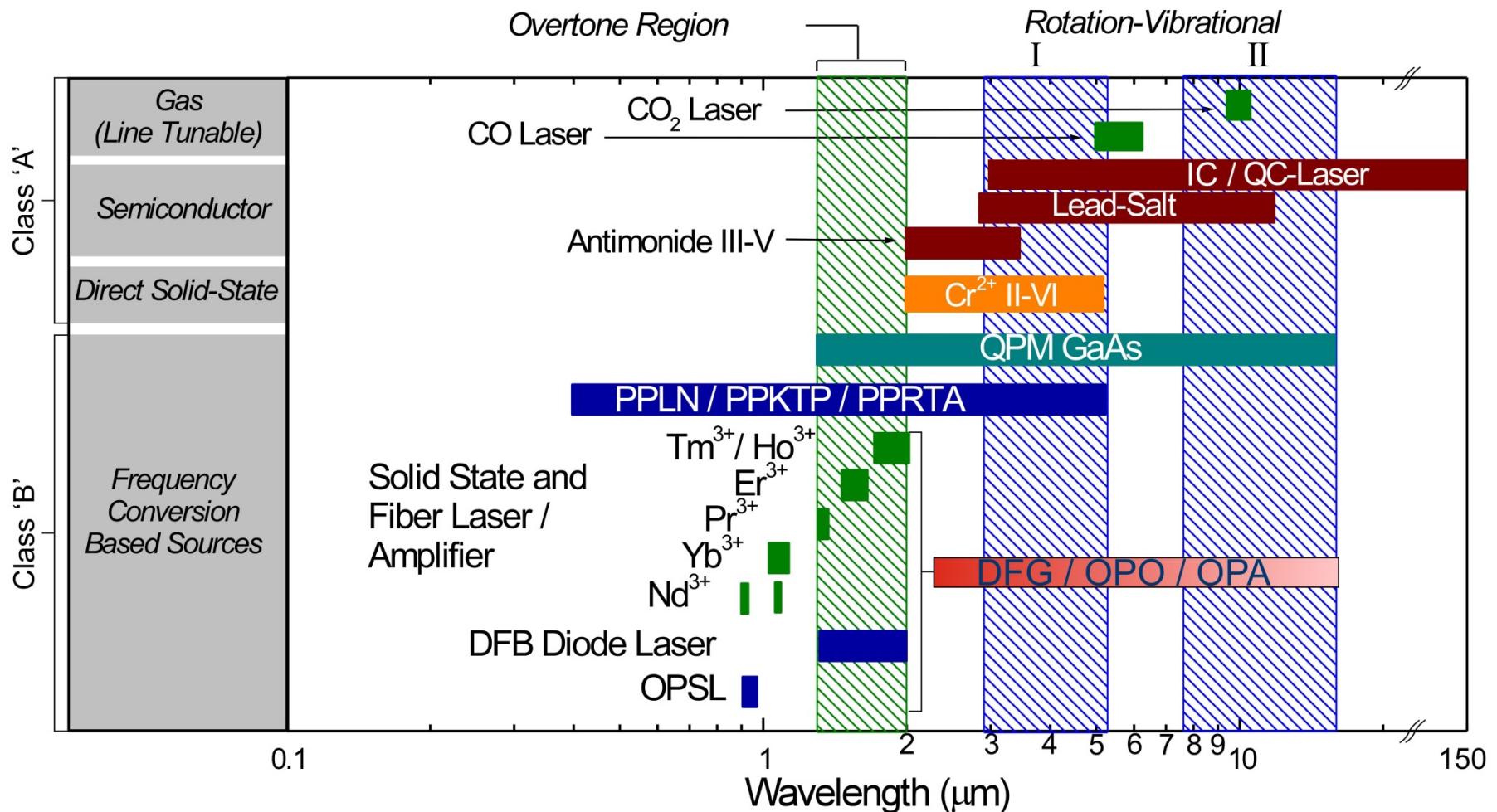
Other Requirements for Laser based Gas Sensors

- Field deployable
- Autonomous, unattended, remote operation and control
- Self calibrating
- Low weight, small size, low power consumption
- Easy to use (avoid complexity)
- Cost of ownership

Laser based Sensor System Considerations

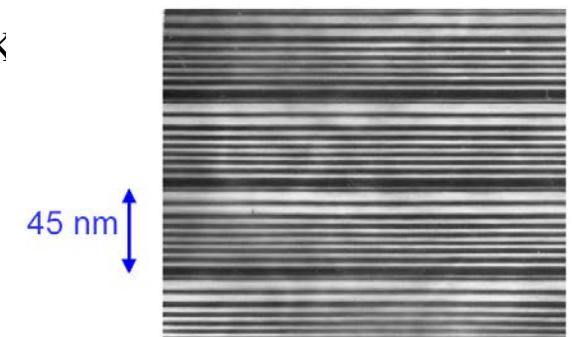
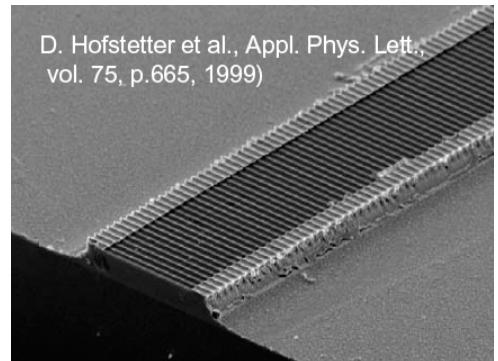
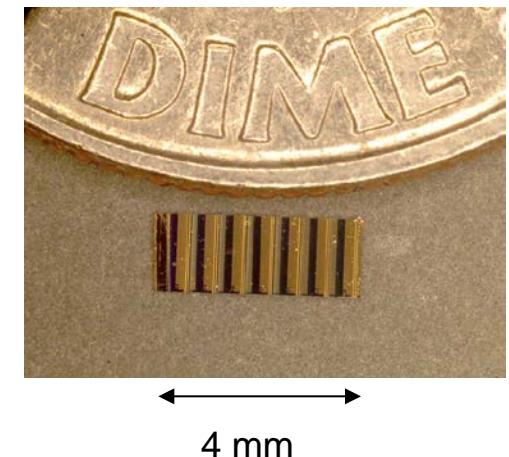
- Design and Architecture (COTS based system)
- Precision and Accuracy (Temperature, Pressure, and Humidity)
- Inlet design (if extractive)
 - Gas sampling (local-remote)
 - Filtering and pre-concentration
 - Calibration
- Data acquisition and signal processing
 - Ethernet and GPS option
- Intercomparison with FTIR or GC-MS

IR Laser Sources and Wavelength Coverage

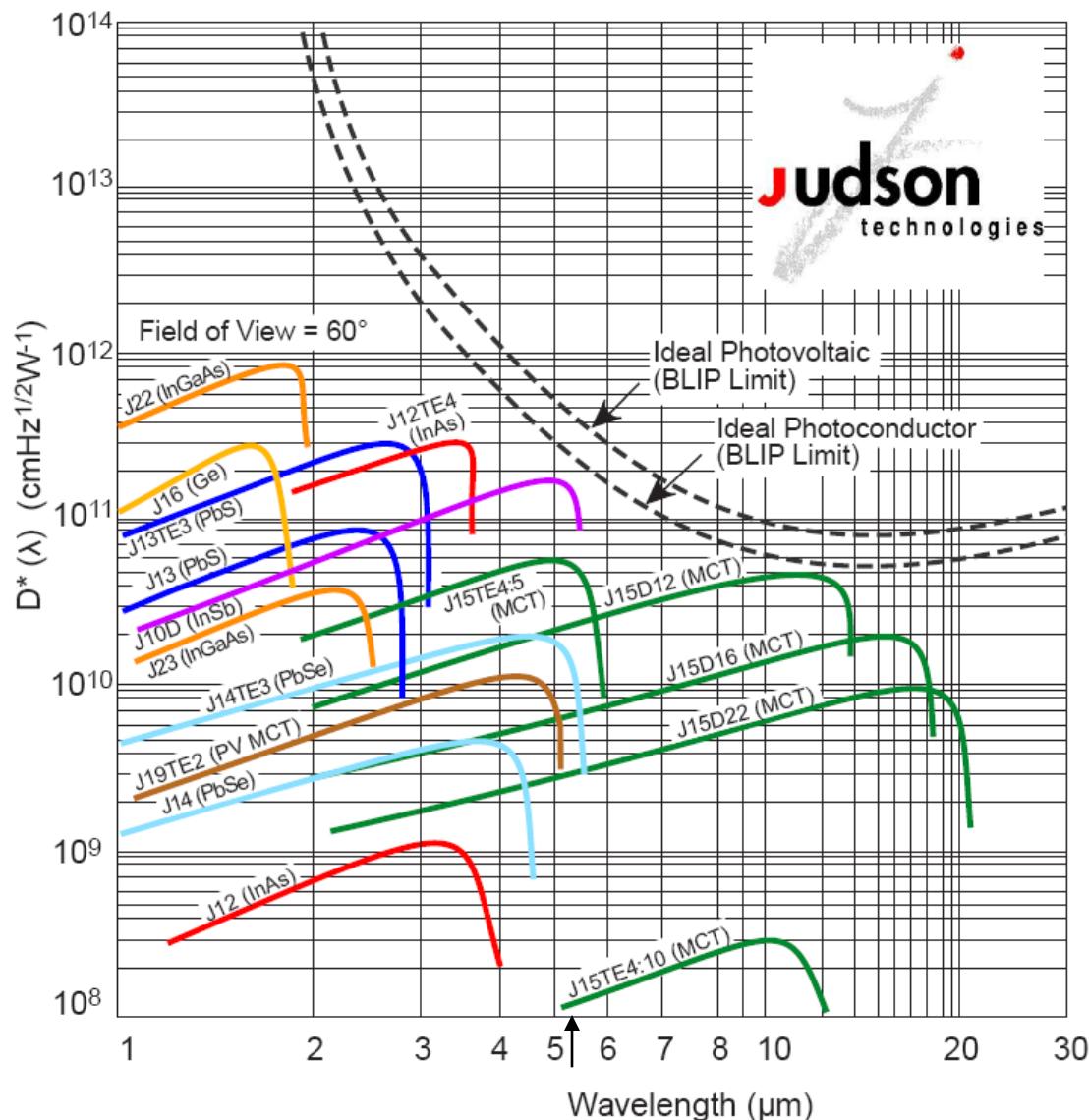


Quantum and Interband Cascade Laser: Basic Facts

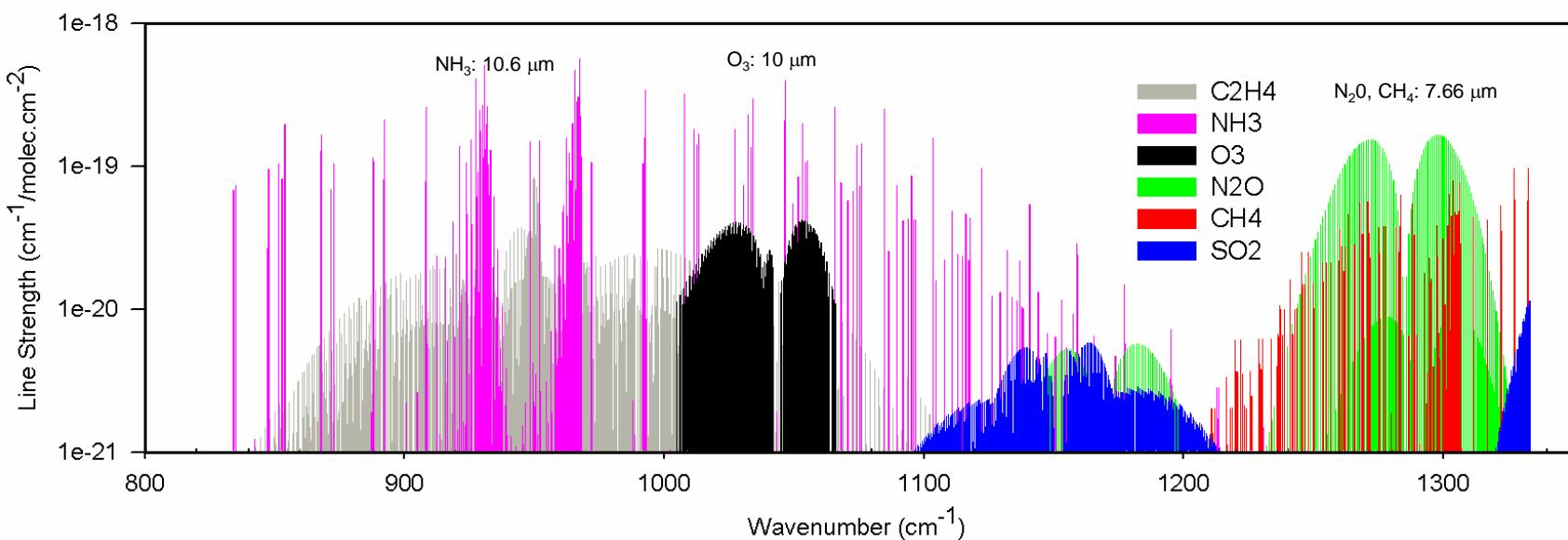
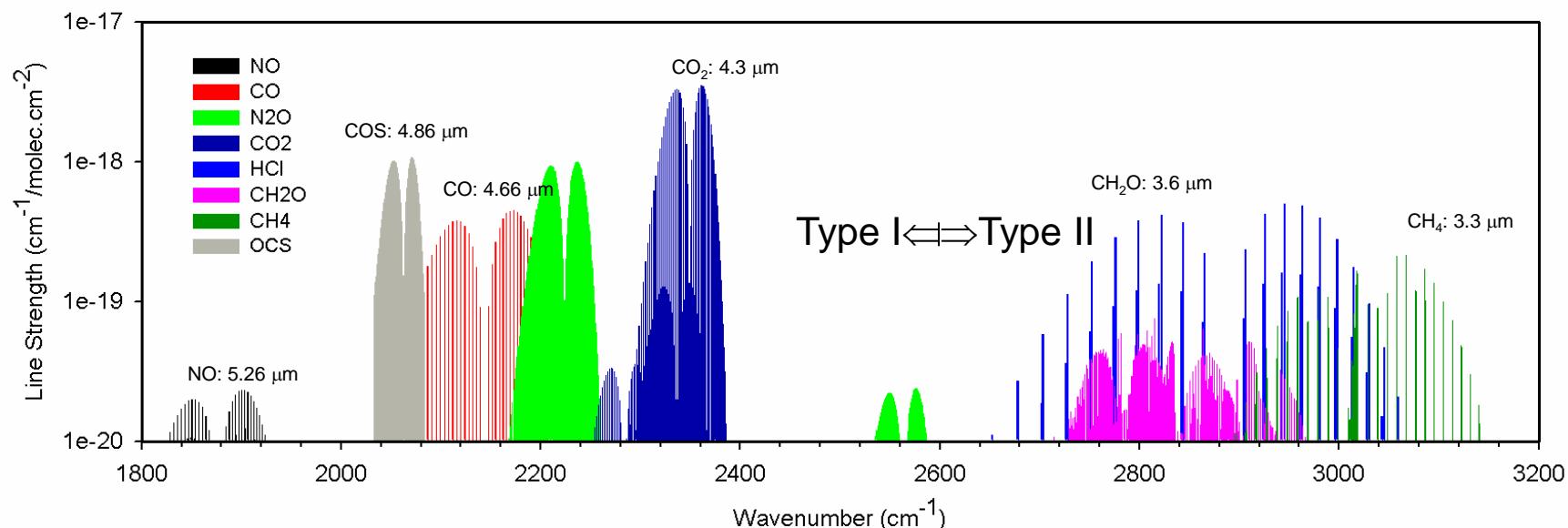
- Band – structure engineered devices (emission wavelength is determined by layer thickness – MBE or MOCVD) QCLs operate from 4 -160 μm (limited by the CB offset on the short wavelength side)
 - Unipolar devices
 - Cascading (each electron creates N laser photons and the number of periods N determines laser power)
- Compact, reliable, stable, long lifetime, commercial availability
- Fabry-Perot (FP) or single mode (DFB)
- Broad spectral tuning range in the mid-IR (4-24 μm for QCLs and 3-5 μm for ICLs)
 - 1.5 cm^{-1} using current
 - 5-20 cm^{-1} using temperature
 - $> 150 \text{ cm}^{-1}$ using an external grating element
- Narrow spectral linewidth cw, 0.1 - 3 MHz & $< 10\text{Khz}$ with frequency stabilization
Linewidth is $\sim 300 \text{ MHz}$ of pulsed QCLs (chirp from heating)
- High output powers
 - Pulsed peak powers of 1.6 W; high temperature operation $\sim 425 \text{ K}$
 - Average power levels: 1-600 mW
 - $\sim 50 \text{ mW}$, TEC CW DFB (Alpes)
 - $> 600 \text{ mW}$ (CW FP) and $> 150 \text{ mW}$ (CW DFB) at 298 K (Northwestern)



Wavelength Coverage of IR Detectors



HITRAN Simulation of Absorption Spectra (3.1-5.5 & 7.6-12.5 μm)



Representative Trace Gas Detection Limits

Species	cm^{-1}	Precision 1 s RMS (ppt)	LOD 100 s (ppt)
NH ₃	967	50	20
NO ₂	1600	80	40
HONO	1700	200	80
CO	2190	120	50
N ₂ O	2240	100	50
HNO ₃	1720	200	80
O ₃	1050	500	200
NO	1905	200	100
CH ₄	1270	400	200
SO ₂	1370	310	120
C ₂ H ₄	960	360	140
HCHO	1765	350	100
H ₂ O ₂	1267	1000	400

