Compact and low-noise quartz-enhanced photoacoustic sensor for sub-ppm ethylene detection in atmosphere

P. Patimisco[a,b]

A. Sampaolo[a], M. Giglio[a], F. Sgobba[a], H. Rossmadl[c], V. Mackowiak[c], B. Gross[c], A. Cable[d], F.K. Tittel[b], and V. Spagnolo[a,b]

[a] PolySense Lab - Dipartimento Interateneo di Fisica, Politecnico and University of Bari, Italy;
[b]Rice University, Department of Electrical and Computer Engineering,, USA;
[c]Thorlabs GmbH, Dachau, Germany
[d]Thorlabs, Inc., Newton, USA

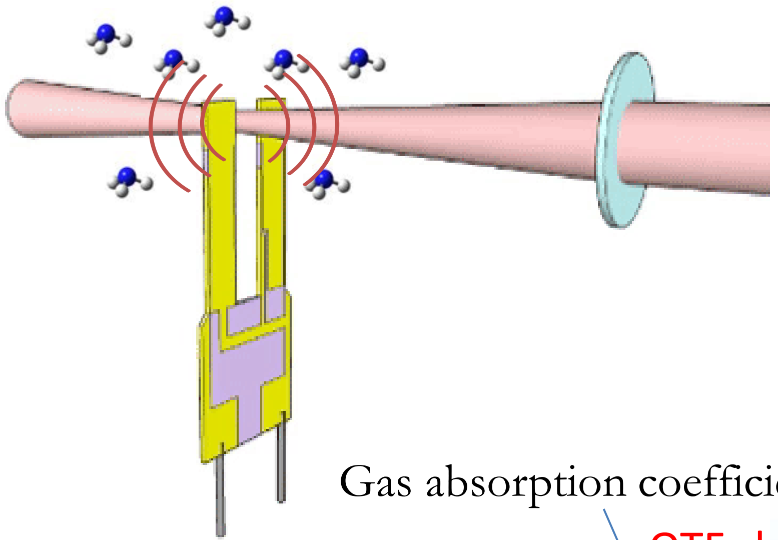
SPIE Photonics West 2018 - San Francisco, California, US, 27 January - 1 February 2018

Outline

- Introduction to **QEPAS**
- Basics on QTFs operating at the **1° overtone mode**
- **QEPAS sensor** for **ethylene** detection
- **Results** and **performance**
- **Conclusions**

Quartz-Enhanced PAS

Physics and Basic Operation



- The laser light is modulated
- A tuning fork detects the sound wave produced by the absorbing gas
- The mechanical vibration converted in an electrical signal

Gas absorption coefficient

QTF-dependent

$$\boldsymbol{QEPAS\ Signal} \propto P \cdot \alpha \cdot Q \cdot \varepsilon$$

Conversion efficiency: optical power → sound

Laser power

QTF Q-factor

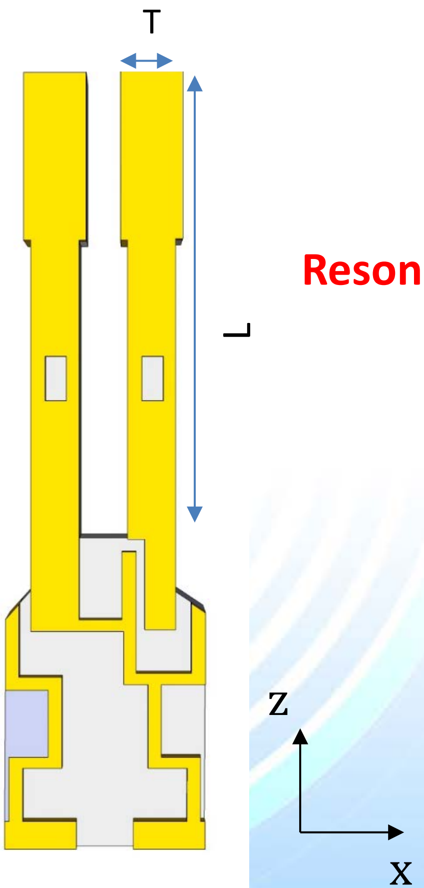
P. Patimisco et al., "Recent advances in quartz enhanced photoacoustic sensing", Applied Physics Review, in press, **2018**

P. Patimisco et al., "Quartz–enhanced photoacoustic spectrophones exploiting custom tuning forks: a review", Advances in Physics X 2, 169-187, **2016**

P. Patimisco et al., "Quartz-enhanced photoacoustic spectroscopy: a review", Sensors, 14, 6165–6206, **2014**.

Quartz tuning fork

Resonance frequency



Euler-Bernoulli beam theory

$$EI_y \frac{d^4 X}{dz^4} + \rho S \frac{d^2 X}{dt^2} = 0$$

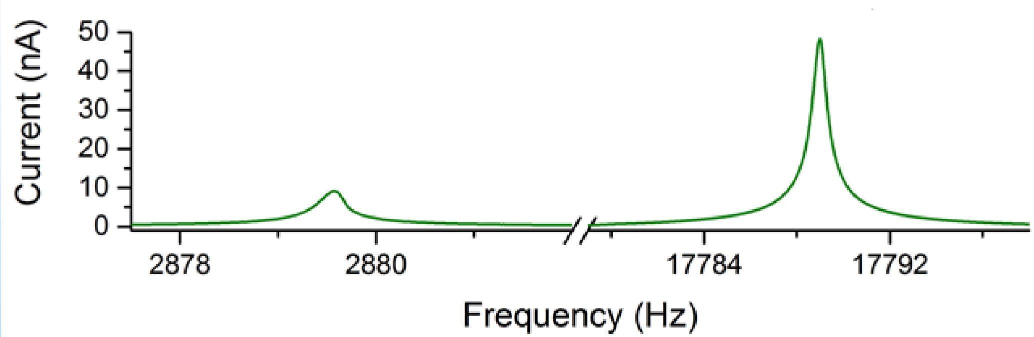
Resonance Frequency, in-plane flexural modes

$$f_n = \frac{\pi \boldsymbol{T}}{8\sqrt{12}\boldsymbol{L^2}} \sqrt{\frac{E}{\rho}} n^2$$

$$\boldsymbol{n_{fund}} = 1.19$$

$$\boldsymbol{n_{overt}} = 3$$

$$f_{1st\ overtone} \sim \mathbf{6.2} \cdot f_{fundamental}$$



P. Patimisco, et al., "A quartz enhanced photo-acoustic gas sensor based on a custom tuning fork and a THz QCL," Analyst 139, 2079, **2014**.

