Quantum Cascade Lasers for Measurement Science

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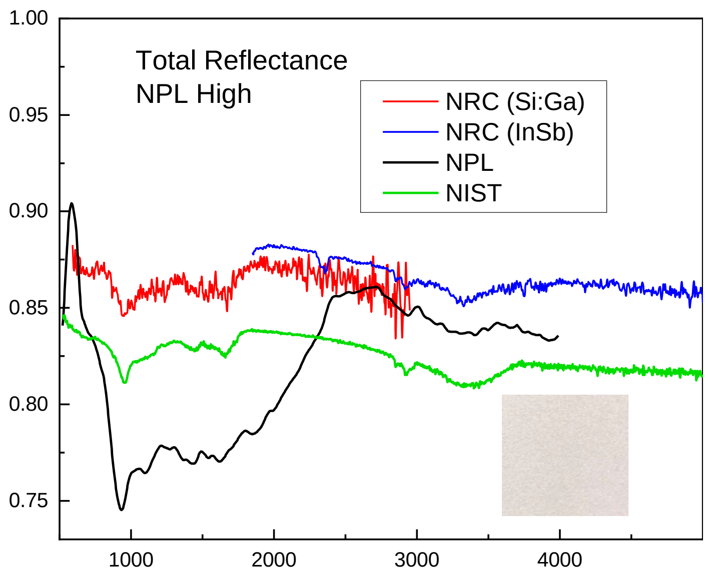
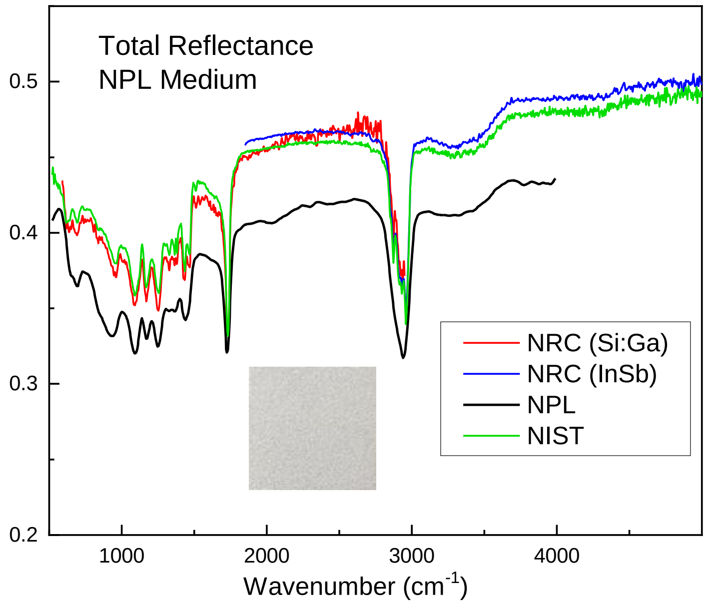
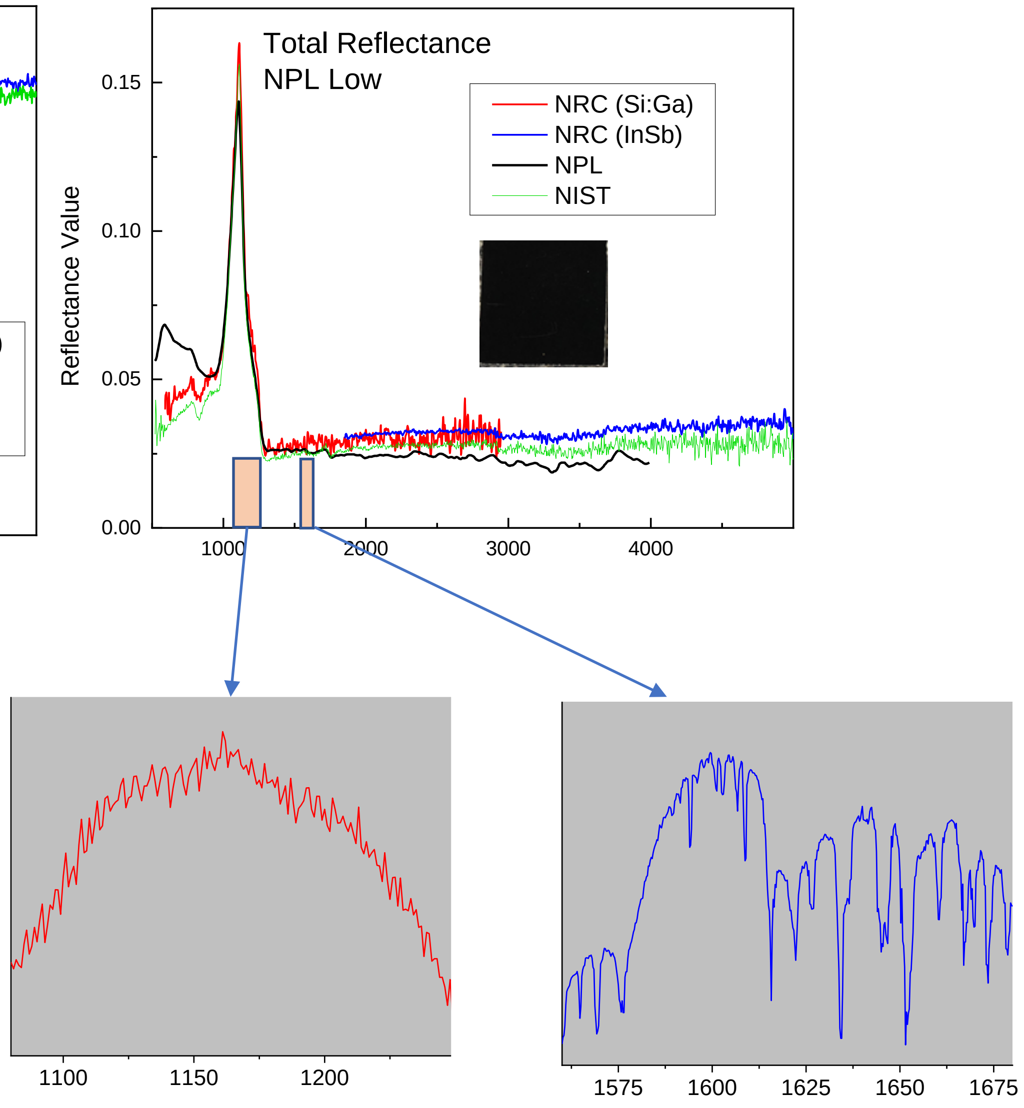
Outline

- **Quantum cascade lasers (QCLs) in infrared (IR) metrology**
- **Three example applications**
- **Total reflectance values (R) with QCLs**
  - Low reflectance diffuse materials - 0.001% < R < 1% for 5.9 to 9.3 µm
  - Tunable IR sources for IR integrating sphere
  - Advantages of QCLs sources - high brightness and collimation
  - Disadvantage - restricted QCL tuning range
- **IR microscopy with QCLs as high brightness sources**
  - Spectrometer-free, hyper-spectral imaging of samples with MCT focal plane array detector
  - Limitation - IR spot size, diffraction limited to tens of micrometers
- **Optical photothermal infrared (O-PTIR) spectroscopy**
  - Combining a QCL with a visible laser in a pump-probe method
  - IR spectra spatially resolved at the visible diffraction limit
  - 20x smaller than the IR limit