Limit of Detection
(LOD) for S/N = 2

Pathlength: 210 m

Typical data acquisition
time: 1-100 s

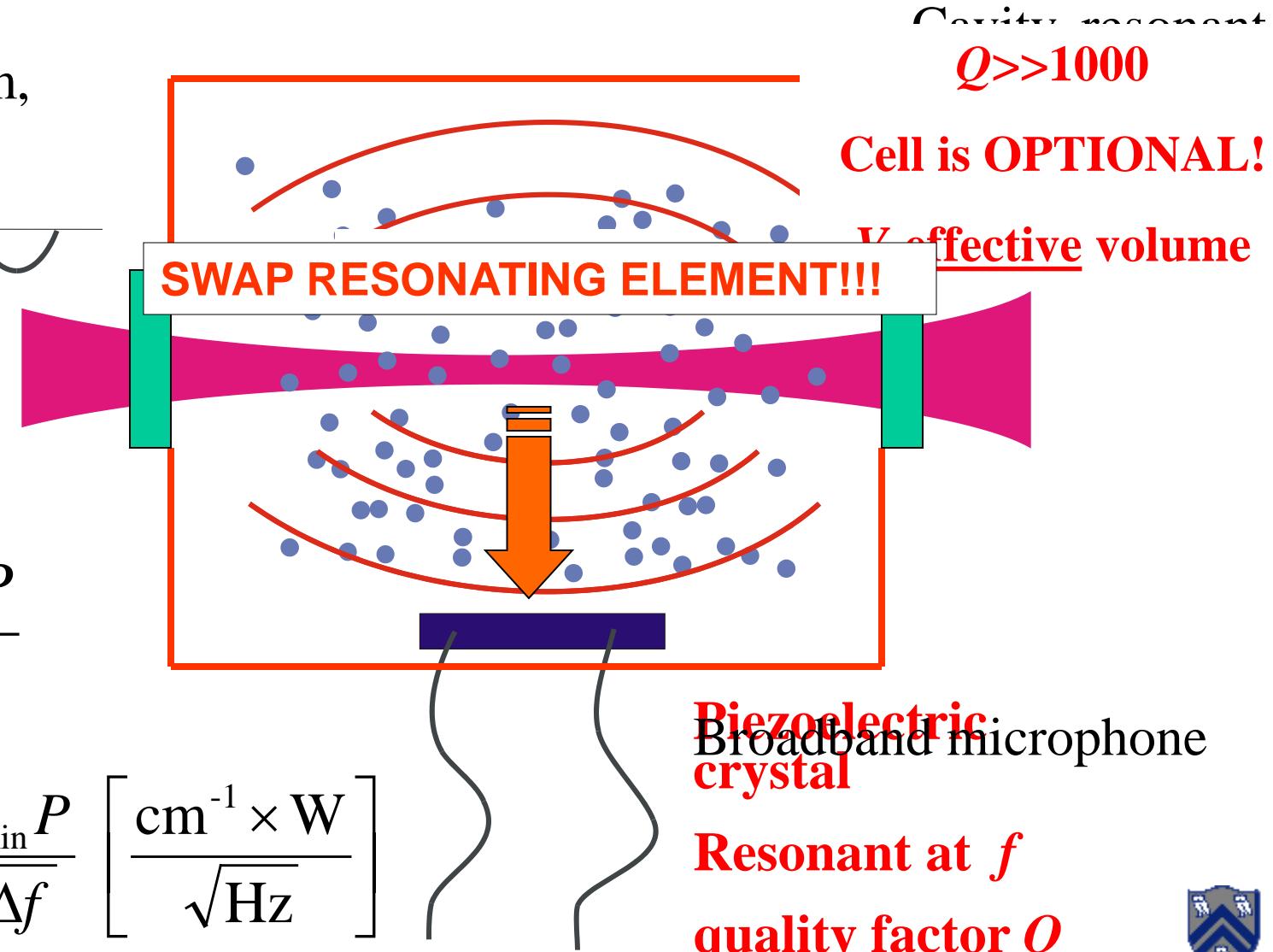
From conventional PAS to QEPAS

Laser beam,
power P

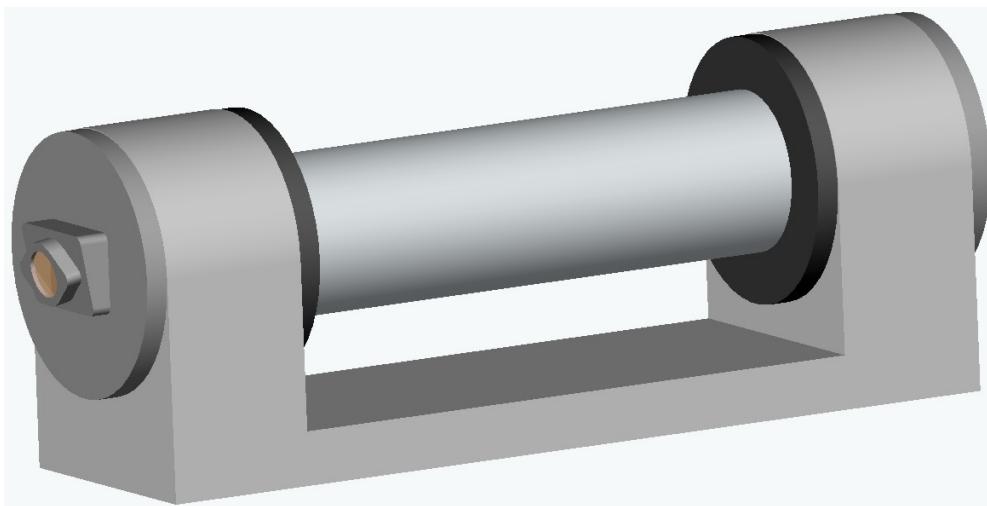
Modulated
(P or λ) at f
or $f/2$

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

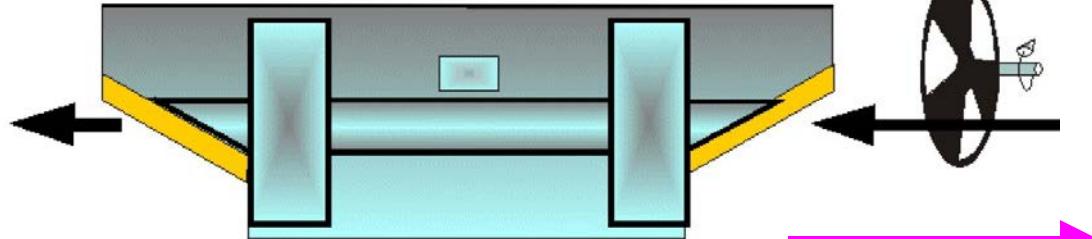
$$NNEA = \frac{\alpha_{\min} P}{\sqrt{\Delta f}} \left[\frac{\text{cm}^{-1} \times \text{W}}{\sqrt{\text{Hz}}} \right]$$



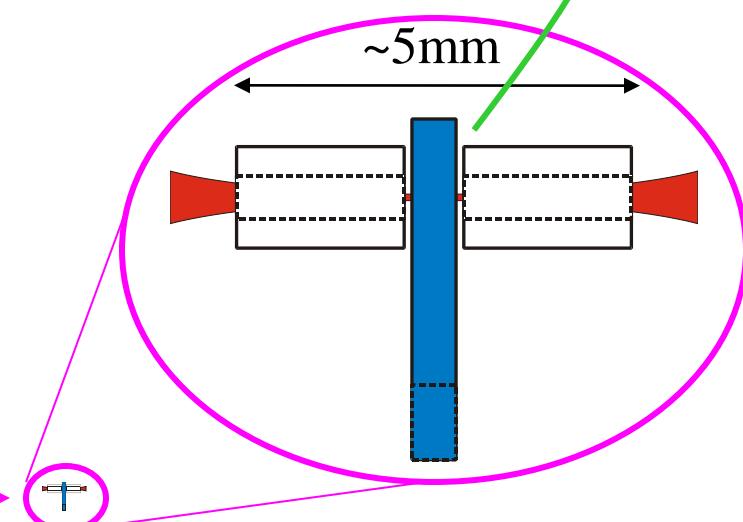
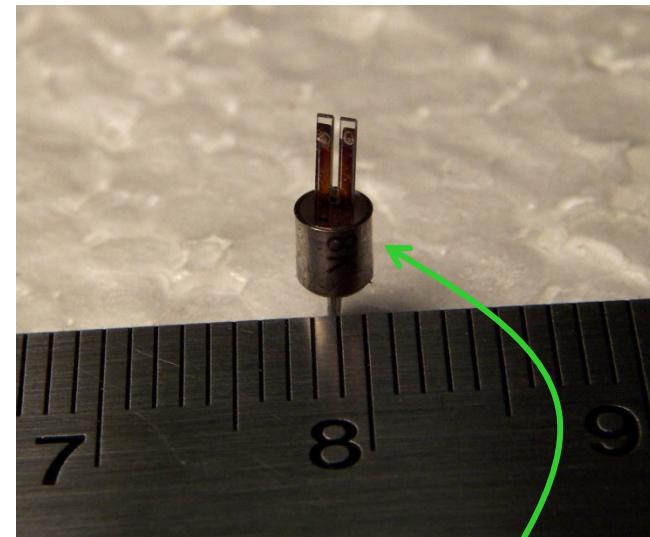
Comparative Size of Absorbance Detection Modules (ADM)



Optical multipass cell (100 m):
 $l \sim 70 \text{ cm}$, $V \sim 3000 \text{ cm}^3$

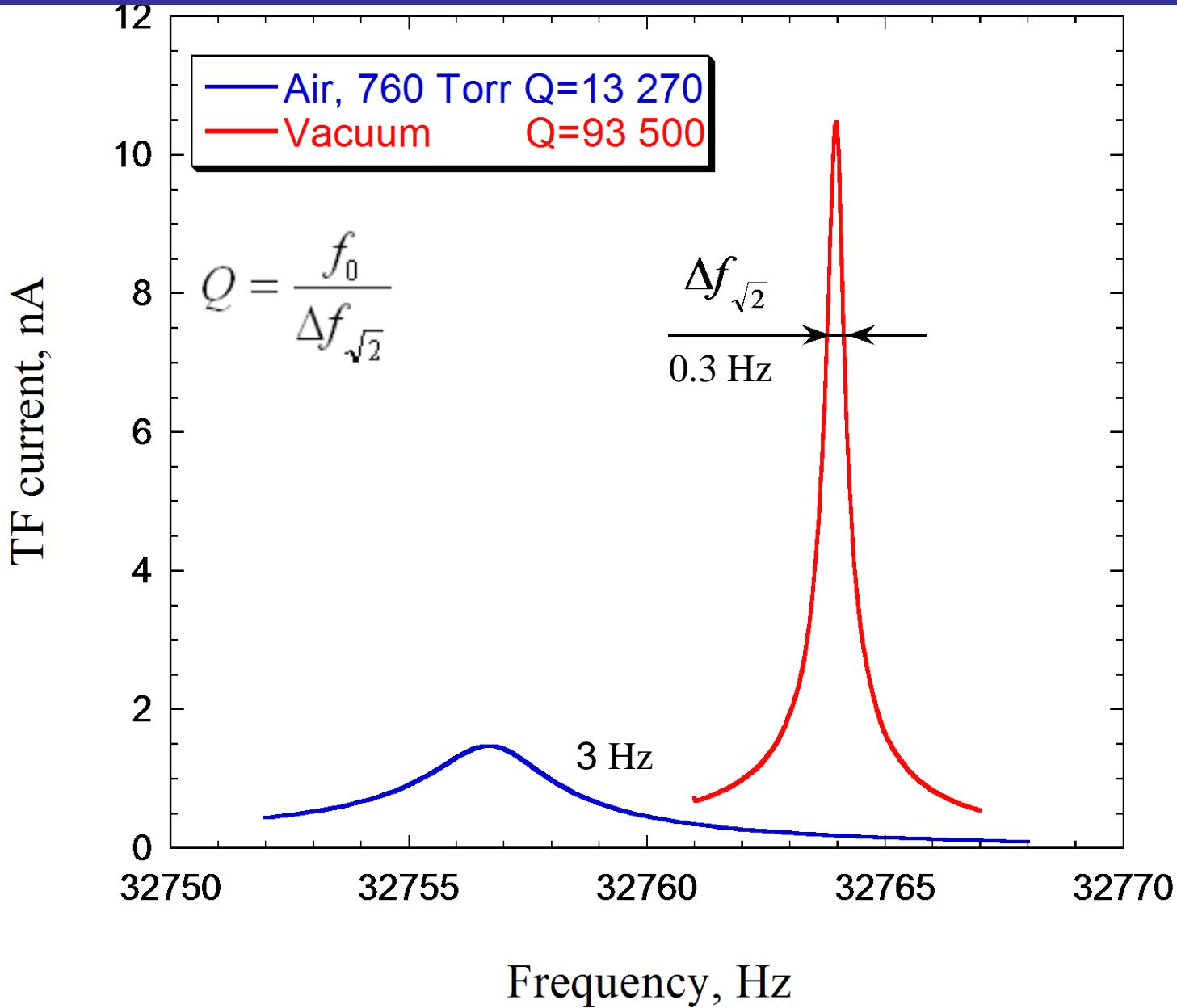


Resonant photoacoustic cell (1000 Hz):
 $l \sim 60 \text{ cm}$, $V \sim 50 \text{ cm}^3$

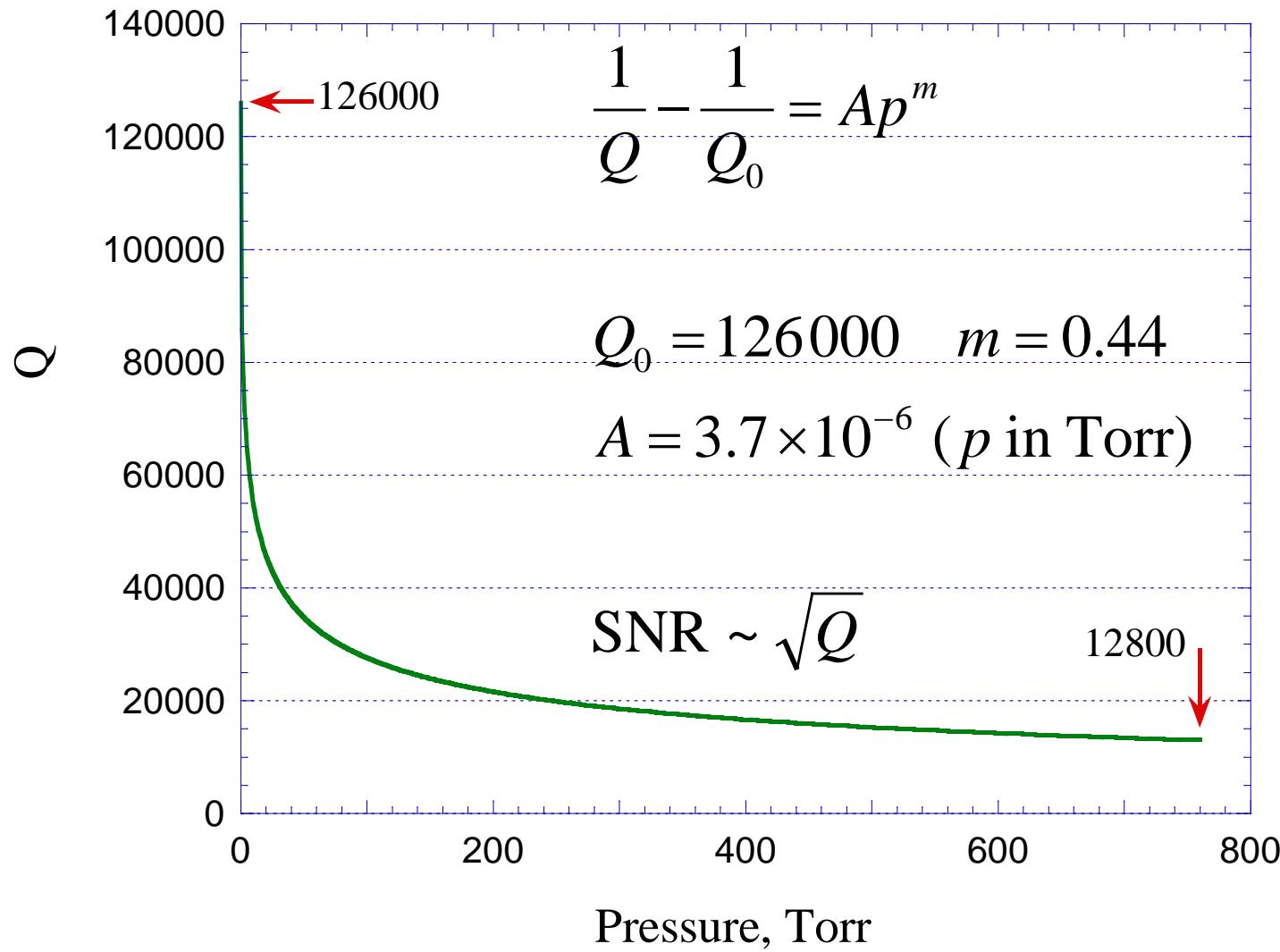


QEPAS ADM:
 $l \sim 0.5 \text{ cm}$, $V \sim 0.05 \text{ cm}^3$

Typical QTF Resonance Curves



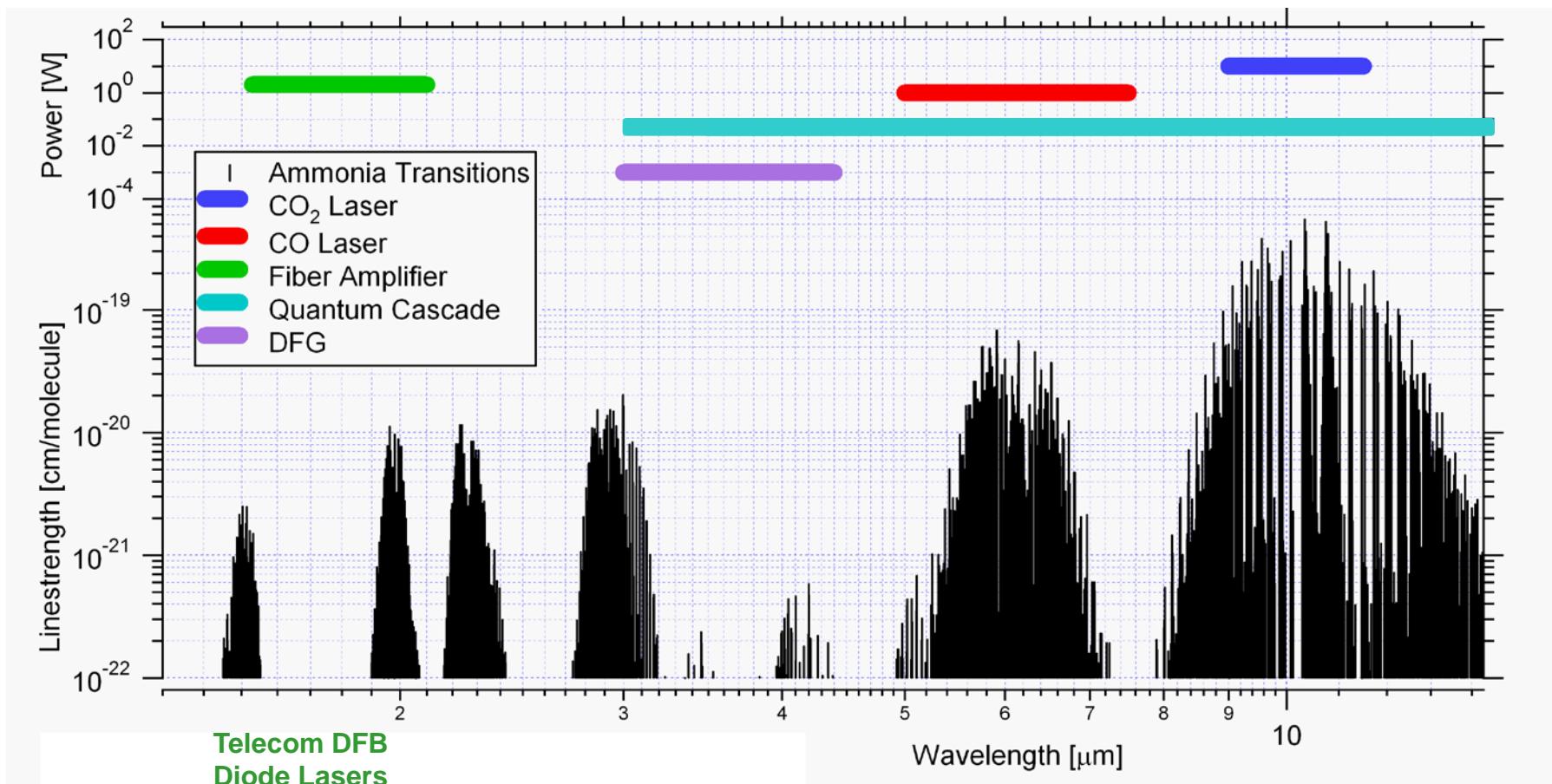
Air Pressure Dependence of Q Factor of a Typical TF