A. Sampaolo, et al., "Quartz-enhanced photoacoustic spectroscopy exploiting tuning fork overtone modes," Appl. Phys. Lett.,107, 231102, **2015**.

P. Patimisco et al., "Analysis of the electro-elastic properties of custom QTFs for optoacoustic gas sensing" Sens. Actuators B Chem. 227, 539, **2016**.

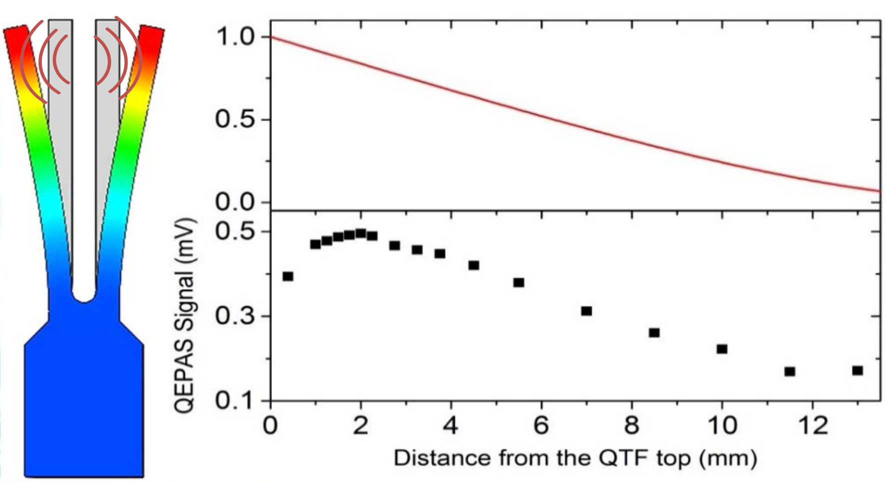
Quartz tuning fork

Why overtone mode?

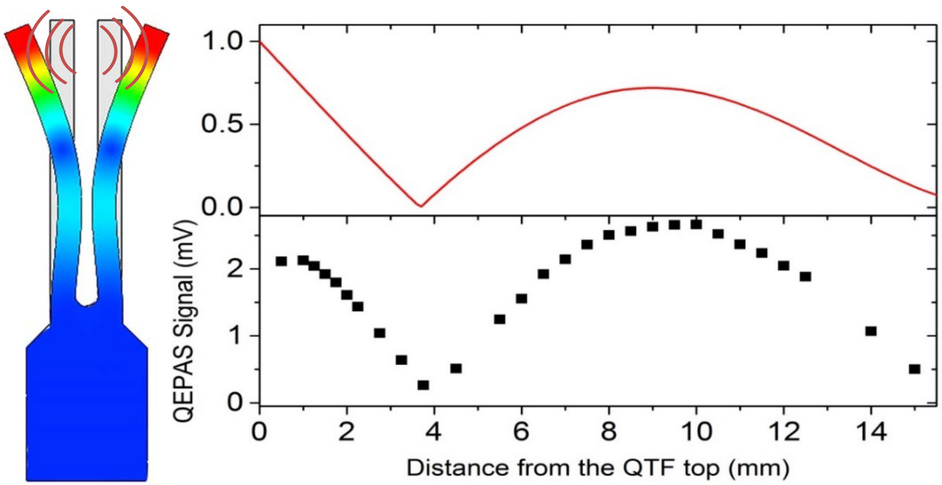
Euler-Bernoulli beam theory

$$X_n(z) = A((cosh(k_n z) - cos(k_n z)) - (\frac{cosh(k_n L) + cos(k_n L)}{sinh(k_n L) + sin(k_n L)})(sinh(k_n z) - sin(k_n z)))$$

Fundamental mode



Overtone mode



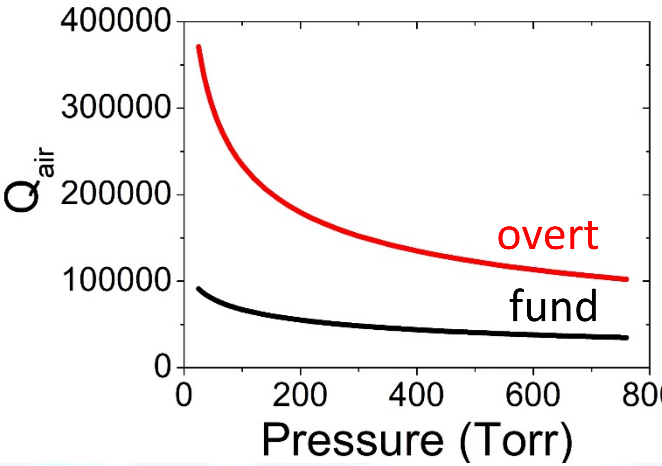
F.K. Tittel, et al., "Analysis of overtone flexural modes operation in quartz-enhanced photoacoustic spectroscopy," Opt. Express, 24, A682, **2016**.

Quartz tuning fork

Quality factor

$$\frac{1}{Q(P)} = \frac{1}{Q_{air}(P)} + \frac{1}{Q_{sup}}$$

L = 17 mm
w = 1 mm
T = 0.25 mm



Air Damping losses

$$Q_{air} = \frac{4\pi\rho T w^2 f_n}{3\pi\mu w + \frac{3}{4}\pi w^2\sqrt{4\pi\rho_{air}\mu f_n}}$$

Support losses

$$Q_{sup} = A_n \frac{L^3}{T^3} \qquad \frac{A_{fund}}{A_{overt}} \sim 12$$

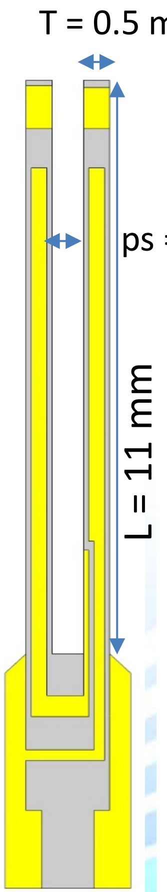
Straightforward approach for reducing support losses is designing QTFs having prongs with large length-to-thickness aspect ratio.