Total/Diffuse Reflectance Reference Materials

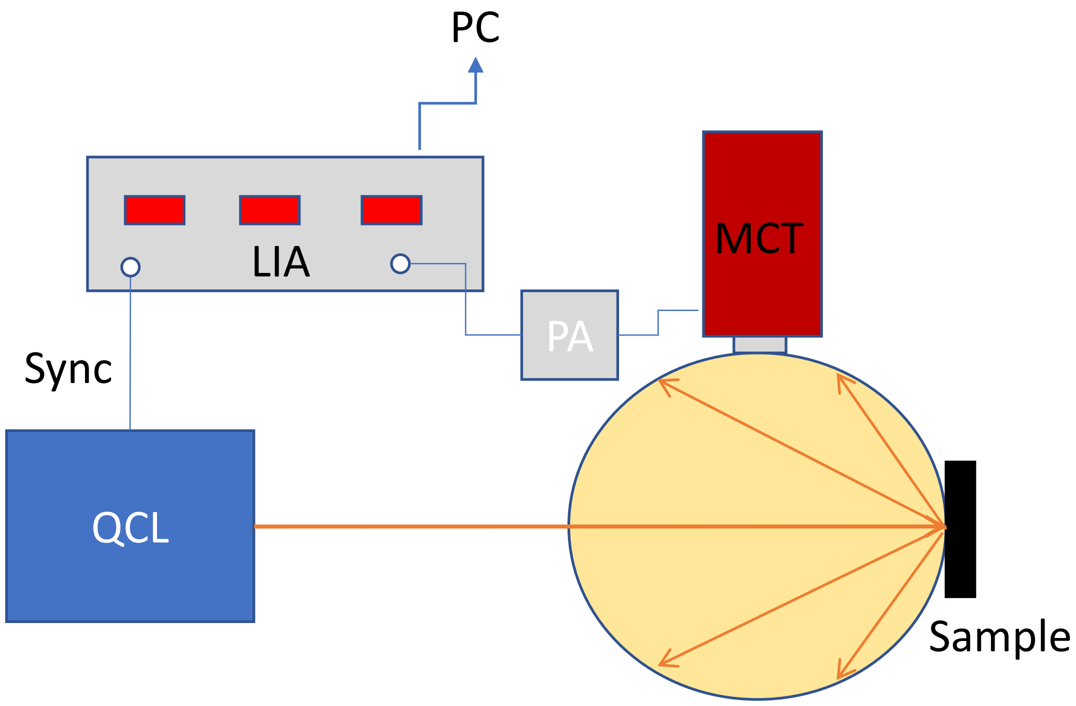


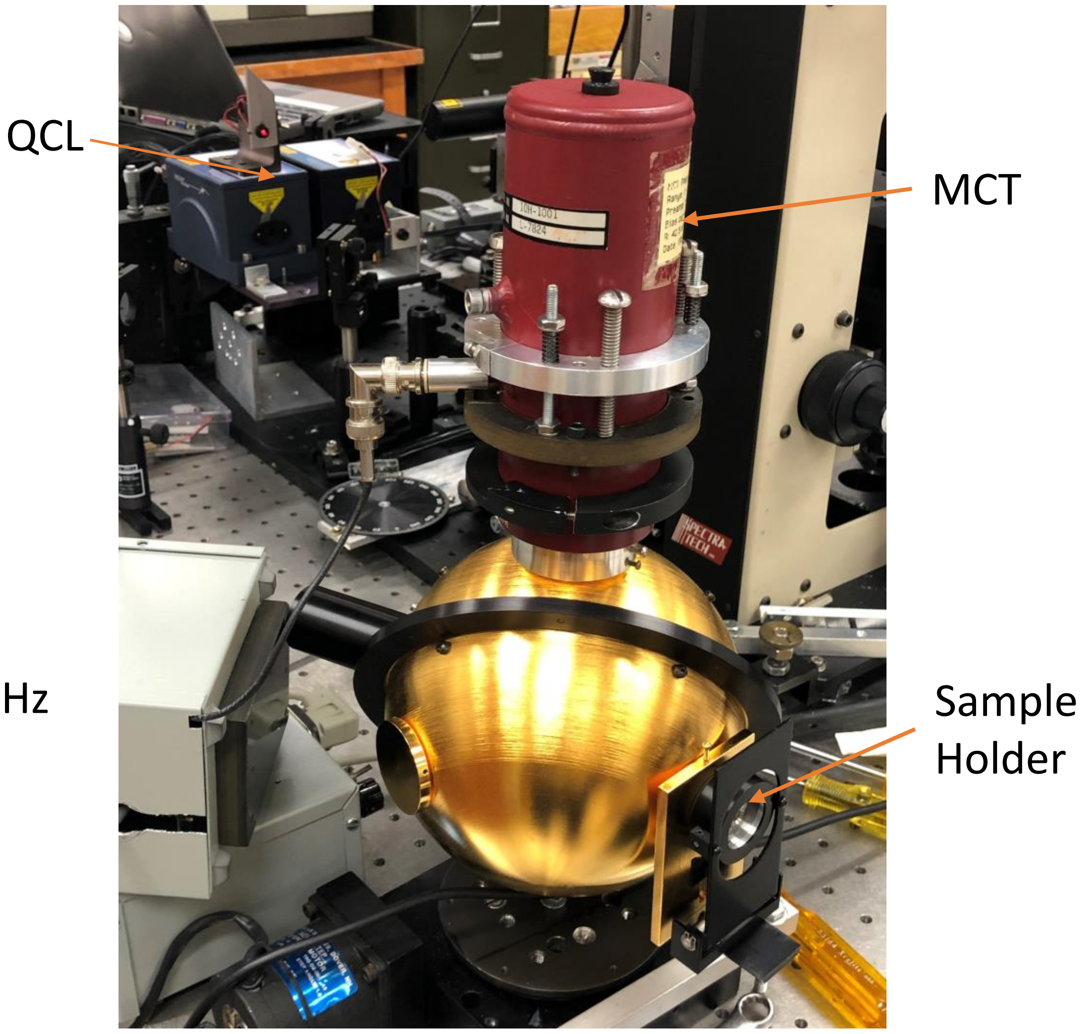




- Concept of Frank Clarke – NPL (UK)
- Flame sprayed aluminum – Low: Nextel, Med: Gray
- Data: NRC, NIST (Leonard Hanssen), NPL
- Differences in measurement geometries
- Concentrate on the low reflectance

QCL and Sphere Apparatus





- QCLs - Daylight Solutions 1075-1260 & 1550-1685 cm^{-1} Pulsed - 15 kHz
- Integrating sphere - Labsphere wall mount - Infragold interior
- Angle of incidence – 15^{o}
- Detector: Judson MCT (77K) on sphere top port
- Lockin Amplifier: Stanford synchronized to QCL
- VB program sets wavenumber and reads LIA
- Substitution with Infragold calibrated on absolute FTIR system
- Specular included

QCL – Sphere Data Processing

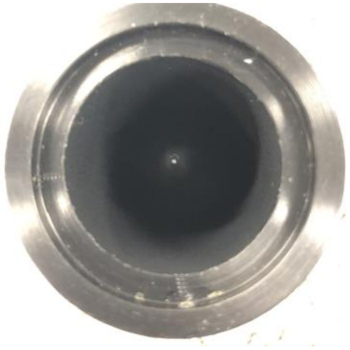
Reflectance of a sample with a simple ratio

$$R = \frac{V_s - V_{op}}{V_{IG} - V_{op}} R_{IG}$$

- V_{op} is the signal obtained with laser on but with an open sample port
- V_s is the signal with the sample in place
- V_{IG} is the signal with a calibrated Infragold sample in place
- R_{IG} is the calibrated reflectance value of the Infragold sample