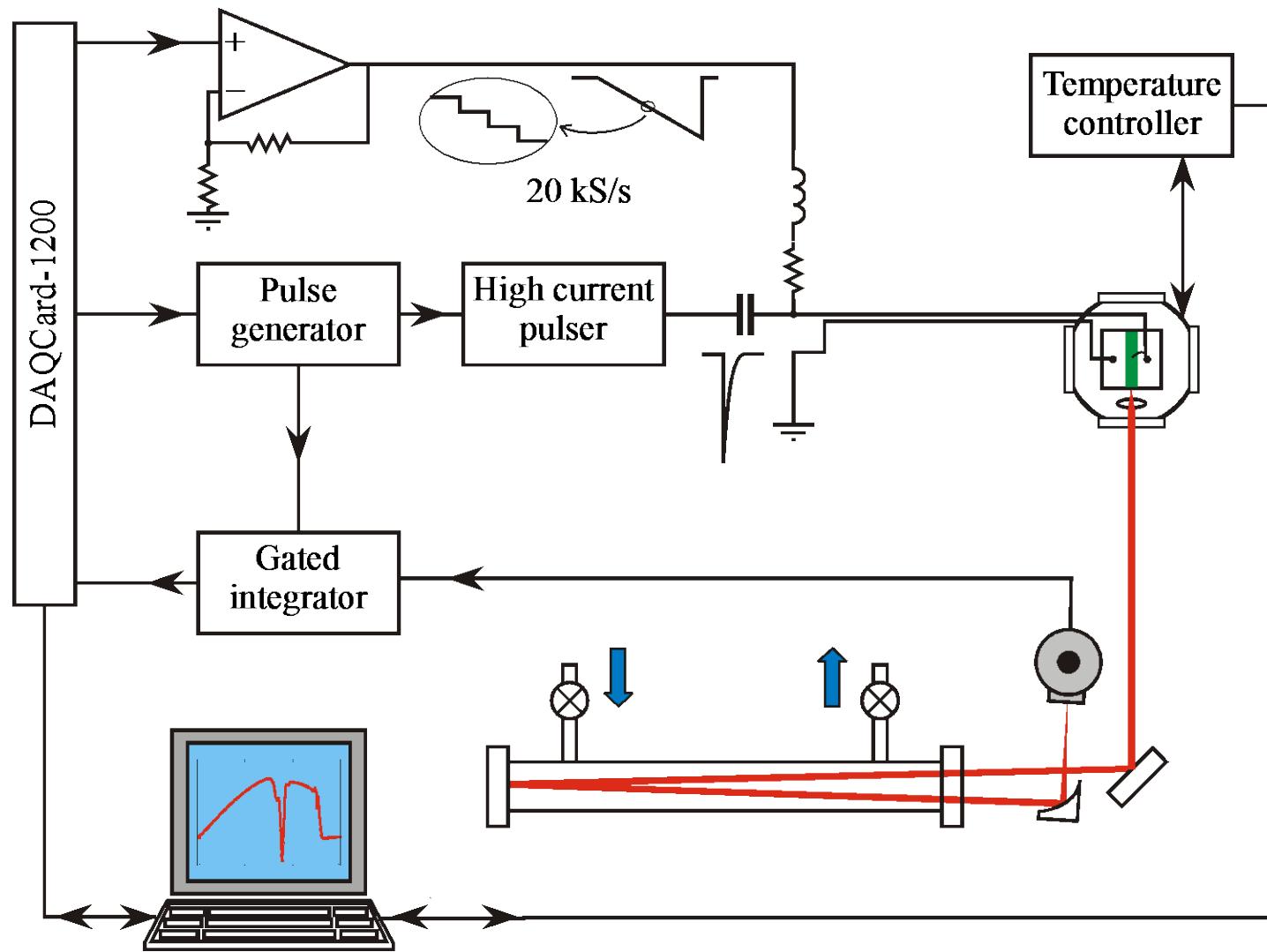
Motivation for NH₃ Detection

- Monitoring NH₃ 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
- Spacecraft related gas monitoring
- Pollutant gas monitoring
- Atmospheric chemistry
- Medical diagnostics (kidney & liver dysfunctions)

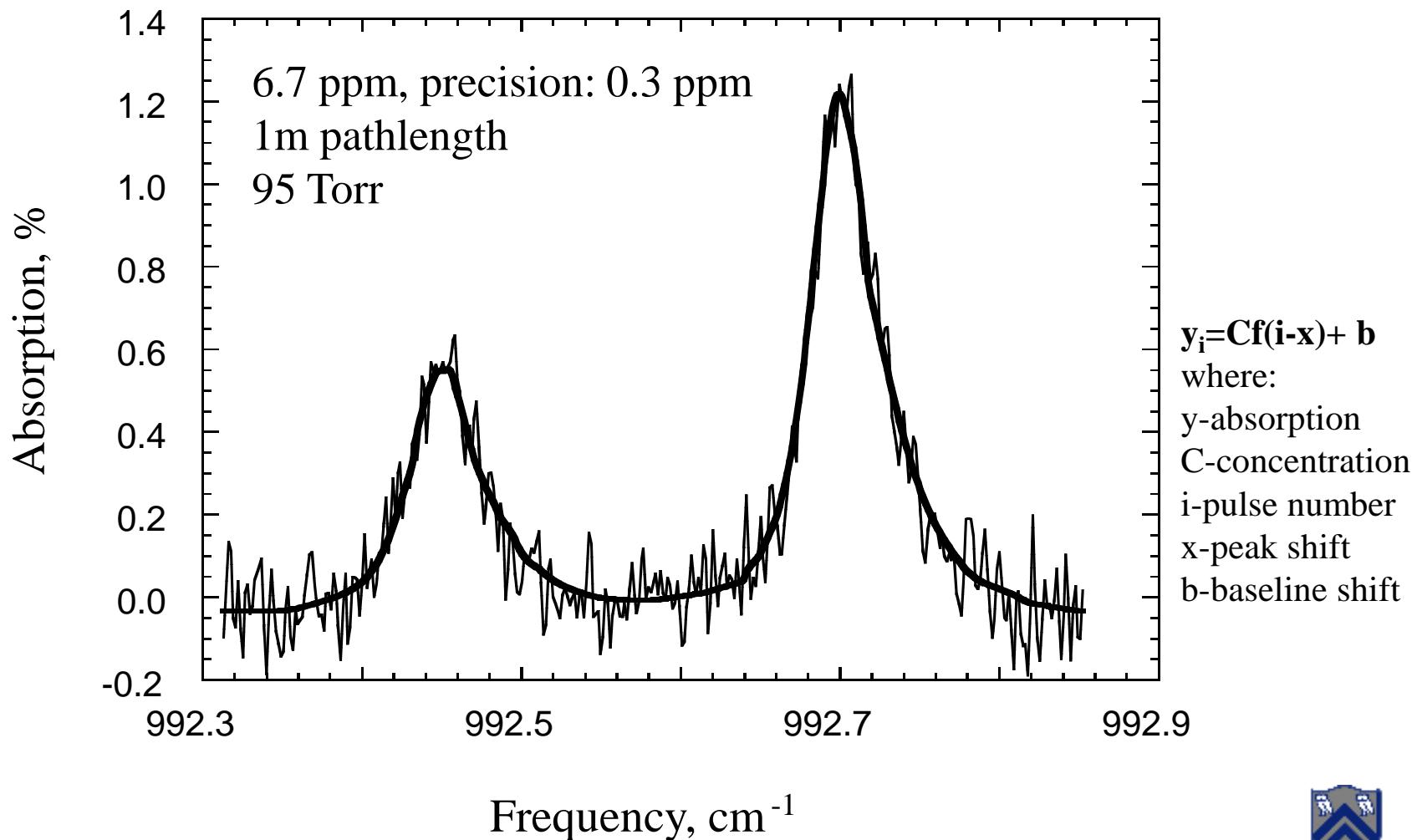
Infrared NH₃ Absorption Spectra



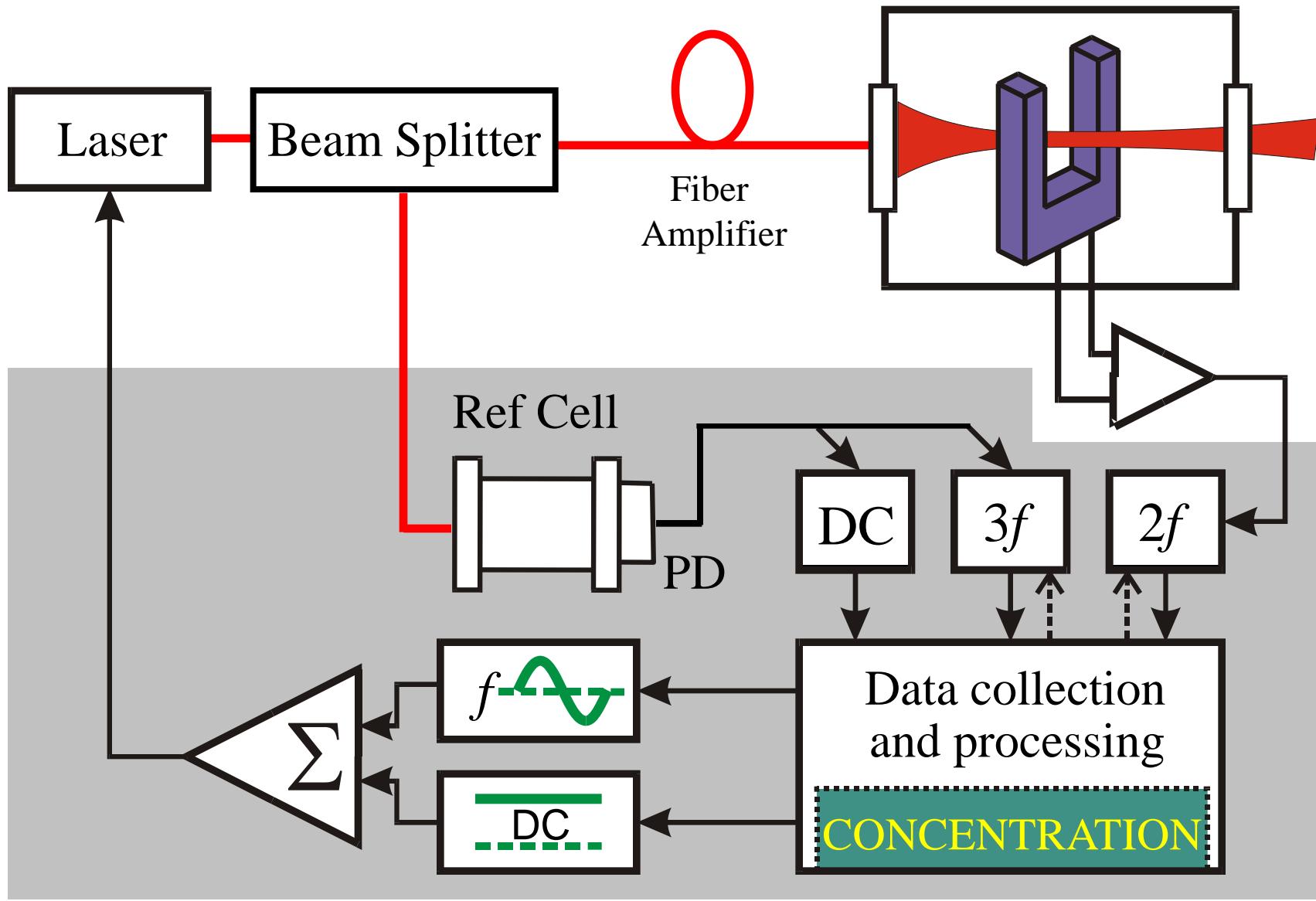
Pulsed QC Laser Based Gas Sensor



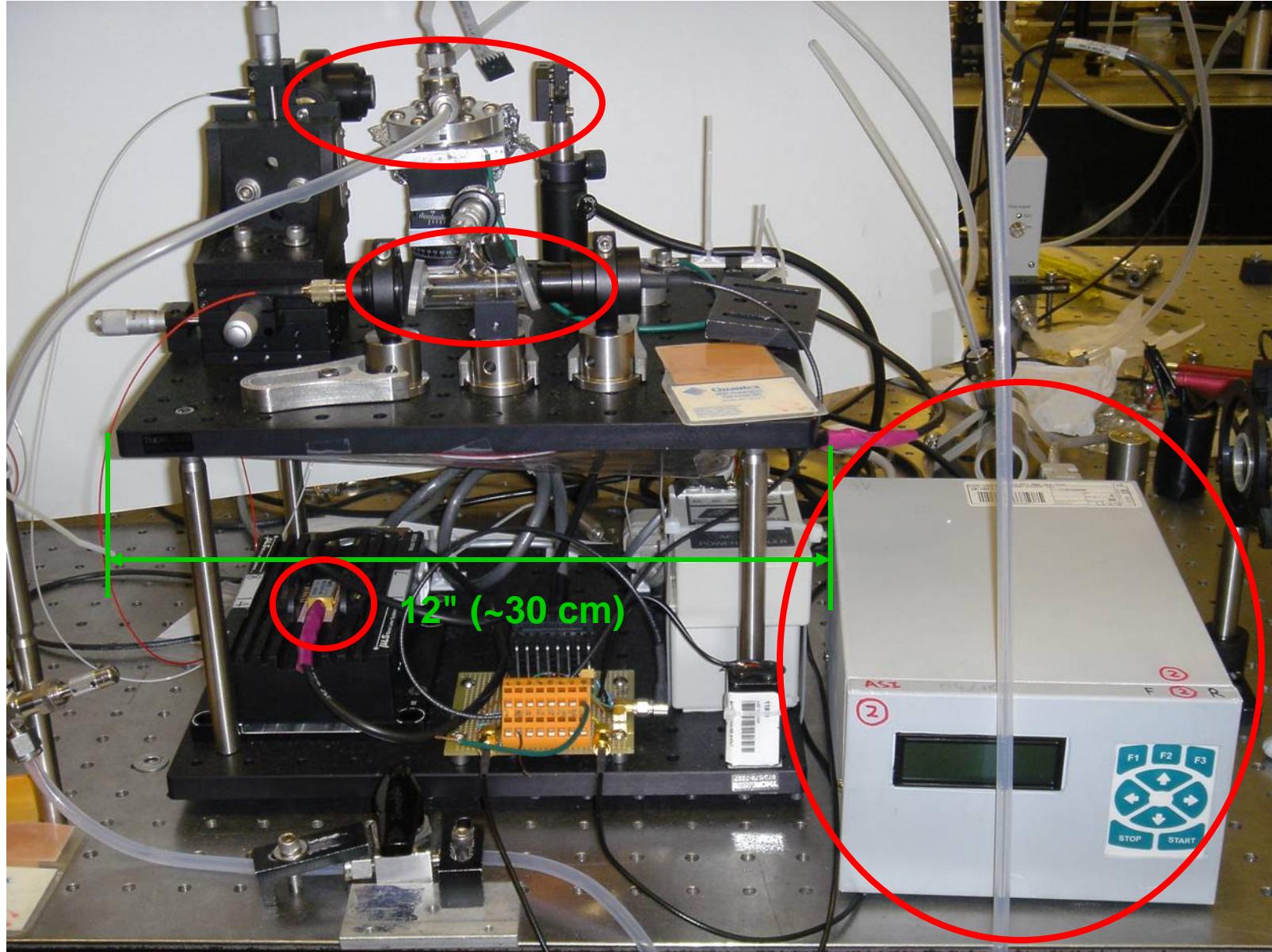
Ammonia Absorption Spectrum @ 993 cm⁻¹



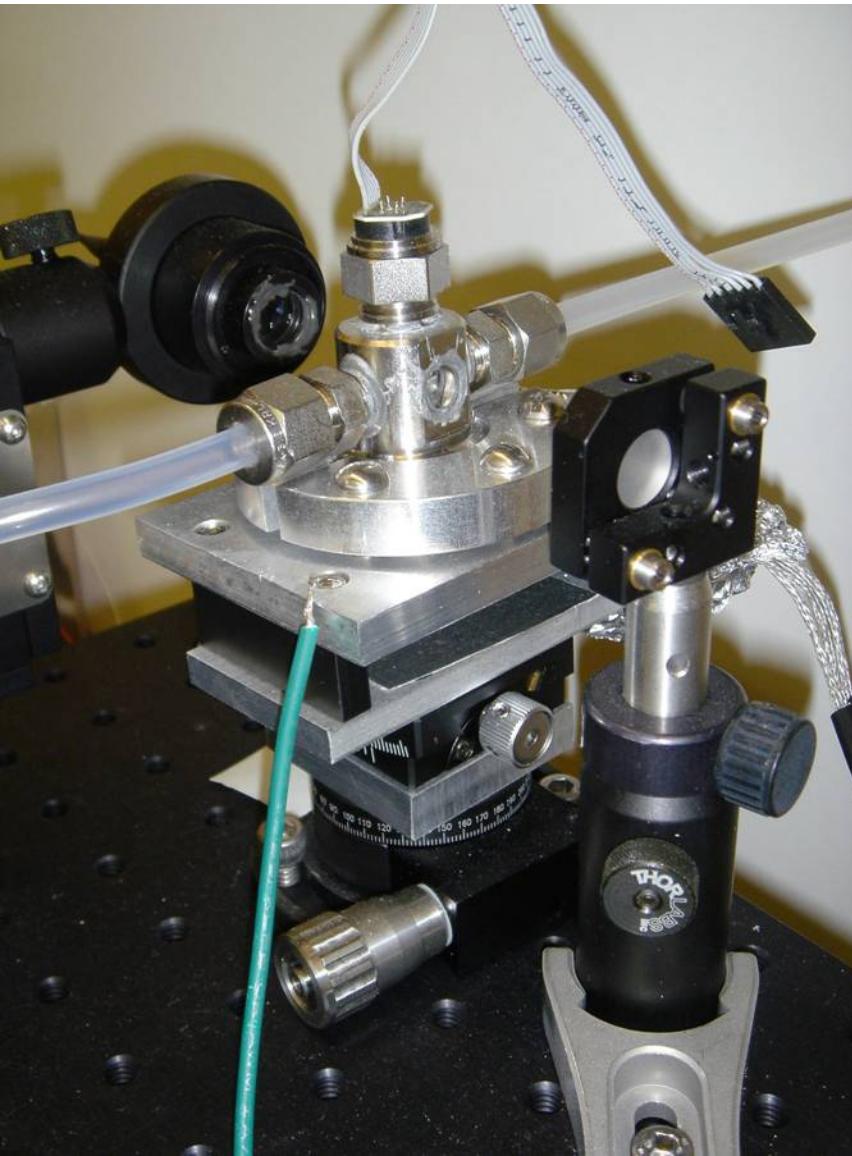
QEPAS Fiber based Gas Sensor Architecture