Z. Hao et al., "An analytical model for support loss in micromachined beam resonators with in-plane flexural vibrations," Sensor. Actuat. A-Phys., 109, 156, **2003**.

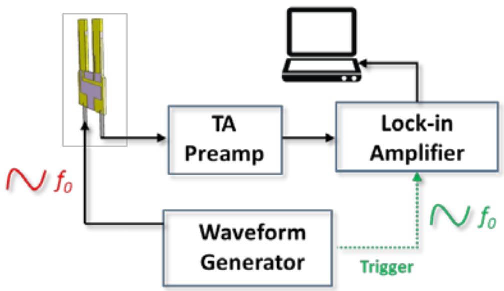
H. Hosaka , K. Itao, S. Kuroda, "Damping characteristics of beam-shaped micro-oscillators," Sensor. Actuat. A-Phys., 49, 87, **1995**.

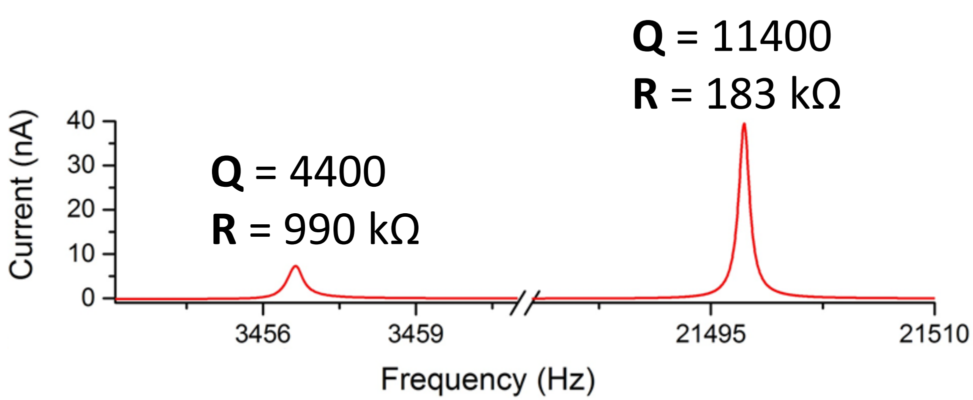
Quartz- tuning fork

Design and test



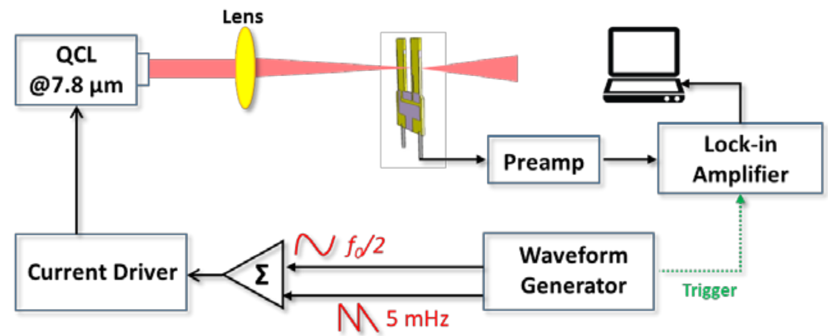
Electrical test

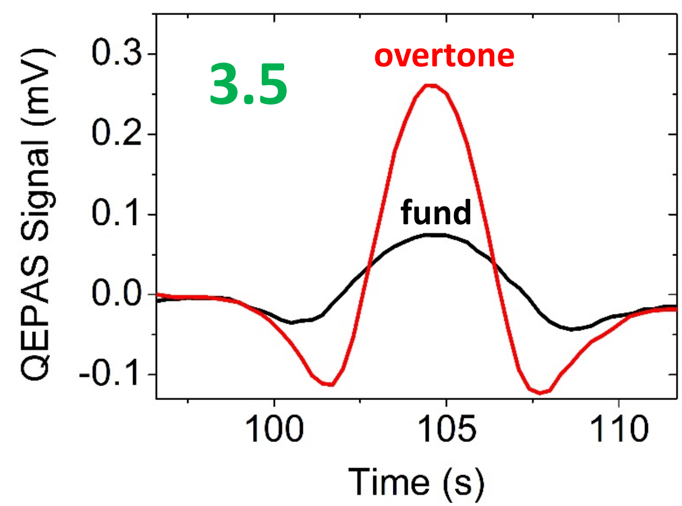




