Near- and MID-IR Semiconductor Laser-Based Sensors for Industrial Process Monitoring

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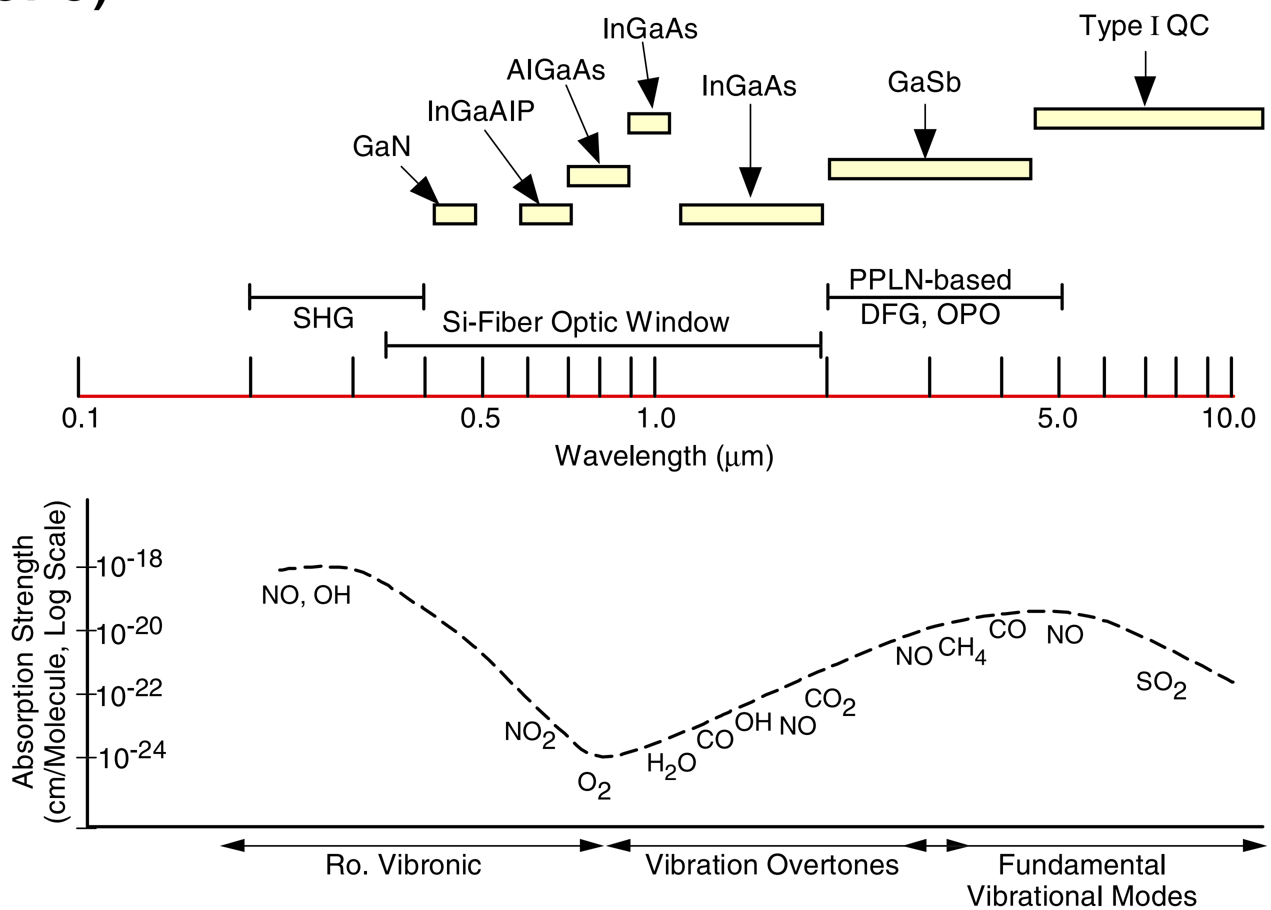
Physical Sciences Inc.

May 2001

Sources and Target Gas Absorptions

VG01-102-1

- **Direct current injection devices from 0.43 → 2.0 μm; 4.6 → 25 μm**
- **Frequency converted devices from 0.22 → 0.43 μm (SHG); 2.3 → 4.5 μm (DFG, OPO)**



E-3757a

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Example of PSI Multi-Wavelength Near-IR TDL Sensor Configuration

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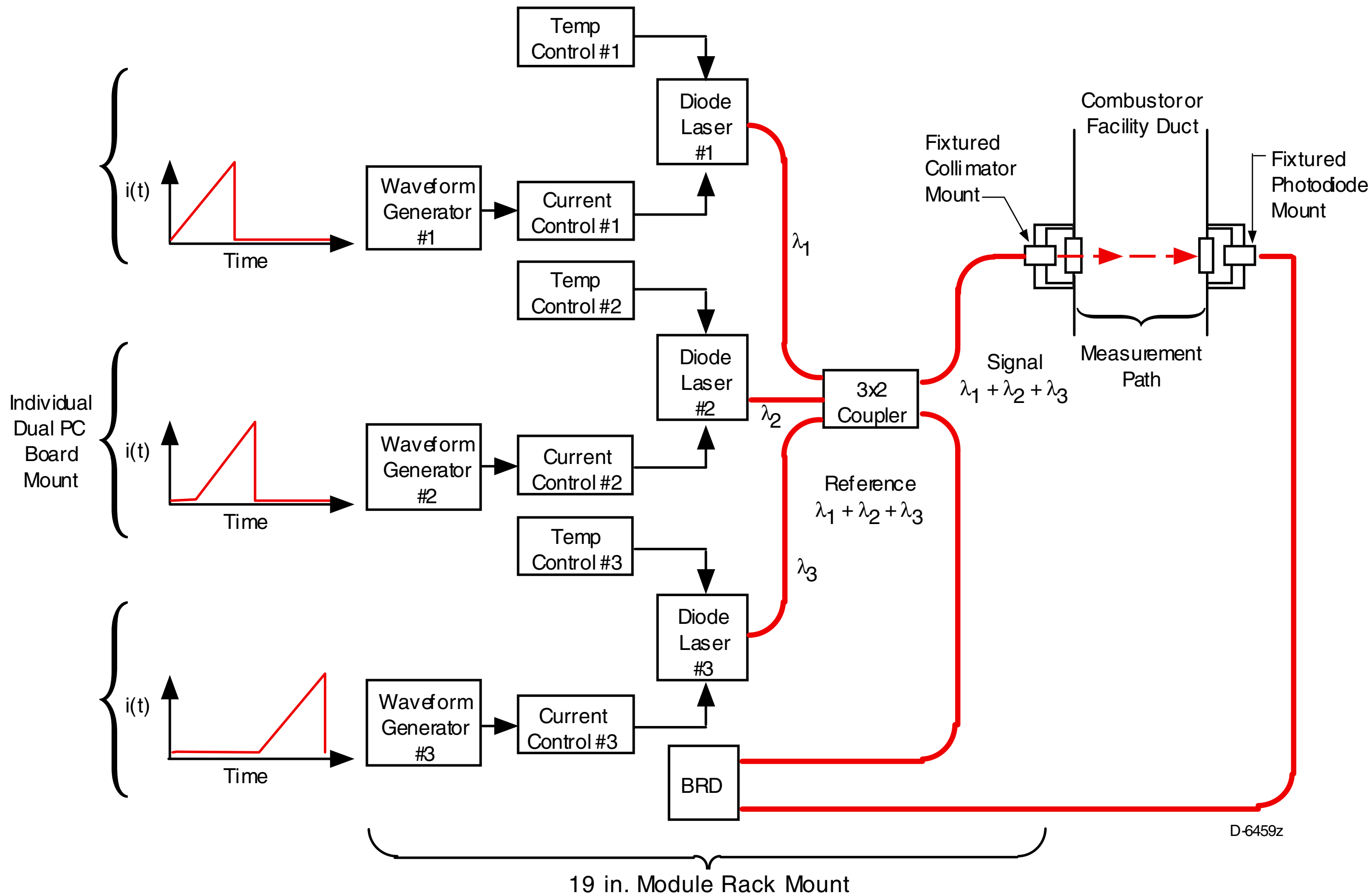

- **Multiple lasers integrated into single instrument module**
- **Fiber/copper transmission of ~ 1000's m to measurement location**

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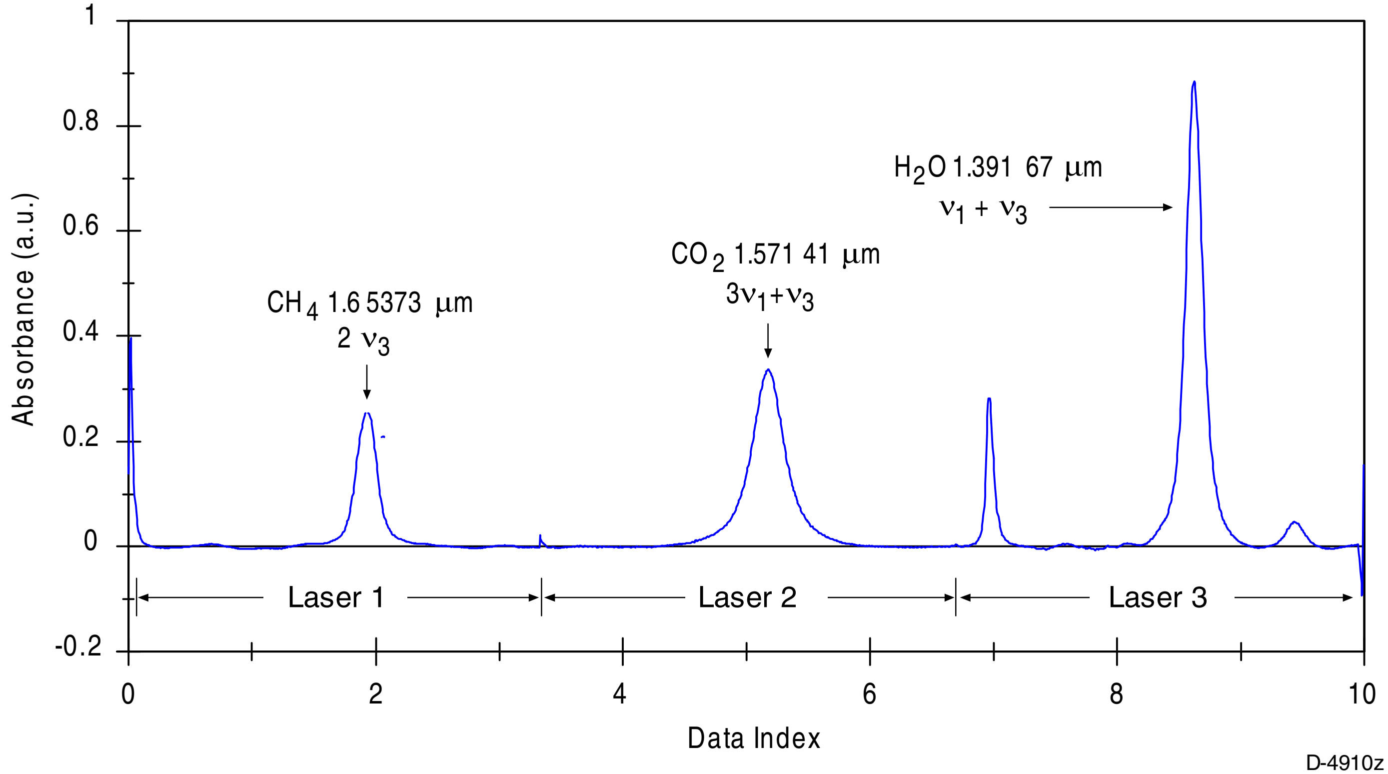
Simultaneous Detection of CH_4, CO_2, and H_2O Using Multi-plexed Diode Laser Sensor

VG01-102-3

- **0.5 Torr CH_4, 68.1 Torr CO_2, 14.1 Torr H_2O**
- **50 cm path, single-pass, room-temperature**



- **10 ms sweep (sum of three lasers), 200 sweep average, 2 second measurement time**

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Grating-Coupler, Sampled Reflector Laser

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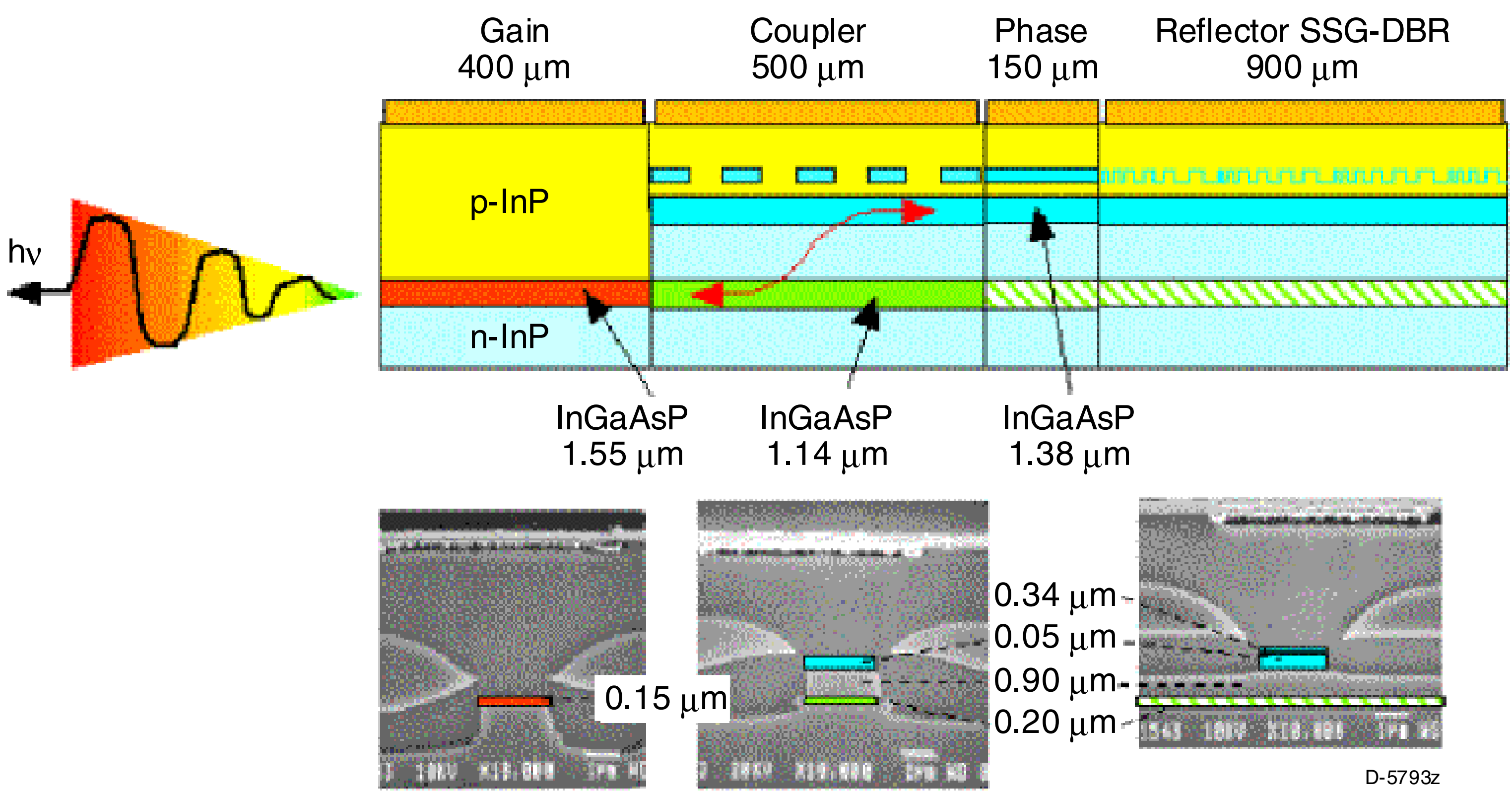

