Compact Single CW-QCL Instrument

Outline of Presentation

Spectroscopy and Principles of Operation

Optics

Data Acquisition

Software – TDLWintel

Thermal Control

Measurement Principle - Absorption Spectroscopy

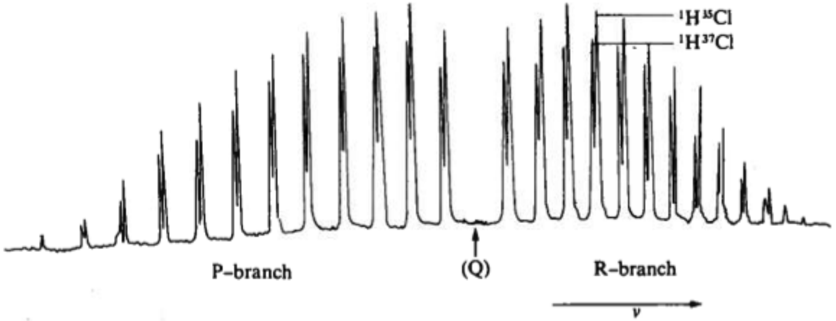


Fig. 17.14. A high-resolution vibration–rotation spectrum of HCl.

Why do we observe a spectrum?

Quantum mechanics explains:

- **-Each molecule absorbs light at specific frequencies**
- **-Absorption adds energy to vibration and rotation levels**
- **-Additional energy must be specific quantity**
- **-Every molecule has unique spectrum**

$h\nu = \Delta E = \hbar\omega$

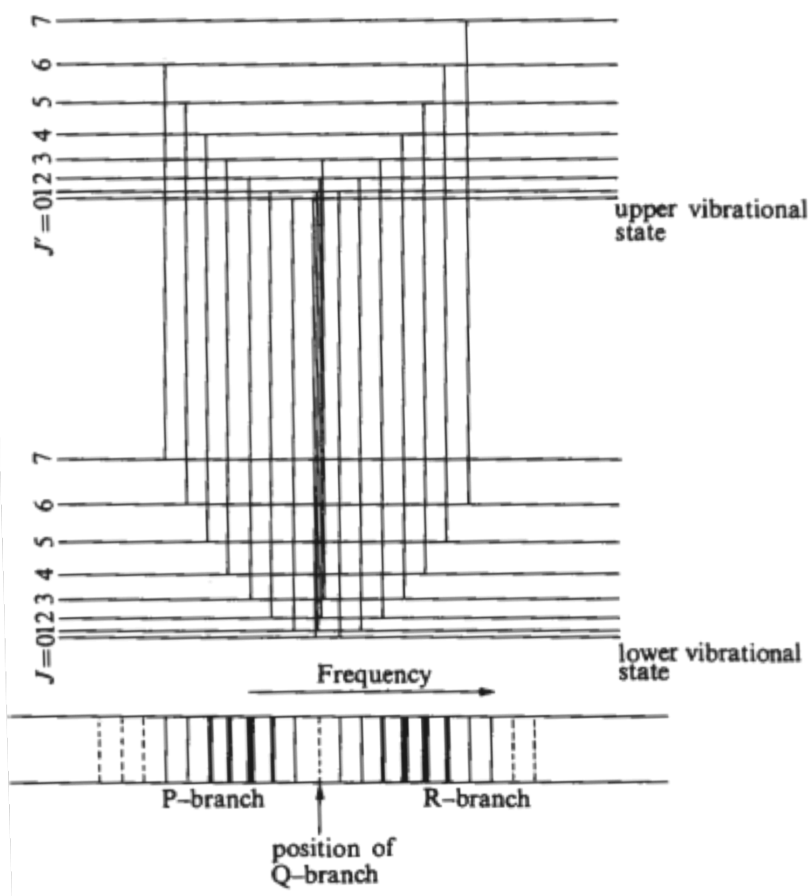


Fig. 17.15. The formation of P, Q, and R branches.

J is rotational quantum number.

Measurement Principle – Beer’s Law

How much light is absorbed by the sample?

$$A = N \times \sigma \times L$$

A is fractional absorption – we measure this

N is number density – we want to know this

σ is the absorption cross section – property of molecule – we want it big

L is the optical path length – we want it big

$$N = A / (\sigma \times L)$$

Measurement of N is absolute – no need for calibration!

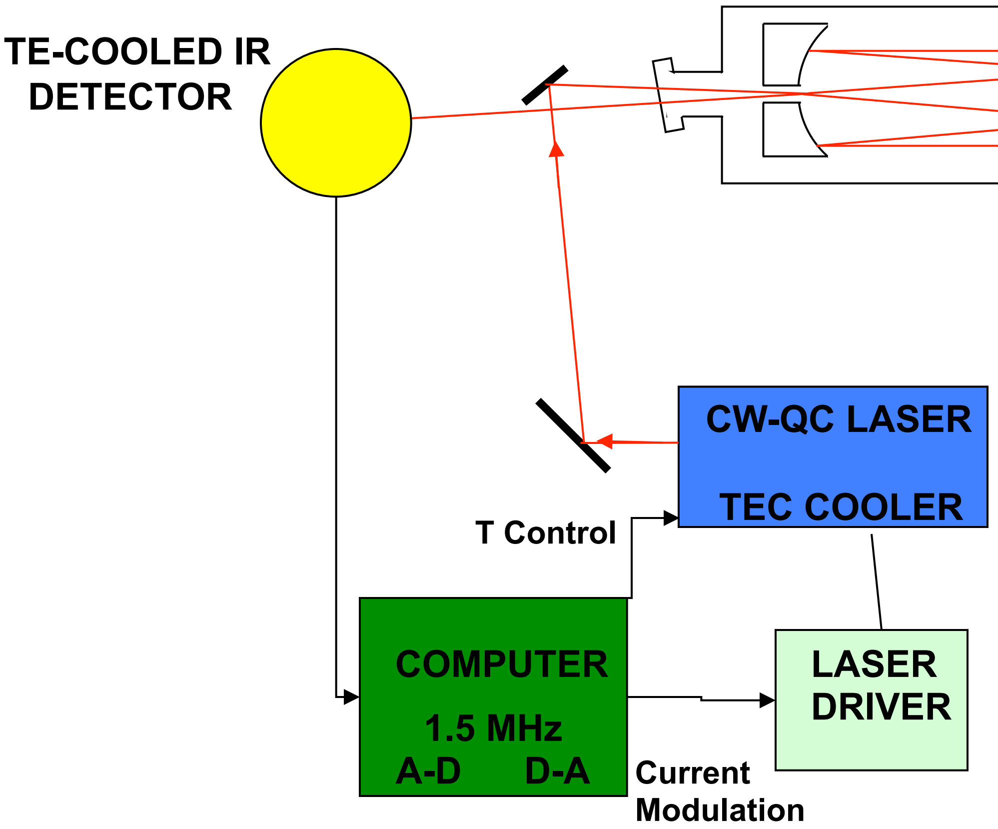
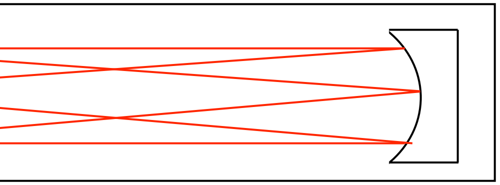
Trace Gas Measurements with Mid-Infrared Quantum Cascade Lasers

Why Laser Spectroscopic Instrumentation?
- Specific and verifiable detection.
- High sensitivity.
- Fast time response.
- Rugged instruments – field ready.

Why Mid-IR?
- Fingerprint spectra for many molecules.
- Strongest absorption, Highest sensitivity.

Why Quantum Cascade Lasers?
- Spectroscopic quality IR lasers at near room temperature.
- High power, continuous operation for high sensitivity.