Optical test

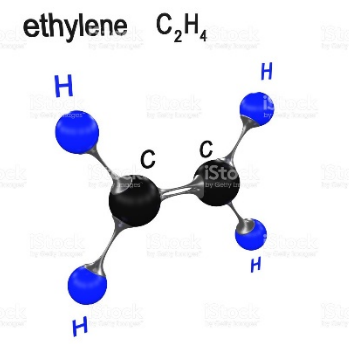
H_2O line @1296.49 cm^{-1}





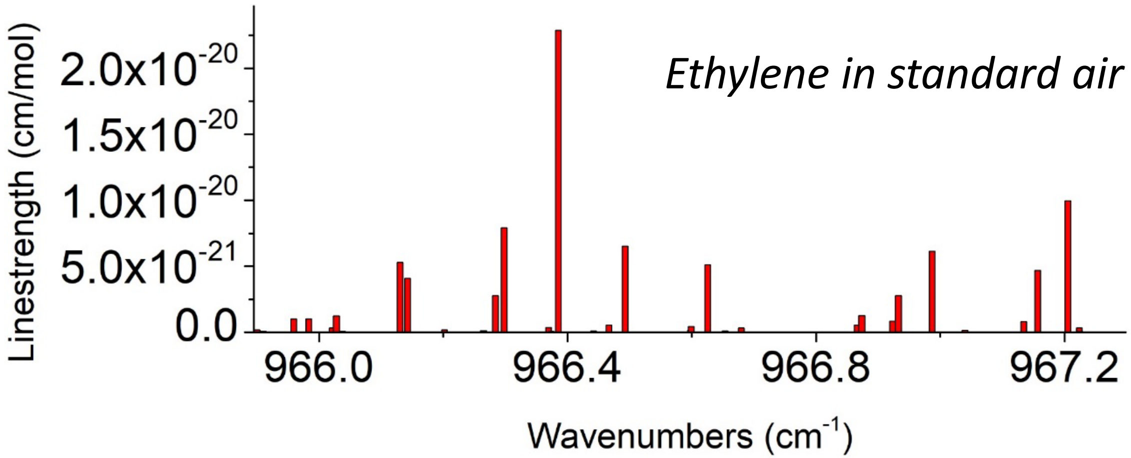
Ethylene detection

Motivations



- Colorless, odorless and flammable **hydrocarbon.**
- Produced by the **petrochemical industry** as a result of steam cracking
- In the **food industry**, measures quantity and the ripeness state of the fruit
- **Human breath analysis** (severity of oxidative stress and metabolic disturbances)

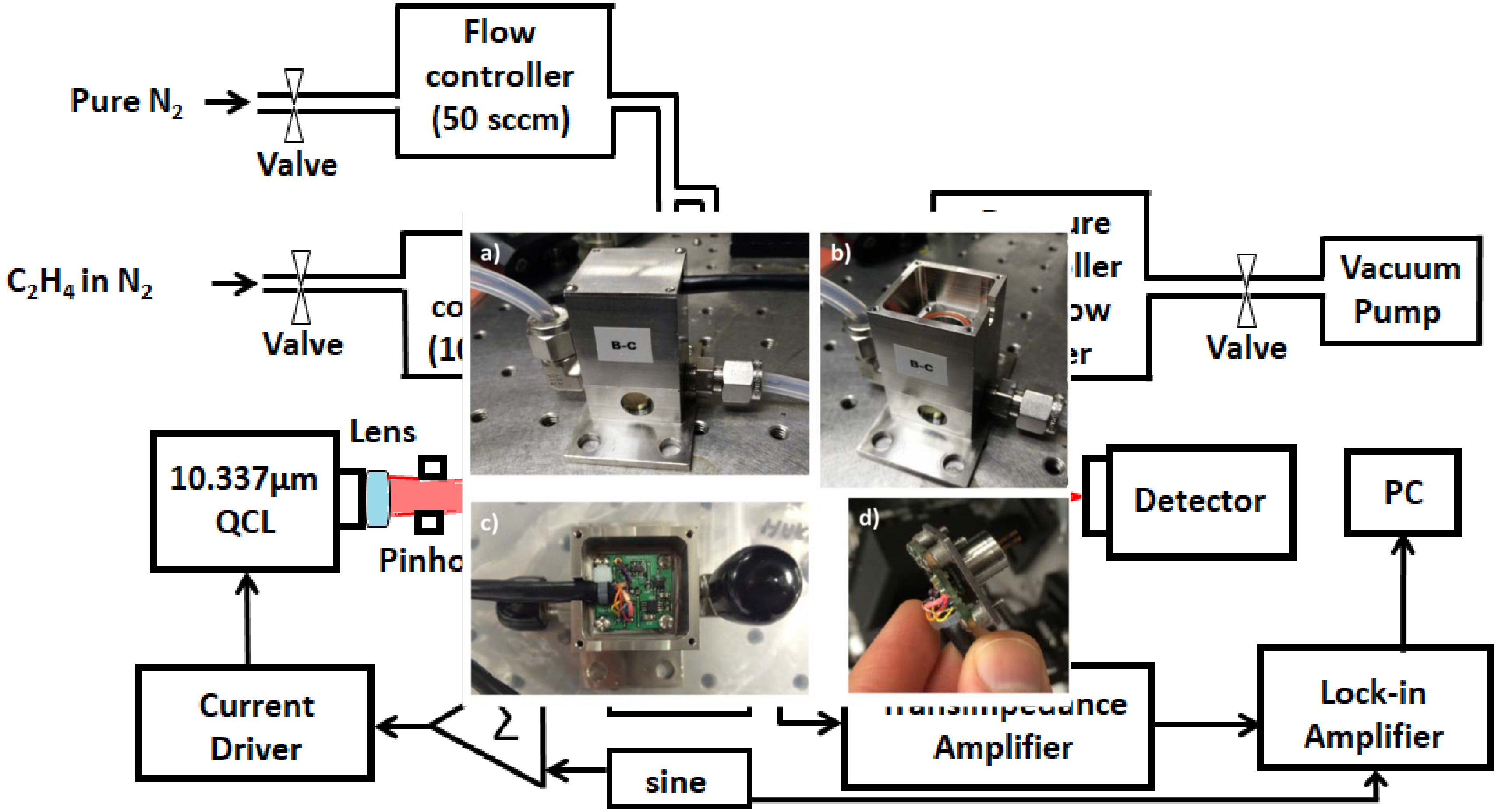
Where I can detect ethylene?