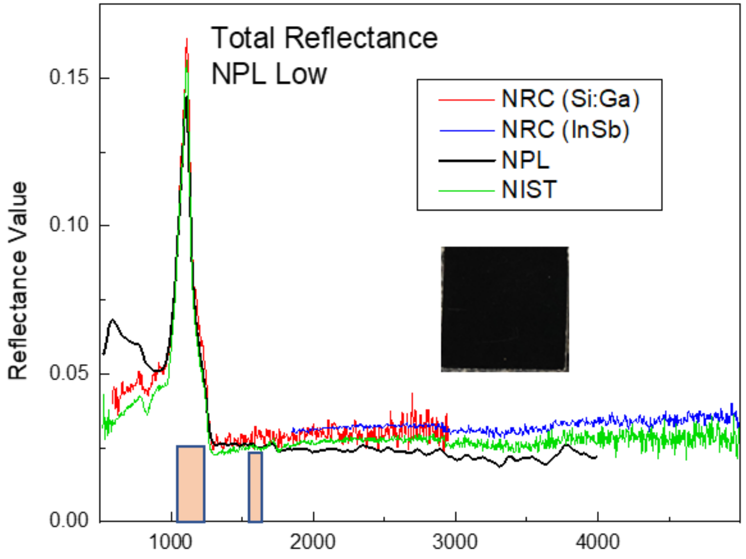
Results for 1600 cm^{-1} unpurged

	V_s (µV)	$V_s - V_{op}$	% R
Light trap	457	91	**0.06**
Black paint	8026	7660	4.82
NPL low	4028	3662	2.30
NPL medium	64178	63813	40.14
NPL high	130537	130171	81.87
V_{op}	366		
V_{IG}	151407	151041	

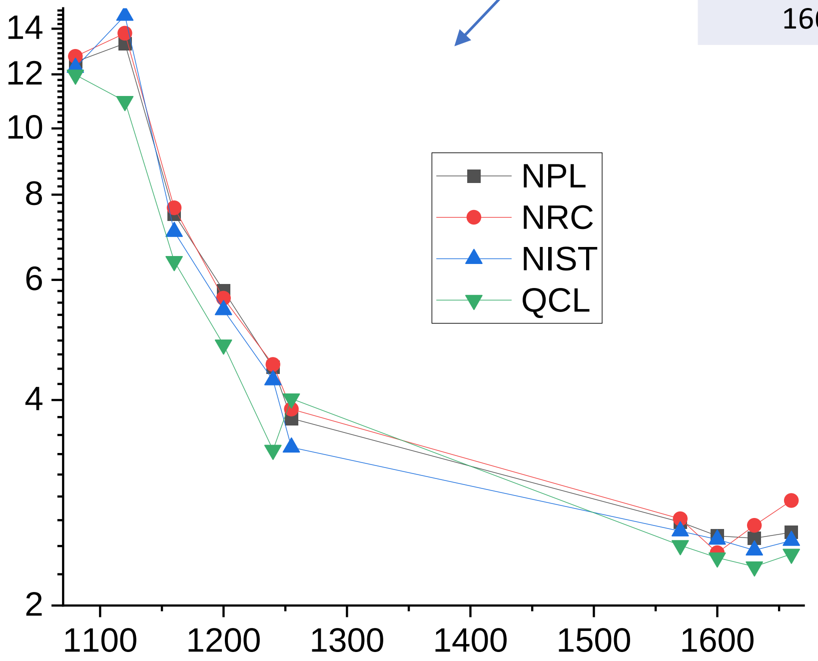


Demonstrates sensitivity below 0.1% with the Labsphere light trap, more than 10x better than previously

Accuracy with NPL Low



Wavenumber	NPL	NRC	NIST	QCL
1080	12.52	12.75	12.28	11.98
1120	13.32	13.79	14.64	10.96
1160	7.49	7.65	7.06	6.39
1200	5.79	5.64	5.42	4.82
1240	4.47	4.51	4.28	3.38
1255	3.76	3.88	3.41	4.02
1570	2.65	2.68	2.57	2.45
1600	2.53	2.39	2.50	2.35
1630	2.51	2.62	2.41	2.28
1660	2.56	2.85	2.49	2.38



Reasons for differences

Ambient vs purge

Spectral resolution – lineshape convolution

Differing geometries

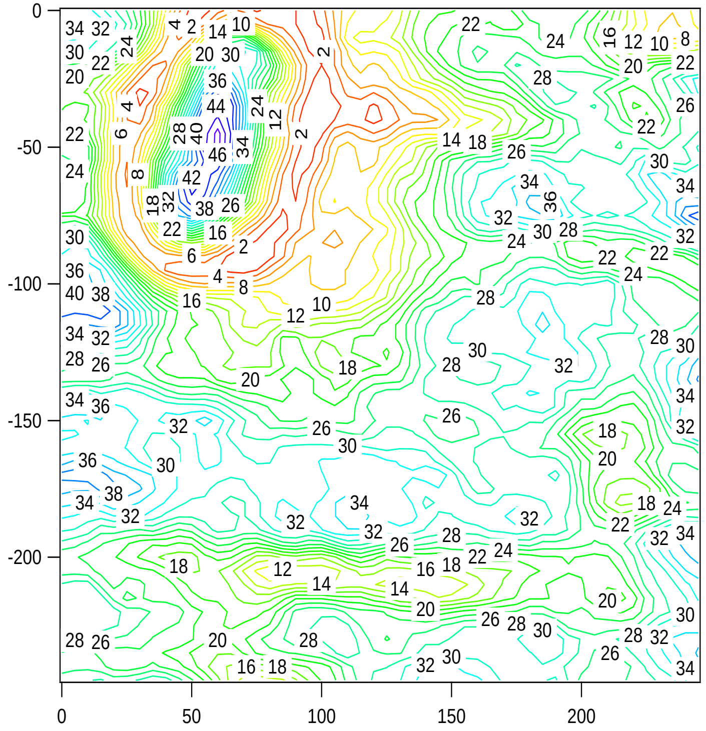
QCL Microscopy

(1) Mapping: Single point MCT detector Spectra Tech IR Plan Microscope
slowest by far for complete "images"

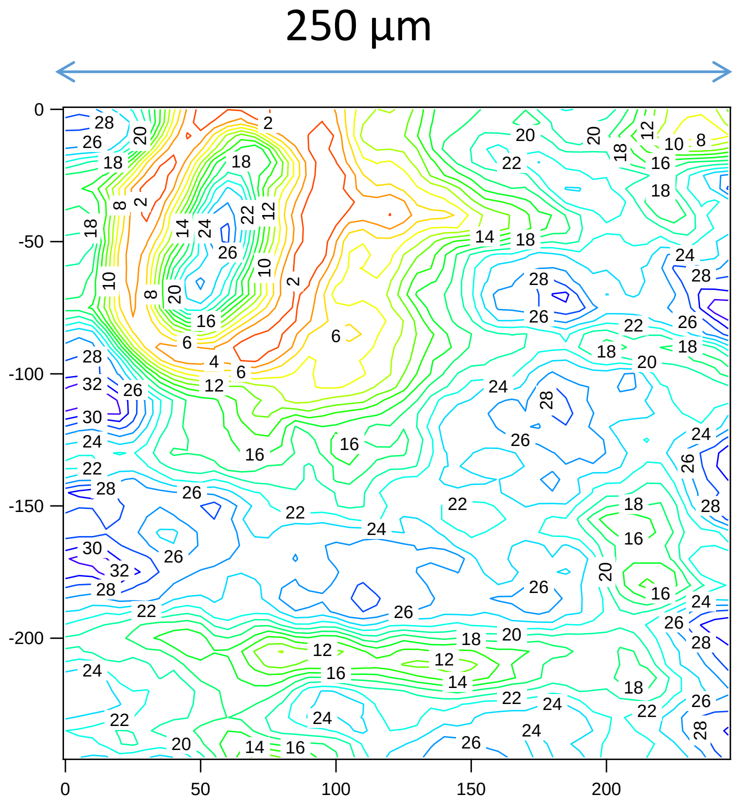
(2) Imaging: QCL source for Bruker FTIR, Hyperion Microscope with MCT FPA
uses OPUS software
rapid imaging
Michelson losses (-50%)