CW-RT QCL Spectrometer

ASTIGMATIC MULTIPASS CELL
76 m (238 passes), 0.5 liters

TE-COOLED IR DETECTOR

CW-QC LASER

TEC COOLER

T Control

COMPUTER
1.5 MHz
A-D D-A

Current Modulation

LASER DRIVER

TDLWINTEL SOFTWARE

Display Mode Field 4

A/D 0 Poly 2 Data Port iter= 18

115.9, 294.0

489.9

[520.8]

-30.9

1 23:21:44 230

382 14.6 Torr 300.0 K
0.998 s 3.8 Hz 384%
606 kHz 2.6 kHz
19799

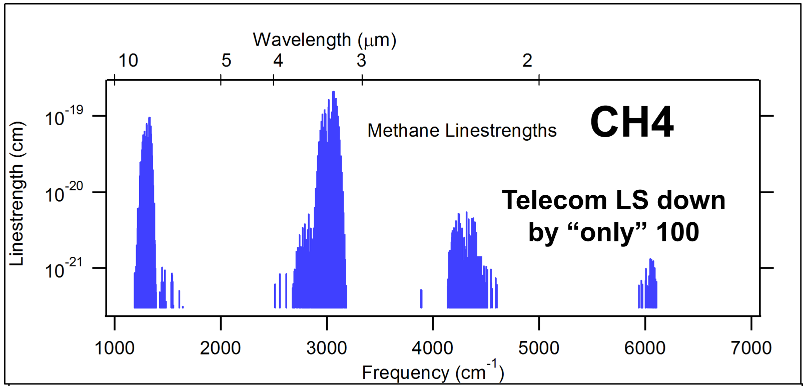
382

NO "DOUBLET"
1900 c m $^{-1}$

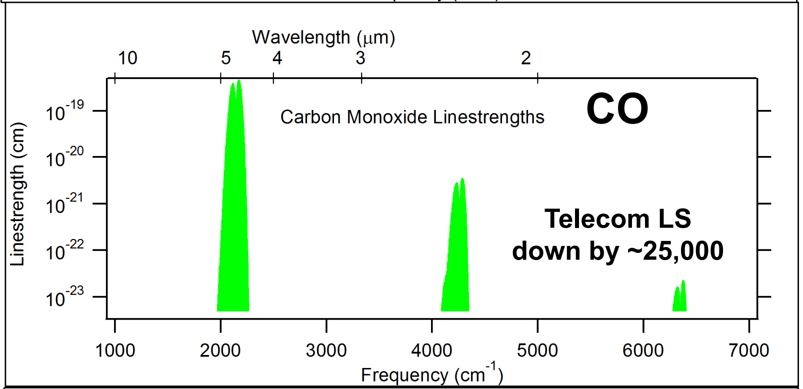
LASER OFF

Advantages of Mid-IR Detection

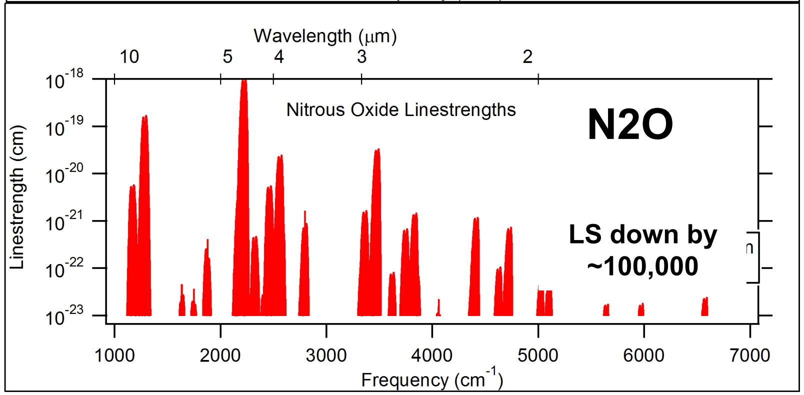
- Linestrengths in mid-IR are **much** stronger than near-IR
- For methane, the loss of 100x in LS is mitigated by superior lasers, optics, detectors and methane’s large ambient concentration (2 ppm)
- For CO and N_2O, telecom detection will be very hard due to lower atmospheric abundance and extremely small linestrengths



Telecom LS down by “only” 100



Telecom LS down by ~25,000



LS down by ~100,000

Wide Variety of Species

- Infrared spectroscopy is a general technique – with the right laser, we can detect any small molecule with a vibrational dipole moment
- For example:
  - CO, CO_2, H_2CO, $HCOOH$, CH_4, C_2H_2, C_2H_4, C_2H_6, acrolein, butadiene
  - H_2O, $HOOH$, HO_2, O_3, O_2
  - NO, NO_2, N_2O, NH_3, HNO_3, $HONO$, N_2H_4, HCN
  - HF, HCl, $HOCl$, HBr, HI
  - COS, SO_2, SO_3, H_2S, H_2SO_4
  - Isotopes of above species as well

Optical System

Laser in the system

Optical paths and layout

What is on the optical table

Touch-up alignment

Alignment aids

The 76 meter multi-pass cell

Compact Single Quantum Cascade Laser Spectrometer Optical System

One IR Laser in the system:

Mid-infrared quantum-cascade laser
with distributed feedback gratings (DFB)
Continuous wave operation
Thermoelectrically cooled
Power ~10 mW
Completely eye-safe

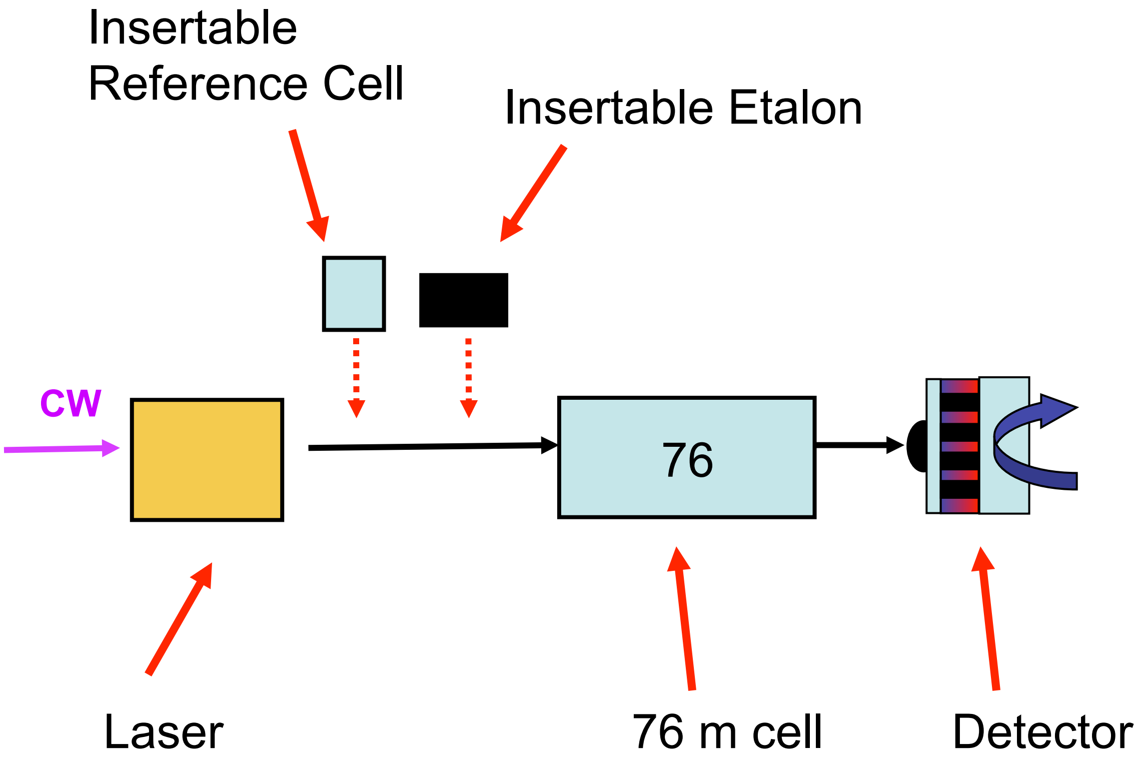
For N2O: 4.5 μm, ~2240 cm^{-1}

One visible trace laser for alignment

~1 mW, 0.67 μm
Eye-safe

* Wavelength (λ) in microns, μm ; Frequency (v) in wavenumbers, cm^{-1}
$v = 10{,}000/ \lambda(\mu m)$