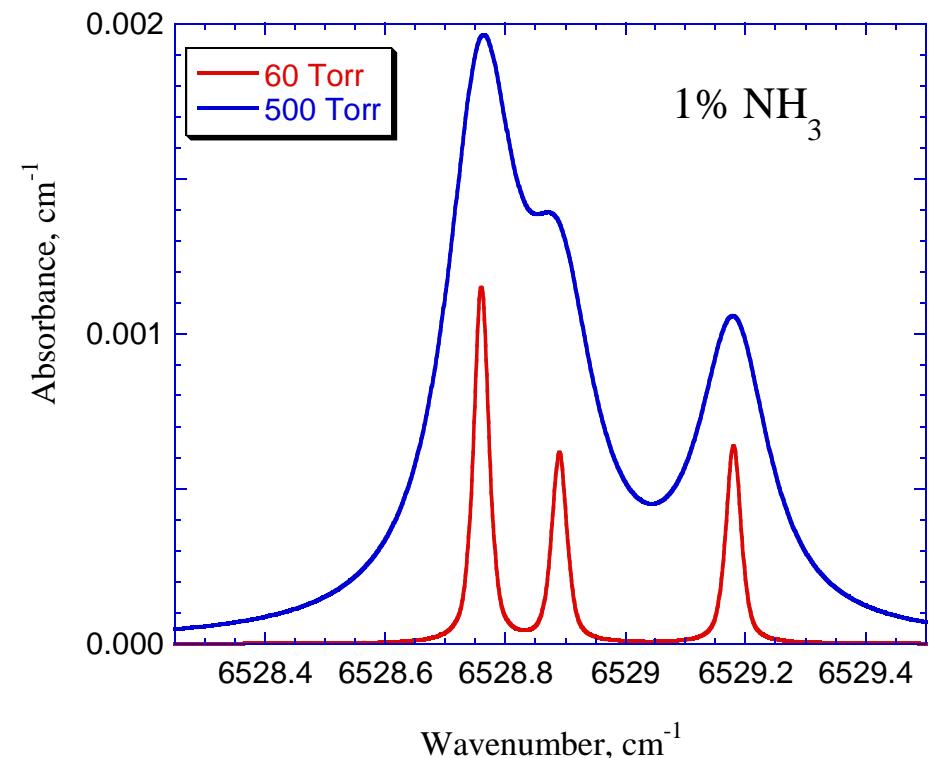
Implementation:a Prototype QEPAS Sensor



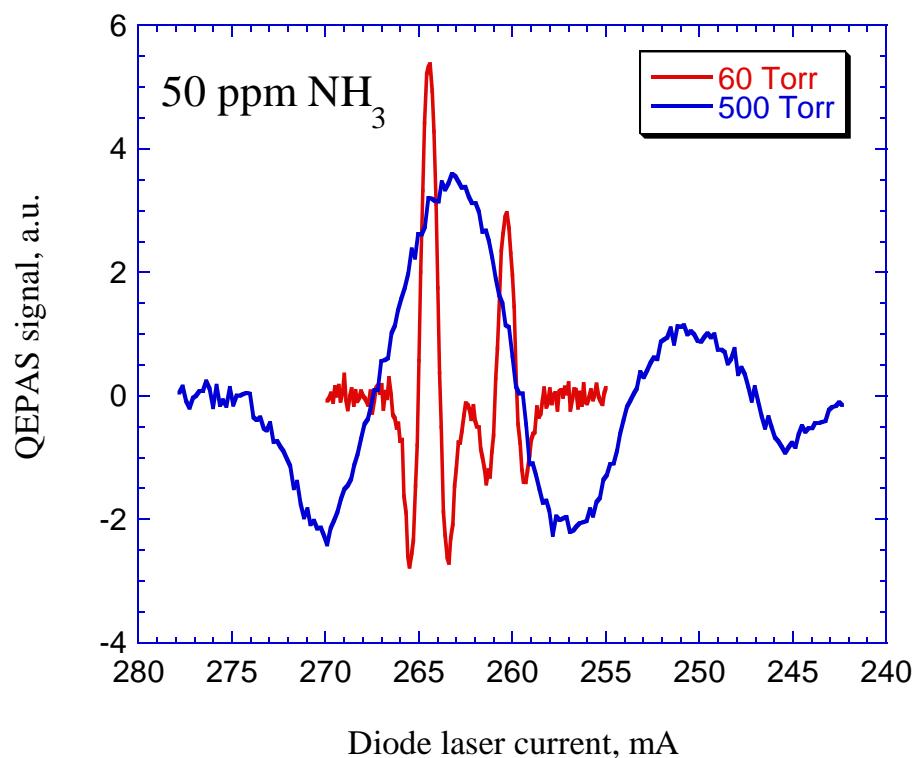
QEPAS Trace Gas Sensing Module



Ammonia Detection using a 1.53 μm Telecom Diode Laser

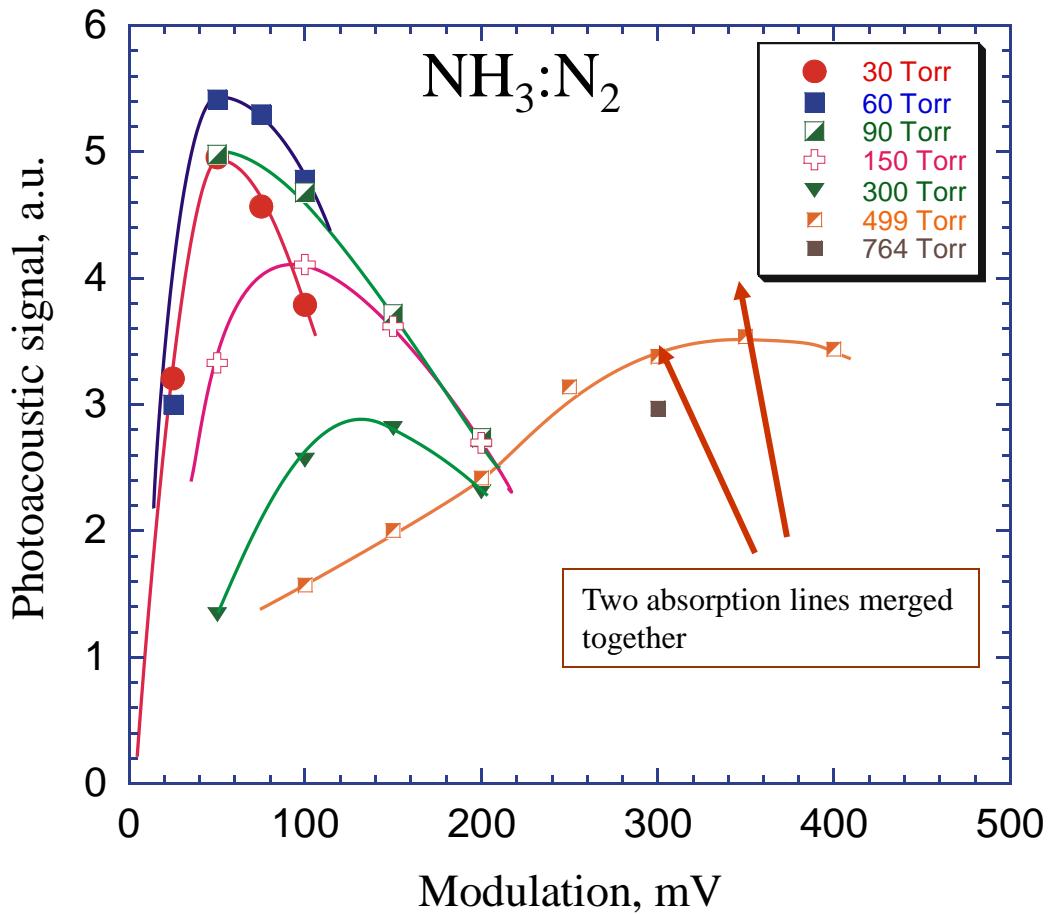


Spectral simulations based on data from
Webber et al., APPLIED OPTICS 40,
2031-2042 (2001)]



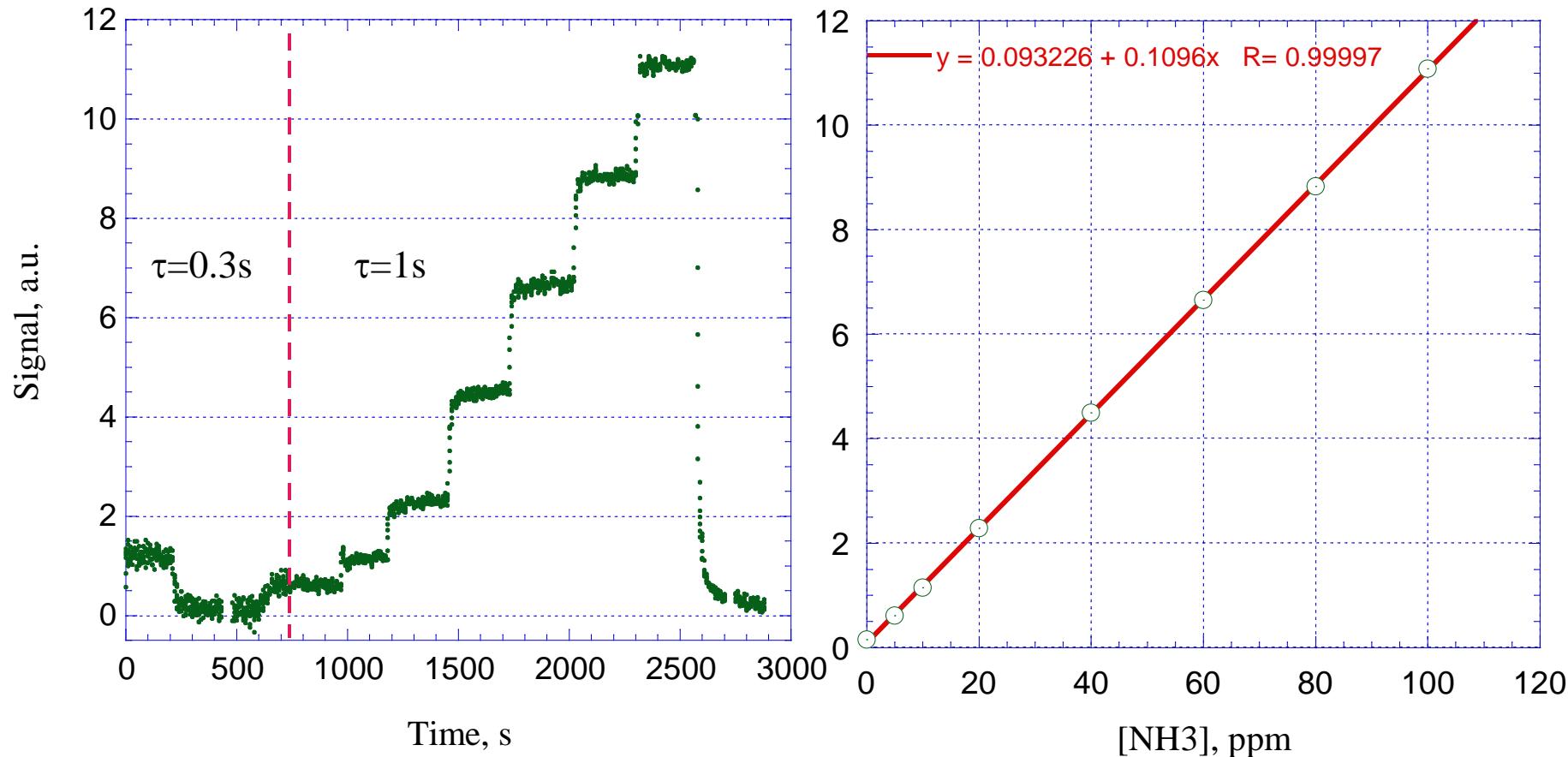
QEPAS spectra at different pressures of
 $\text{NH}_3:\text{N}_2$ gas mixture; $t=0.3\text{s}$, 38 mW diode
laser excitation power at 6529 cm^{-1}

Pressure Dependence of QEPAS Sensitivity



- Peak optical absorption varies with pressure
- Q-factor decreases at higher pressure
- V-T relaxion is faster at higher pressure
- Acoustic resonator enhancement factor changes with pressure

Calibration and Linearity of QEPAS based NH₃ Sensor



Noise –equivalent (1s) concentration (NEC). for $\tau=1\text{s}$ time constant is 0.65 ppmv for 38 mW excitation power

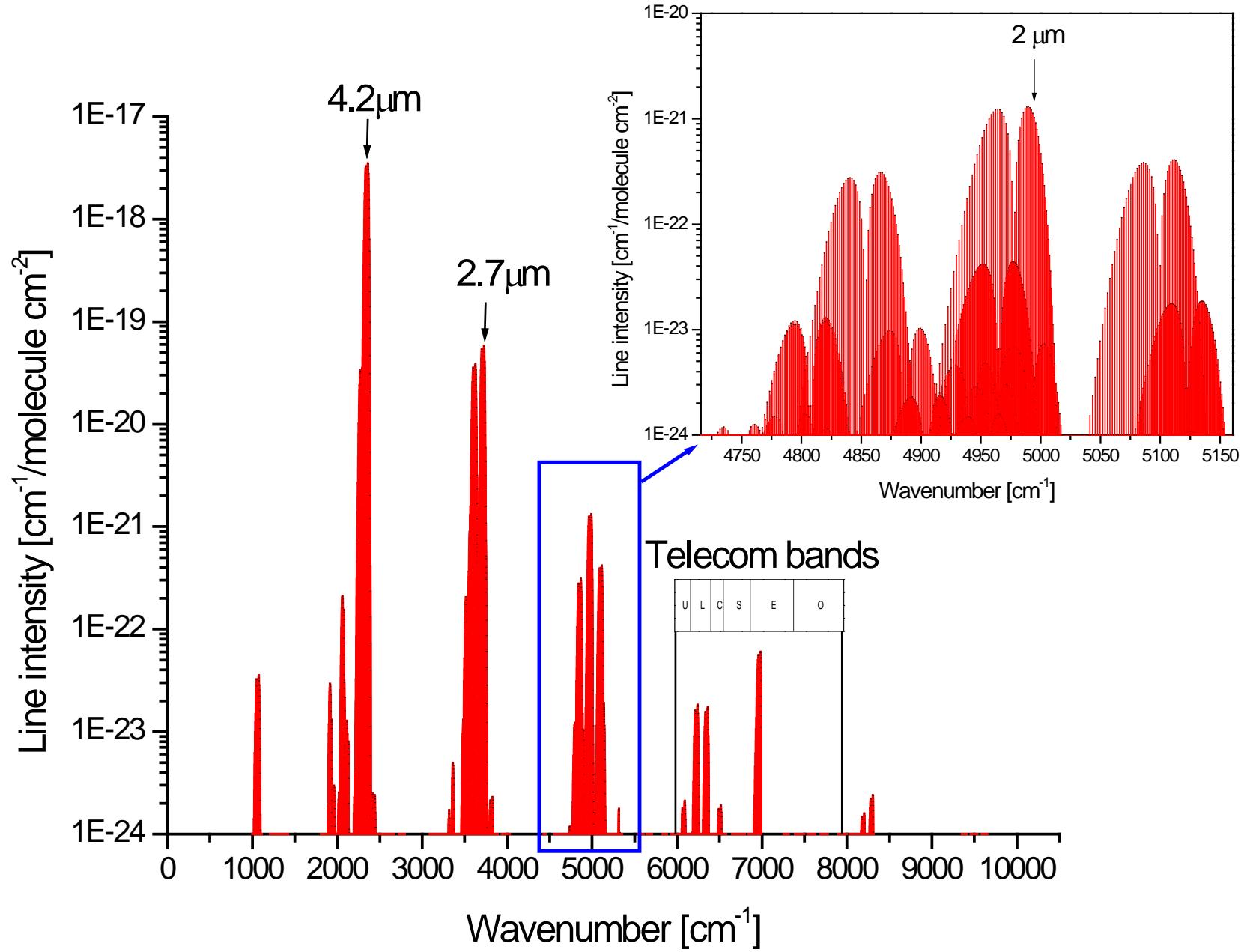
Noise-equivalent absorption (NEA) coefficient $k=7.2\times 10^{-9} \text{ cm}^{-1}\text{W}/\text{Hz}^{1/2}$

90 last points of each step averaged

(Traditional PAS* – $1.5\times 10^{-9} \text{ cm}^{-1}\text{W}/\sqrt{\text{Hz}}$)

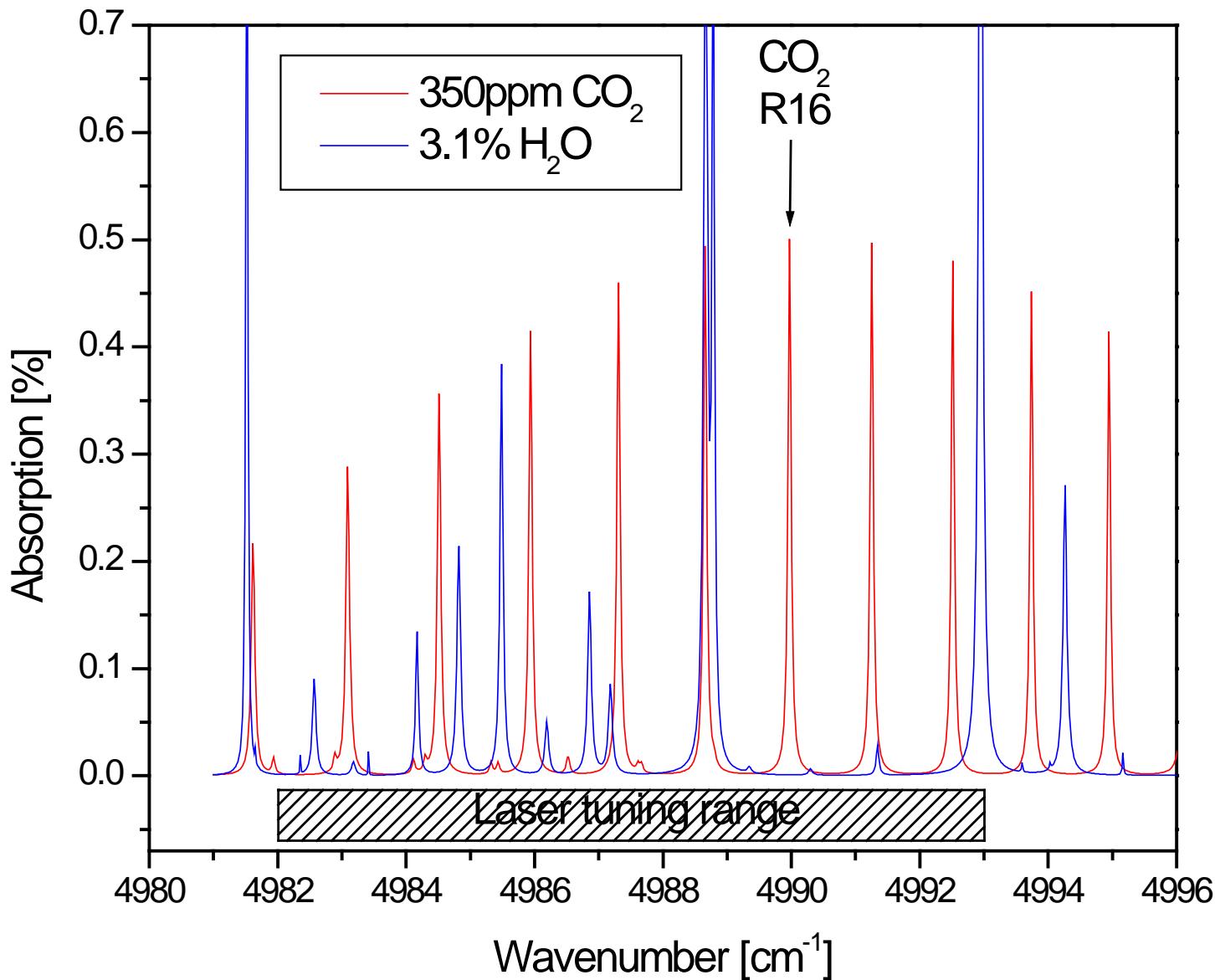
*Webber et al., APPLIED OPTICS April 2003 Vol. 42, No. 12, p.2119