(3) Imaging: QCL with direct access to SBRC FPA
utilizes full laser brightness
most rapid imaging
significant software development required
relies on laser wavenumber calibration

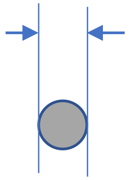
QCL Microscopic Mapping



1650 cm^{-1}



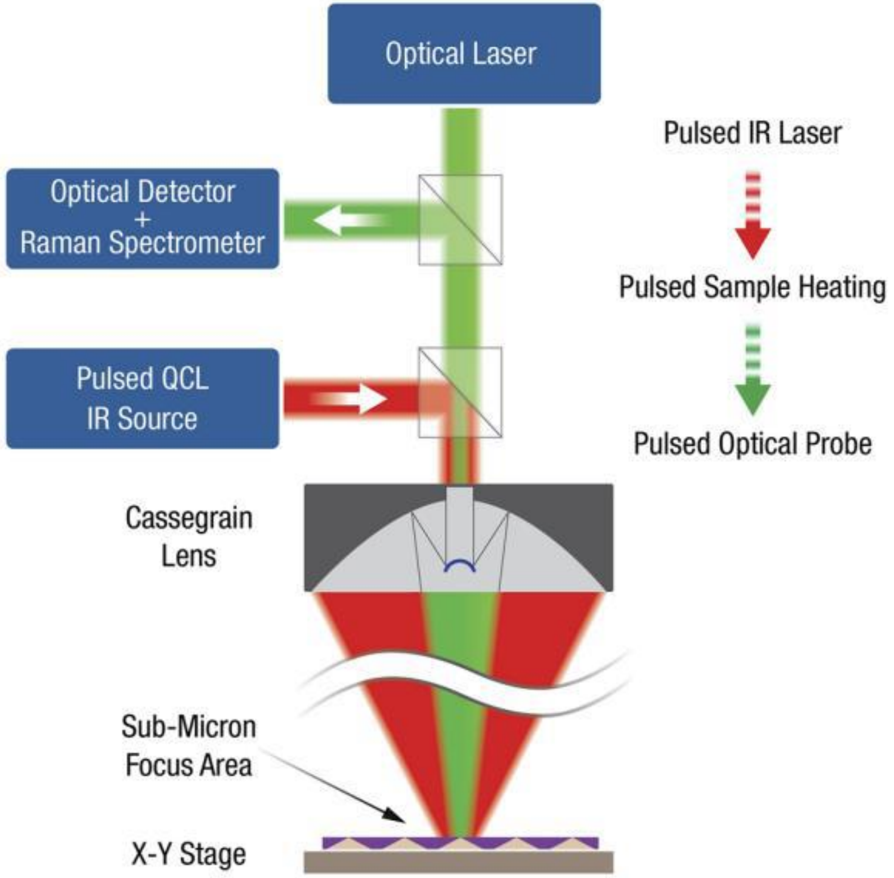
1600 cm^{-1}



IR Resolution
12 µm

Organic sample
Resolution ± 16 µm
Total 250 x 250 µm

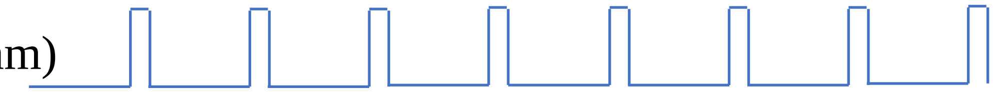
QCLs in O-PTIR



Measures IR absorption spectrum by detecting changes in thermal changes in sample
Pump with pulsed tunable QCL and probed with visible probe
IR absorption spectrum acquired by measuring the modulated optical probe from the pulsed IR heating of the sample
Transmission-like IR spectra free from IR dispersion effect obtained from IR reflectance spectroscopy

How does O-PTIR work

- Example: Semi infinite bulk polystyrene
- Probe: Visible reflectance at 532 nm
- Pump: Pulsed laser at 1600 cm^{-1} (6250 nm)
- IR Absorption during laser pulse
- Temporary change in temperature within the IR absorption volume
- Change in refractive index at 532 nm from thermo-optic effect
- Change in visible reflectance in phase with pump

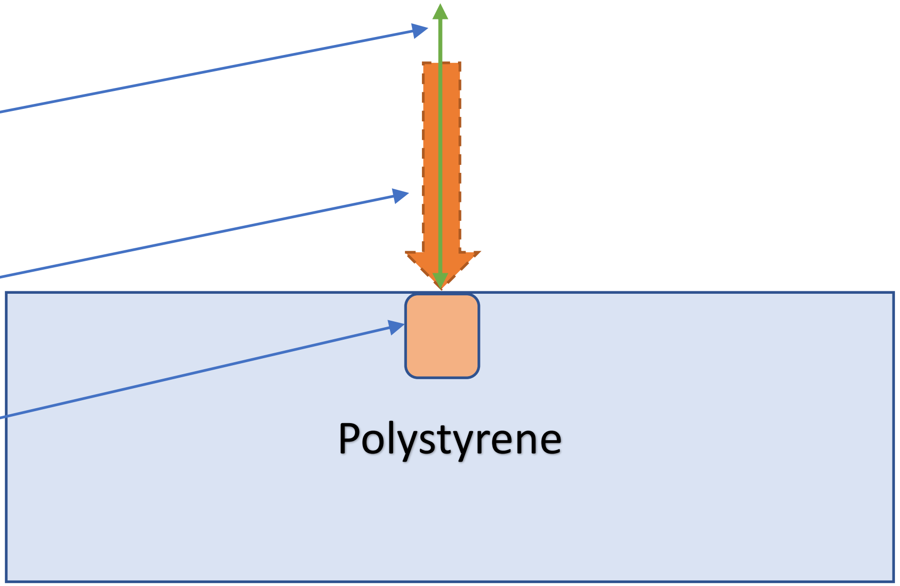


$$R = \left[\frac{n-1}{n+1}\right]^2 \qquad \frac{\Delta R}{R} = \frac{4}{\left[n^2-1\right]^2}\Delta n = 1.67\Delta n$$