- **Current tuning is de-coupled from gain**

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Example GCSR Tuning Surface from ADC

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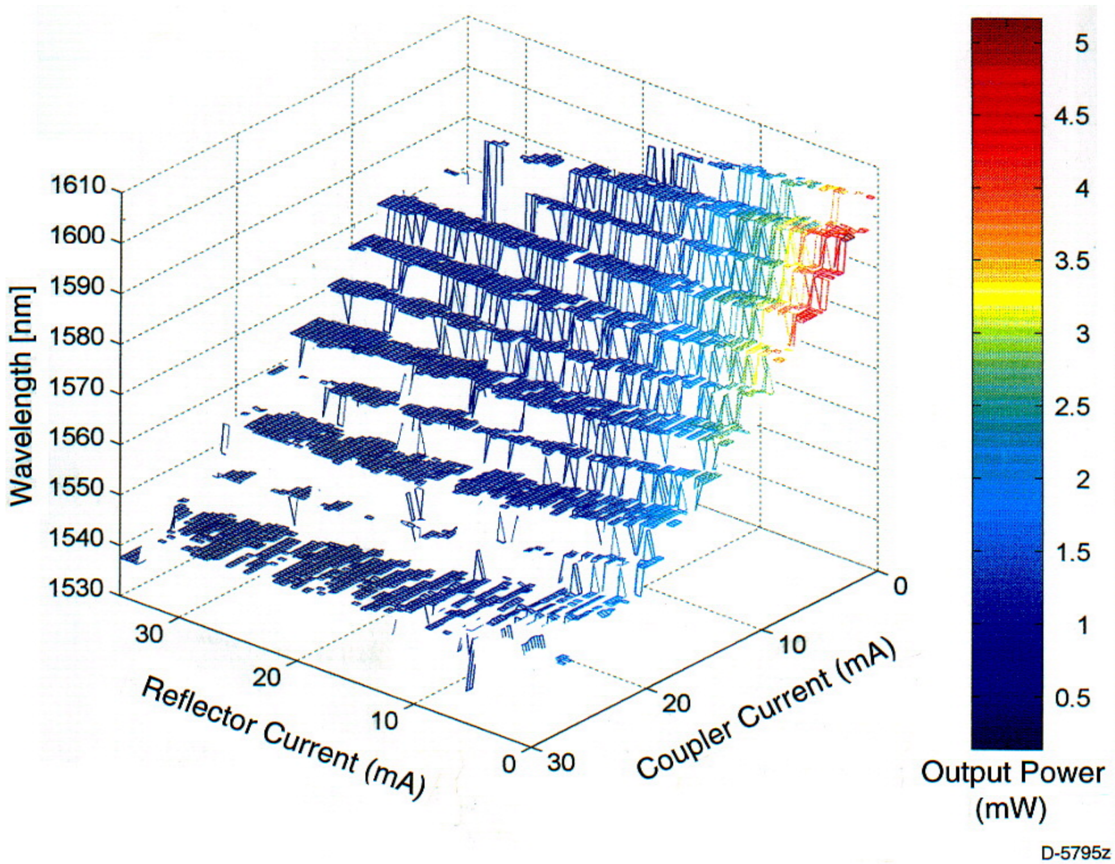
- **Provided by manufacturer**



- **Any point on this surface can be selected with ~1 μs or less**
- **This single laser can access CO, CO_2, OH, H_2O, N_2O, ...**

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GCSR Scan of Laser Mix

4% CO, 9% CO_2, Balance He, N_2, H_2, atm Pressure

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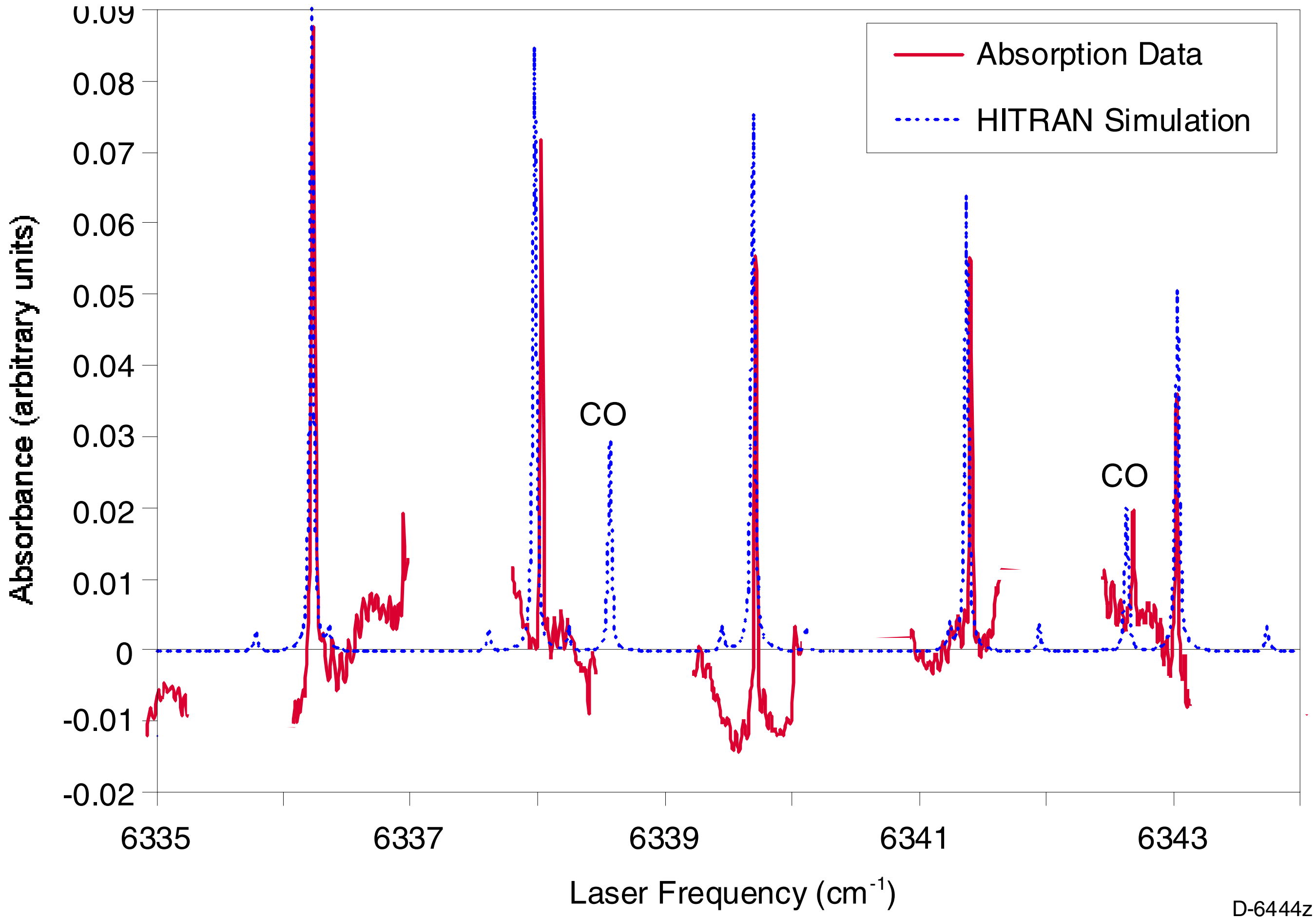

D-6444z

- **SSG reflector scan at fixed coupler, gain currents**
- **No phase current control (open circuit), resulting in mode-hops**

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Example TDL Sensor Dynamic Range: Atmospheric Trace NO_2 Monitor

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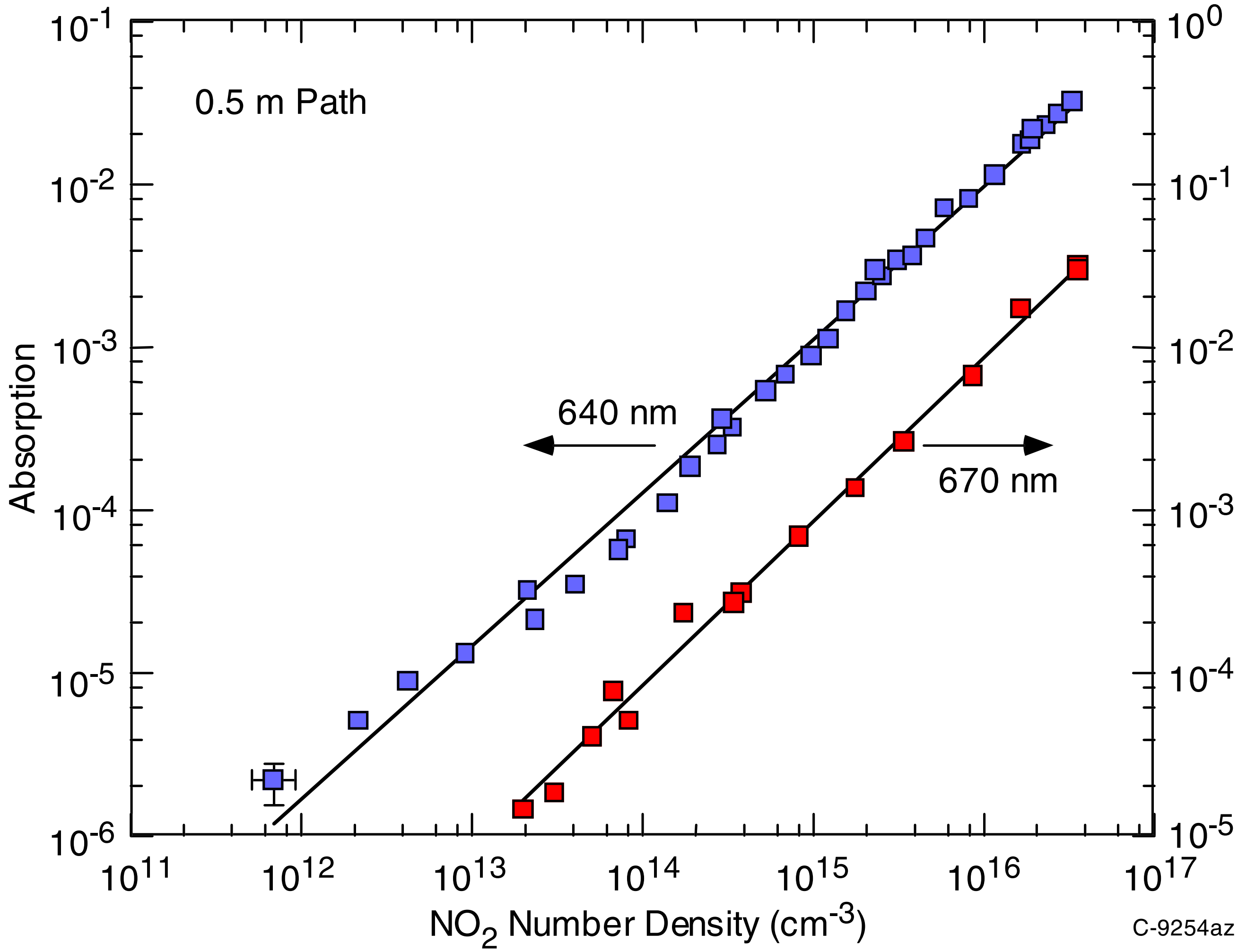

- **More than 10^4 linear dynamic range**

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Example Near-IR Diode Laser Species Sensors

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- **Demonstrated sensitivity in 1 m path at STP**

Species	Wavelength (μm)	Sensitivity (ppm-m)
H_2O	1.31, 1.39	40, 1
CO	1.57	5
CO_2	2.0	0.5
CH_4	1.65	0.1
NO	1.79	3
NO_2	0.67	0.02
N_2O	1.52	1
HCN	1.54	0.02
O_2	0.76	10
$HC\ell$	1.2, 1.7	0.01
NH_3	1.54	0.2

- **Others added frequently**

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Comparison of Measured and Equilibrium CO Concentrations

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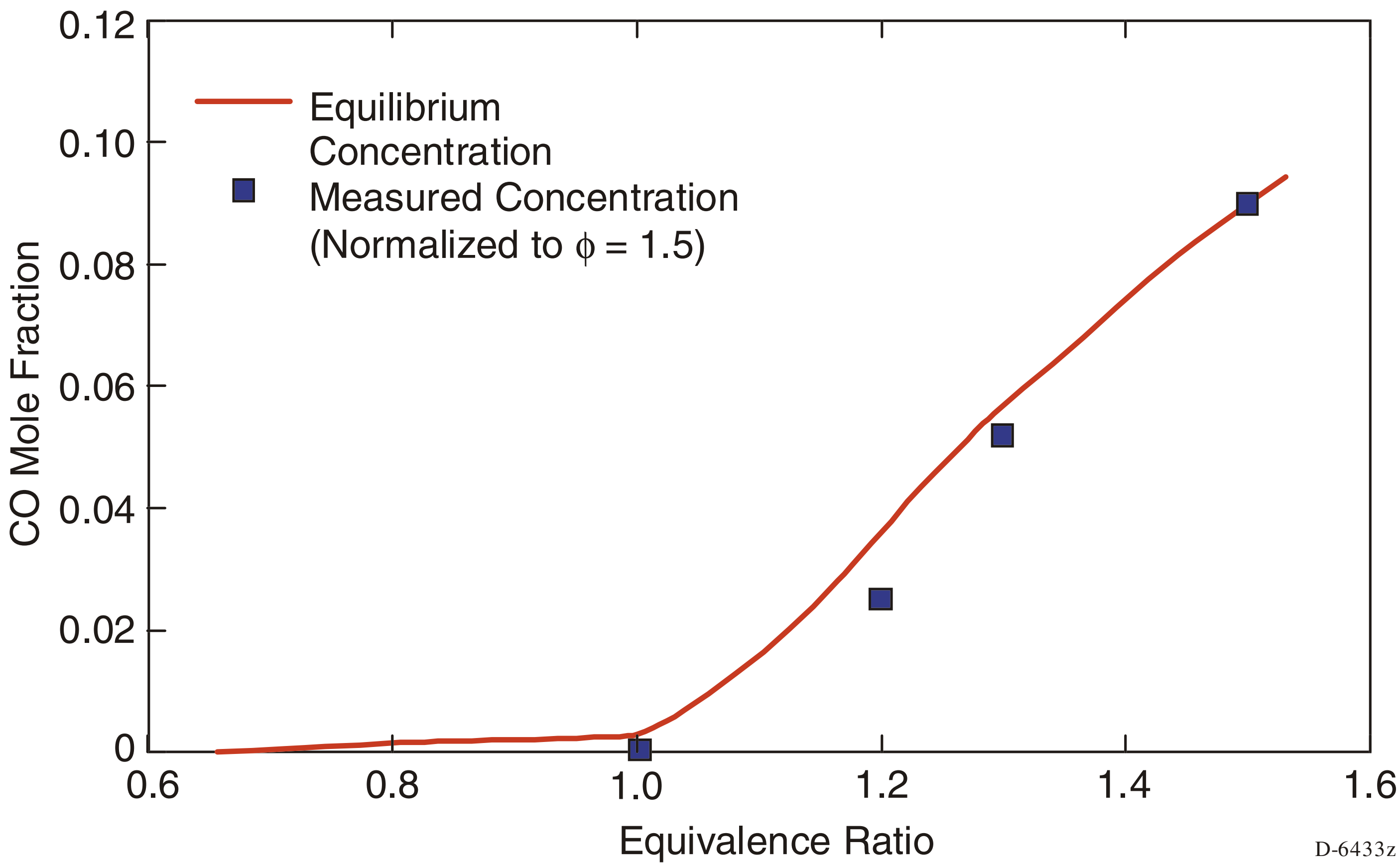
- **Methane air flame, atmospheric pressure**