Detection of CO₂ at 2 μm



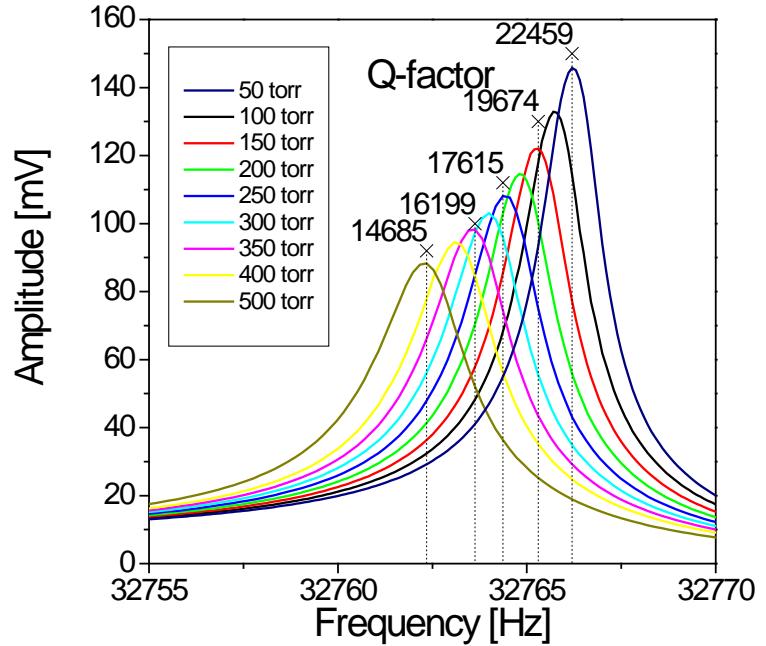
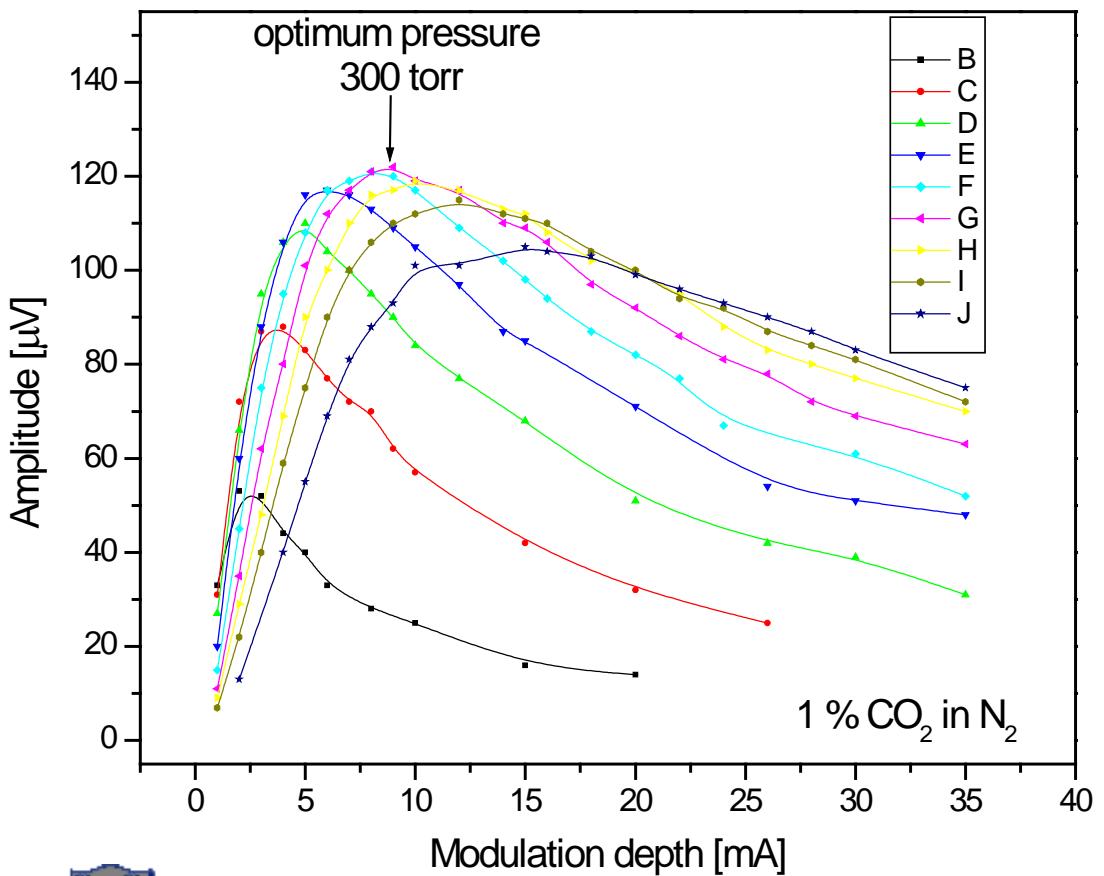
RICE

Detection of CO₂ at 2 μm



RICE

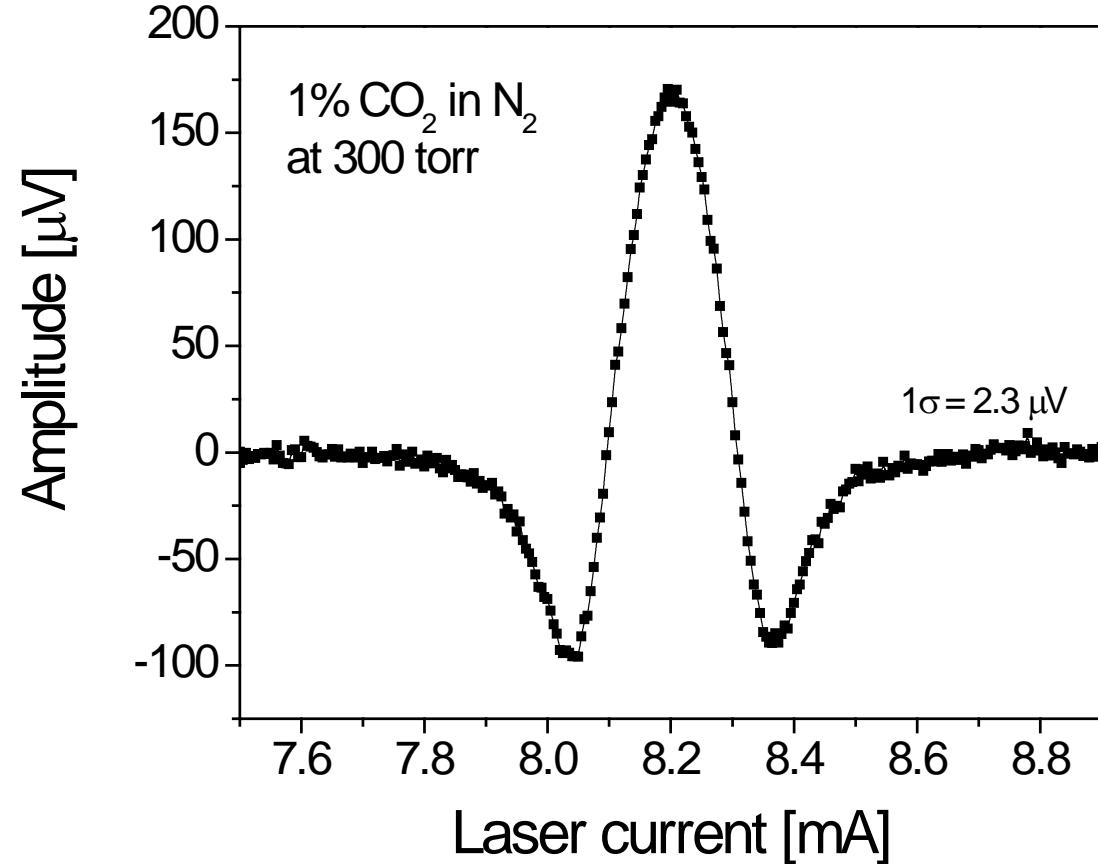
Optimization of QEPAS CO₂ detection



Parameters, which influence
QEPAS sensitivity:

- Q-factor (decreases with pressure)
- V-T relaxation (faster at higher pressure)
- Modulation depth (optimum when equal to $\sim 2 \times$ FWHM of the absorption line at given pressure)

CO₂ detection limit



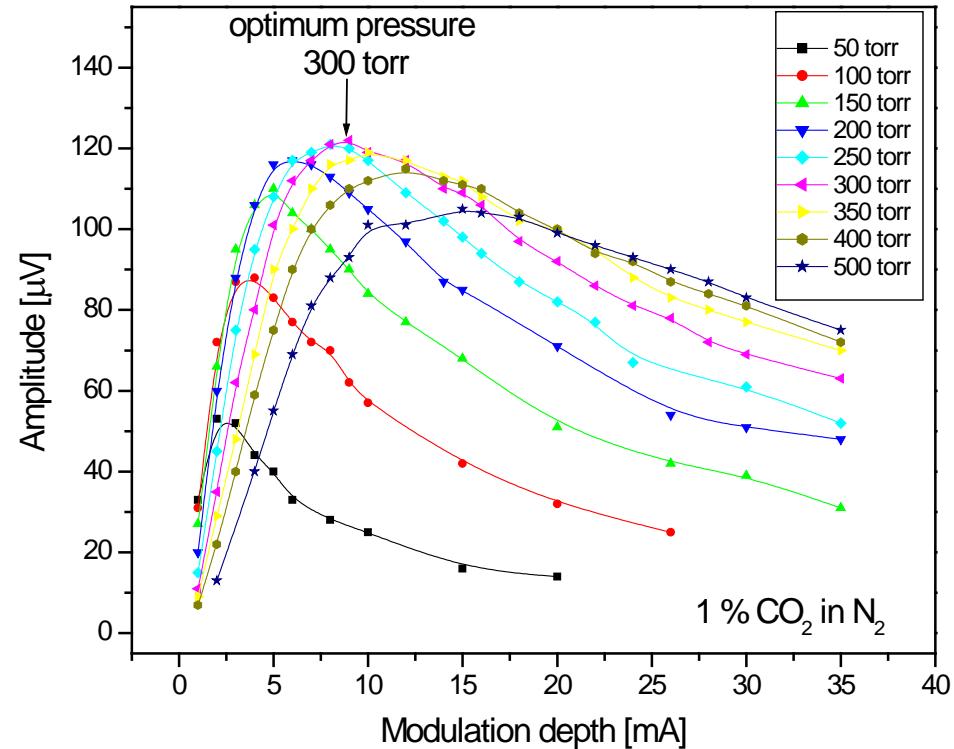
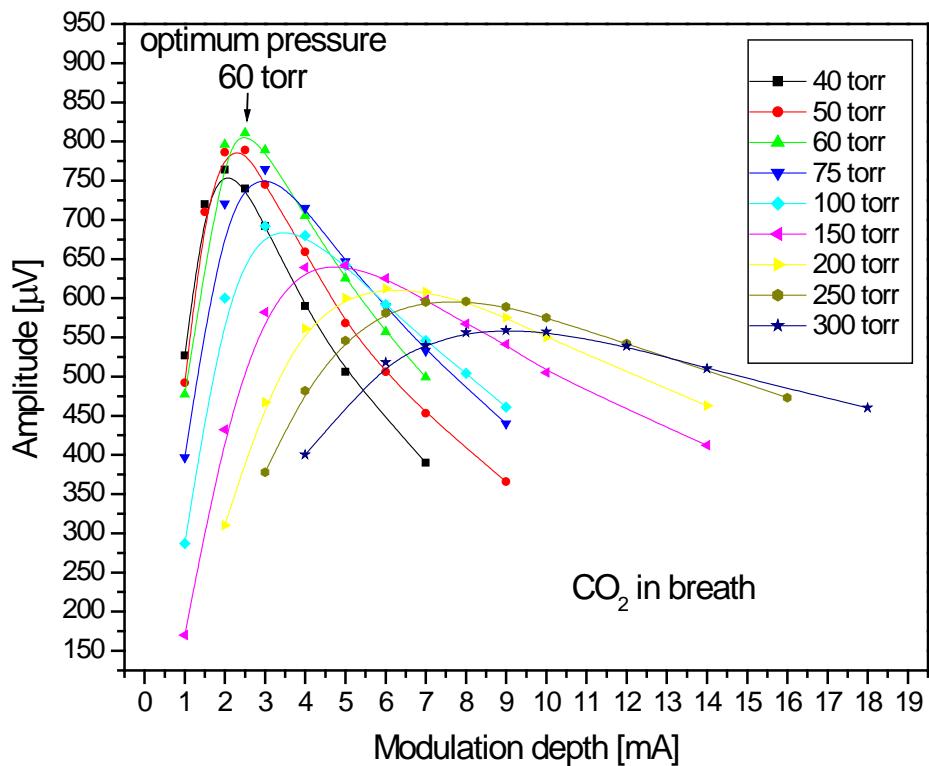
- SNR : ~74 (~ 135 ppm of CO₂)
 - Laser power: ~ 4.6 mW
 - Lock-in time constant: 1 s
 - Peak absorbance: $\sim 1.4 \times 10^{-3} \text{ cm}^{-1}$
-
- Normalized noise equivalent sensitivity for CO₂ in N₂:

$$\text{NES} = 1.54 \times 10^{-7} \text{ cm}^{-1} \text{ W} / \sqrt{\text{Hz}}$$



RICE

Effect of H₂O on V-T relaxation



High concentration of H₂O in breath causes instantaneous thermalization of the absorbed laser power. In such a situation the optimal conditions depend mainly on the Q factor and absorption within the gas sample

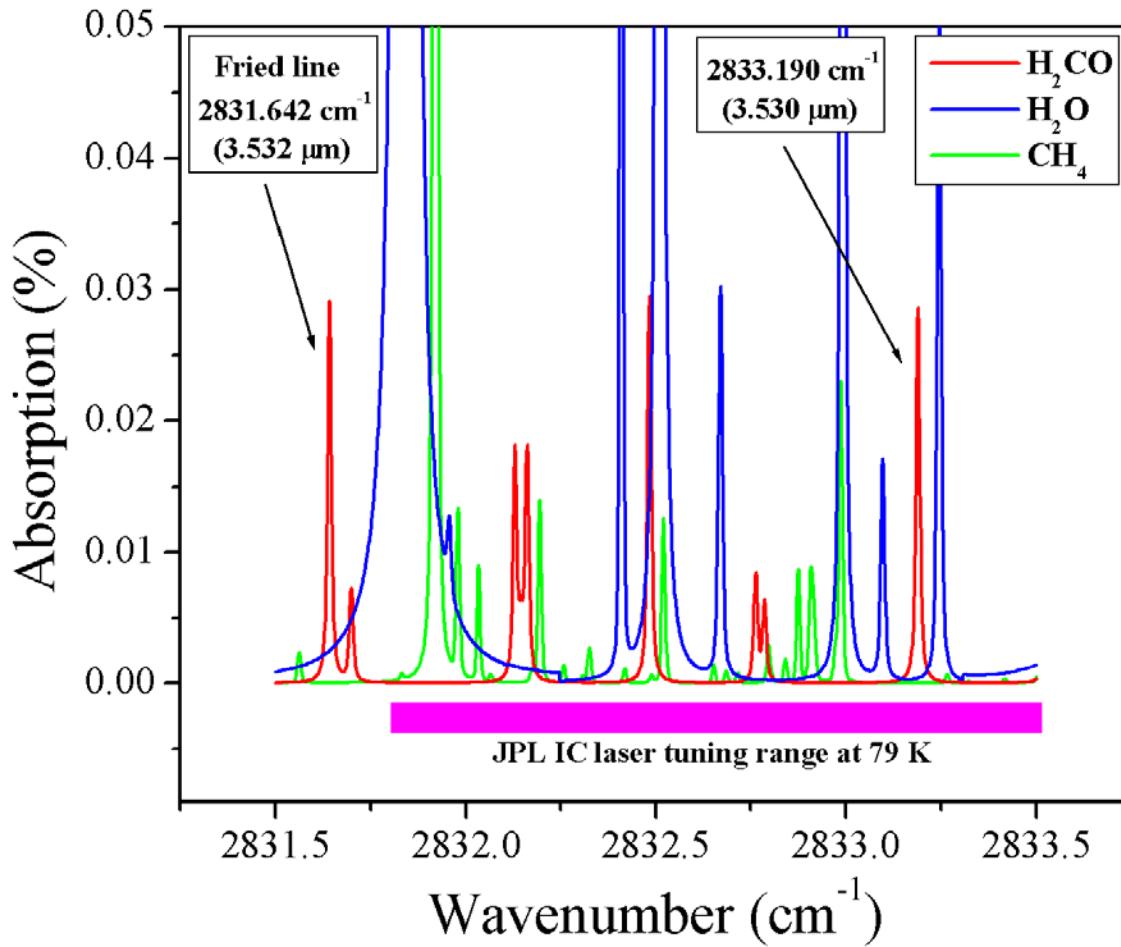


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Motivation for Precision Monitoring of H₂CO

- Pollutant due to incomplete fuel combustion processes
- Potential trace contaminant in industrial manufactured products
- Precursor to atmospheric O₃ production
- Medically important gas

HITRAN Based Simulation of a H₂CO-H₂O-CH₄ Spectrum in Tuning Range of a 3.53μm IC Laser



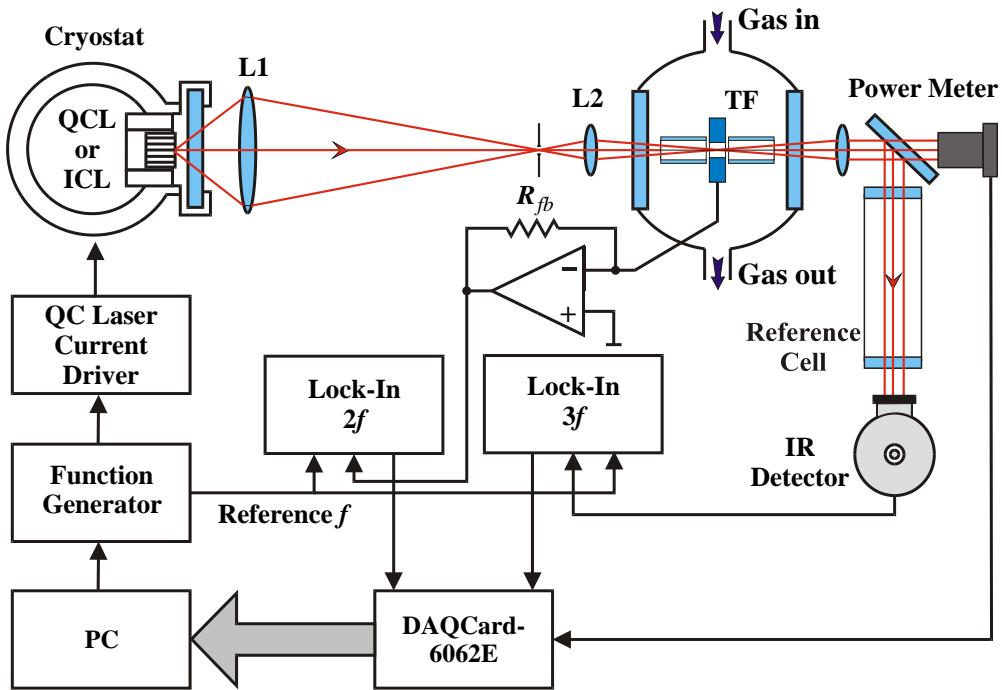
- H₂CO : 10 ppb
- H₂O : 3%
- CH₄ : 2 ppm
- Optical path: 100 m
- Total pressure: 30 Torr