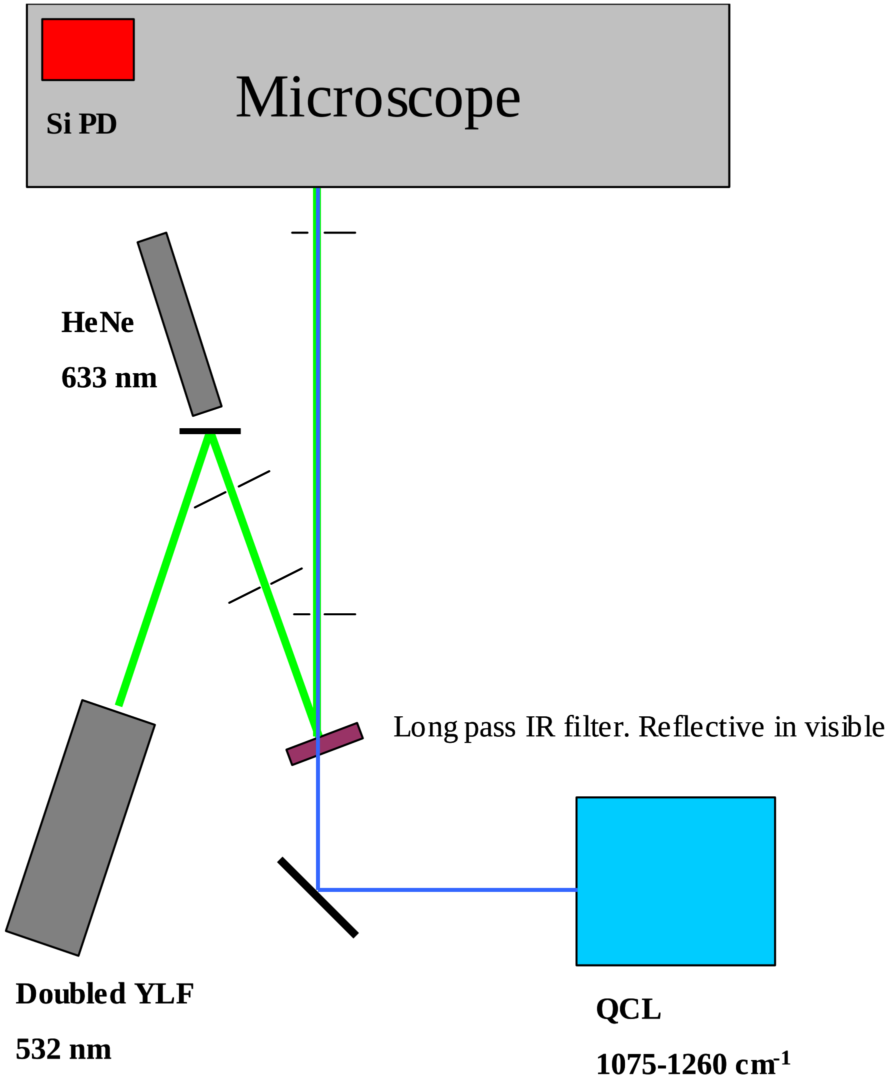
Energy, E_p, is 1x10^{-7} Joules per pulse
Volume heated is 1.3x10^{-15} m^3
Thermal heat capacity: $c_p m\Delta T = E_p$ $\qquad \Delta T = 57.4$ K

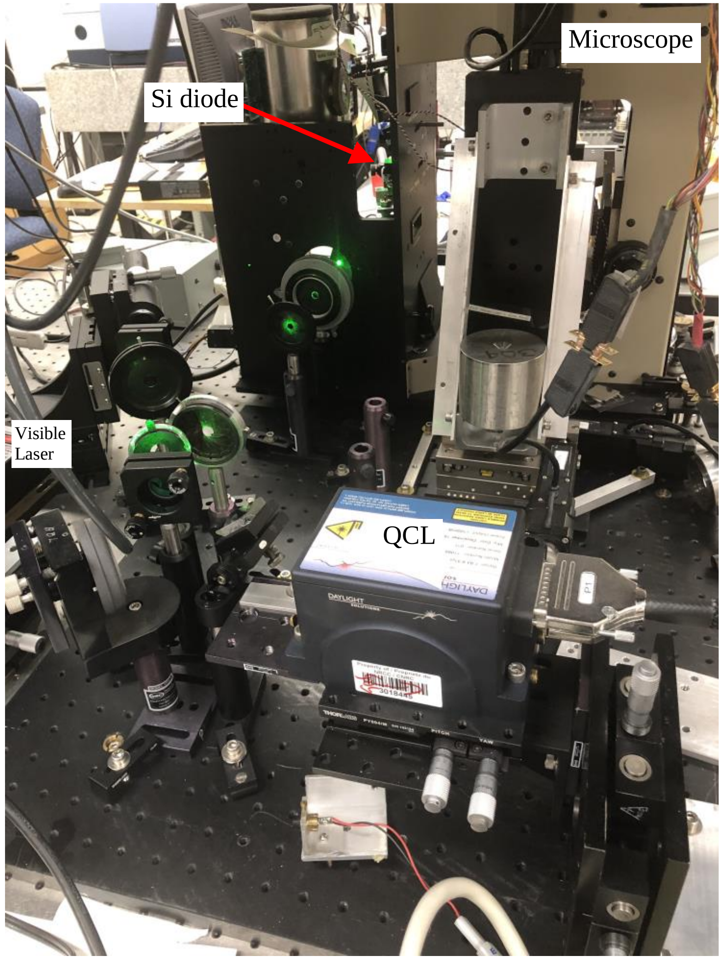
$$\Delta n = \Delta T\frac{dn}{dT} = -6.9x10^{-3}$$