HITRAN Database

QEPAS Sensor

Experimental Setup



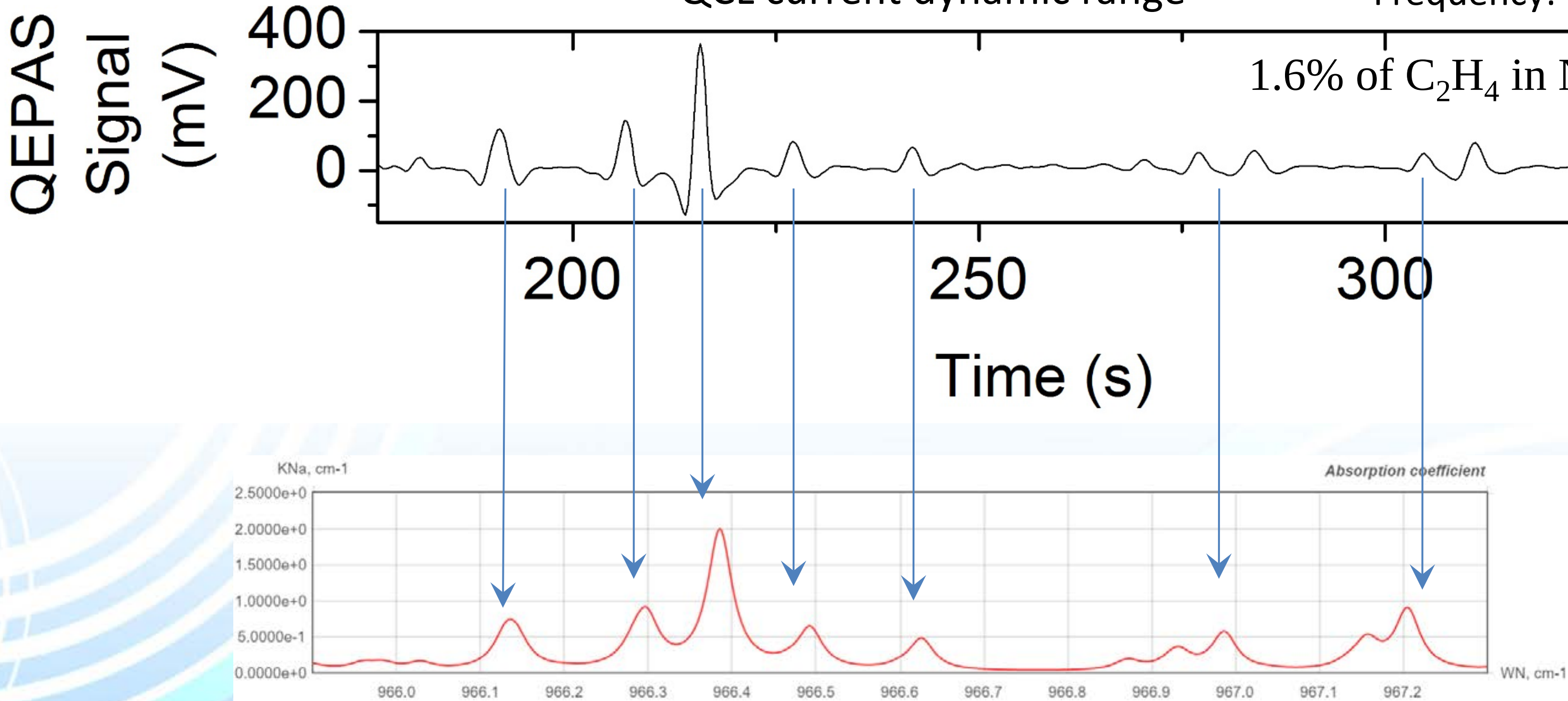
QEPAS Sensor

Preliminary investigation

Pressure: 120 Torr
Flow: 20.8 sccm

Modulation:
Amplitude: 23 mV
Frequency: 10751.59 Hz

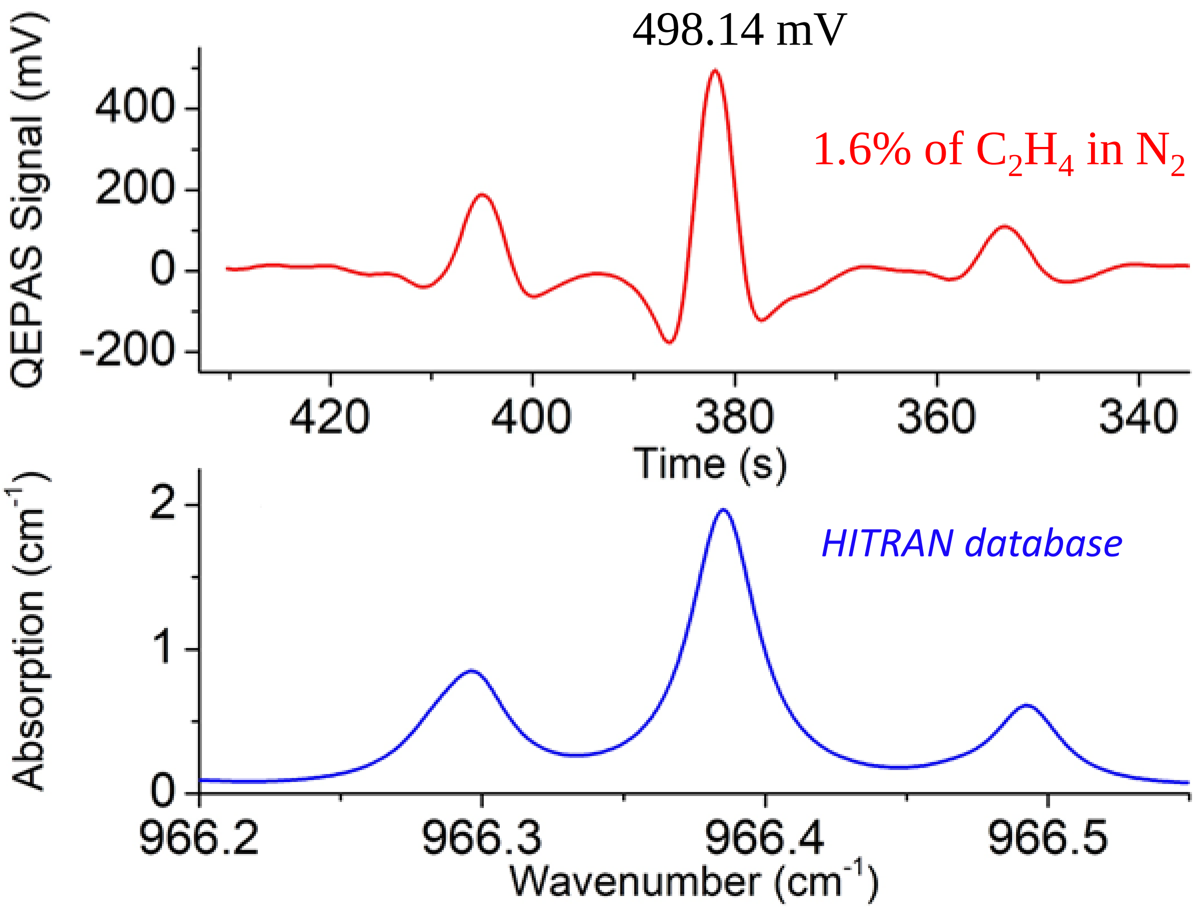
QCL current dynamic range



HITRAN database simulation

QEPAS Sensor

Preliminary investigation



Pressure: 120 Torr
Flow: 20.8 sccm

Selected absorption line
Wavenumber: 966.38 cm^{-1}
Linestrength : 2.2 10^{-20} cm/mol