“Cartoon” of the instrument: the most basic description



Insertable
Reference Cell

Insertable Etalon

CW

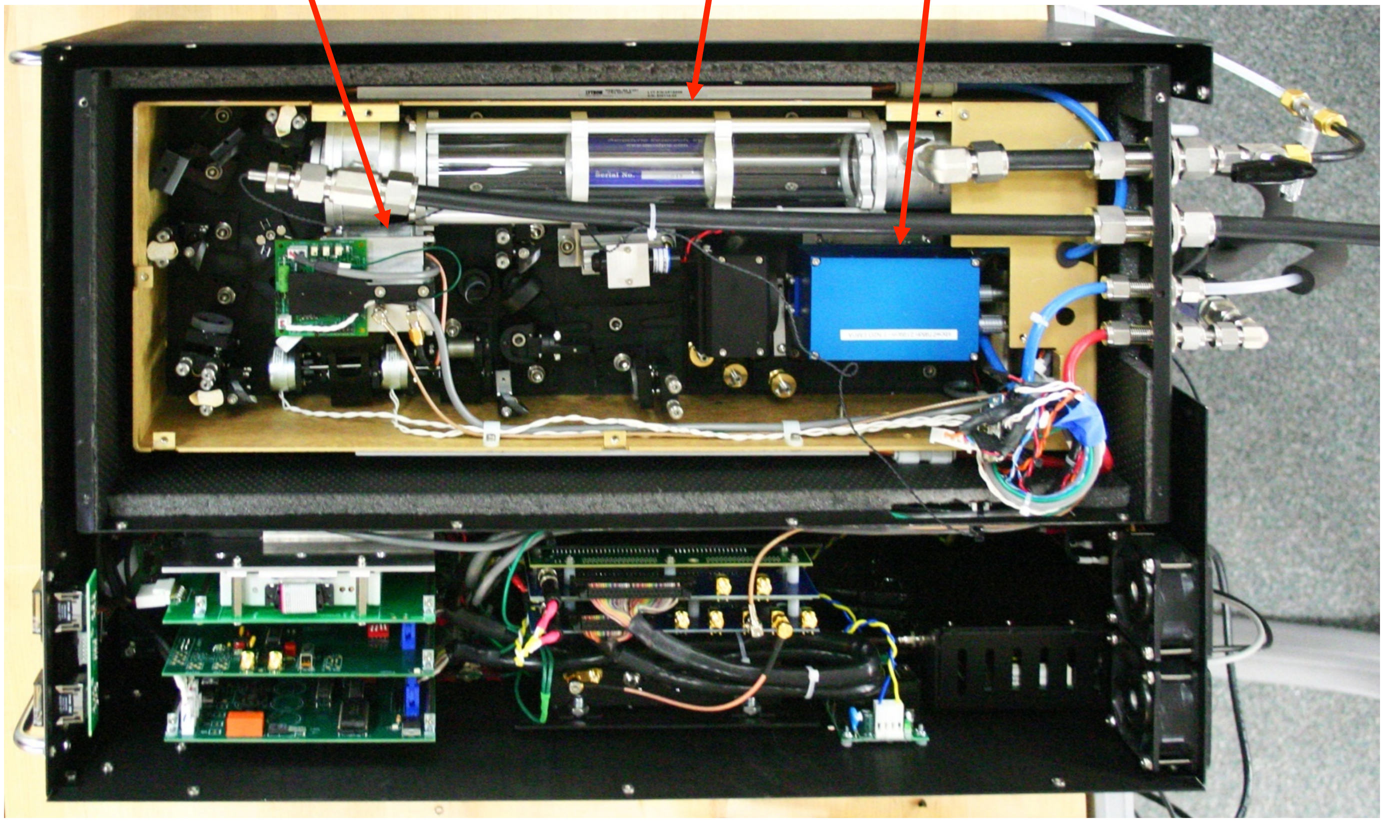
76

Laser

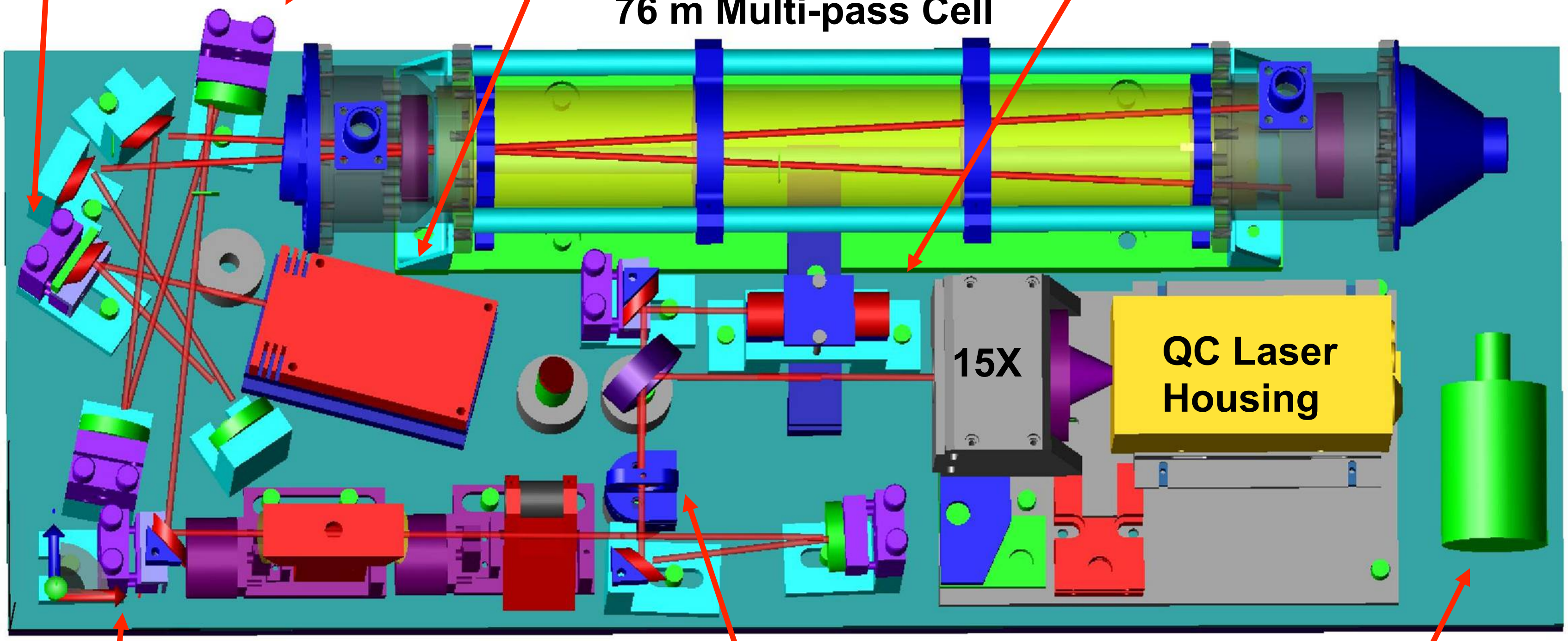
76 m cell

Detector

Instrument top view, outer and inner covers removed



Optical Module Diagram



Mirror to aim at detector

Mirror to aim at cell hole

IR Detector

Red Trace Laser

76 m Multi-pass Cell

Flip-in Ref. Cell

Flip-in Etalon

15X, X-Y-Z Adjust

Pressure Sensor

Mirror to aim cell pattern size

Pinhole

Optical Table: From basic concepts to the hardware

Instrument Optical Outline:

One QC laser is sent through 76 meters of optical path, to give enough absorption for a high sensitivity measurement.