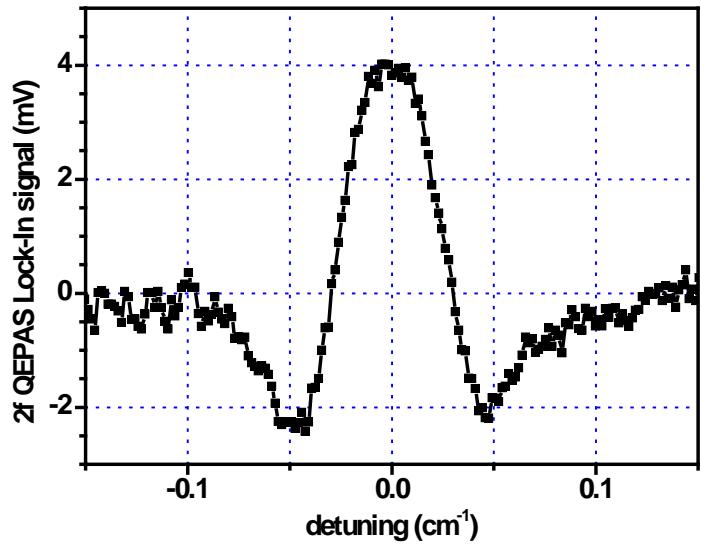
JPL



QCL based Quartz-Enhanced Photoacoustic Sensor



2f-QEPAS based H₂CO signal at 3.53 μm (2832.48 cm⁻¹)



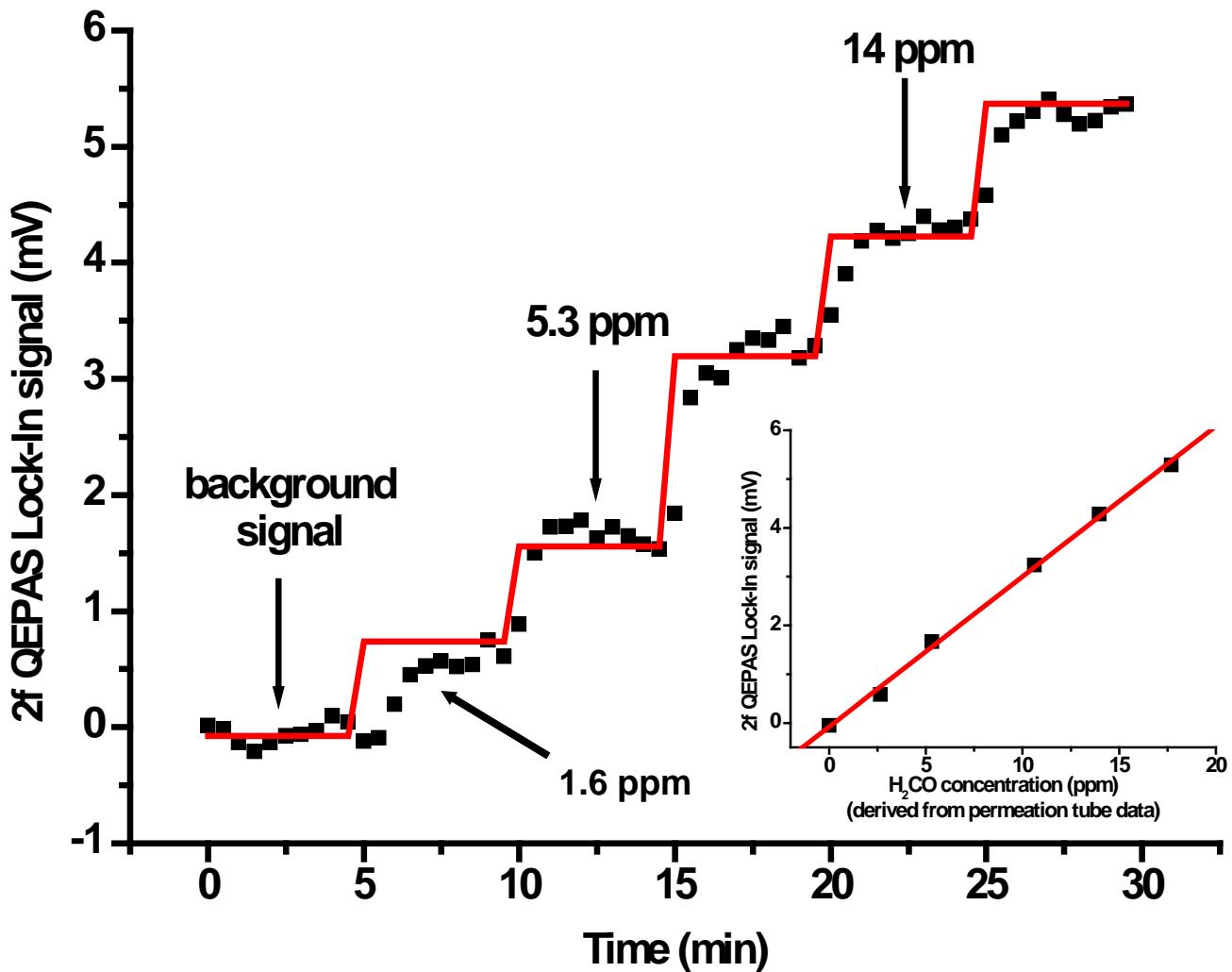
- [H₂CO]: 13.27 ppm
- **QEPAS NNEA Sensitivity:**
 $2.2 \times 10^{-8} \text{ cm}^{-1} \text{ W}/\sqrt{\text{Hz}}$;
- **$4.6 \times 10^{-9} \text{ cm}^{-1} \text{ W}/\sqrt{\text{Hz}}$** as of June 10, 2005
For comparison:
QEPAS Sensitivity for NH₃ :
 $5.4 \times 10^{-9} \text{ cm}^{-1} \text{ W}/\sqrt{\text{Hz}}$



JPL



IC Laser based Formaldehyde Calibration Measurements with a Gas Standard Generator



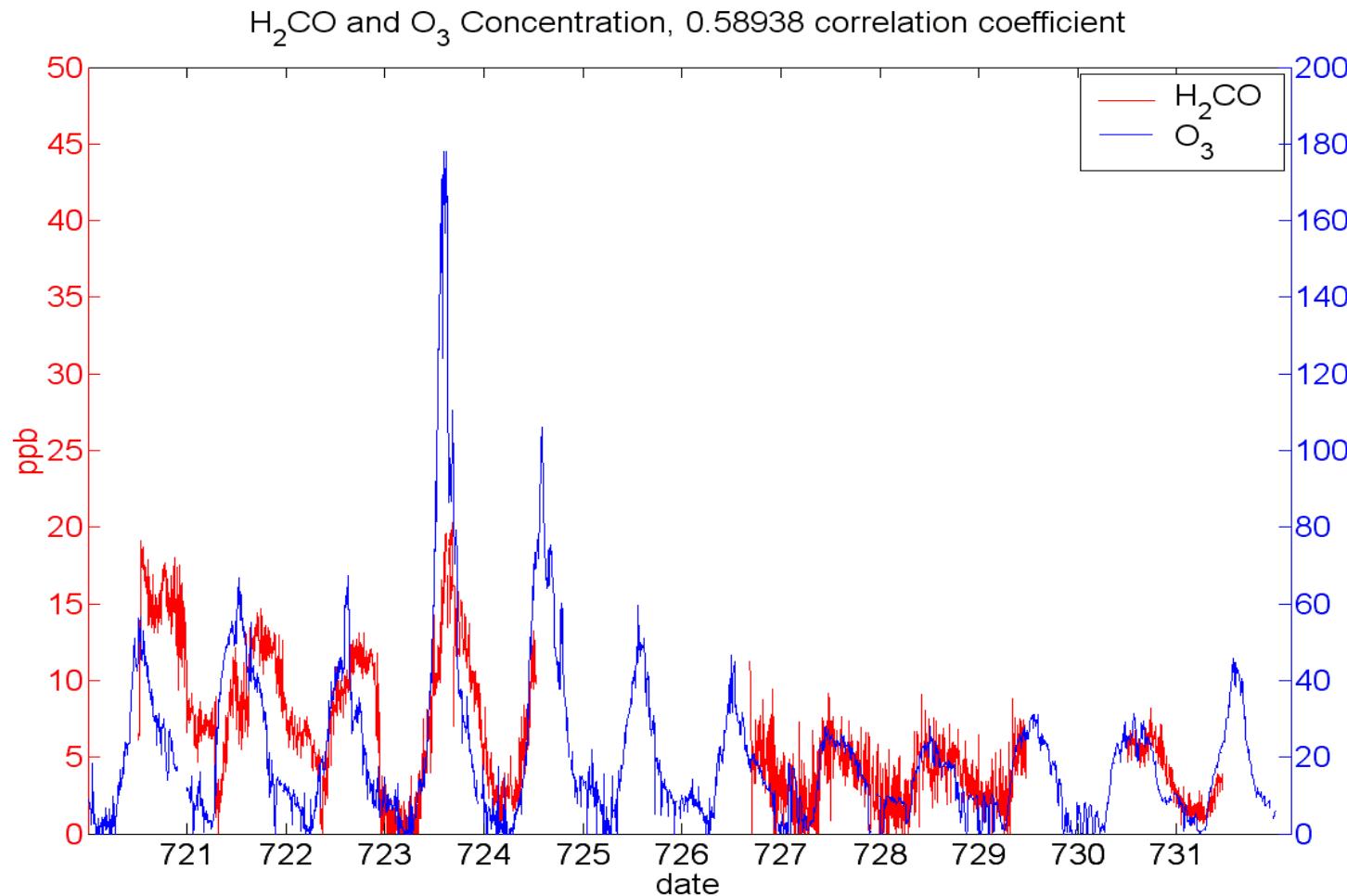
- H_2CO absorption frequency: 2832.5 cm^{-1}
- Lock-In time constant: 10 s
- QEPAS parameters
 - Resonance frequency: 32.760 KHz
 - Q-factor: 17336
 - Pressure: 200 Torr
 - Gas Flow: 75 sccm
 - IC laser power: 6 mW
- Sensitivity: $2.2 \times 10^{-8} \text{ cm}^{-1} \text{ W}/\sqrt{\text{Hz}}$
For comparison:
Sensitivity for NH_3 :
 $5.4 \times 10^{-9} \text{ cm}^{-1} \text{ W}/\sqrt{\text{Hz}}$
- MDC is 550 ppbv



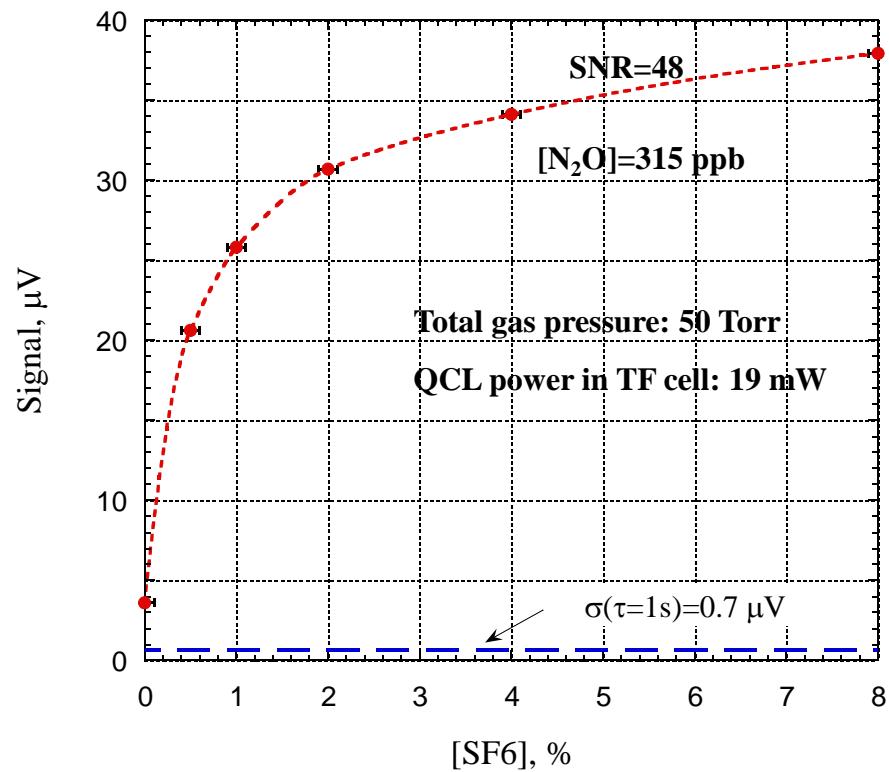
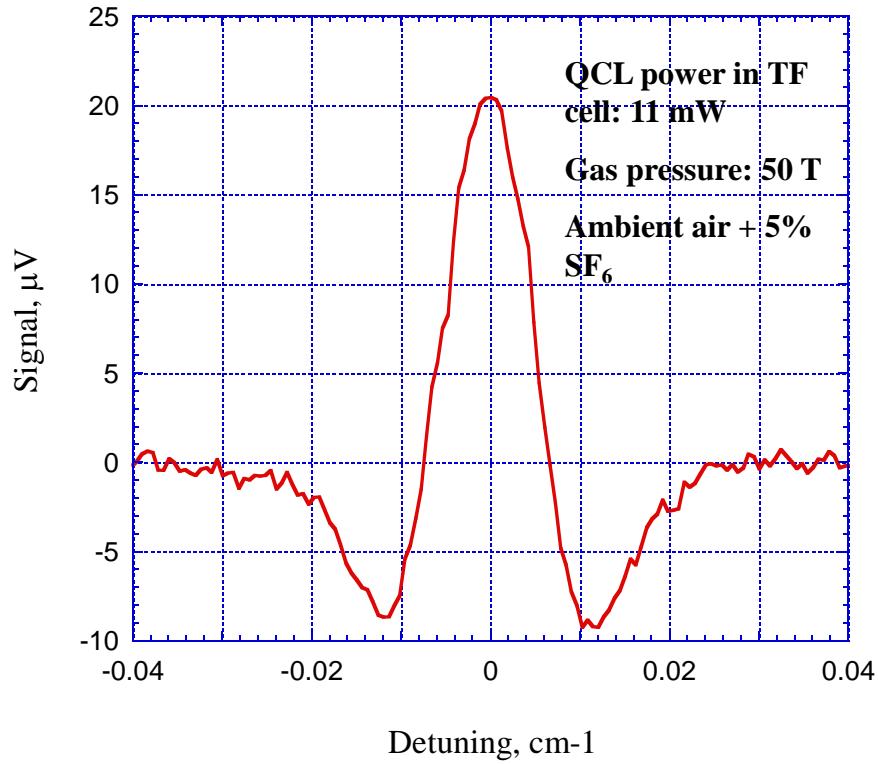
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H_2CO and O_3 Concentrations at Deer Park, TX for July 20-31, 2003



N_2O Detection in Ambient Air at $4.55 \mu\text{m}$ (2195.6 cm^{-1})



Noise-equivalent absorption coefficient is $1.5 \times 10^{-8} \text{ cm}^{-1}\text{W}/\text{Hz}^{1/2}$ for 5% SF_6 with a noise equivalent concentration of 4 ppbv for $\tau = 3$ sec

Merits of QE Laser-PAS based Trace Gas Detection

- High sensitivity (ppm to ppb gas concentration levels); proportional to laser power, Q and excellent dynamic range
- Immunity to ambient and flow acoustic noise, laser noise and etalon effects, which allows applications that involve a harsh operating environment
- Required sample volume is very small. The volume is ultimately limited by the gap size between the TF prongs, which is 0.34 mm^3 for the presently used QTF.
- No spectrally selective elements are required
- Applicable over a wide range of pressures, including atmospheric pressure
- Finite time of the energy transfer rate from vibrational to translational degrees of freedom strongly influences amplitude and phase of the photoacoustic response, and hence the sensitivity of photoacoustic gas sensing.
- Ultra-compact, rugged and low cost compared to LAS that requires a multipass absorption cell and infrared detector(s)
- Potential for optically multiplexed concentration measurements

QEPAS Performance for 10 Trace Gas Species (Sept'05)

Molecule (Host)	Frequency, cm ⁻¹	Pressure, Torr	NNEA, cm ⁻¹ W/Hz ^{1/2}	Power, mW	NEC ($\tau=1s$), ppmv
H ₂ O (N ₂)**	7181.17	60	2.1×10^{-9}	5.8	0.18
HCN (air: 50% hum) **	6539.11	60	$< 2.6 \times 10^{-9}$	50	0.1
C ₂ H ₂ ((N ₂))**	6529.17	75	$\sim 2.5 \times 10^{-9}$	~40	0.06
NH ₃ (N ₂)*	6528.76	60	5.4×10^{-9}	38	0.50
CO ₂ (exhaled air)	6514.25	90	1.0×10^{-8}	5.2	890
CO ₂ (N ₂) ***	4990.00	300	1.5×10^{-7}	4.6	130
CH ₂ O (N ₂) *	2832.48	100	4.6×10^{-9}	4.9	0.20
CO (N ₂)	2196.66	50	5.3×10^{-7}	13	0.5
CO (propylene)	2196.66	50	7.4×10^{-8}	6.5	0.14
N ₂ O (air+5%SF ₆)	2195.63	50	1.5×10^{-8}	19	0.007

* - Improved microresonator

** - Improved microresonator and double optical pass through QTF

*** - Without microresonator

NNEA – normalized noise equivalent absorption coefficient.

NEC – noise equivalent concentration for available laser power and $\tau=1s$ time constant.