- **Acquired with GCSR laser**

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Diode Laser Sensors for Control of Oxygen-Enriched Furnaces

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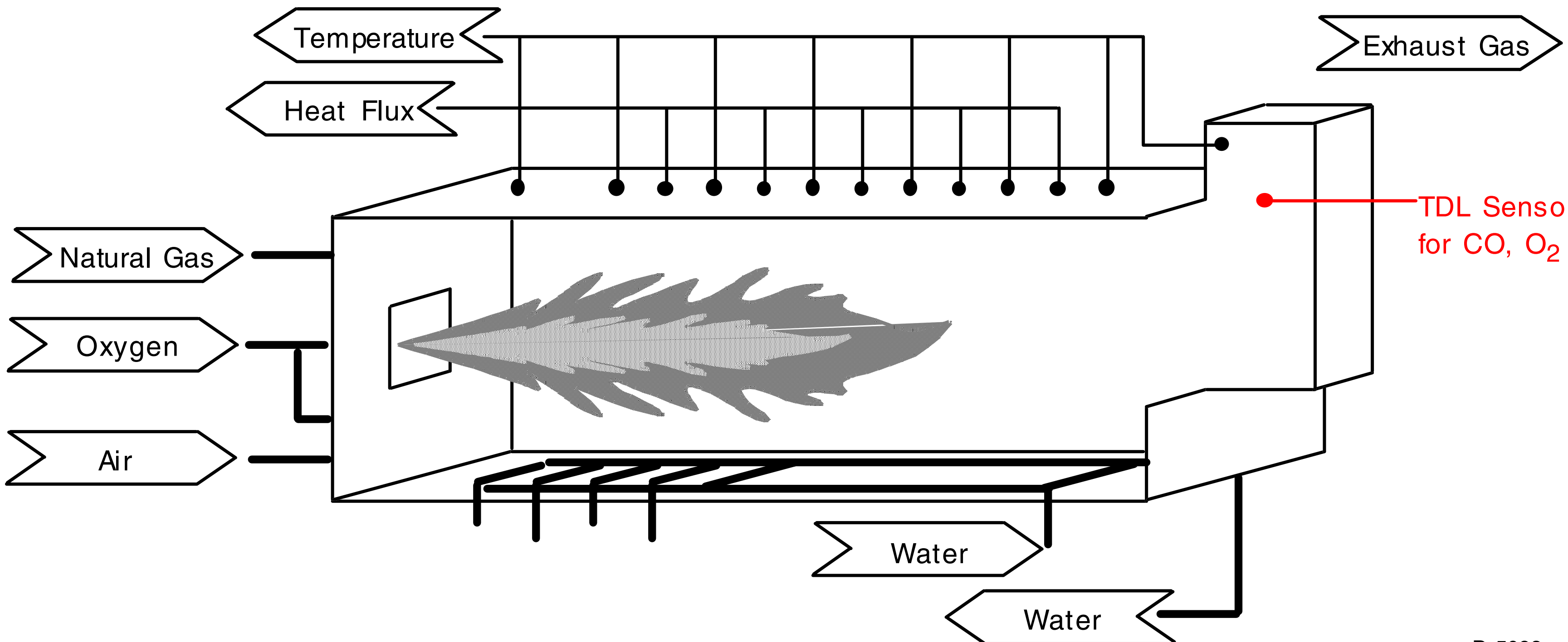

D-5098z

- **In partnership with AirLiquide for pulsed oxy-fuel furnace control**

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O_2 Measurement Comparison

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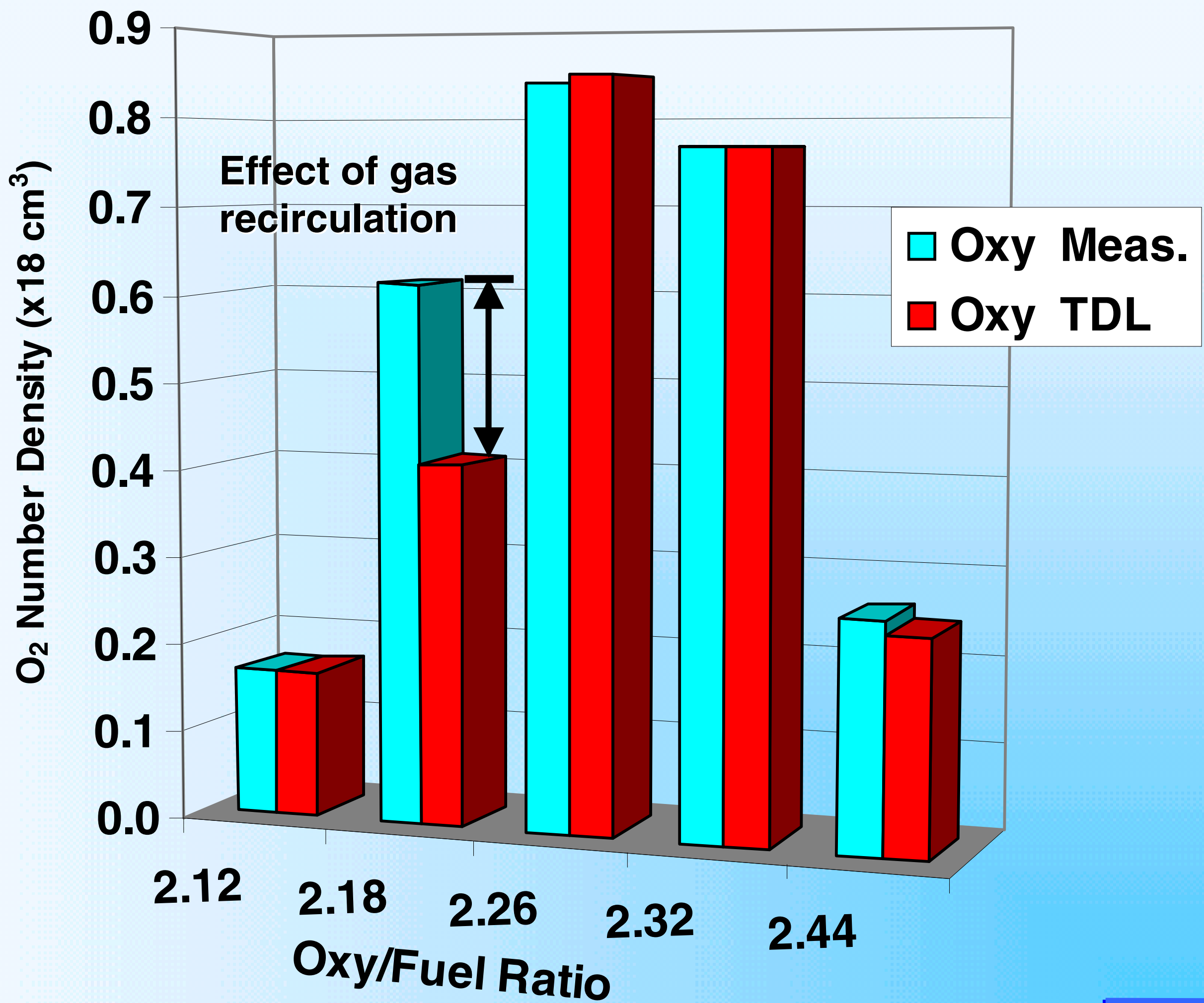
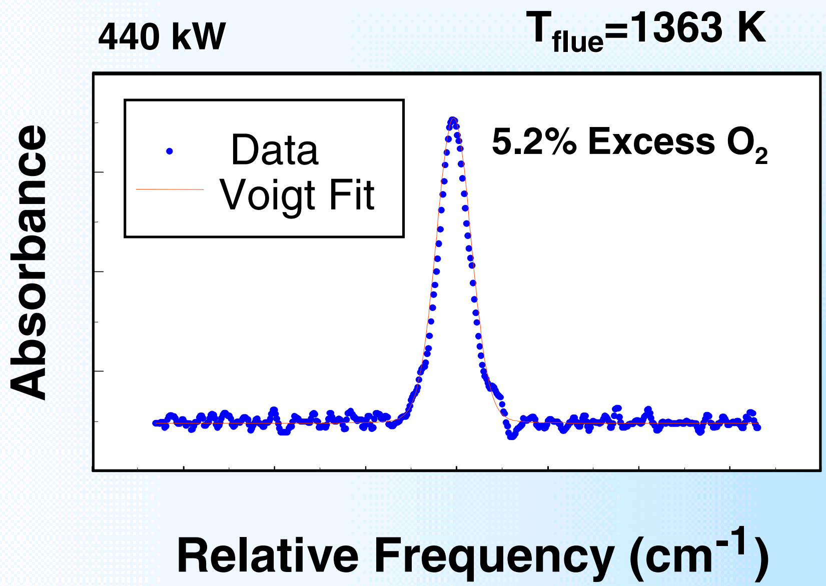
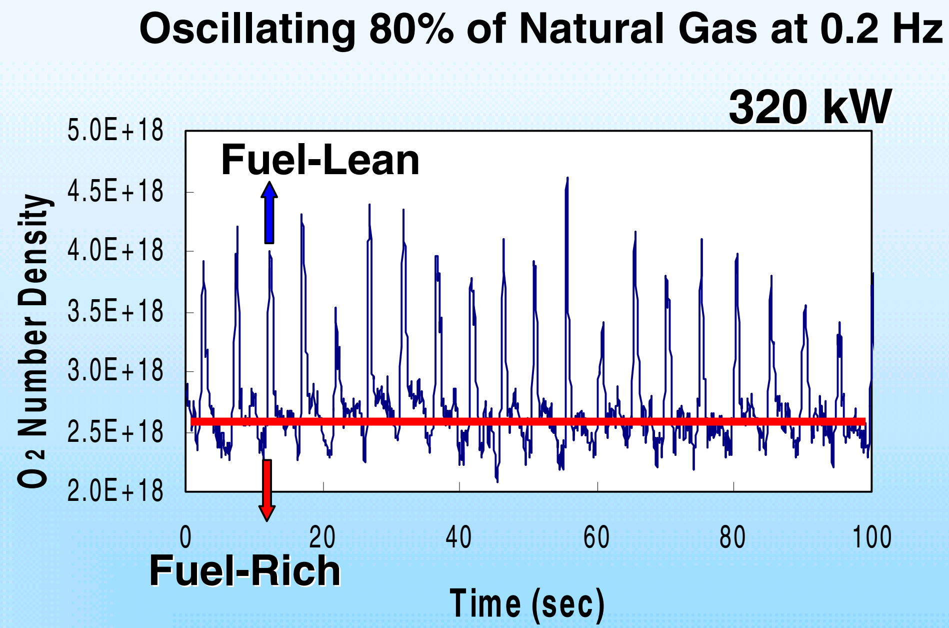


O_2 Monitoring Demonstration

VG01-102-12





- O_2 sensor response in pulsed fuel operation mode

- ***Noise contributions dominated by radiative emission fluctuations in furnace***

AIR LIQUIDE
RESEARCH AND DEVELOPMENT

CO Monitoring Demonstration

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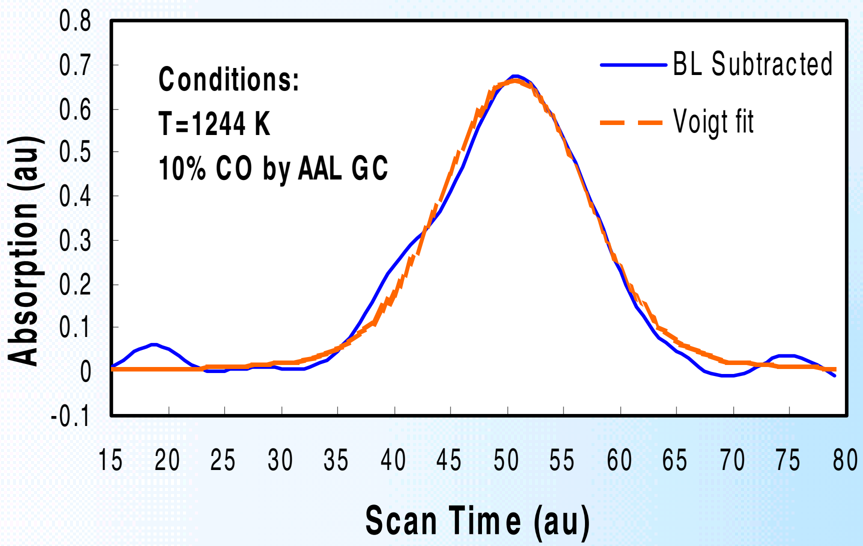
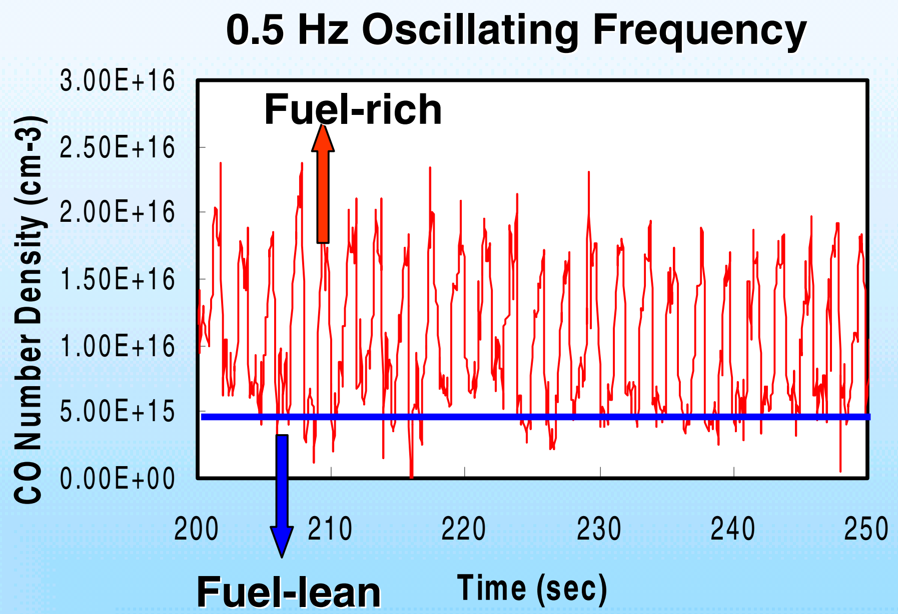


Isolated CO lineshape at 1244 K, 30 cm path (R14 transition, (3,0) band)