The light passing through the main sample cell is detected with a thermoelectrically-cooled infrared detector, and is seen in “Field 1” on the monitor.

A “flag” is flipped electrically to insert either the Reference Cell (for frequency locking) or the Etalon form occasional determination of the laser tuning rate.

Frequency Lock spectrum always appears in “Field 4”

Compact Quantum Cascade Laser Spectrometer Optical System

What else is on the optical table:

Flip-in elements:	Etalon
	Gas Reference Cell
Optical alignment aids:	Trace laser
	(Drop-in) Eyepiece
Gas handling devices:	Pressure sensor (100 Torr Baratron)
Thermal control:	No Heaters
	T Controlled by circulating liquid
	Insulation
	Water lines to lasers & detectors

Compact Quantum Cascade Laser Spectrometer Optical System

Alignment

Thermal changes or transport may cause the optical components to shift slightly, causing a reduction in optical power at the detector, and a reduction in sensitivity.

Small adjustments in a few components should be all that is needed to bring the power back to optimum.

Re-alignment should only be done when the instrument is warmed-up.

Try doing a “touch up” alignment – this is usually all that is needed.

For very serious misalignment a more elaborate procedure is used.

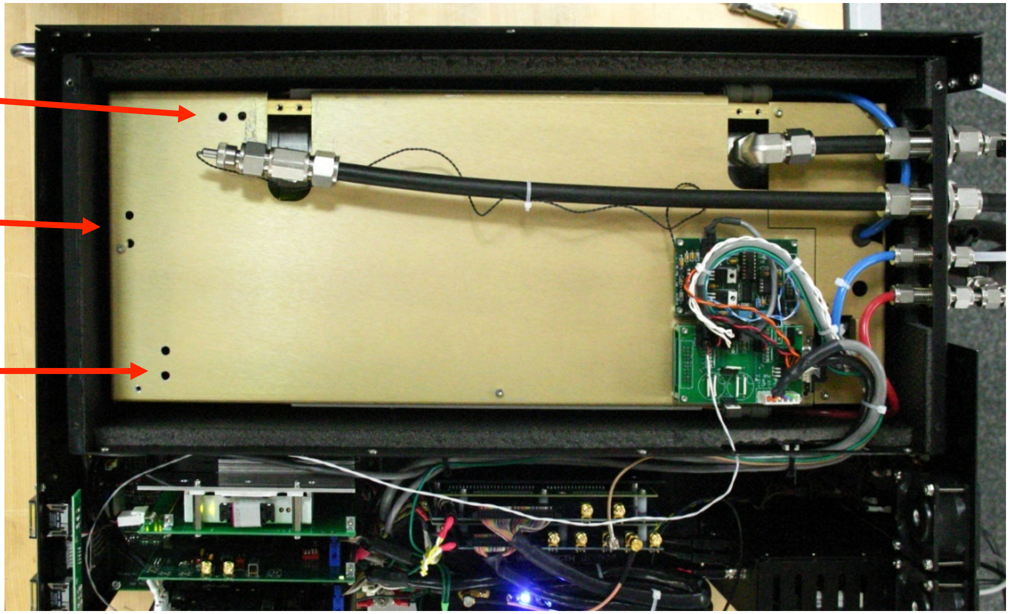
Optics Alignment

“Touch-up” Alignment: **Maximize power**
Minimize Fringes

Open outer cover.
Put instrument in “Quick” mode [control-Q].

Sequentially make small adjustments to 3 optical mounts, both horizontal and vertical, for aiming at detector, cell hole, and cell pattern size.

Use a 3/32” hex-driver inserted through the holes in the instrument inner cover.



Mirror to aim at cell hole

Mirror to aim at detector

Mirror to aim cell pattern size

Optics Alignment

"Serious" Alignment: **After transport, if light is very low**

Open outer and inner covers.
Put instrument in "Quick" mode [control-Q].

Adjust Laser Through Pinhole:
- **Flip up pinhole.**
- **Maximize signal with mirror aiming at detector.**
- **Maximize power sequentially, and in several cycles with 15X X-Y-Z adjustors.**
- **Re-Maximize signal with mirror aiming at detector.**
- **Flip down pinhole!**

Adjust 3 downstream mirrors as in the minor adjustment,
- **Cell-hole aim-in,**
- **Cell pattern size aim,**
- **Detector aim.**

Optical System Alignment: The Pinhole

The “pinhole” is the anchor of the optical system.

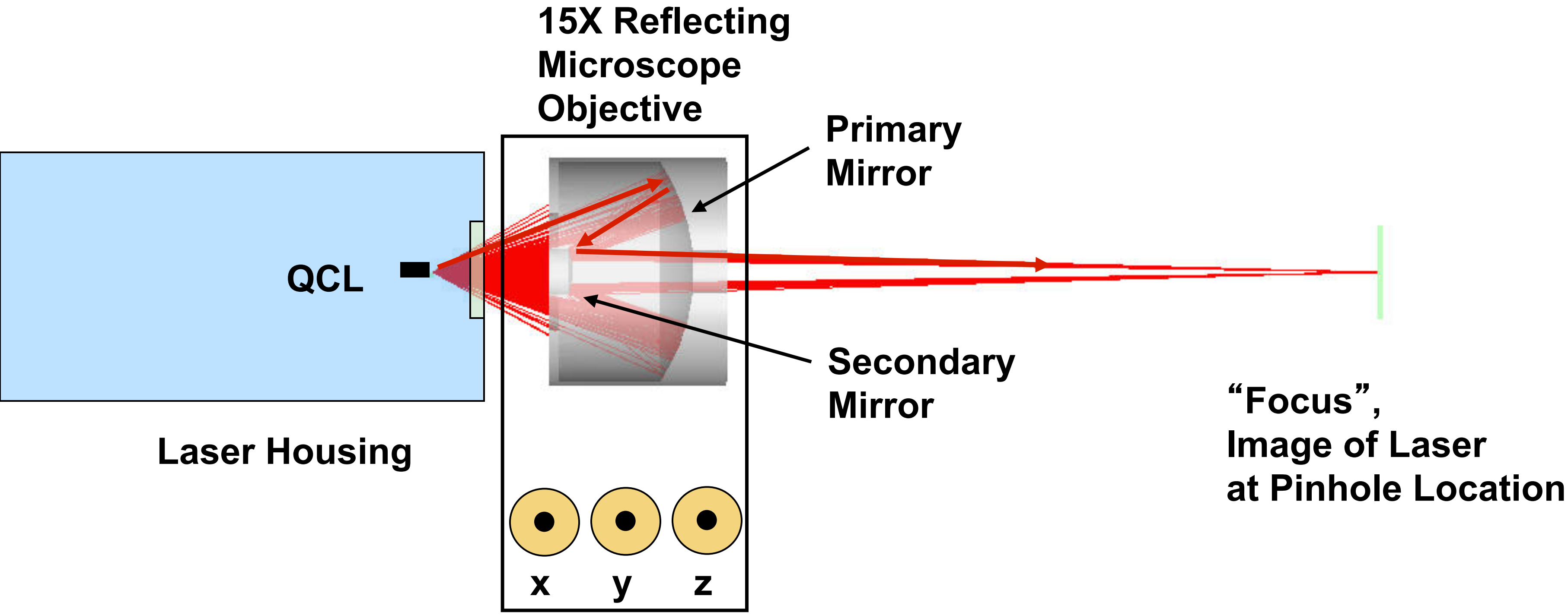
It is a 200 µm (0.2 mm) hole in a small steel sheet, mounted near the middle of the optical table, made so it can flip in or out of the beam.