Relative reflectance change at 532 nm in phase with the IR pump is -1.15x10^{-2}

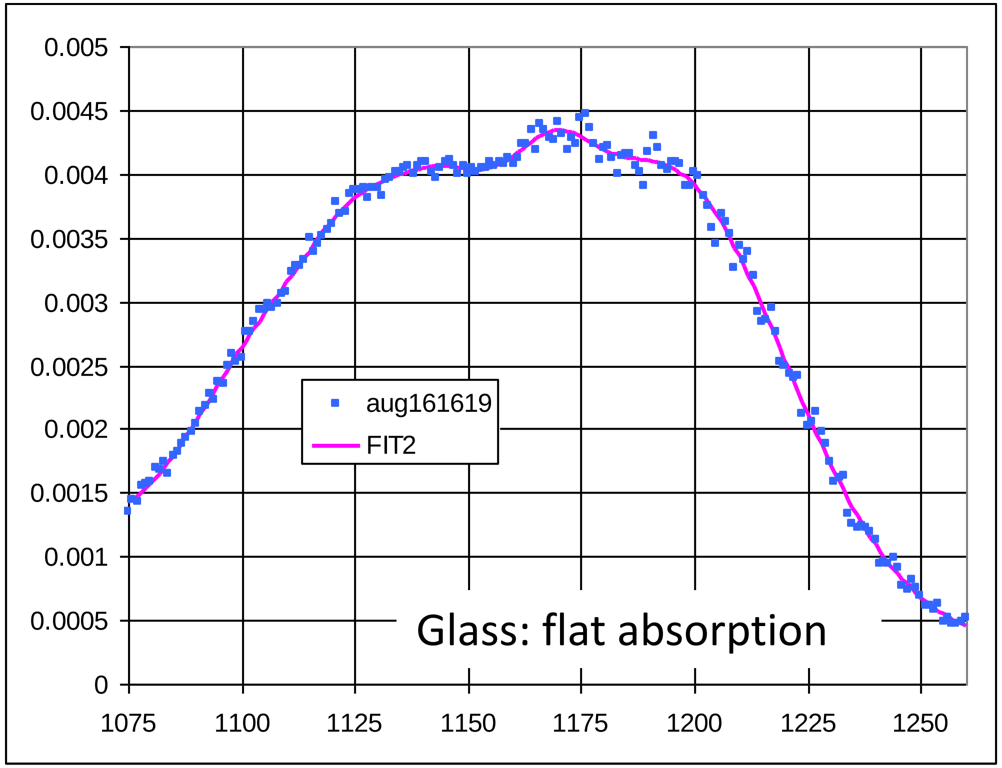


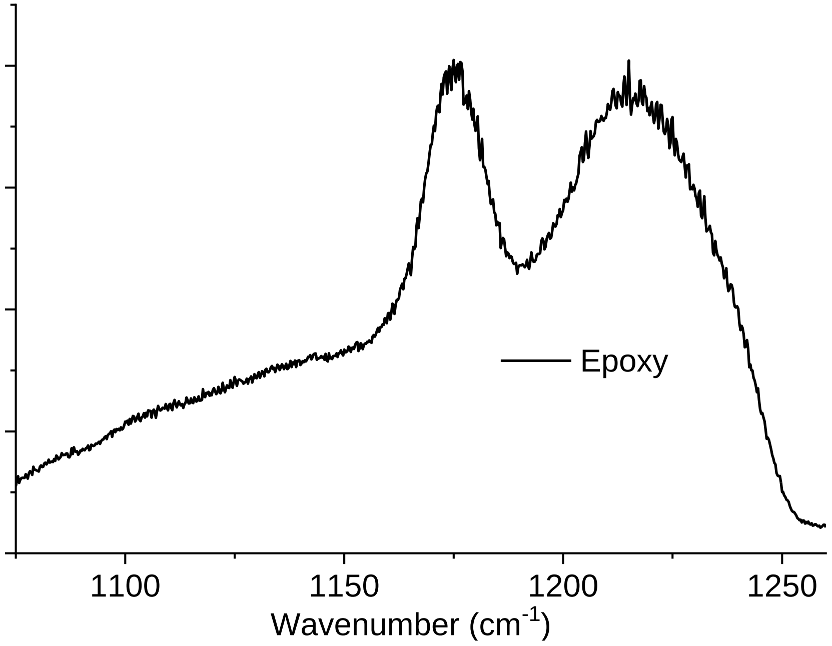
Our apparatus

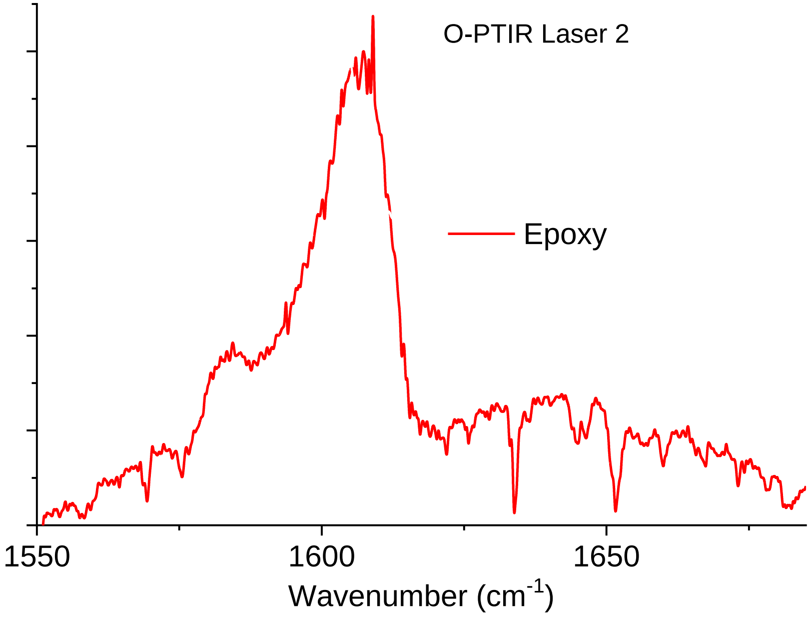




Bulk samples

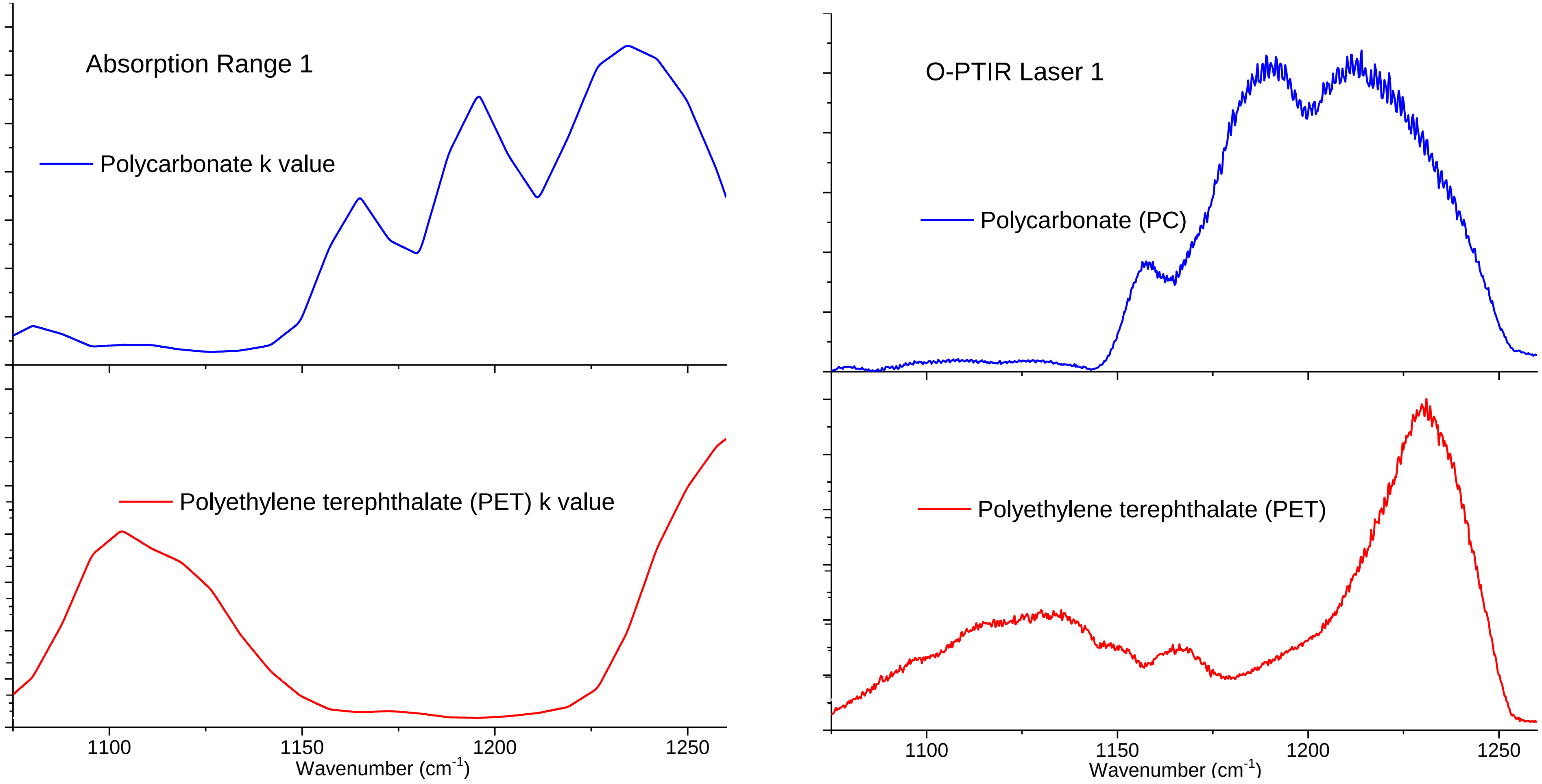