CO sensor response in pulsed fuel operation mode

- **Noise levels reduced compared to O_2 data due to smaller detector aperture**
- **rms CO noise level ~ 160 ppm-m**

Two-Line Temperature Measurements

VG01-102-14

- **Measure integrated absorbance from two transitions and define their ratio, R, as:**

$$R \equiv \frac{\int S_{(T)} g_{(\omega)} N \, d\omega \big|_1}{\int S_{(T)} g_{(\omega)} N \, d\omega \big|_2}$$

$$= \left(\frac{S_1}{S_2}\right)_{T_o} \bullet \exp\left[-\frac{hc(E_1 - E_2)}{k}\left(\frac{1}{T} - \frac{1}{T_o}\right)\right]$$

where T_o is an arbitrary reference temperature

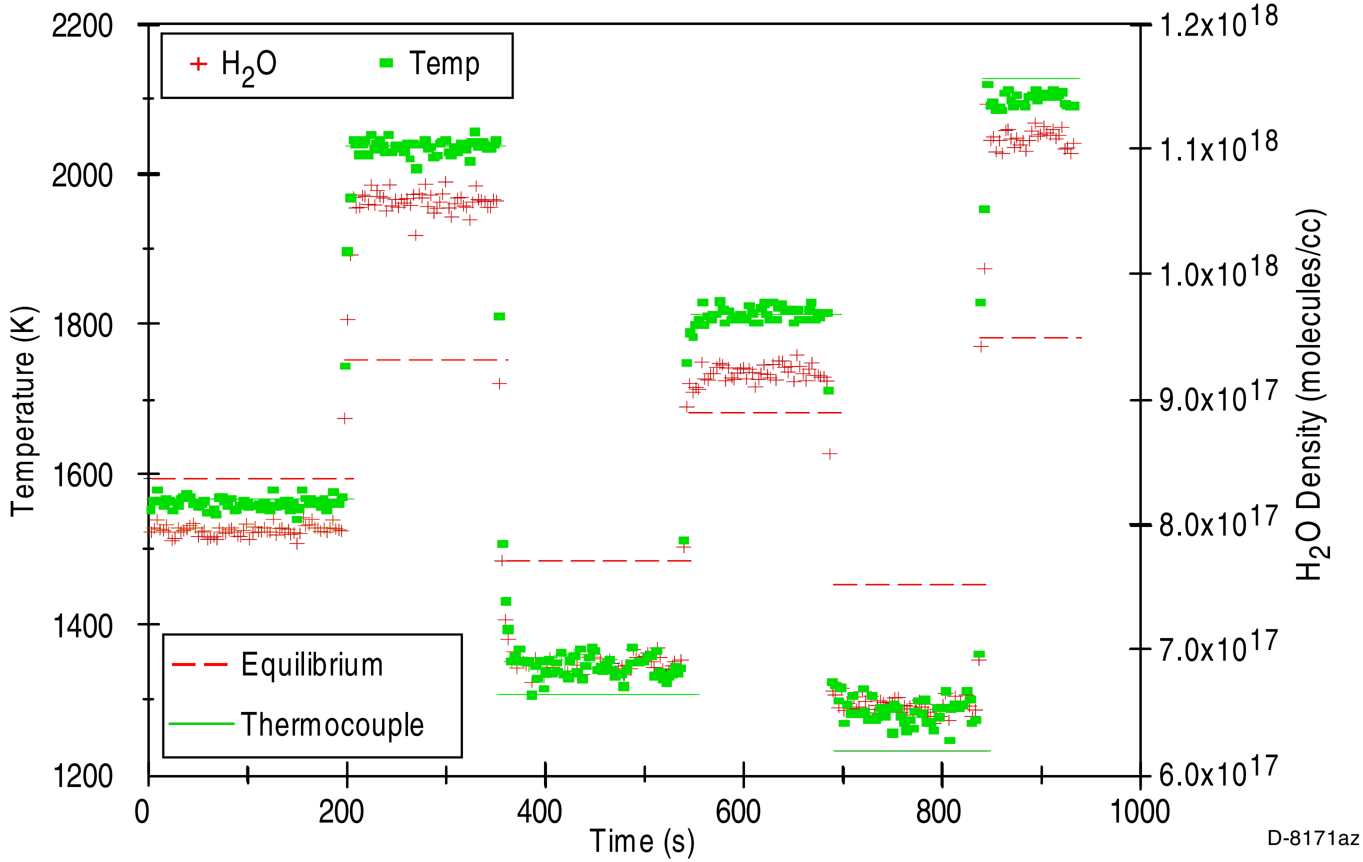
- **Sensitivity of the temperature measurement depends on the choice of absorption lines**

- **Accuracy of the temperature measurement depends on the accuracy of the measured ratio**

Simultaneous Water Vapor Density and Thermometry

H_2-air flame, 70 cm pathlength, 3 second time constant

VG01-102-15



- **Temperature precision ±15 K, thermocouple disagreement < 50 K**
- **Density precision ±2 x 10^{16} cm^{-3} (<2%)**

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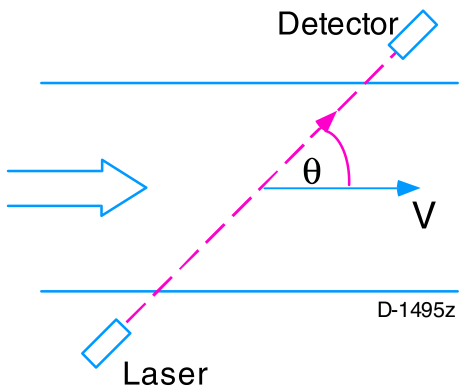
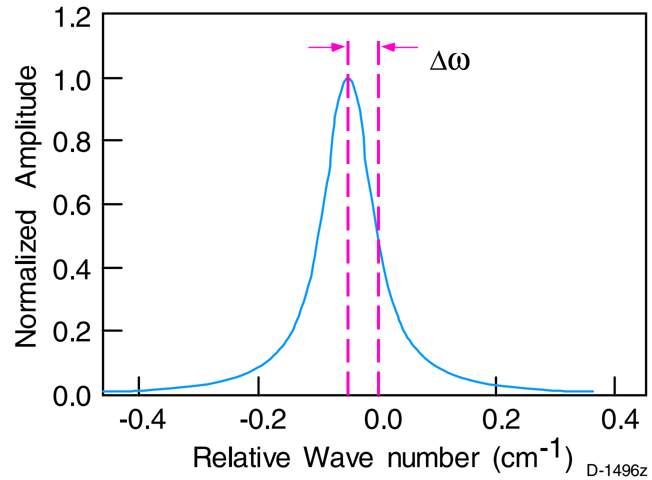
Optical Mass Flux Sensor Basics

VG01-102-16

Basic physics:





$$u = \frac{c\Delta\omega}{\omega_o \cos\theta} \qquad N = \frac{\int_o^{\omega} \ln(I/I_o)\, d\omega}{S\ell} \qquad \dot{m} = u \cdot \rho$$

Technology:

- **Velocity sensitivity to ~ 1 m/s at atm pressure**
  - $\Delta\omega_v/\Delta\omega_a \sim 10^{-4}$

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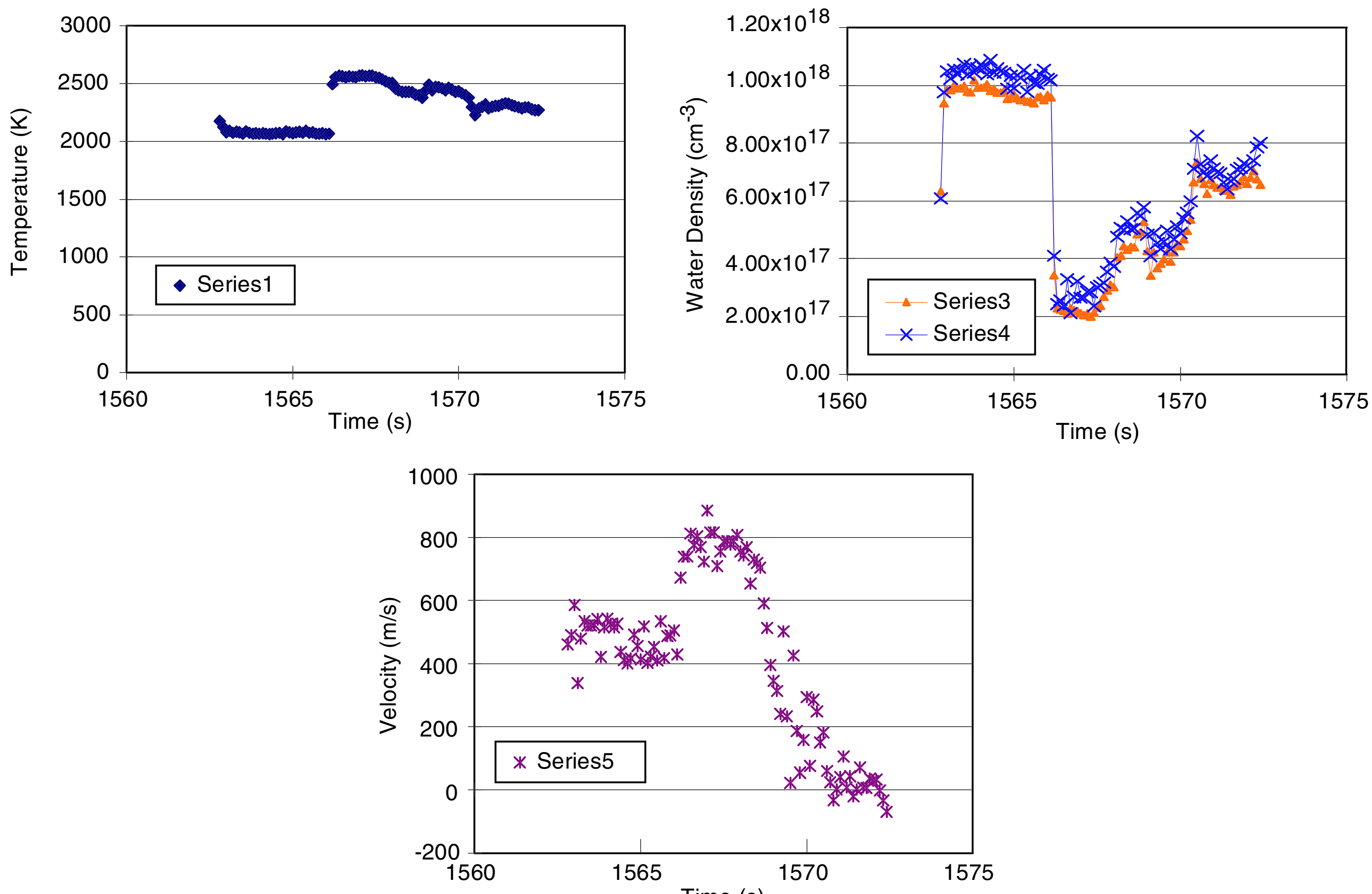
Continuous Gasdynamic Sensing in Supersonic Combustion

VG01-102-17

- **In collaboration with Tohoku University, Sendai, Japan**



E-7683

- **10 Hz sensor response in blowdown SCRAMJET model**

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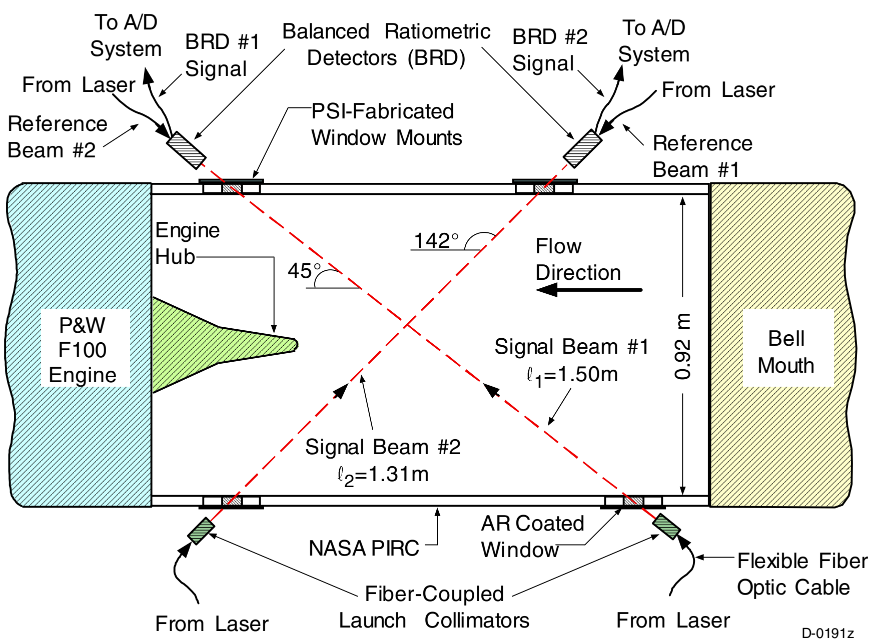
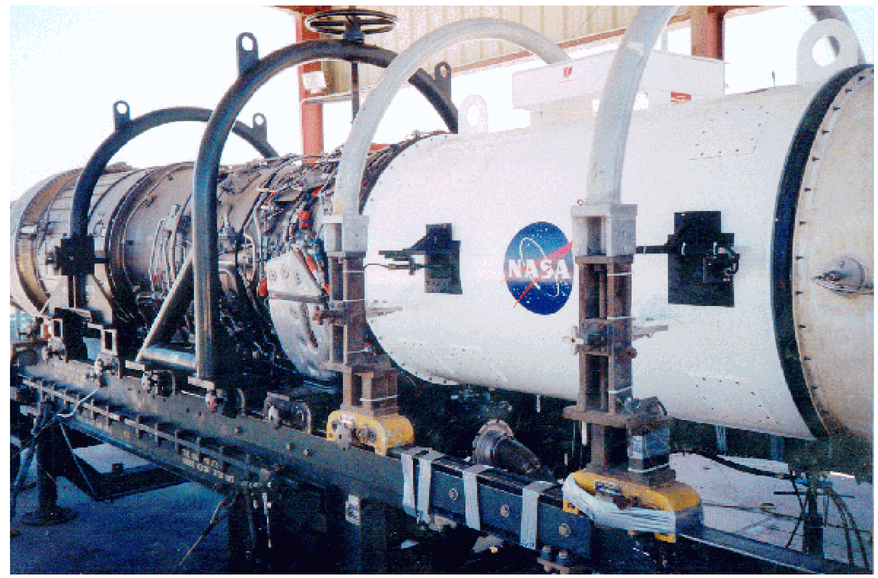
NASA Dryden Full-Scale Engine Tests

VG01-102-18





V-2272

P&W Engine

ρ = 0.9 to 1.0 kg/m3

u = 0 to 170 m/s

$\dot{m}$ = 0 to 100 kg/s

Measurement Standards

Inlet pitot-static probes (ρ, u, $\dot{m}$)

DEEC ($\dot{m}$)