It is only flipped into the beam to check alignment, never for measurements.

The QC laser beam is focused carefully through the pinhole.

The visible trace laser beam is also focused through the pinhole,
thus guaranteeing that the visible and IR will coincide at the detectors.

Front End Optics:
Collecting laser light and forming a narrow beam



15X Reflecting Microscope Objective

Primary Mirror

QCL

Secondary Mirror

Laser Housing

"Focus", Image of Laser at Pinhole Location

x y z

XYZ Adjuster moves the 15x Objective relative to the QCL, causing the focus to move at the pinhole location.
"x" = horizontal, "y"= vertical, "z"= focus.

Front End Optics: Alignment Adjustment

Make the instrument ready for alignment:

- Go from “Stream Data” to “Current Signal”.
- Change to 10 Hz, Control-Q
- Note the “Range” number on the screen

Flip up the pinhole

- Note the ratio of range with/without the pinhole.
- The ratio is typically ~1/2 when well aligned.

Make small adjustments to the XYZ controls on the objective,

- maximizing power (Range) with each control.
- Cycle through the XYZ controls several times to reach the maximum range.

Check that the trace laser beams go through the pinhole,

- and produce a smooth red dot on mirror M1.
- Adjust the trace laser tilt mounts if needed.

Return the instrument to measurement mode:

- Flip out the pinhole.
- 1 Hz, Control-O
- Field 1, Stream Data

Downstream Optics: Alignment Adjustment

Make the instrument ready for alignment:

Go from “Stream Data” to “Current Signal”(optional).
Change to 10 Hz, Control-Q
Note the “Range” number on the screen

Follow “No More Tears Procedure” :

Sequentially make *very* small adjustments to 3 optical mounts, both horizontal and vertical, for aiming at detector, cell hole, and cell pattern size.

Return the instrument to measurement mode:

1 Hz, Control-O
Stream Data

“No More Tears” Aim-in Optics For Multi-Pass Cells

We aim the high F# IR beam into the cell using a particular arrangement that allows independent aiming through the entrance hole and unto the back mirror, without either adjustment influencing the other.

A focusing mirror is placed before the cell entrance at a distance to produce images of the back mirror and input hole on the two upstream adjustment mirrors.

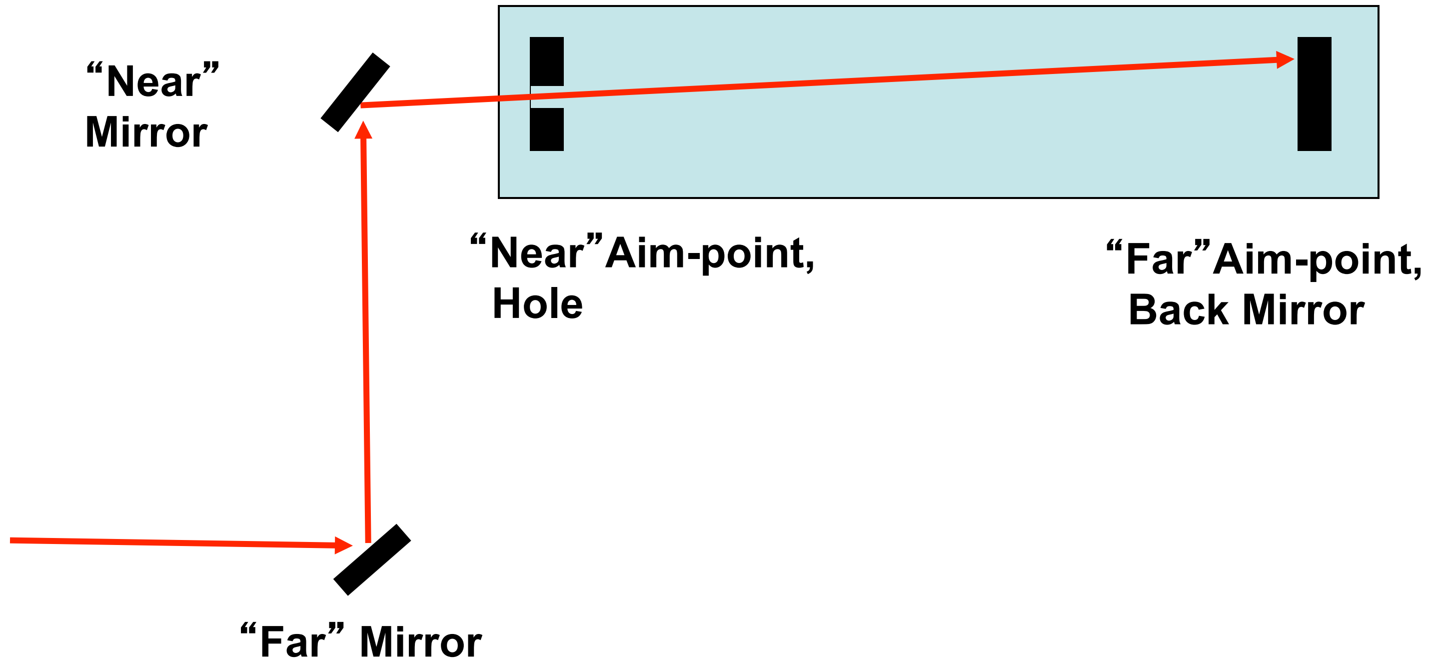
Adjusting the laser beam direction on the mirror with either image leaves the position of the beam stationary on the imaged optic.

Thus, we may adjust aiming at the input hole with the mirror where the back cell mirror is imaged, without affecting the beam position on the back mirror.

Similarly, we may adjust aiming at the back cell mirror with the mirror where the input hole is imaged, without affecting the beam position on the input hole.

This arrangement greatly simplifies optimizing the beam coupling into the cell.