Laser setpoints:
Temperature: 15°C
Current: 442.1 mA

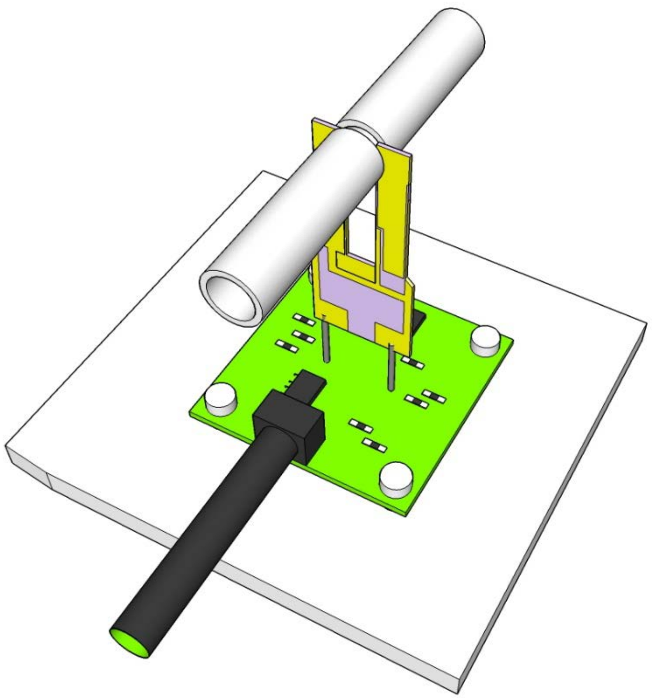
Modulation:
Amplitude: 23 mV
Frequency: 10751.59 Hz

Ramp:
Amplitude: 100 mV
Frequency: 5 mHz

Integration time: 100 ms

QEPAS Sensor

Micro-resonator tubes

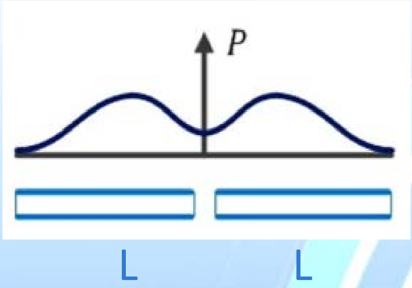


Internal Diameter

QTF frequency	prong spacing (ps) (mm)	ID (mm)	$\frac{ID}{ps}$
32.7 kHz	0.3	0.6	2
7.5 kHz	0.8	1.3	1.6
4.2 kHz	1.0	1.5	1.5