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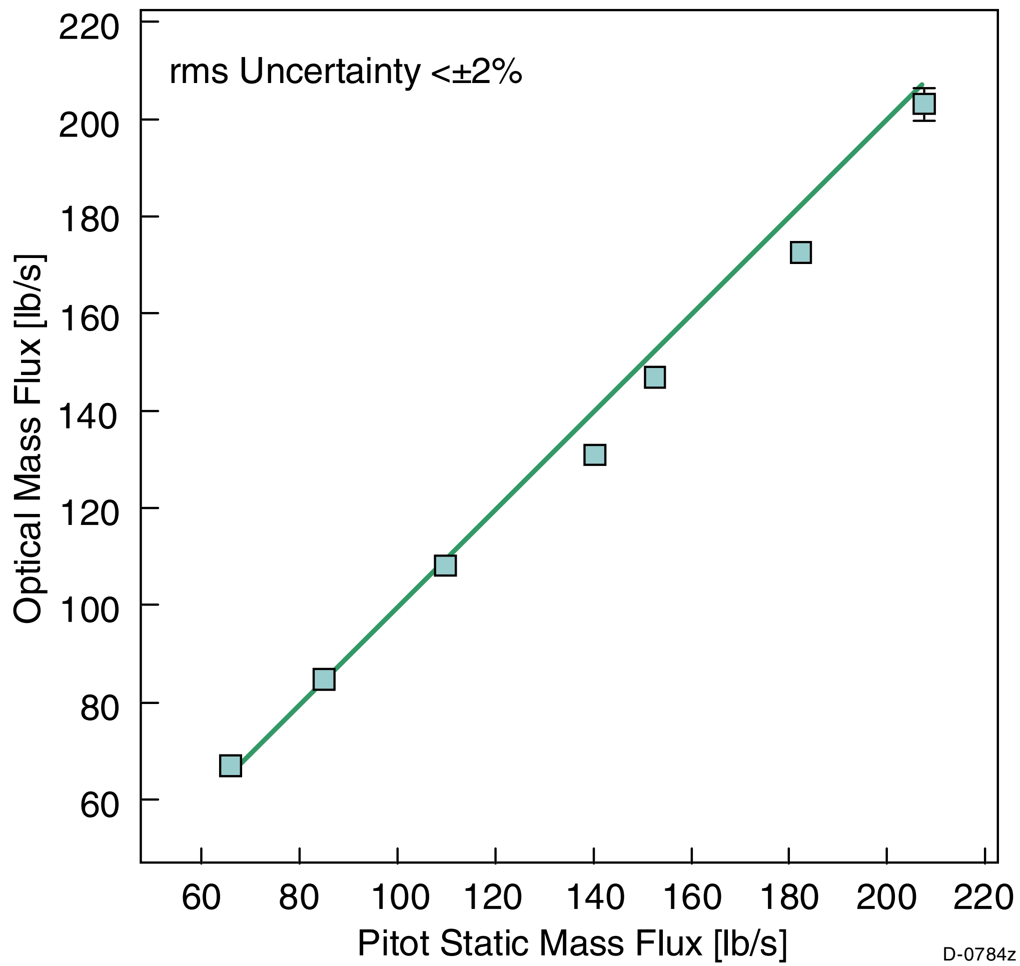
Mass Flux Measurements
NASA Dryden Full-Scale Engine Tests

VG01-102-19



- **TDL sensor accuracy equivalent to or better than test facility standard**

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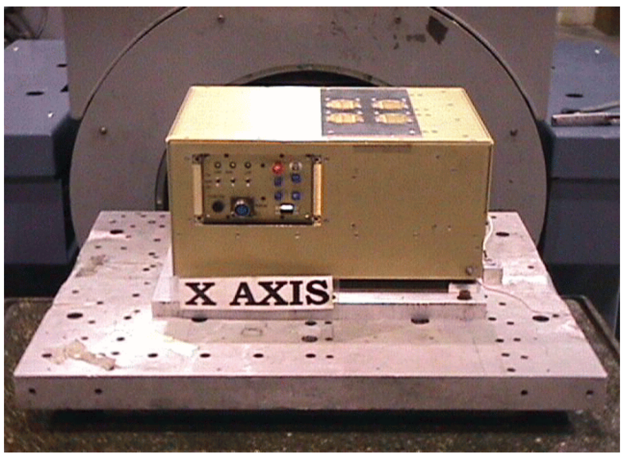
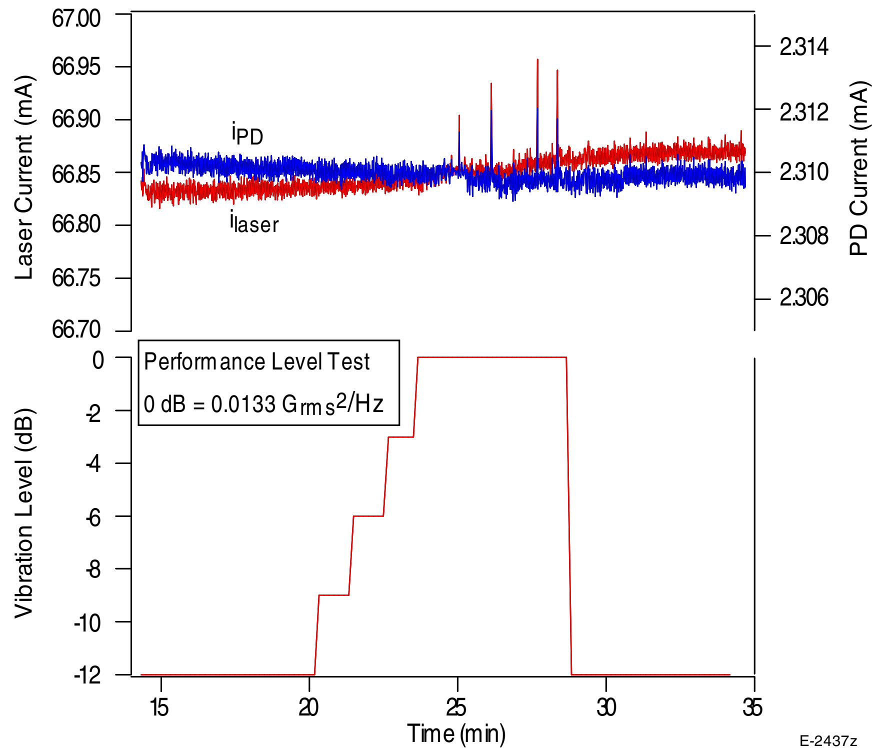
Aeroengine Flight Mass Flux Sensor

VG01-102-20

- **Ground testing on F-100 engine showed ± 2% uncertainty from idle to mil-spec power**



Flight sensor module on vibration test stand



Example "shake & bake" test result

- **Sensor package (including optical interfaces) passed environmental tests and awaiting early 2001 flight**
  - vibration requirements for F-18 operability exceed Pegasus launch requirements

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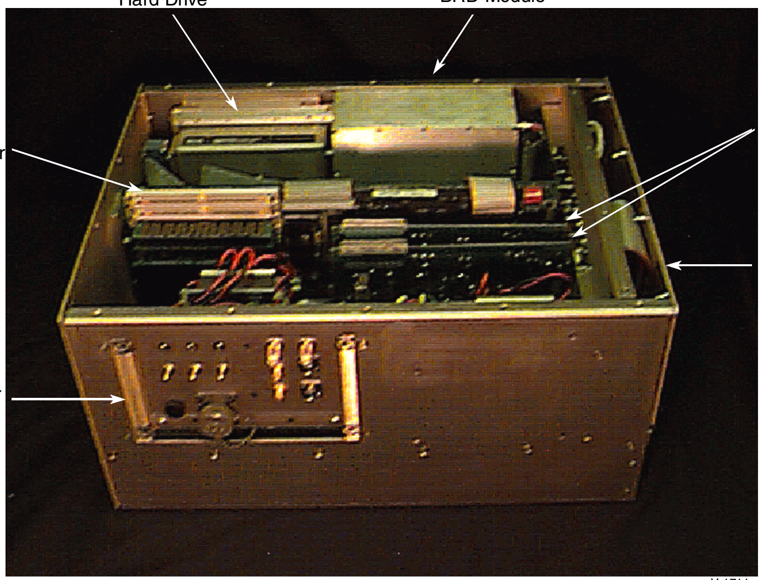
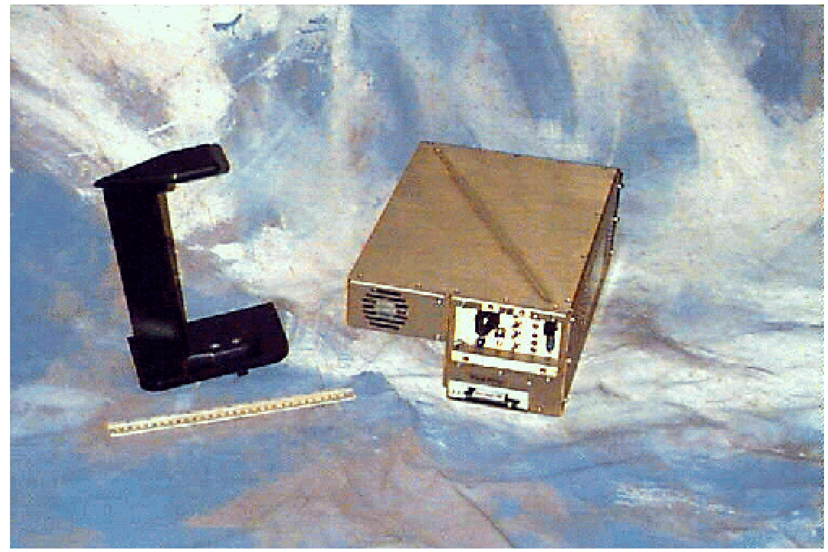
PSI Airborne Diode Laser Sensors

VG01-102-21





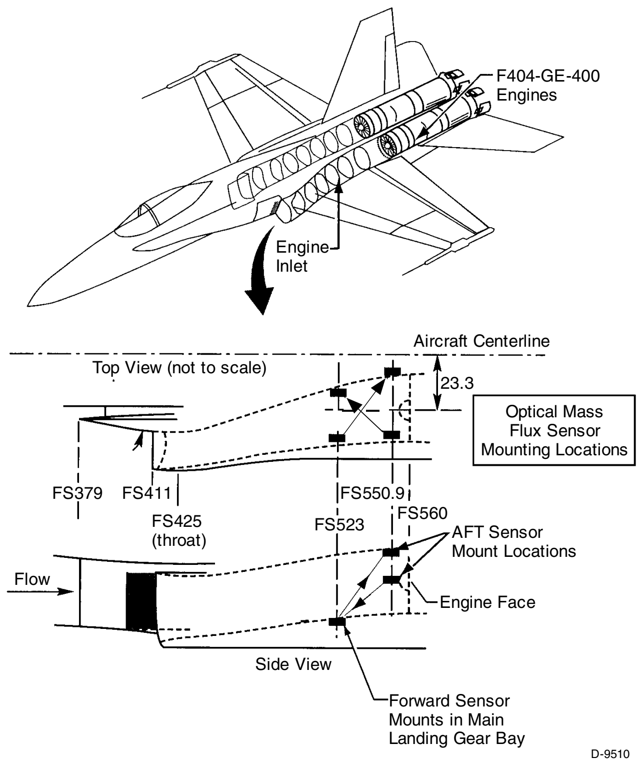
(a) PSI/NASA O_2 Mass Flux Sensor

(b) PSI/DOE UAV Hygrometer

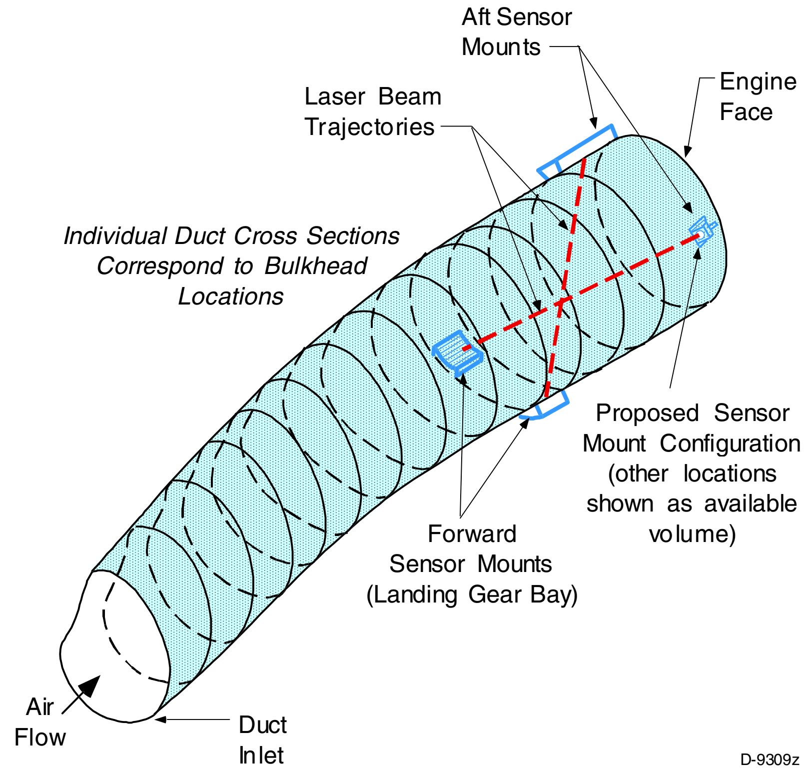
Flight Mass Flux Sensor Engineering

VG01-102-22

- **Supported by NASA Dryden Flight Research Center for Engine Control Applications**



Schematic of F-18 installation



3-D view of optical interface layout

- **Optical interface hardware installed and flying for ~ 8 months**

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Compact, Airborne Laser Multigas Sensor

VG01-102-23

Program Goals

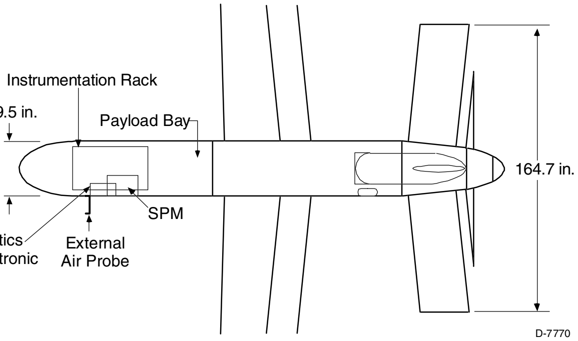
- **Develop diode laser sensor for in-situ measurement of trace gas species from aircraft for atmospheric research on global climate change**
- **Develop capability for multiple species measurement using several lasers and fiber-optic network**
- **Automate and size sensor for deployment on new generation of research aircraft: Unmanned Aerial Vehicles (UAVs)**

Altus UAV

V-1316

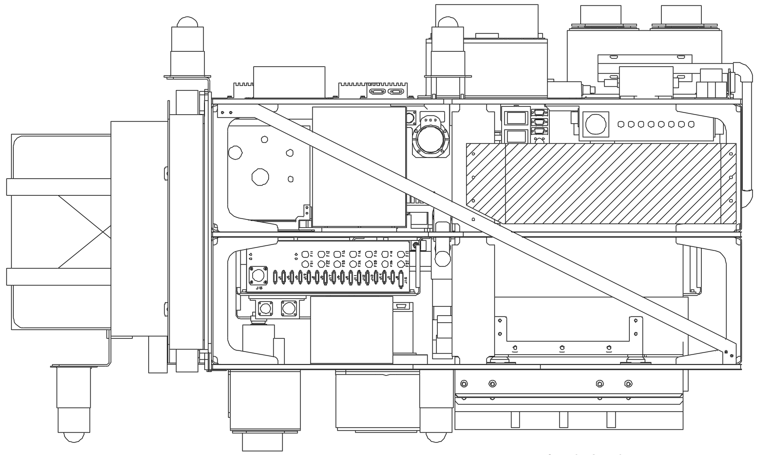
- **External air probe provides true in-situ sampling**
- **Probe remotely mounted from processor module**