Usual Cell Aim In: Iteration of two aiming mirrors



“Near” Mirror

“Near”Aim-point, Hole

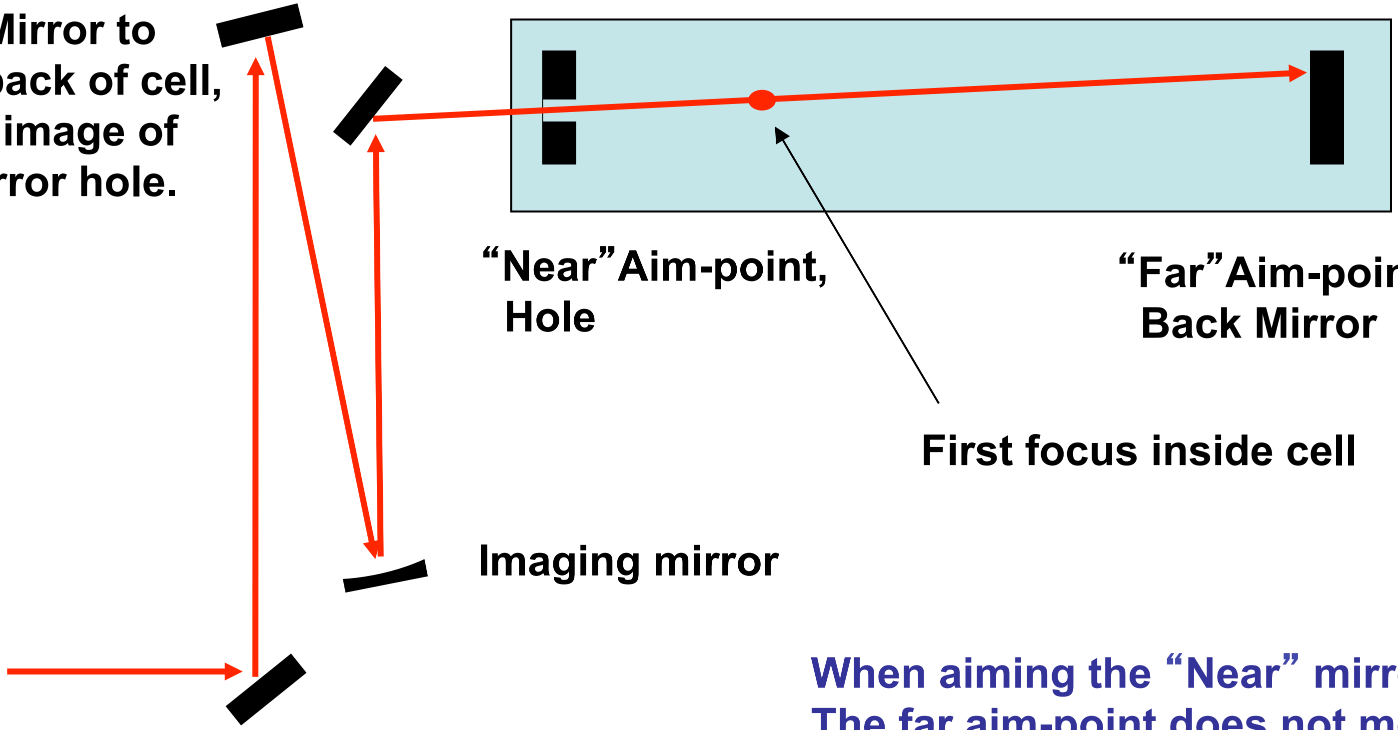
“Far”Aim-point, Back Mirror

“Far” Mirror

Iteratively aim:

“Far” mirror aims toward “near” point.
“Near” mirror aims toward “far” point.

“NMT” Cell Aim In: Independent aiming mirrors



“Near” Mirror to aim at back of cell, w/ Back image of front mirror hole.

“Near” Aim-point, Hole

“Far” Aim-point, Back Mirror

First focus inside cell

Imaging mirror

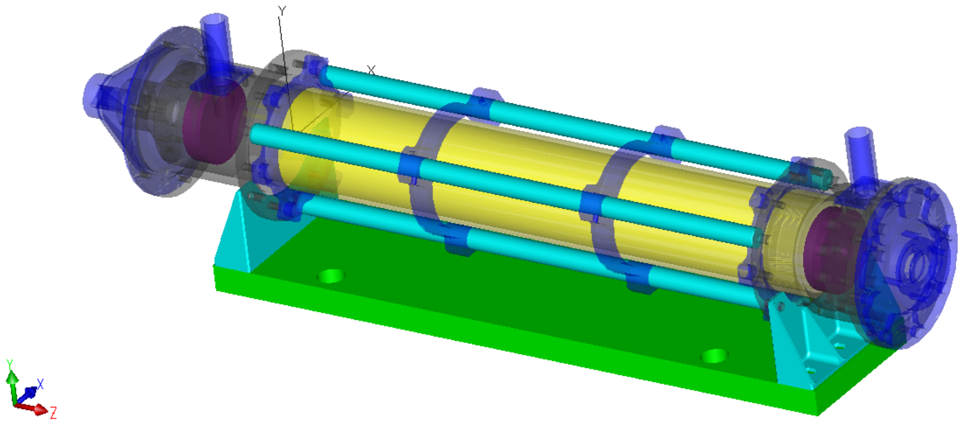
“Far” Mirror to aim at hole, w/ Back image of Back mirror

When aiming the “Near” mirror, The far aim-point does not move.

When aiming the “Far” mirror, The near aim-point does not move.

76 meter multi-pass absorption cell

Base length: 0.32 m
Passes: 238
Mirror diameter: 38 mm
Volume: 0.5 liter



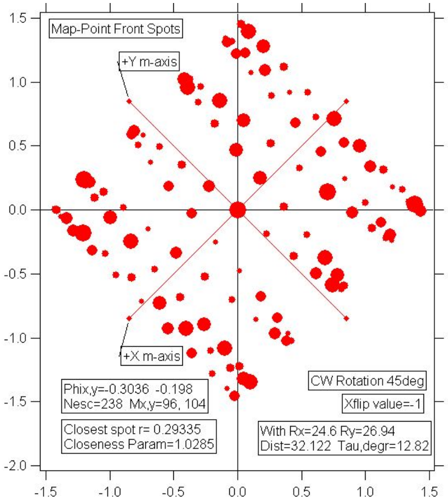
A long absorption path length is achieved by folding the optical path so that the beam transits the 0.32 m base path 238 times before exiting.

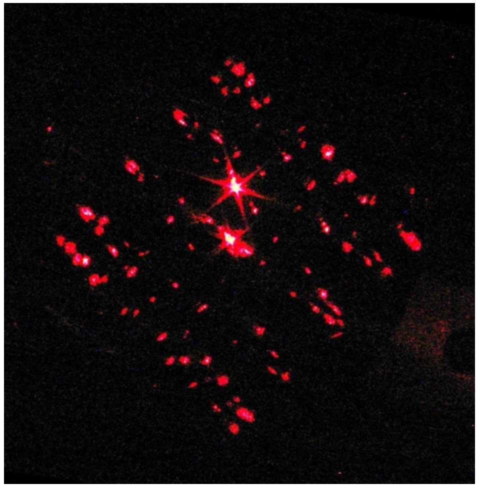
The high number of reflections means that the mirror reflectivity must be high, and the mirrors must be clean. Dirt and dust on the mirrors reduces transmission.

If the ratio of: (Field 1 range)/(Field 2 range) is too low, the mirrors could be dirty.

76 meter multi-pass absorption cell:
Some Details

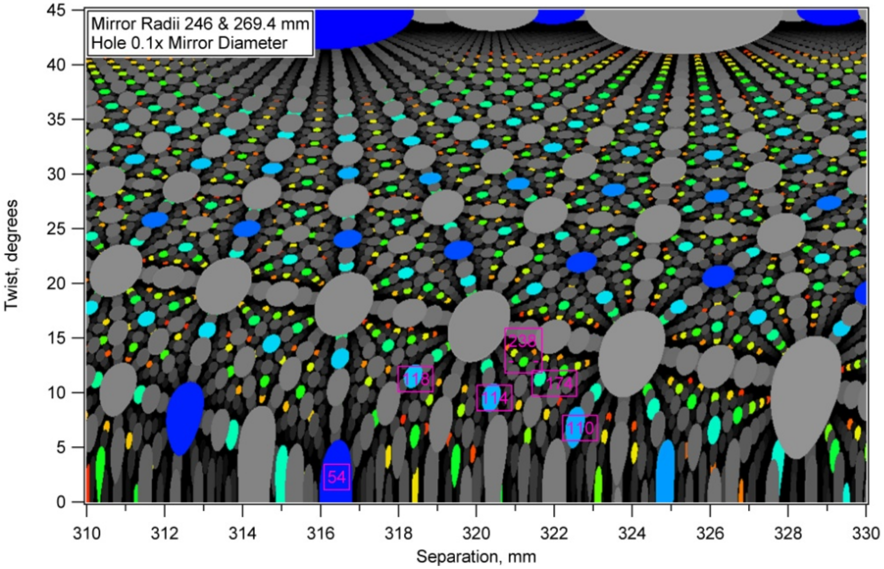
Beam spot pattern on the cell mirror (front), computed and observed.