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

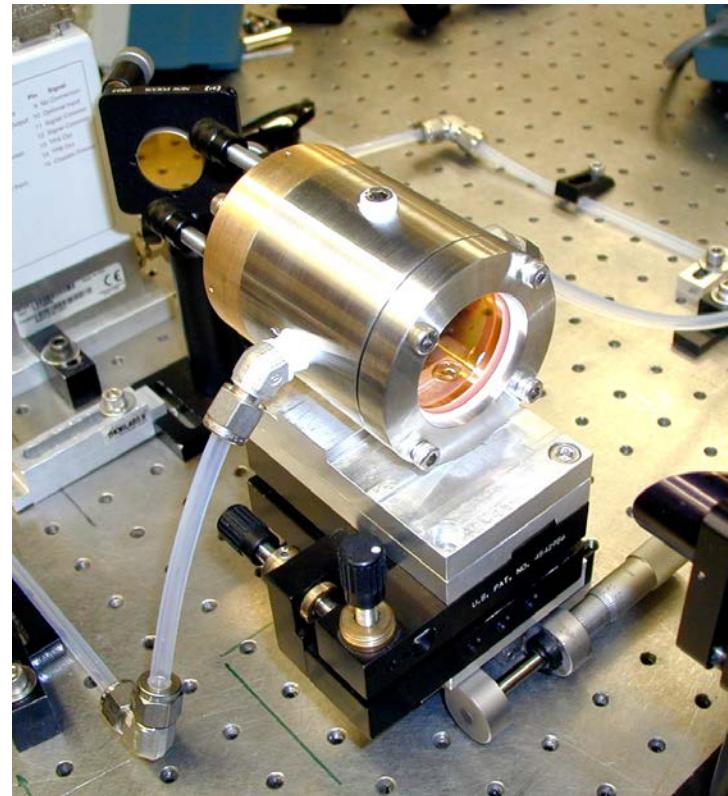
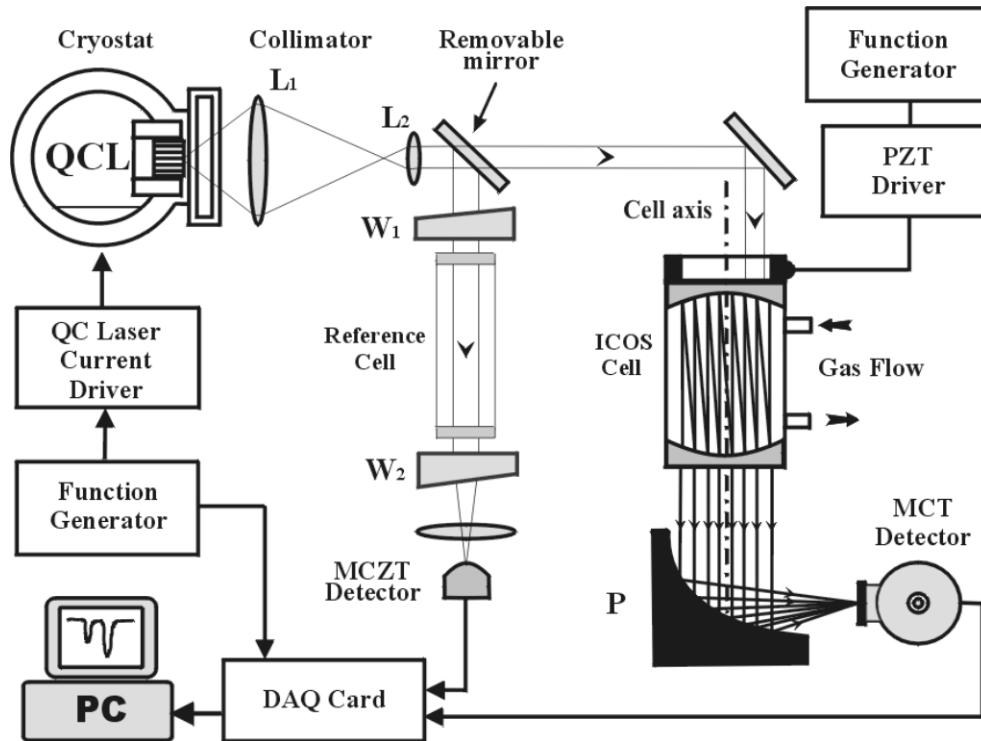
* M. E. Webber, M. Pushkarsky and C. K. N Patel, Appl. Opt. 42, 2119-2126 (2003)



Important Biomedical Target Gases for Breath Analysis

Molecule	Formula	Biological/Pathology Indication
Pentane	$\text{CH}_3(\text{CH}_2)_3\text{CH}_3$	Lipid peroxidation, oxidative stress associated with inflammatory diseases, transplant rejection, breast and lung cancer
Ethane	C_2H_6	Lipid peroxidation and oxidative stress, lung cancer
CO_2 isotope ratio	$^{13}\text{CO}_2 / ^{12}\text{CO}_2$	Marker for Helicobacter pylori infection, Gastrointestinal and hepatic functions
Carbonyl Sulfide	COS	Liver disease and acute rejection in lung transplant recipients (10-500 ppb?)
Carbon disulfide	CS_2	Schizophrenia
Ammonia	NH_3	Liver and renal diseases, fasting response, hepatic encephalopathy
Formaldehyde	HCHO	Cancerous tumors, breast cancer (400-1500 ppb)
Nitric Oxide	NO	Inflammatory and immune responses (e.g., asthma) and vascular smooth muscle response (6-100 ppb)
Hydrogen Peroxide	H_2O_2	Airway Inflammation, Oxidative stress (1-5 ppb)
Carbon Monoxide	CO	Smoking response, CO poisoning, vascular smooth muscle response, platelet aggregation (400-3000 ppb)
Ethylene	$\text{H}_2\text{C}=\text{CH}_2$	Oxidative stress, cancer
Acetone	CH_3COCH_3	Fasting response, diabetes mellitus response, ketosis

Off-Axis Integrated Cavity Output Spectroscopy (ICOS) Based Gas Sensor



- Novel compact gas cell design of length: 3.8 – 5.3 cm and cell volumes < 80 cm³;
- Low loss mirrors (ROC 1m):~60-250 ppm, R~99.975, L_{eff}=170-800 m
- Rapid eNO concentration measurements during a single breath cycle are feasible

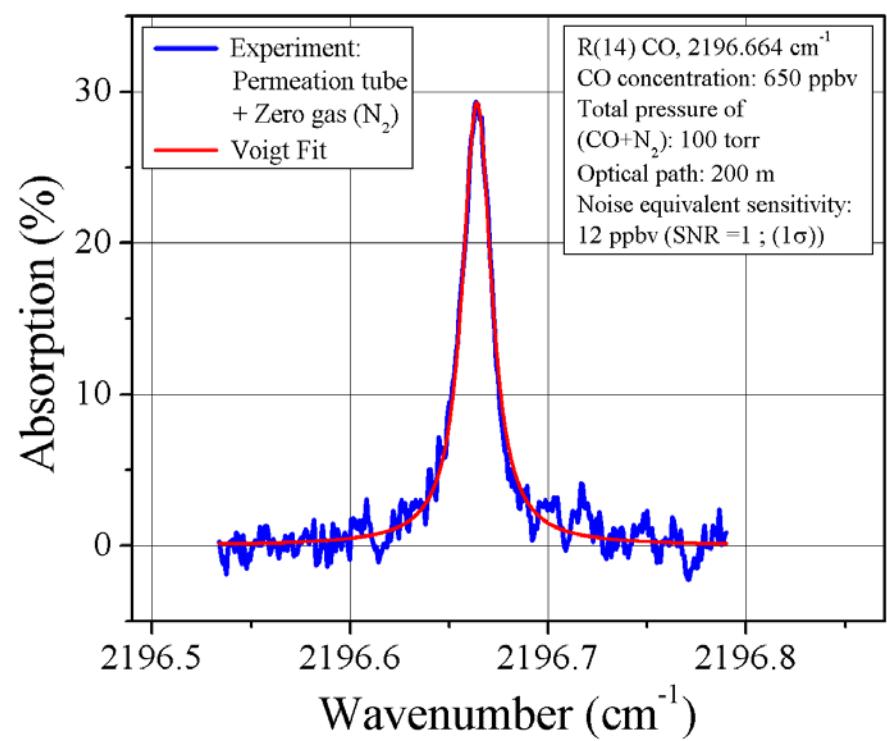
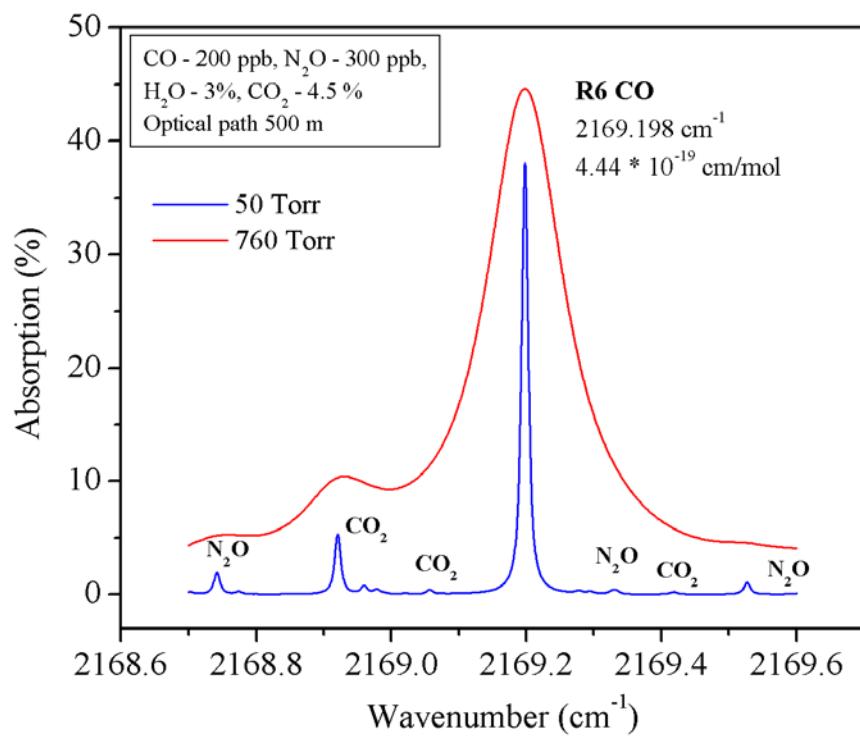
PSI

PHYSICAL SCIENCES INC.

LGR

RICE

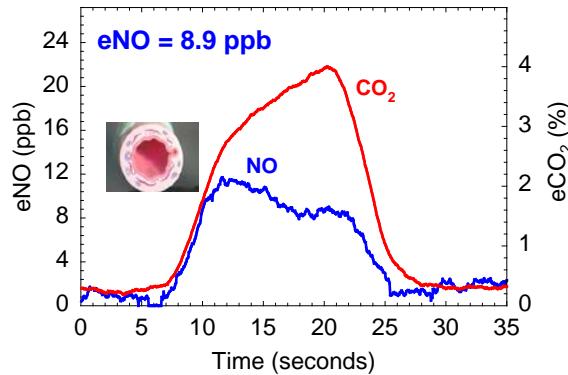
OA-ICOS based CO Concentration Measurements at 2196.66 cm^{-1}



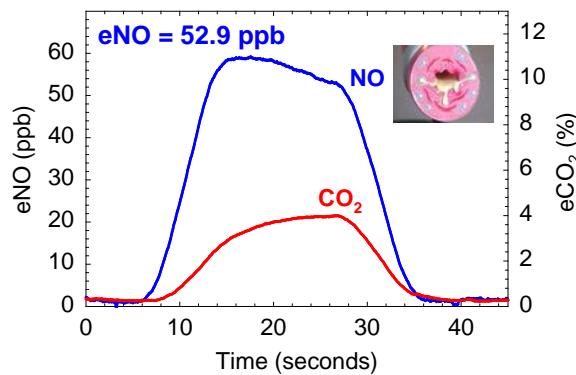
Nitric Oxide & Asthma



Age 5, Female, Mild-Persistent asthmatic, Corticosteroid Treated

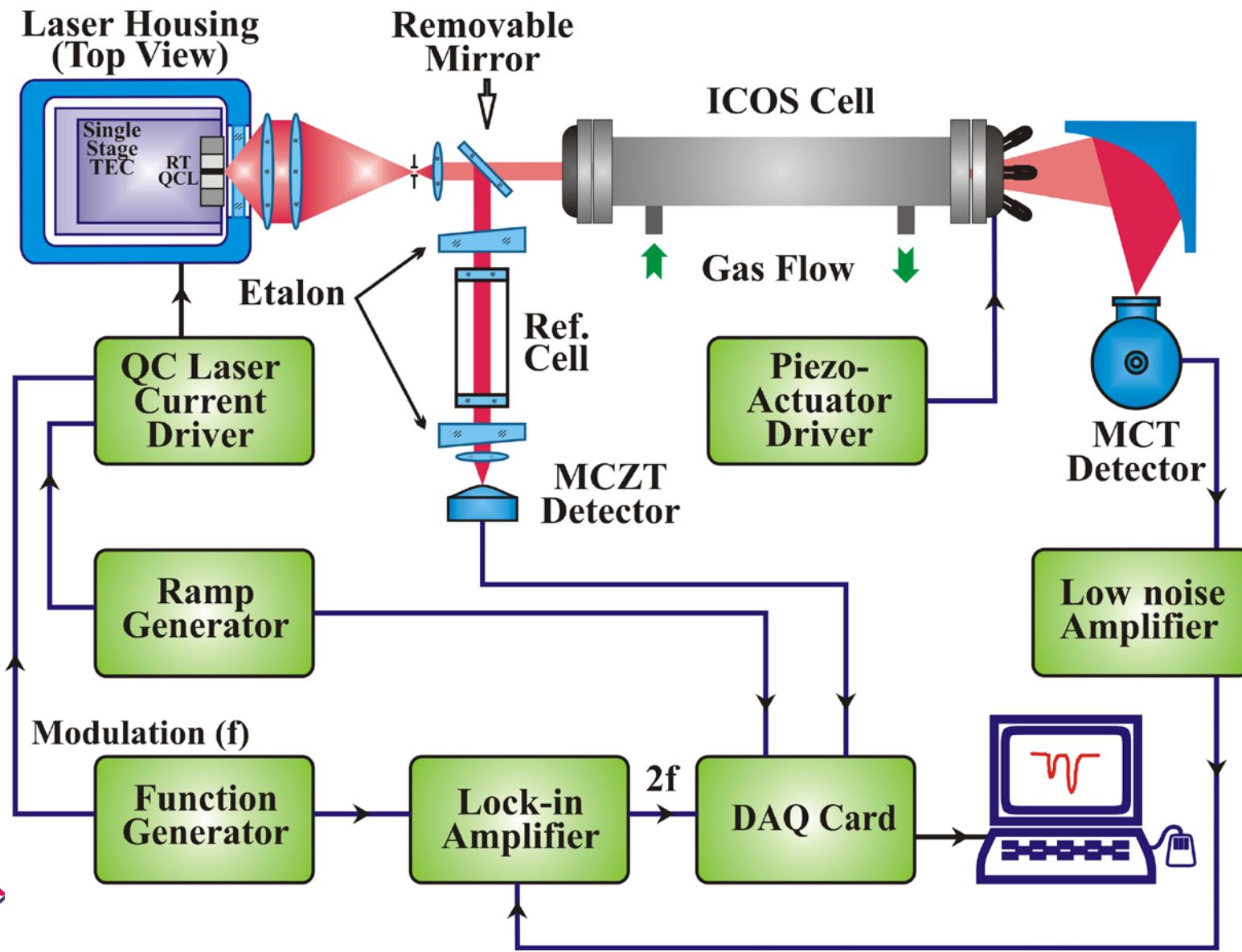


Age 14, Male, Mild-Persistent asthmatic, Non-Treated

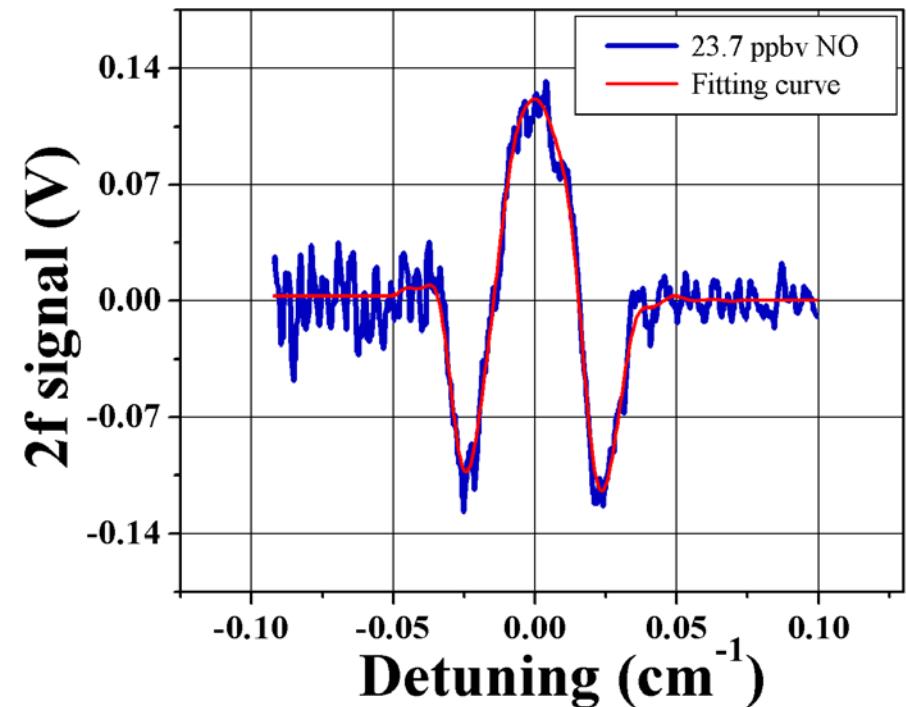


eNO measurements are useful for asthma screening, diagnosis, and therapy monitoring

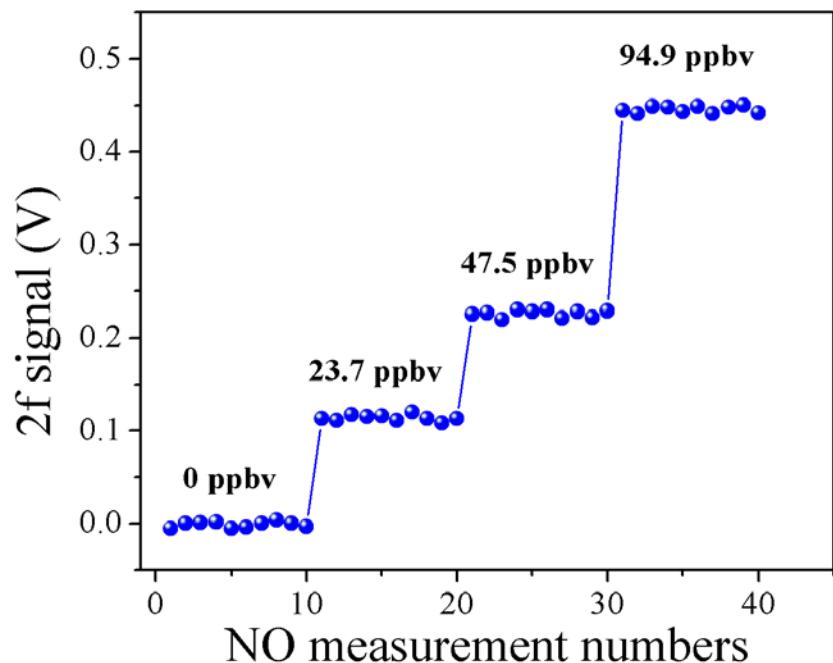
TEC – CW-DFB QCL based Nitric Oxide OA-ICOS Sensor