Sensor Integration



D-7770

- **UAV payload parameters**
  - volume: 16 x 6 x 6 in.
  - weight: 10 kg with probe
  - power: 120 W
- **Expandable to multiple lasers**



Scale 0.250

E-1100

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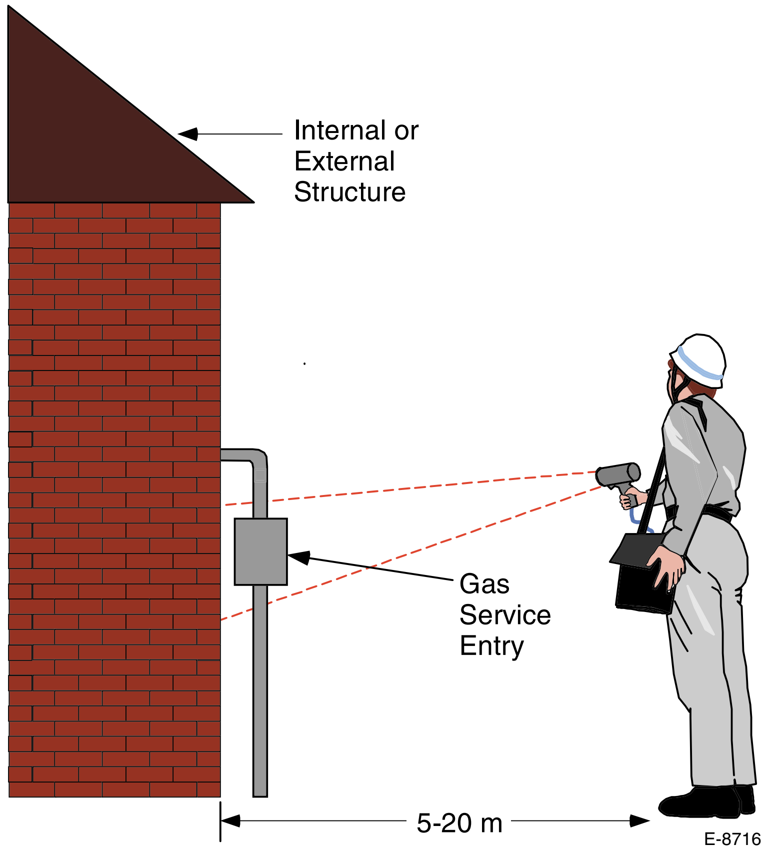
Hand-Held TDL Gas Plume Sensor

VG01-102-24

- **Collect topographic backscatter to check for gas absorption in illuminated region**



- **Presently under development for hazardous gas leak detection (HF, H_2S, CH_4) in petro-chemical processing facilities**
  - 10 ppm-m sensitivity

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Recent Advances in Room-Temperature Mid-IR Lasers

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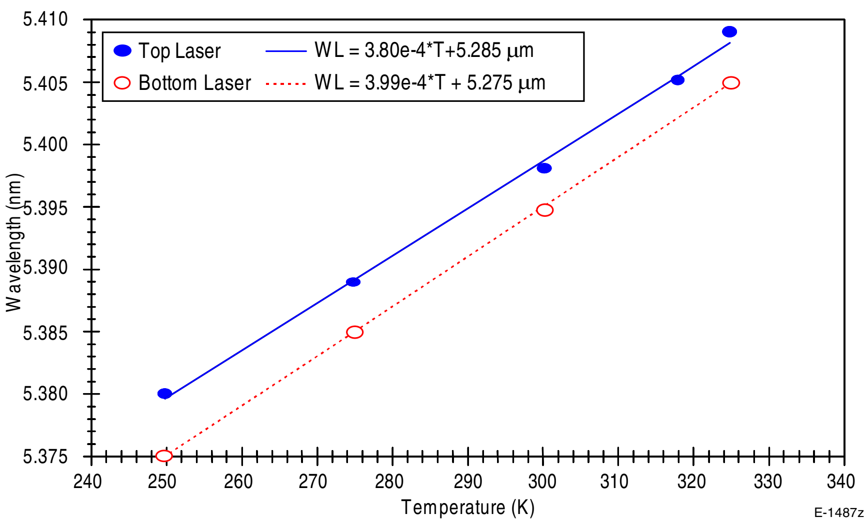
- **MQW devices on GaSb substrates**
  - MIT Lincoln Lab, Univ. of Houston, Sarnoff, Univ. Montpellier
  - multi-longitudinal mode, Fabry-Perot cavity
  - quasi-CW peak power ~ 10 to 100 mW at room temperature

- **Type II intersubband cascade on GaSb**
  - Univ. of Houston, AOI, Northwestern, NRL
  - multi-longitudinal mode, Fabry-Perot cavity
  - quasi-CW peak power ~ 100 mW, but only $T \leq 250K$

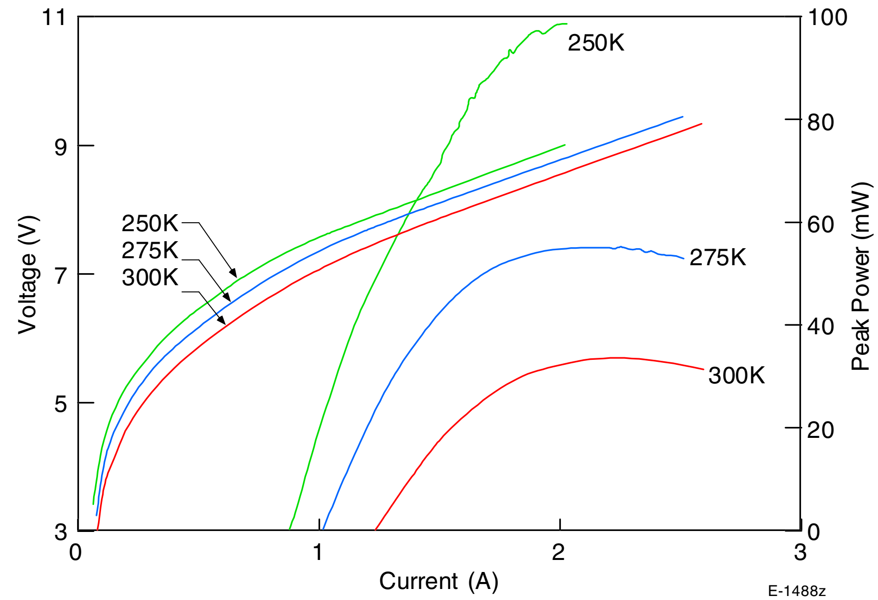
- **Type I intrasubband quantum cascade on InP**
  - Lucent
  - Fabry-Perot and DFB, single-mode
  - quasi-CW peak power ~ 10's mW at $T \lesssim 350$ K

Example Tuning, L-I, and V-I Characteristics of 5.4 μm DFB QC Device

VG01-102-26



Temperature Tuning



L-I, V-I Curves

- **Temperature measured at cryostat mount**
- **Data obtained at Lucent using liquid-N_2 mount**

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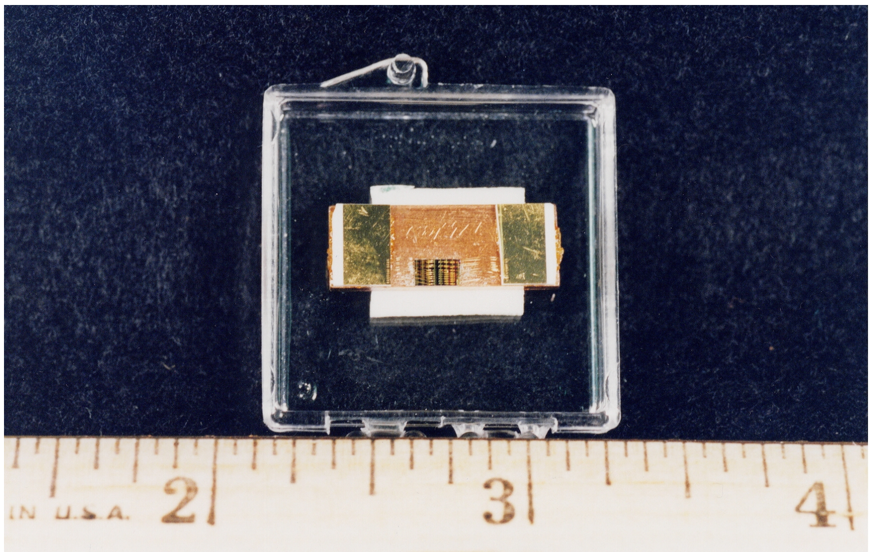
Lucent QC Laser Package

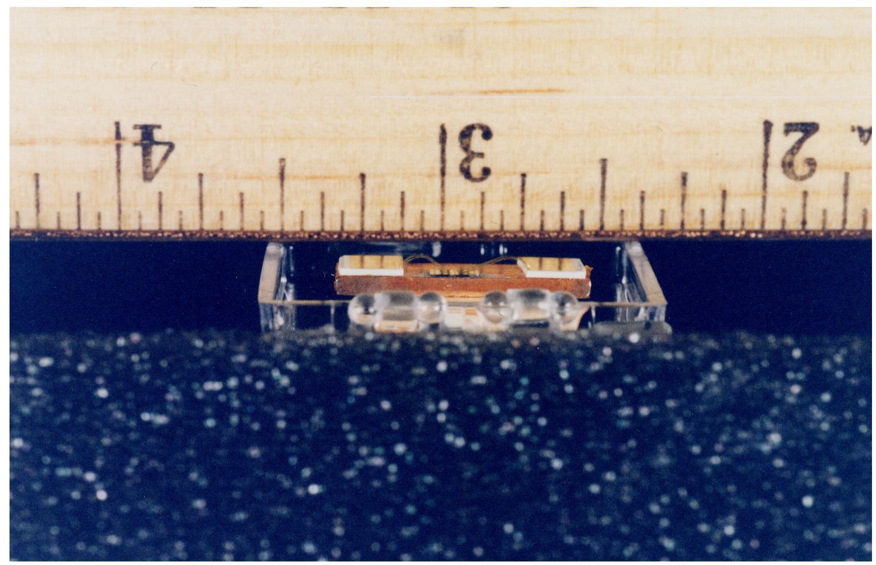
VG01-102-27

- **Each chip contains six lasers, two of which are wired**



V-2496

Top View



V-2497

Front View

Motivation

- **Increasing regulatory pressure for pollutant and particulate omissions control on land and airborne gas turbine systems**
  - CO, NO levels < 5 ppm
  - particulate levels ~ 10^{-4} g/m^3
- **In-situ monitoring of ~ 1 ppm levels of CO, NO difficult to accomplish with near-IR absorption**
- **MWIR emission/absorption offers possibilities for in-situ surveys of gaseous, particulate emissions**
  - FTIR: major species concentrations, temperature, some trace species
  - QCL: high sensitivity measurement of CO, NO, SO_2

Example Detectivity Improvements Using Mid-IR Sensor

VG01-102-29

- **Detection limits per meter absorption**

CO

	Demonstrated Limit Near-IR	Potential Limit Mid-IR	
		2.3 μm	4.7 μm
300 K	3 ppm	20 ppb	0.1 ppb
Flames	100 ppm	0.7 ppm	5 ppb

NO

	Demonstrated Limit Near-IR	Potential Limit Mid-IR	
		2.7 μm	5.2 μm*
300 K	30 ppm	600 ppb	30 ppb
Flames	140 ppm	3 ppm	200 ppb

*Sensitivity of 80 ppb demonstrated at 5.41 μm

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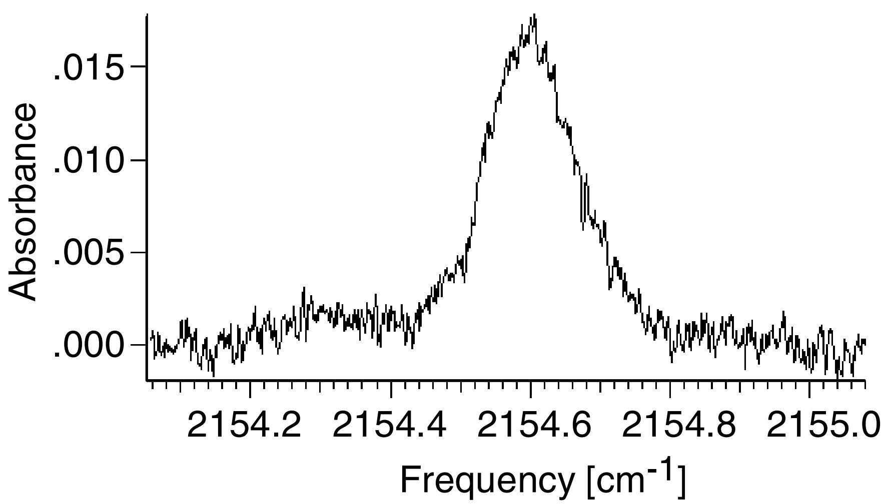
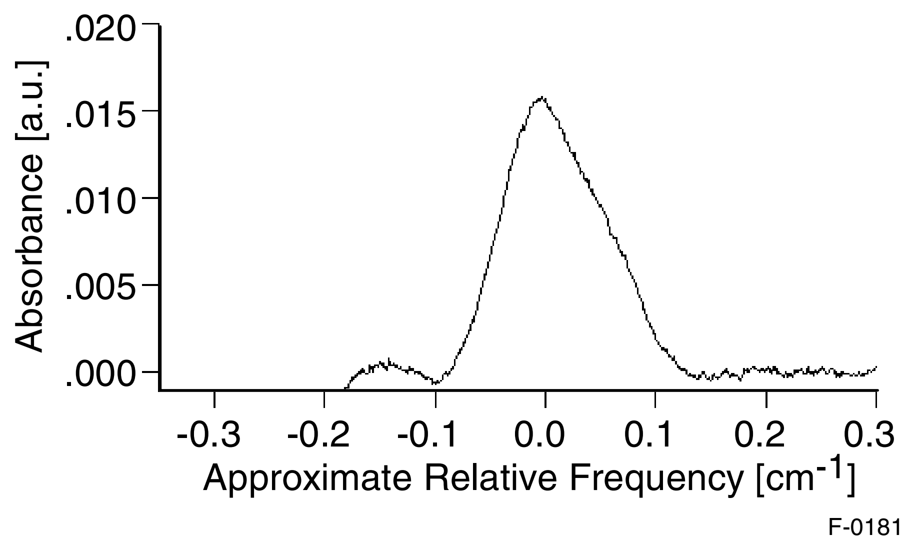
Ambient CO Measurements Using QCL