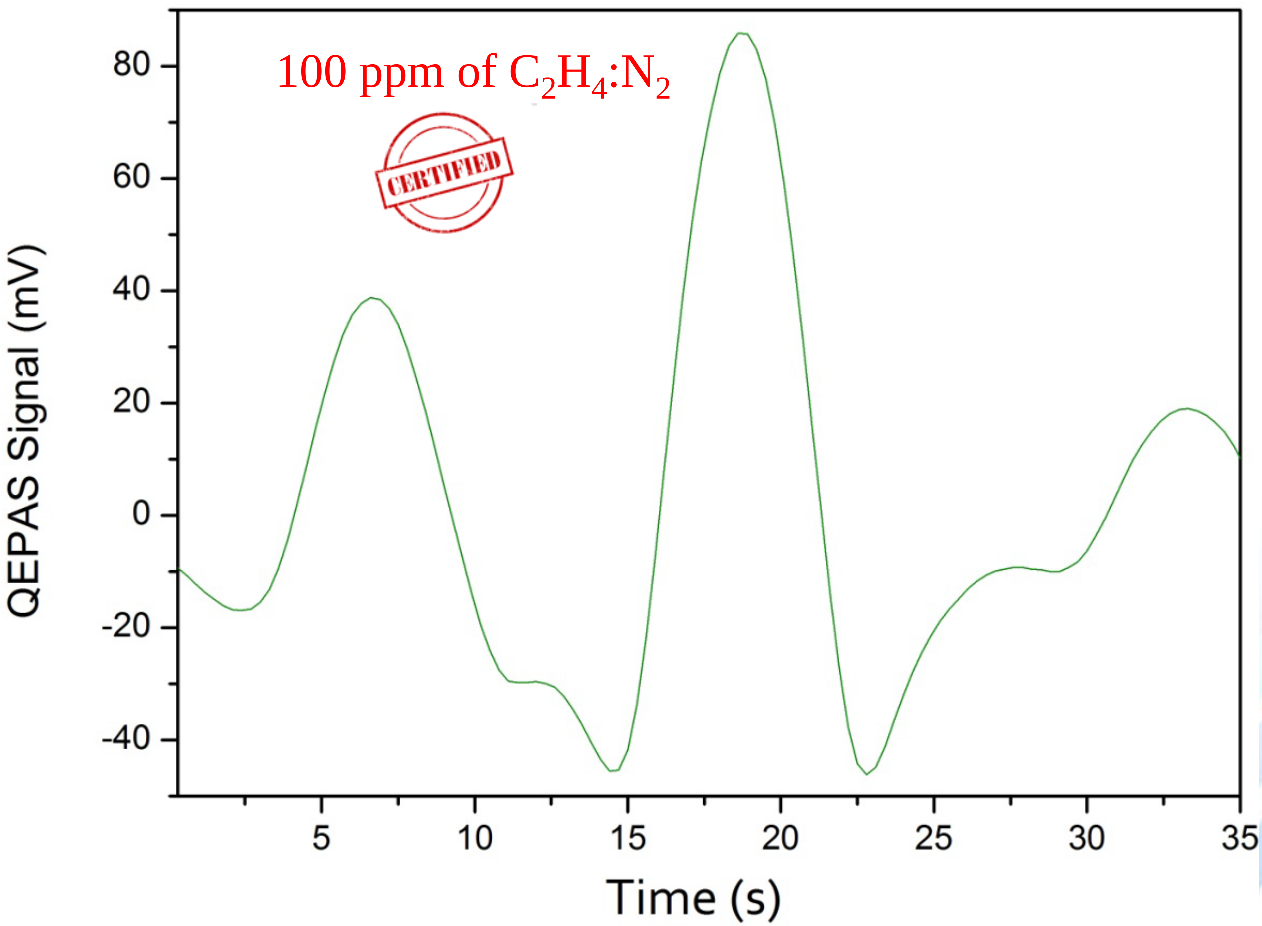
ps = 0.6 mm ⇒ ID = 0.85 mm

Tube length



$$\frac{\lambda}{4} \leq L \leq \frac{\lambda}{2} \Rightarrow L = \frac{\lambda}{4} = 4\text{mm}$$

QEPAS Sensor Results



Pressure: 120 Torr
Flow: 20.8 sccm

Selected absorption line
Wavenumber: 966.38 cm^{-1}
Linestrength : 2.2 10^{-20} cm/mol

Laser setpoints:
Temperature: 15°C
Current: 442.1 mA

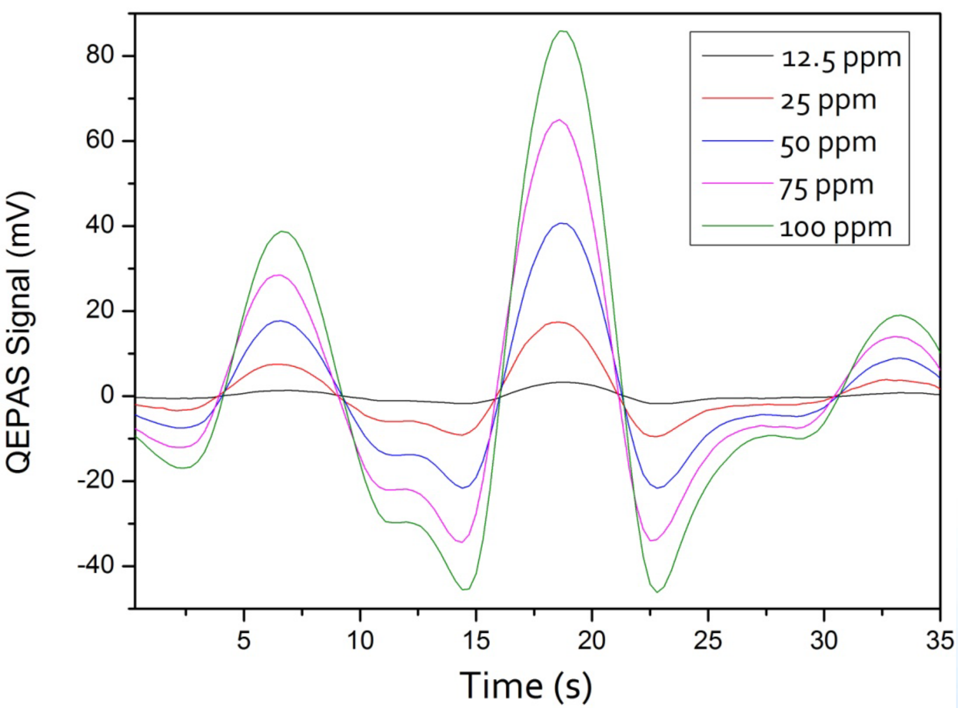
Modulation:
Amplitude: 23 mV
Frequency: 10751.59 Hz

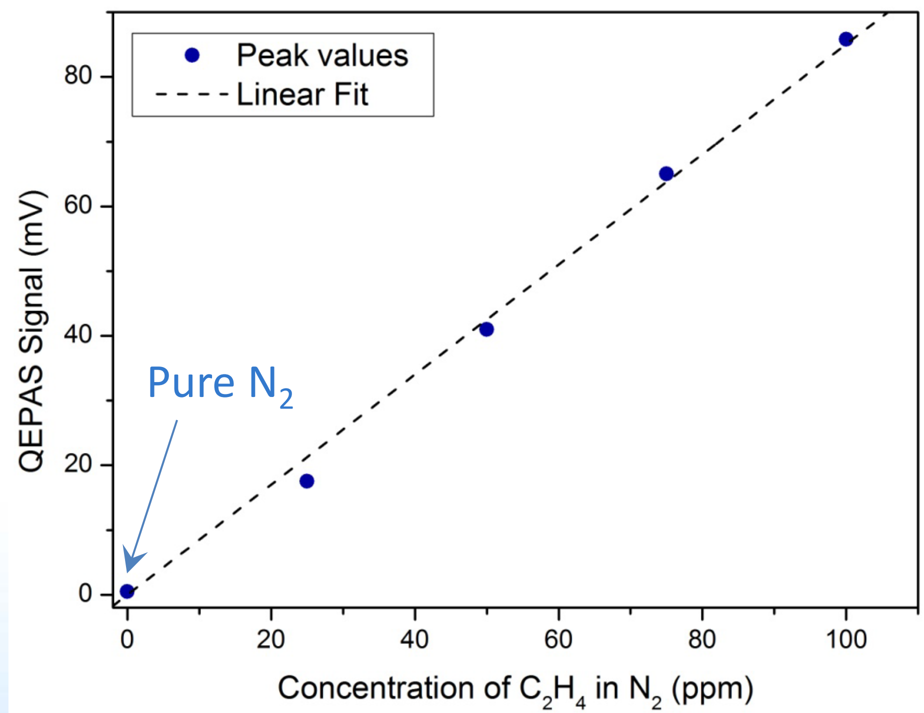
Ramp:
Amplitude: 100 mV
Frequency: 5 mHz