2f NO Absorption Signal at 1835.57 cm^{-1}



NO:N₂ mixture @ 100 Torr
Effective L= 700 m

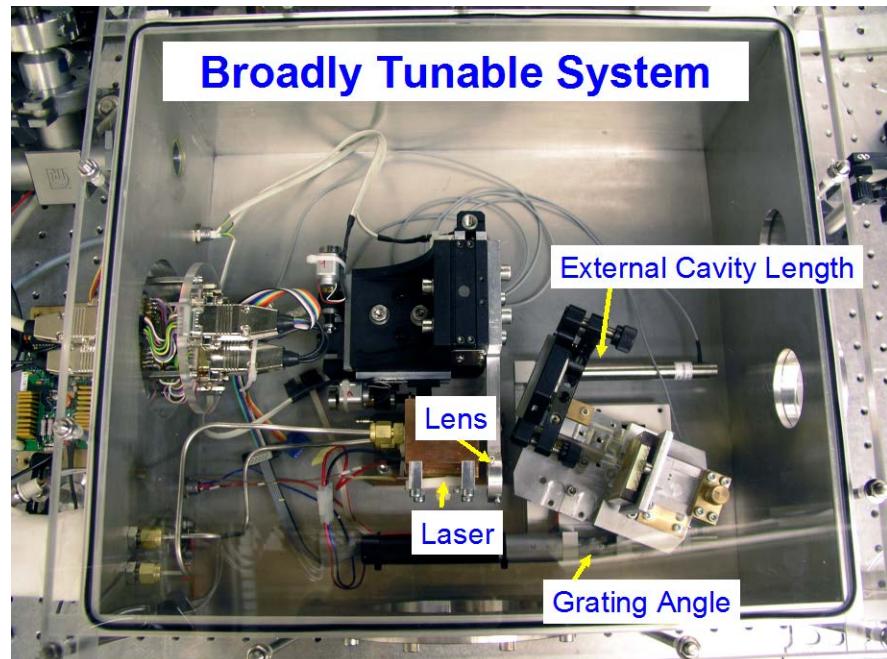
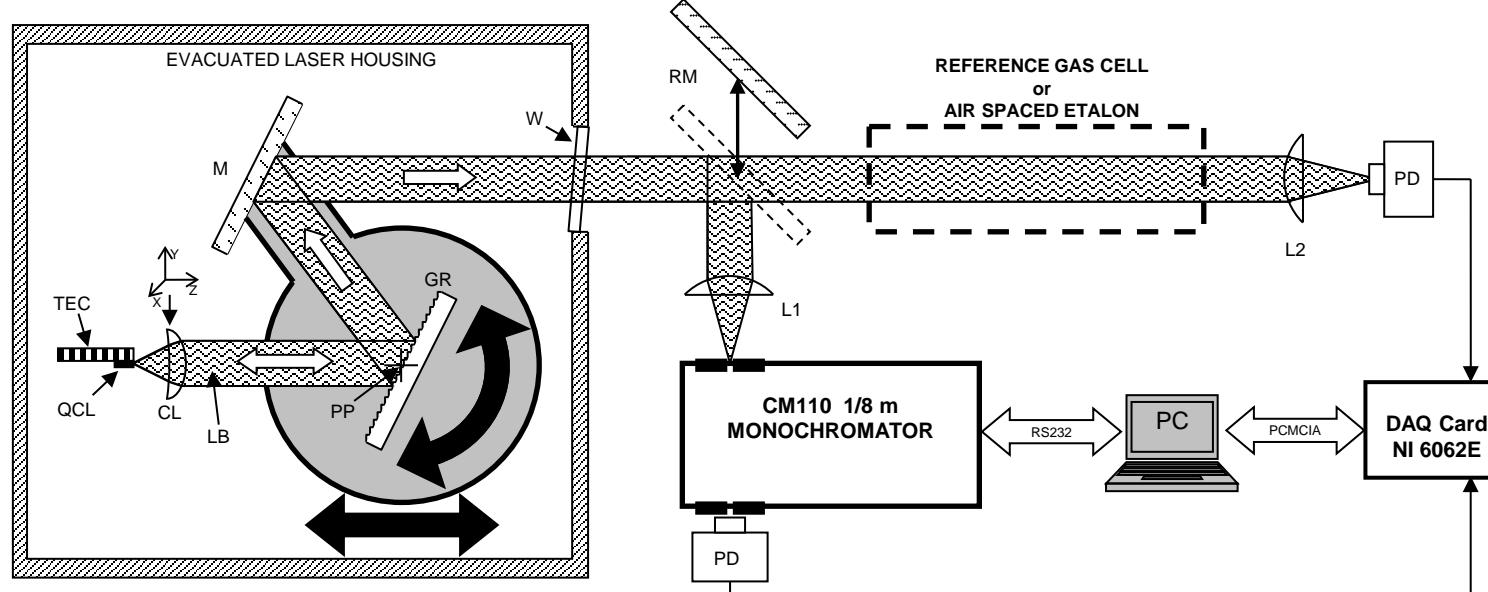


Noise equivalent sensitivity:
0.7 ppbv (1 σ)

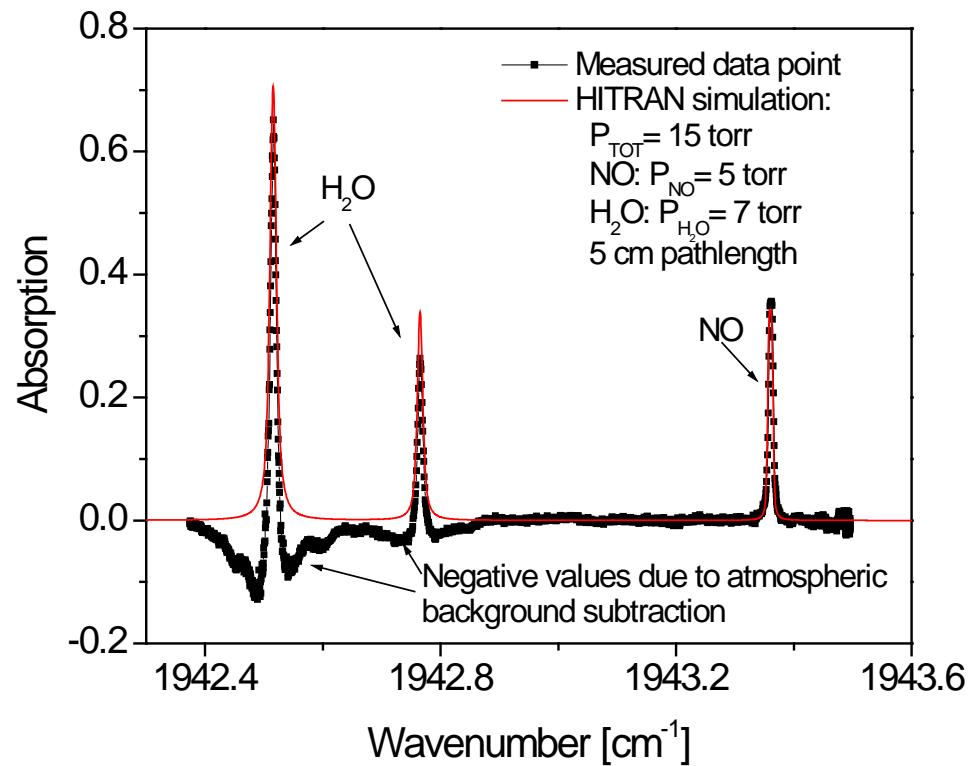
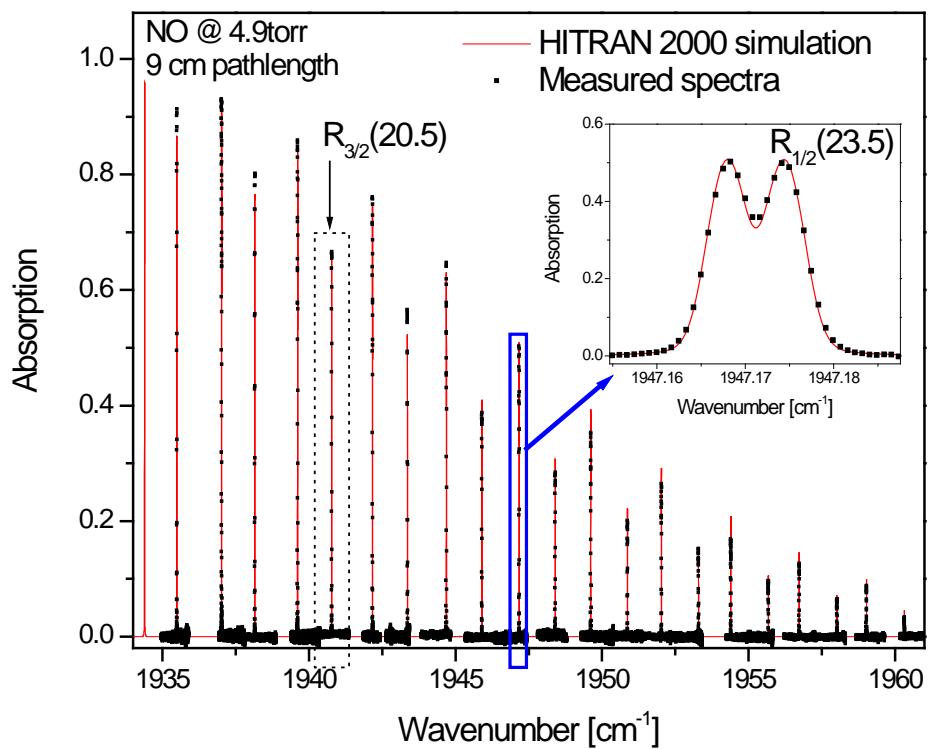
Tunable External Cavity QCL System

- QCL – quantum cascade laser
TEC – thermoelectric cooler
CL – collimating lens (1" diameter, f/0.6,
Ge AR-coated 3-12 μm) mounted on
a motorized 3D translation stage
LB – laser beam
GR – diffraction grating (150 gr/mm blazed for 5.4 μm)
PP – pivot point of the rotational movement
M – mirror (mounted on the same platform with GR)
W – CaF₂ window (thickness 4mm, tilted ~50°)
RM – removable mirror
PD – photodetector (Hg-Cd-Zn-Te, TE-cooled,
Vigo Systems, PDI-2TE-6)
L1, L2 – ZnSe lenses

Coupled cavities:
Laser Chip ~ 15 GHz, External Cavity ~ 1.5 GHz

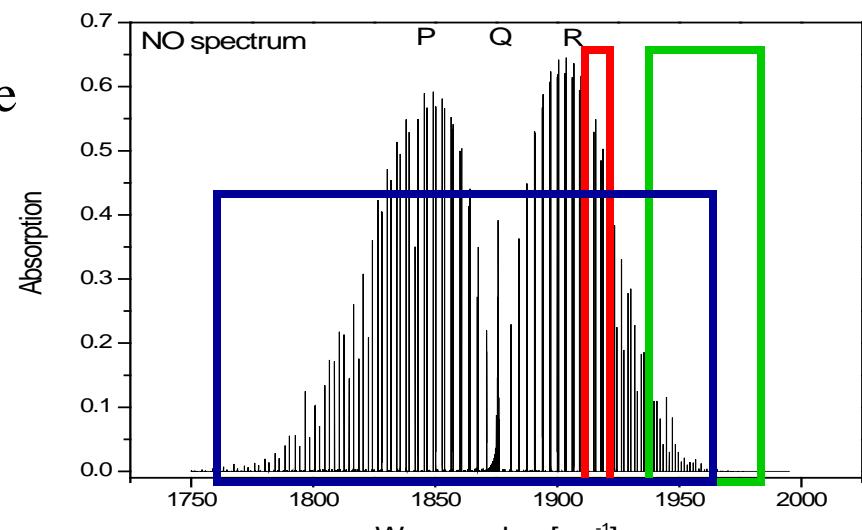
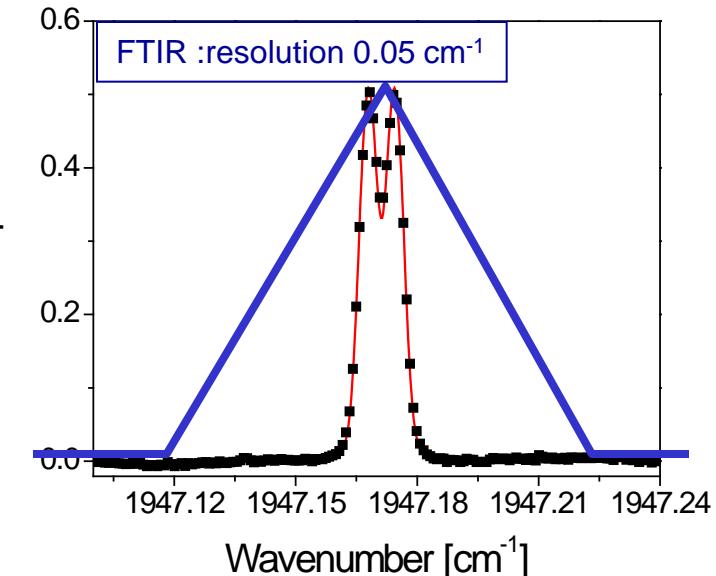


Mid-IR NO Absorption Spectra Acquired with a Tunable TEC QCL

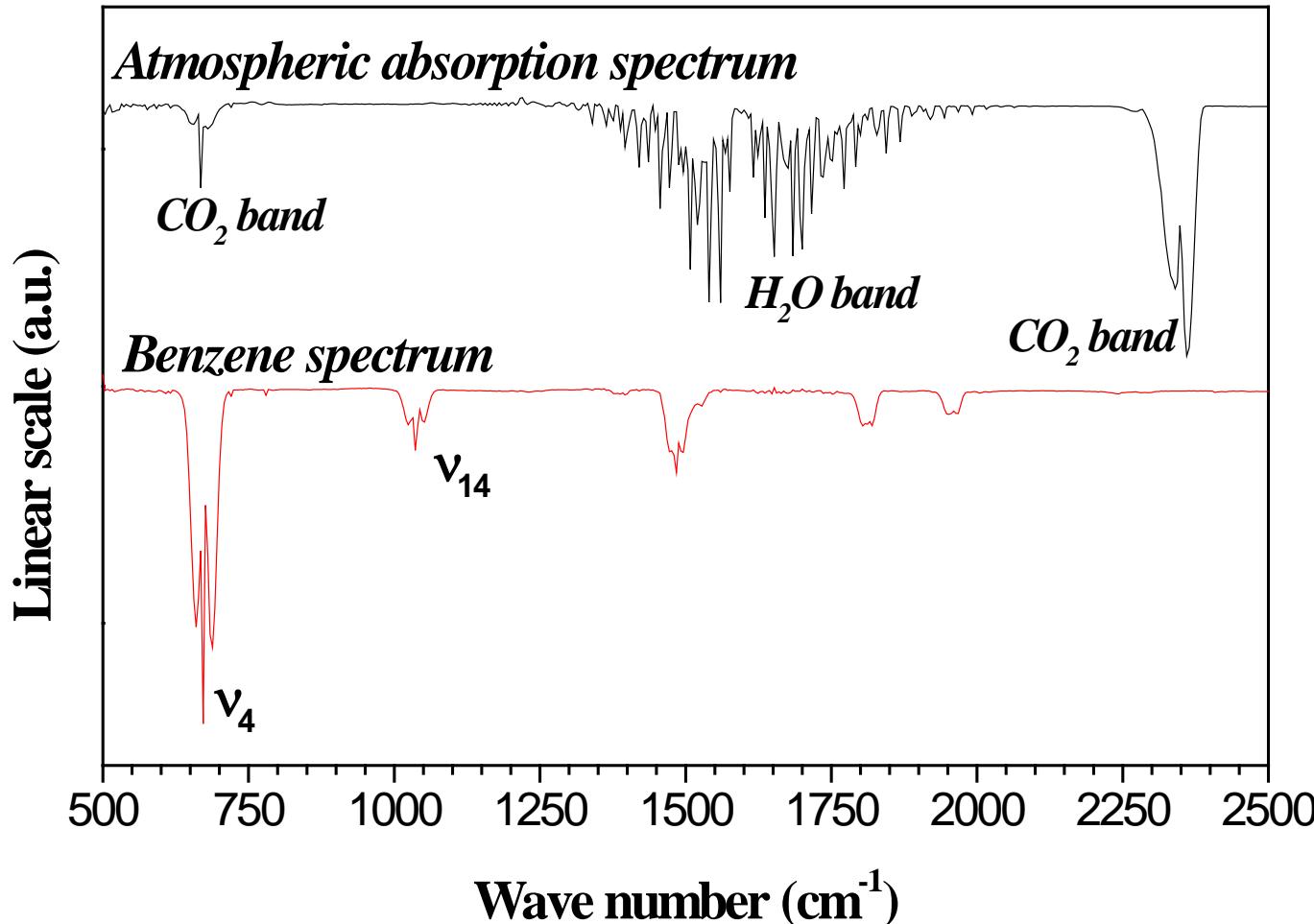


Important facts of novel EC-QCL Technology

- Laser spectroscopy provides superior resolution compared to other techniques e.g. FTIR
- Single mode operation of the laser is required
- Wavelength tunability of single mode (DFB) mid-IR semiconductor lasers is $\sim 10\text{cm}^{-1}$
- Demonstrated wavelength tunability of the Rice EC QCL is $\sim 35 \text{ cm}^{-1}$ (limited by the gain chip properties and not by the designed EC configuration)
- Gain chips, which can provide tunability of $>200 \text{ cm}^{-1}$ were already reported in the literature



FT-IR survey absorption spectrum of benzene vapor (C_6H_6)



Conclusions and Future Directions

- **Laser based Trace Gas Sensors**

- Ultra compact ($\sim 0.2 \text{ mm}^3$), robust & low cost sensors based on QE L-PAS
- QEL-PAS is immune to ambient noise. The measured noise level coincides with the thermal noise of the QTF
- Best to date demonstrated QEPAS sensitivity is $2.1 \times 10^{-9} \text{ cm}^{-1}\text{W}/\sqrt{\text{Hz}}$ for $\text{H}_2\text{O}:\text{N}_2$
- QEPAS exhibits a low $1/f$ noise level, allowing data averaging for more than 3 hours
- Detected 14 trace gases to date: NH_3 , CH_4 , N_2O , CO_2 , CO , NO , H_2O , COS , HCN , C_2H_4 , C_2H_2 , $\text{C}_2\text{H}_5\text{OH}$, SO_2 , H_2CO and several isotopic species of C, O, N & H

- **Applications in Trace Gas Detection**

- Environmental & Spacecraft Monitoring (NH_3 , CO , CH_4 , C_2H_4 , N_2O , CO_2 and H_2CO)
- Medical Diagnostics (NO , CO , COS , CO_2 , NH_3 , C_2H_4)
- Industrial process control and chemical analysis (NO , NH_3 , H_2O)

- **Future Directions and Collaborations**

- QE L-PAS based applications using novel thermoelectrically cooled cw and broadly wavelength tunable quantum and interband cascade lasers
- Investigate QTFs with lower resonant frequencies
- Investigate amplitude modulation QEPAS potential and limitations
- New target gases, in particular VOCs and HCs
- Development of optically multiplexed gas sensor networks based on QE L-PAS