VG01-102-30

- **atm pressure, 21 m path, 300 K**
- **$R_{(2)}$ transition**



Gated Integrator



BRD

- **Measured level ⇒ 200 ppb**
- **BRD-based detection limit ~ 5 ppb**

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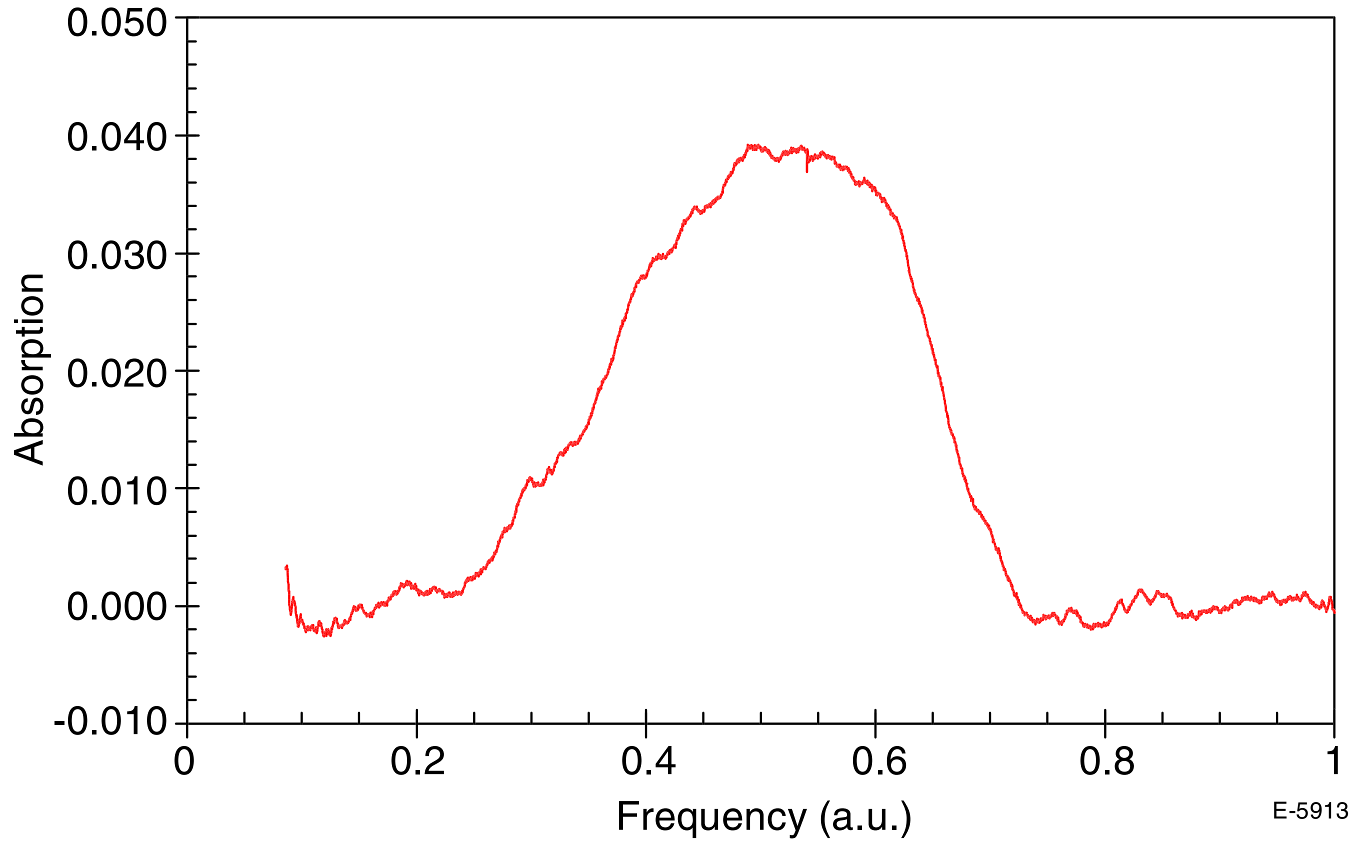
Example NO Detection with QCL

VG01-102-31

- **32 mTorr NO in 50 cm cell**



- **Unresolved doublet**
- **550 ppb-m detection limit**

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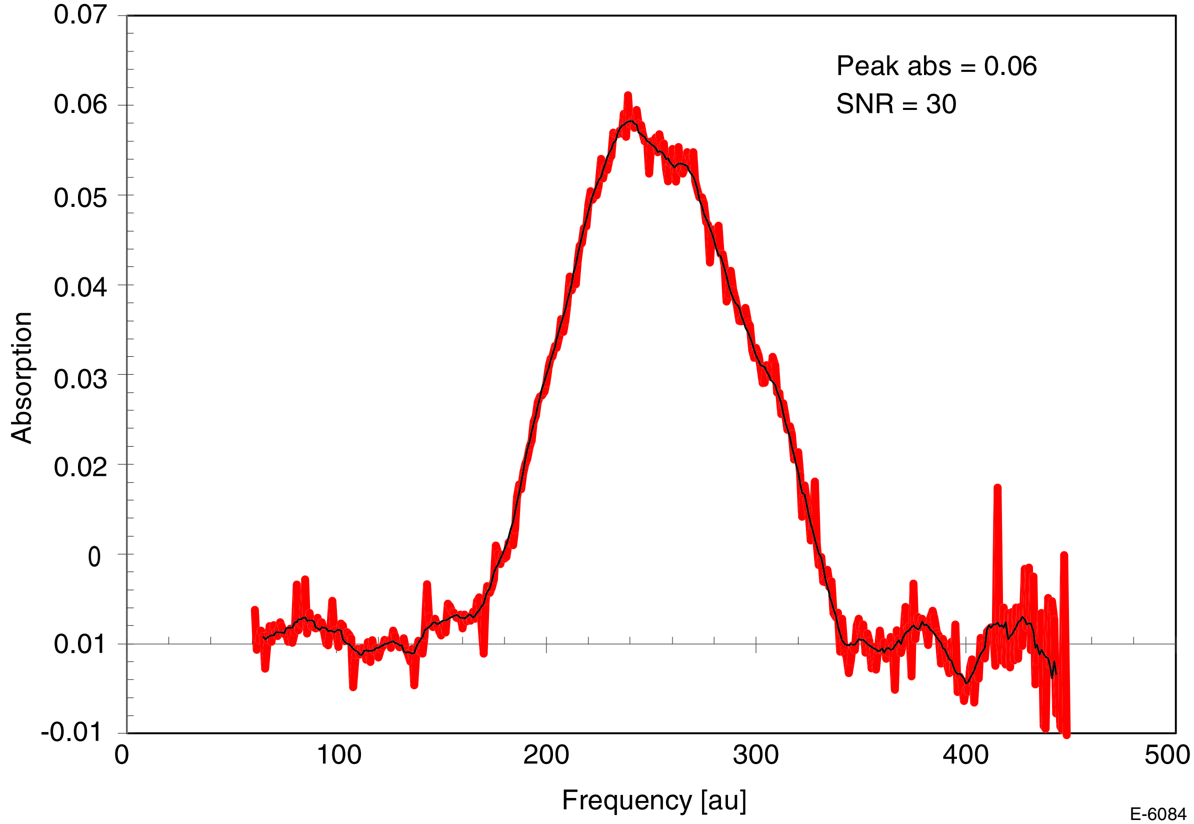
Example SO_2 Detection with QCL

VG01-102-32

- **0.5 Torr SO_2 in 50 cm cell**



- **10 ppm-m detection limit → extend to 100 ppb-m using sensitive detection techniques**
- **H_2O vapor interferences will be important in combustion exhaust applications**

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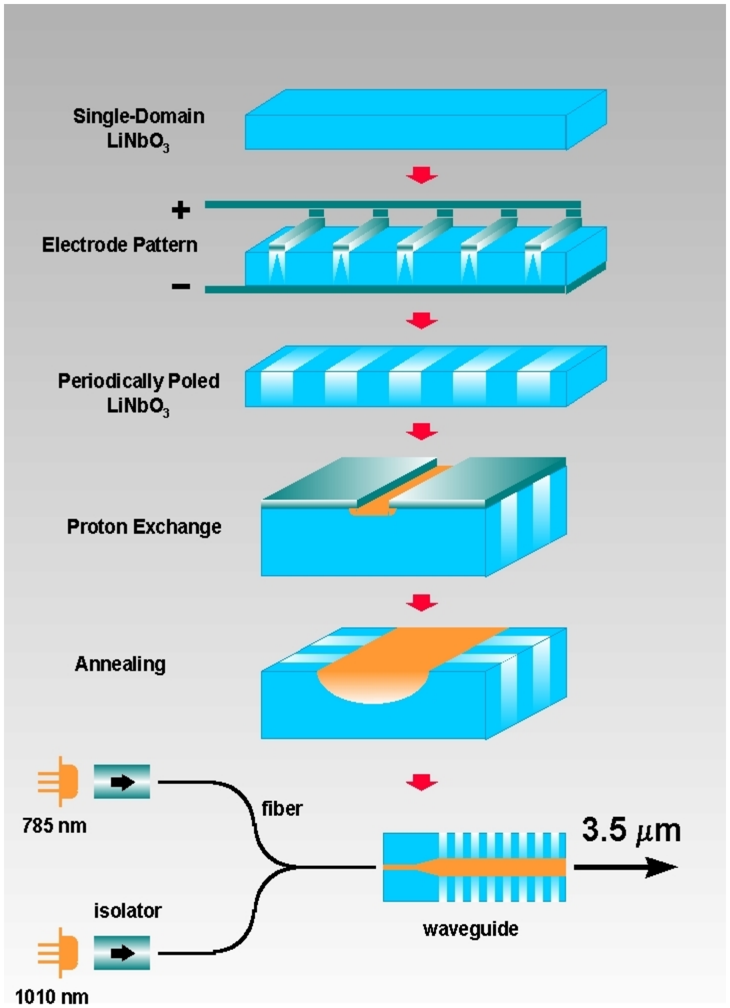
Other QC-Laser Based In-Situ Sensors Under Development

VG01-102-33

- **SO_2 and SO_3 measurements from combustion sources**
  - 7 to 9 µm region
  - project 10 ppm-m detection limits at 600 K
- **H_2CO and CO measurements in ambient troposphere**
  - 5.6 and 4.6 µm
  - project ~10 ppb sensitivity with 100 m Herriot Cell
- **NO and CO measurements in combustion gases**

Frequency-Converted Diode Laser Sources in the MID-IR

VG01-102-34



- **Built around PPLN chip containing APE waveguides**
- **Two near-infrared diode lasers for input**
- **Difference-frequency generation (DFG) to produce tunable mid-IR output radiation**
- **Tapered structures used to excite single mode of highly-multi-moded waveguide**

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Advantages of Guided-Wave DFG Source

VG01-102-35

- **PPLN has high nonlinear coefficient**
- **Near-IR input lasers lead to room temperature operation, low cost, portability**
- **DFG process leads to broad wavelength coverage using tunability of near-IR lasers, engineering of PPLN**
- **Waveguides increase the conversion efficiency**

  $P_1 = \eta_{dev} P_2 P_3$

  if η_{dev} = 10%/W, two 100 mW lasers yield 1 mW

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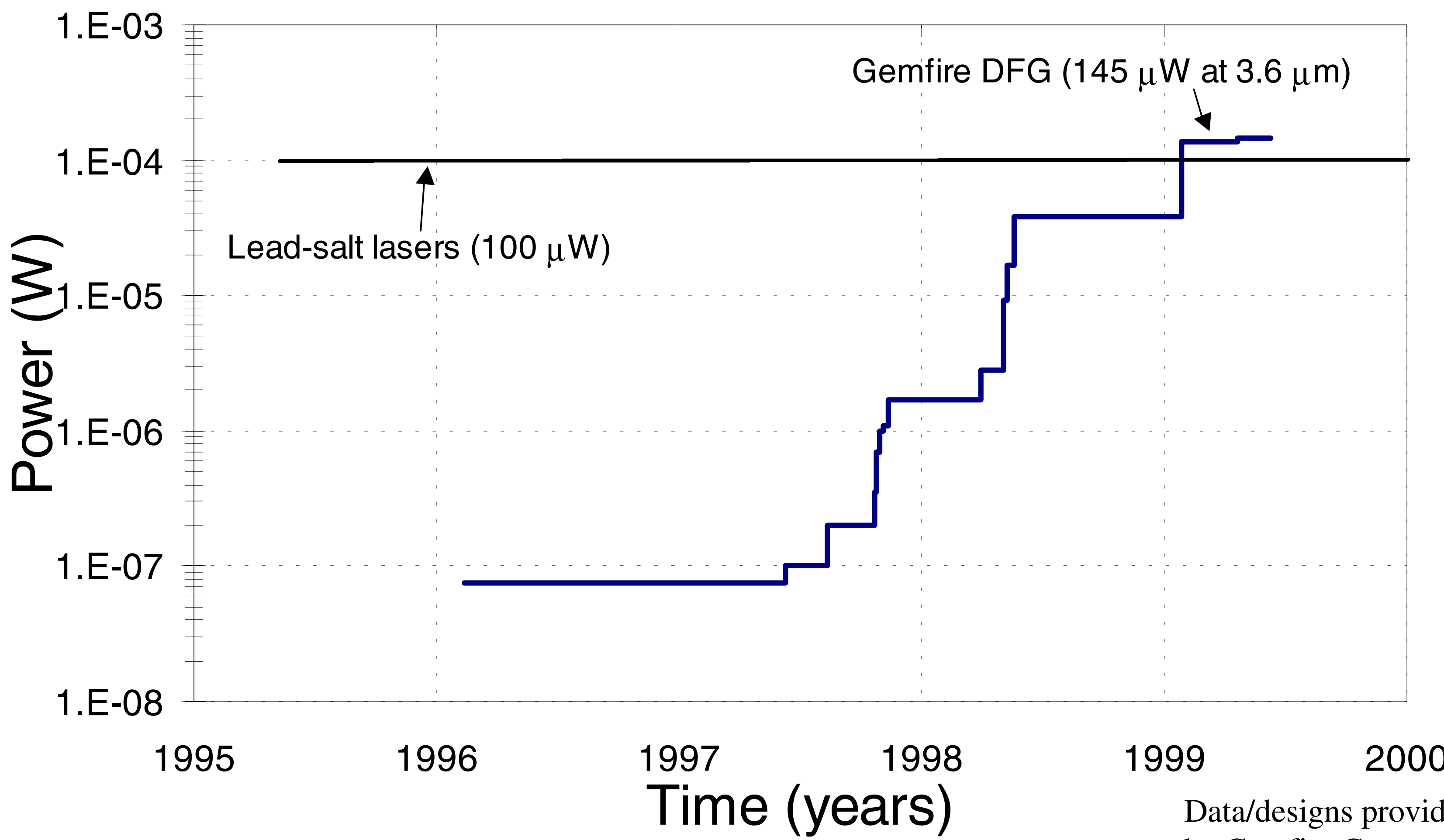
Waveguide DFG Power vs. Time