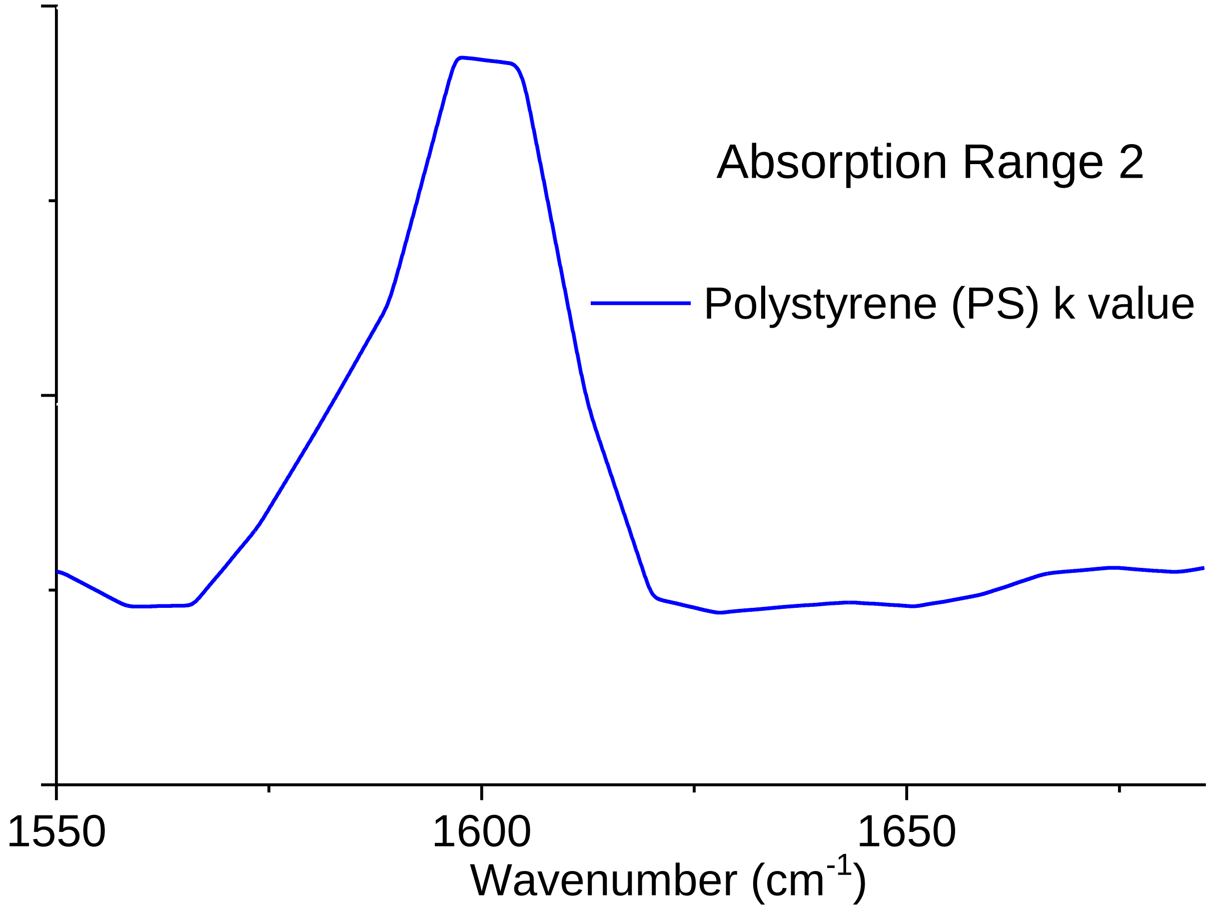
Polymer Absorption Spectra 1

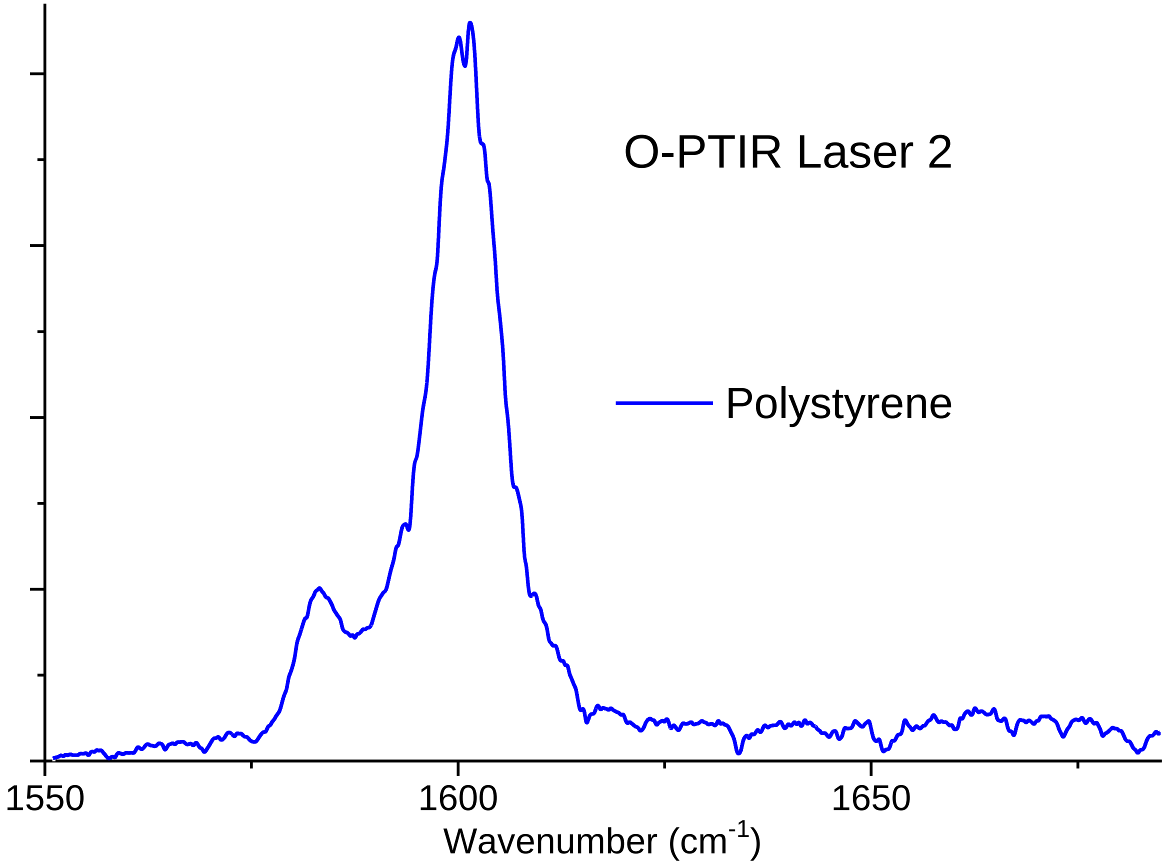
Range: 1075 to 1260 cm^{-1} (9302 to 7937 nm)



Polymer Absorption Spectra – Laser 2

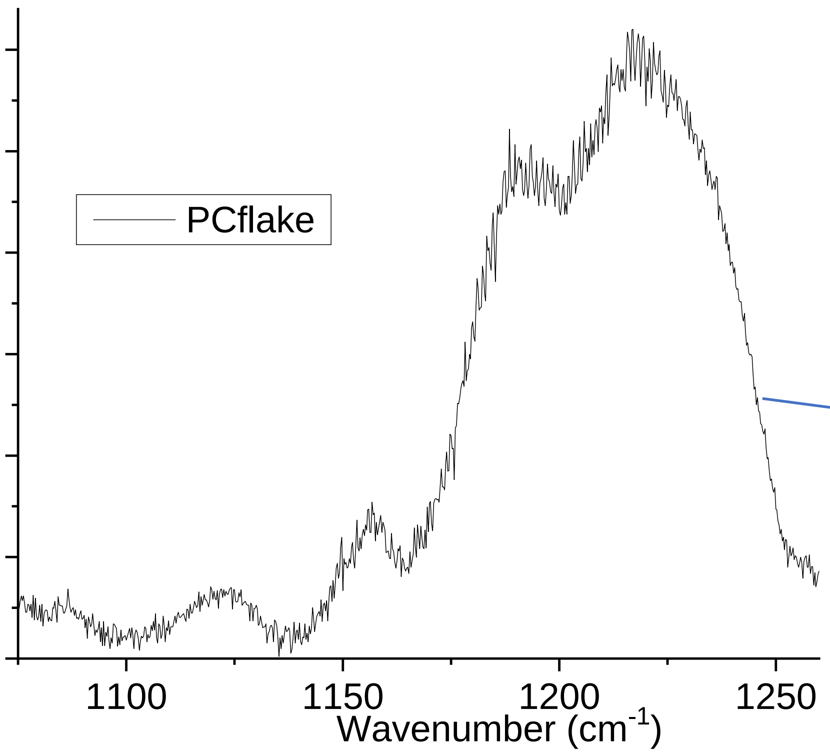
Range: 1550 to 1685 cm^{-1} (6452 to 5935 nm)