In the astigmatic Herriott cell, different circulation patterns are achieved by adjusting mirror spacing and twist.

Shown here is a map of pass number versus spacing and twist.



Data Acquisition – Laser T and I

- The laser sub-mount is held at a constant temperature (+/- 1 mK) by a TEC and PID loop

- For a fixed laser current, I, this determines the output frequency of the laser

- But, we do NOT fix the laser current. Instead we rapidly sweep the laser current which rapidly sweeps the laser frequency

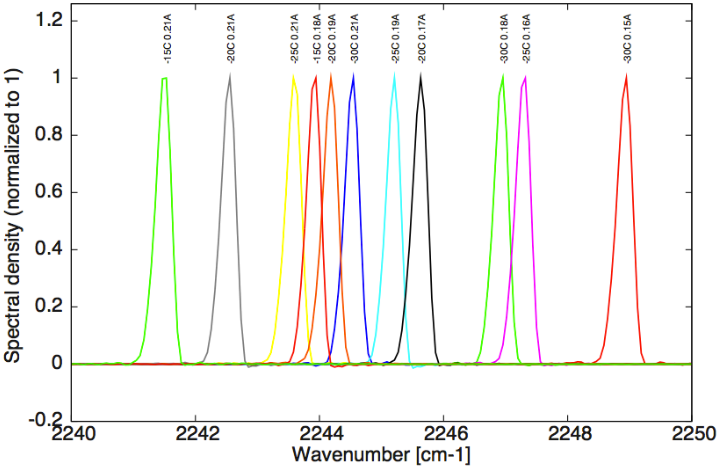
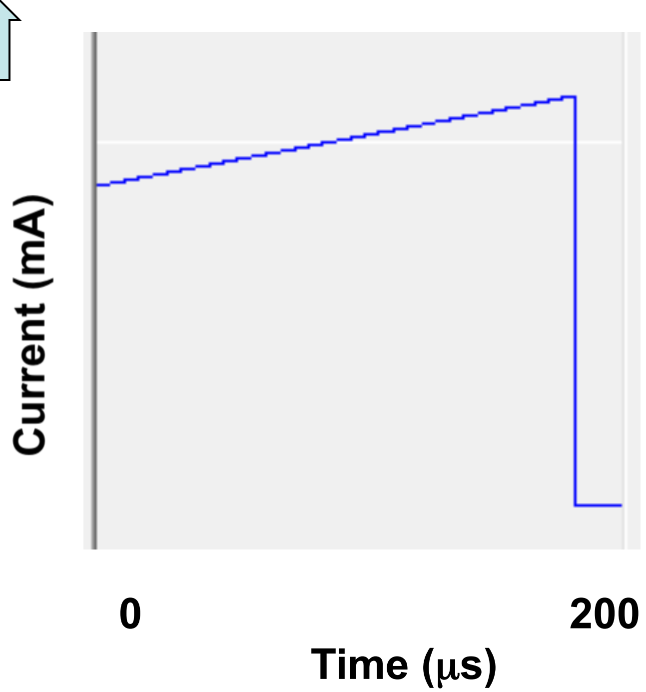
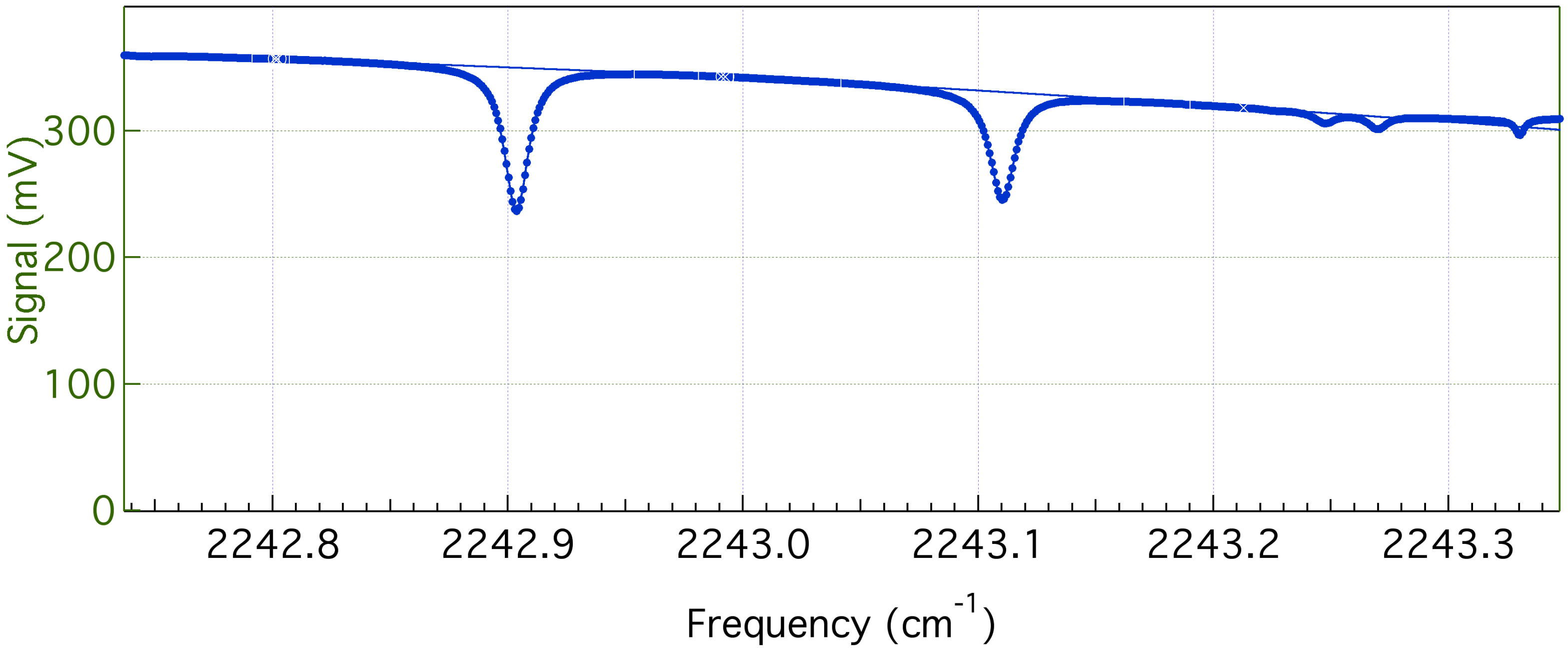


Figure 3: spectra at different temperatures for various DC currents



Data Acquisition – Scanning the Laser



- Laser is rapidly scanned over spectral window (~ 1 cm^{-1})
- Spectral window contains several lines
- Each spectrum has ~200 points; one spectral point is measured in ~1 μs; a full spectrum is captured in ~200 μs.
- 5000 spectra are averaged to produce 1 Hz data
- X axis is *points* or *time* or *frequency (cm^{-1})*

“TDLWINTEL”

Laser Control and Signal Processing Software

Laser Drive Control:

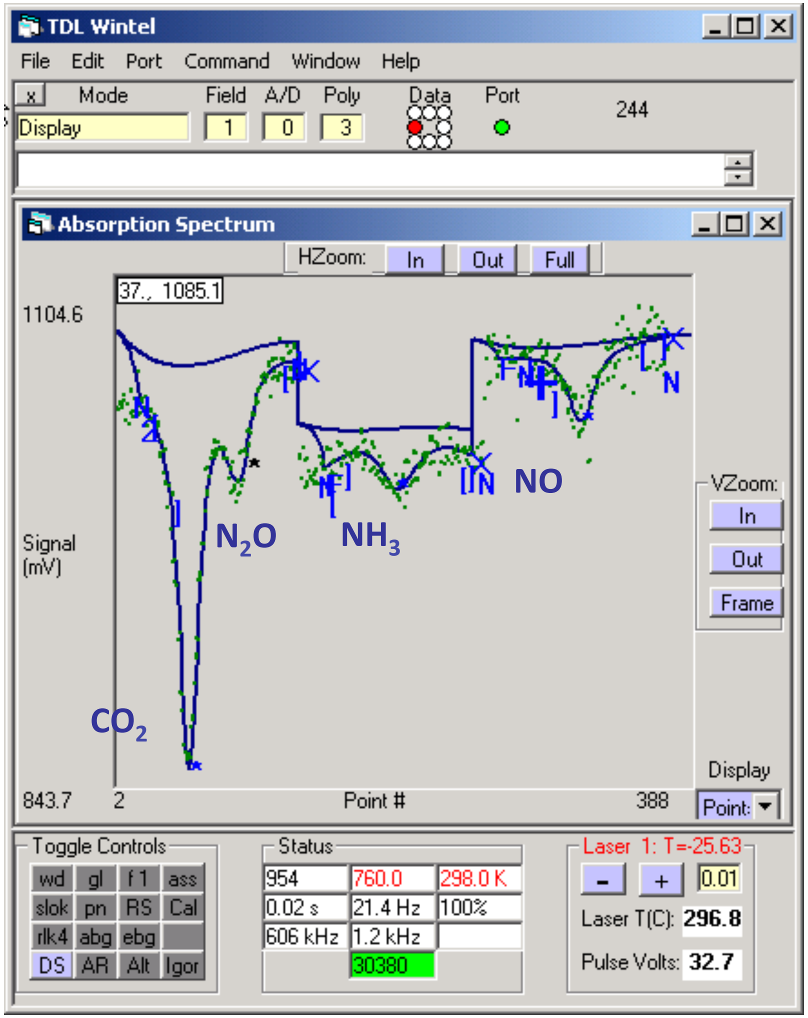
- Pulsed or CW lasers
- Tuning ramp & gate
- Multiplex up to 4 lasers
- Lock to reference lines

Signal Processing:

- Direct absorption
- Power normalization
- Spectral background correction
- Rapid fitting [to 25 Hz]
- HITRAN based spectral fits
- Multi-gas concentrations (up to 16)
- Spectral reanalysis options
- Flexible data output formats
- Absolute Concentrations

Features:

- Multiple calibration gas valves on timers
- Long-term unattended operation
- External command language
- Automatic startup
- Remote Control



Mode: Display
Field: 1
A/D: 0
Poly: 3
244

37., 1085.1
1104.6
843.7
2
388

954	760.0	298.0 K
0.02 s	21.4 Hz	100%
606 kHz	1.2 kHz	
	30380	

Laser 1: T=-25.63
0.01
Laser T(C): 296.8
Pulse Volts: 32.7

Data Sample: 3 lasers, 4 gases, 20 ms

TDLWintel Features

- Automatic start up
- Laser frequency lock
- Reports dry air MR by measuring water
- Corrects for ambient absorption in optics box
- Automatic background spectrum correction
- Automated valve schedule and mask
- Automatic pressure and flow control
- Data outputs: Hard drive, Network, RS232
- Batch re-analysis using “Playback”

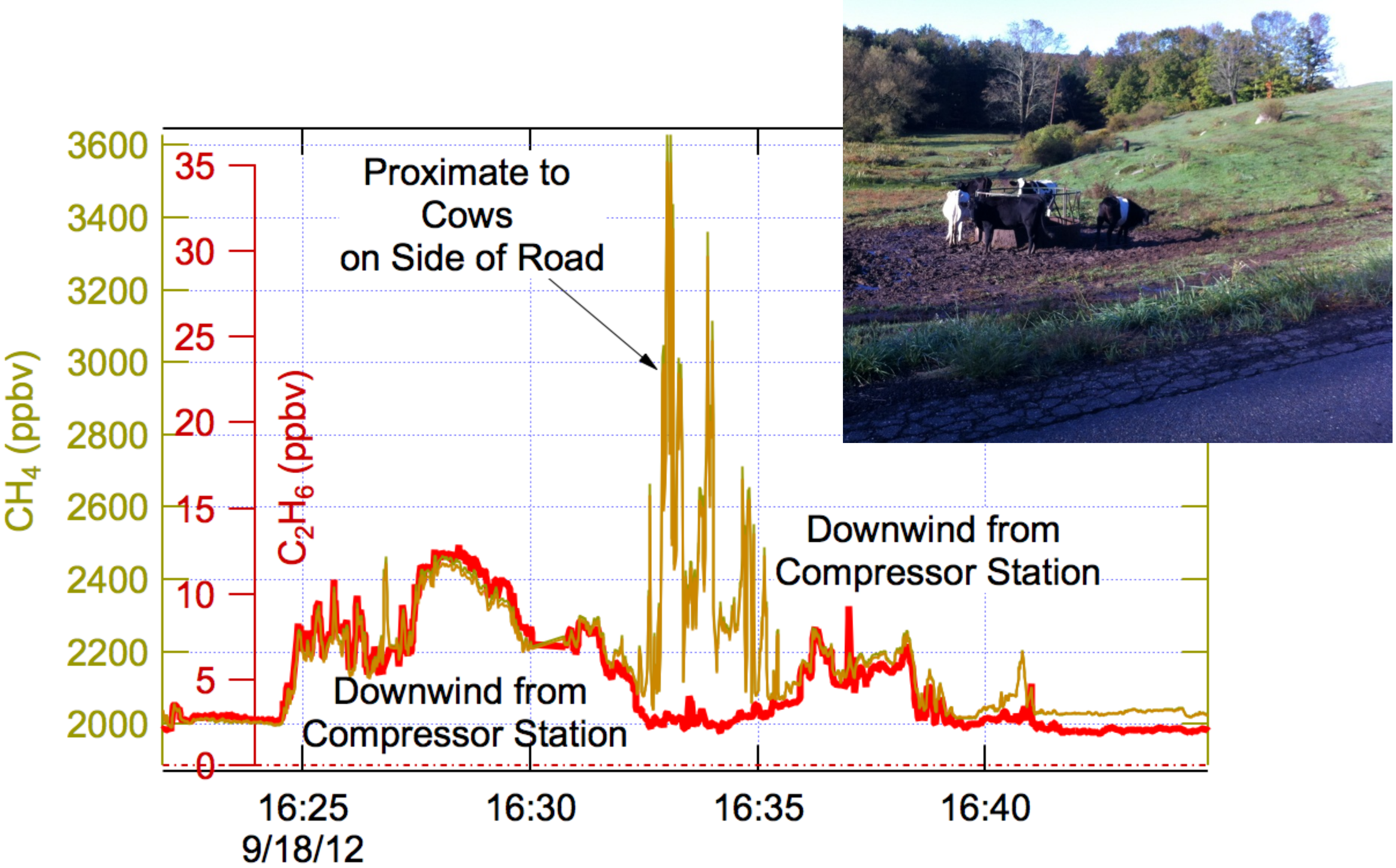
Thermal Control

- The optical bench is temperature stabilized using a constant temperature recirculating fluid (20% ethanol).
- Circulation and T control provided by Thermocube
- Layers of insulation provide good thermal isolation from environmental fluctuations.

Future plans

- Better and better isotopes
- Two lasers in a mini QCL
- More use of 3 micron lasers – ethane and methane isotopes
- Improvements to software to promote experimental integration

Ethane measurement assists methane source attribution



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