VG01-102-36



Data/designs provided
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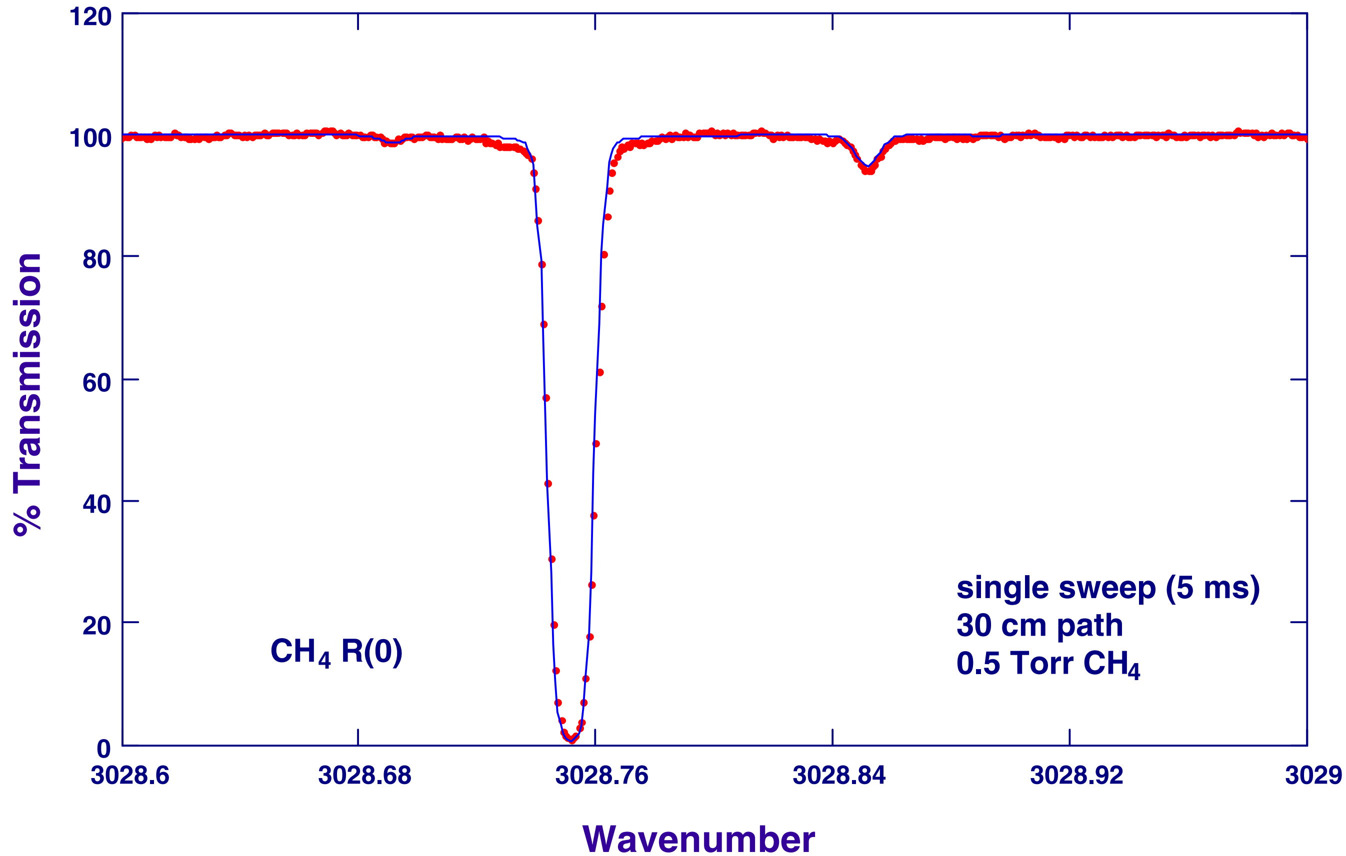
Methane Absorption Spectrum

VG01-102-37



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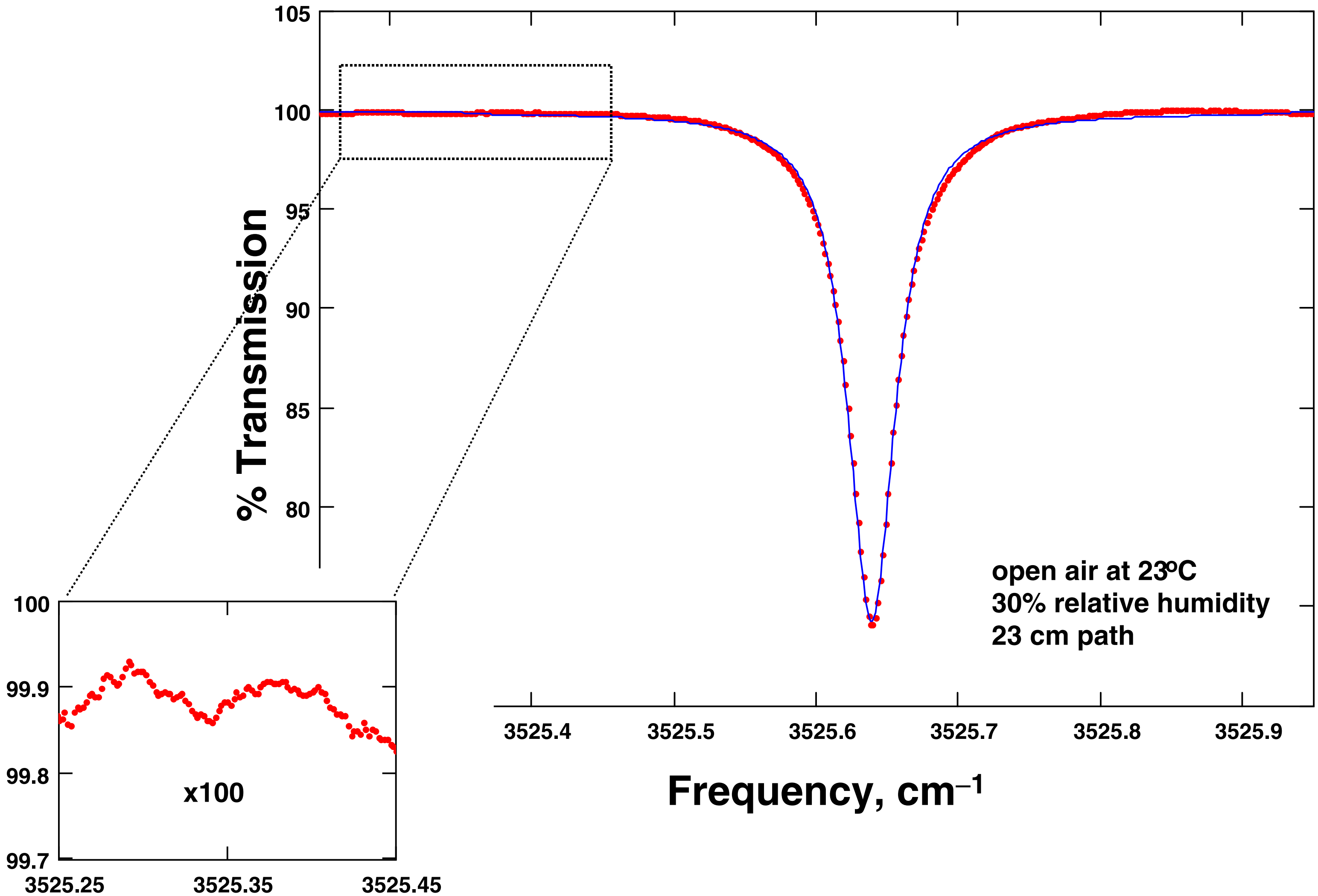
Spectrum of Water Vapor in Ambient Air

VG01-102-38

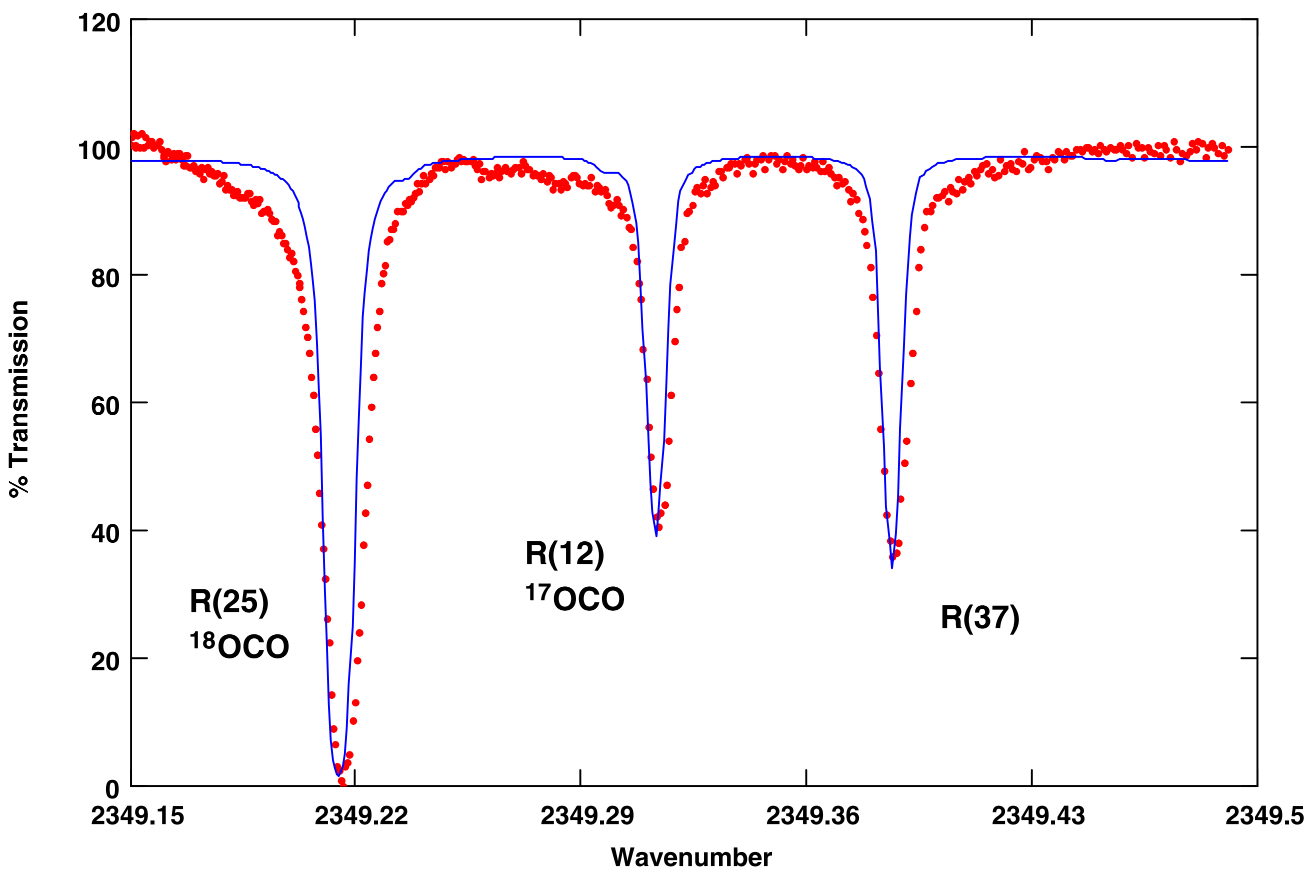


Data/designs provided by
Gemfire Corporation

PHYSICAL SCIENCES INC.

CO_2 ISOTOPES AT 4.3 μm

VG01-102-39

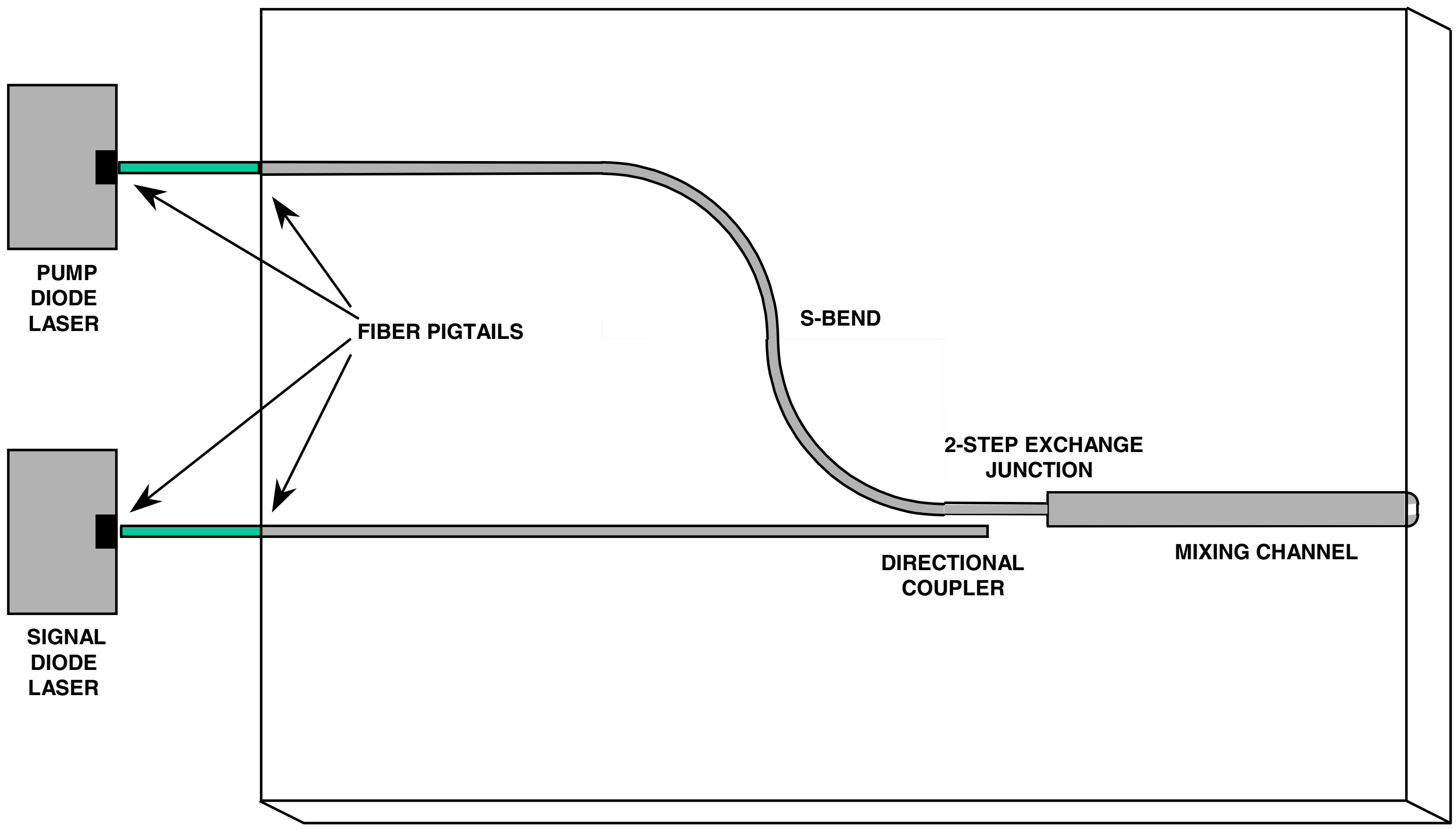


Data/designs provided by
Gemfire Corporation

PSI
PHYSICAL SCIENCES INC.

The Next Level of Integration

VG01-102-40



Data/designs provided by
Gemfire Corporation

PSI
PHYSICAL SCIENCES INC.

Summary

VG01-102-41

- **PSI has nearly a decade of experience in diode-laser-based gas sensors**
  - multi-million $ commercial spin-off company
  - over 70 custom units delivered to research customers in the U.S., Europe, and Asia
  - partnerships established with major industrial companies for eventual high volume applications
- **Present research activities moving to advanced current-pumped mid-IR sources for DIAL and *in-situ* sensor applications**
  - partnerships established with Lucent and AOI/University of Houston
  - licensed technology from Gemfire and growing capabilities in engineered non-linear optical materials for frequency-converted sources