Integration time: 100 ms

QEPAS Sensor

Calibration





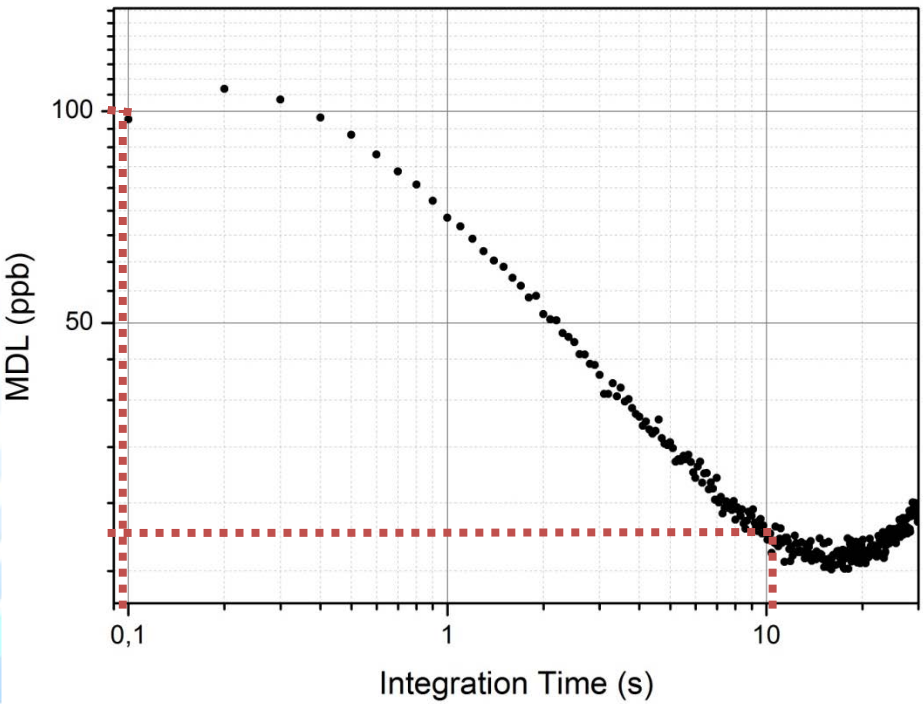
Linear response
Slope: 0.85 mV/ppm

Signal enhancement
X 27

QEPAS Sensor
Allan Deviation Analysis

@100 ms Integration time:
1σ-Noise: 0.086 mV
Peak signal: 85.84 mV

Signal to Noise Ratio (SNR): 1000
Minimum detection limit (MDL): 100 ppb

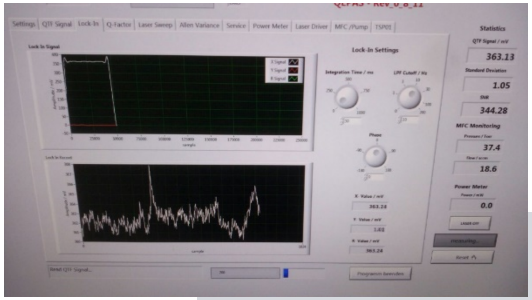


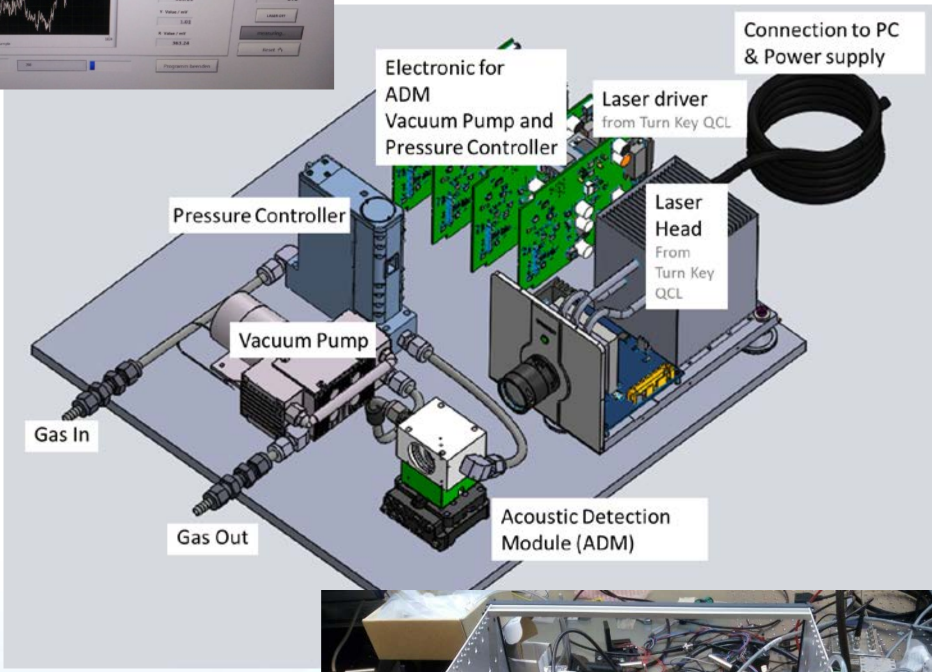
@10 s Integration time:
1σ-Noise: 0.026 mV

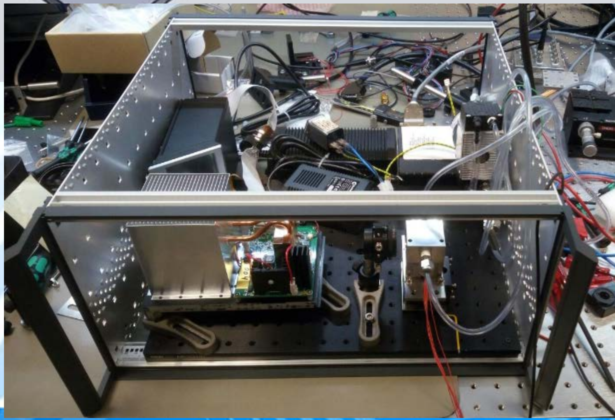
MDL: 30 ppb

QEPAS Sensor

Let's compact it!







Visit us at:

Thorlabs

Booths: 707-807

Conclusions

- **High-Q** QTF operating at **overtone mode**
- QEPAS Sensor for **ethylene** detection
- Detection limit of **30 ppb** at 10 s integration time

Future Perspectives

- New generation of QTF
- Single-tube micro-resonator system