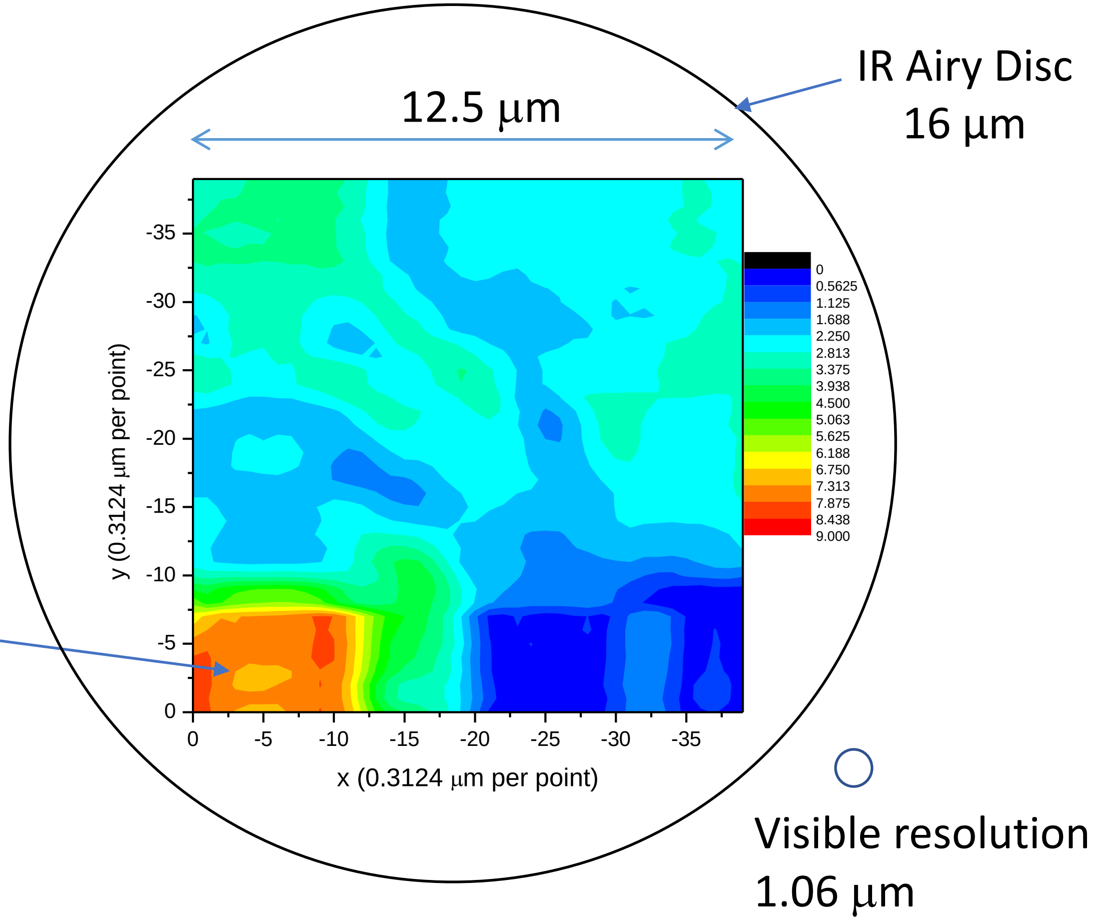


Spatial Resolution

Polycarbonate flakes on glass, map for 1210 cm^{-1}.

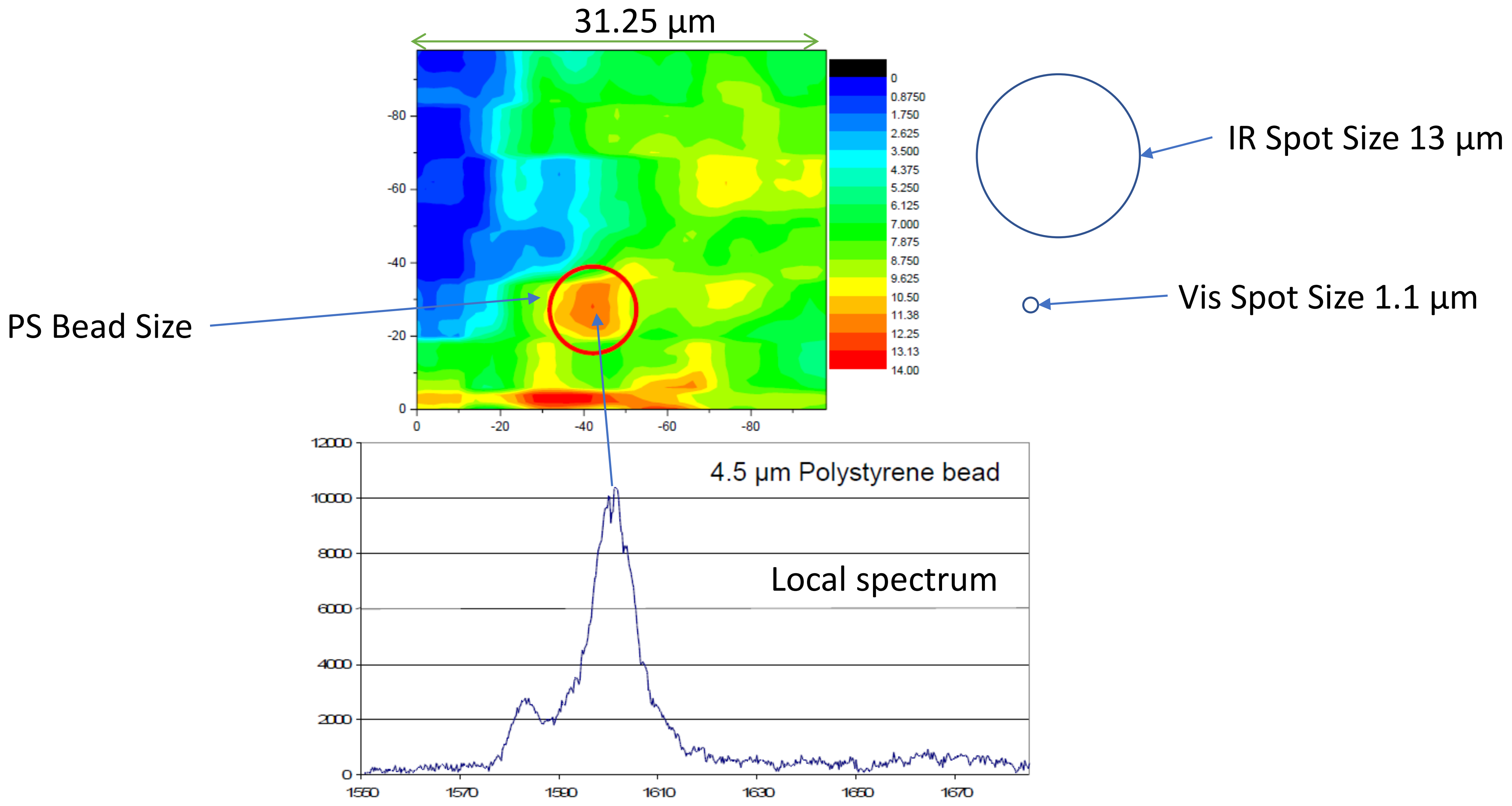


Range is 1075 to 1260 cm^{-1} (9302 to 7937 nm)



- Flake is 3 x 3 μm
- Edge resolved within 1 μm

Polystyrene Beads



Range: 1550 to 1685 cm^{-1} (6452 to 5935 nm)

Summary

Quantum cascade lasers (QCLs) in infrared (IR) metrology

Three example applications

1. Total reflectance values with QCLs
    Low reflectance values below 0.1%
2. IR microscopy with QCLs as high brightness sources
3. Optical photothermal infrared spectroscopy
    Spatial resolution 15x greater