UNIVERSITÀ DEGLI STUDI DI BARI ALDO MORO

DIPARTIMENTO INTERATENEO DI FISICA "M. MERLIN"

PhD SCHOOL OF PHYSICS XXXI CYCLE

Second year report

INNOVATIVE SPECTROSCOPIC TECNIQUES FOR GASEOUS TRACES DETECTION

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PolySenSe

OUTLINE

- **2° year PhD Research activities:**
  - **Quartz Enhanced Photoacoustic Spectroscopy employing custom Quartz Tuning Forks**
  - **Optical coupling of tapered Hollow-Core Waveguides with interband and quantum cascade lasers in the mid-infrared spectral range**
- **3° year PhD goals**
- **List of publications, conference proceedings and talks**

1- Study, realization and characterization of QEPAS gas sensors employing custom QTFs

- **Ethylene detection at sub-ppm concentrations;**

- **Detection of a set of absorption lines of methane and nitrous oxide employing a broad band laser source;**

- **New spectrophone configuration: single-tube on beam (SO-) QEPAS.**

2- Study of the coupling conditions of tapered Hollow-core waveguides with laser sources in the mid-infrared spectral range and experimental demonstration of low-loss single mode laser beam delivery within the range 3.5 - 7.8 µm.

RICE

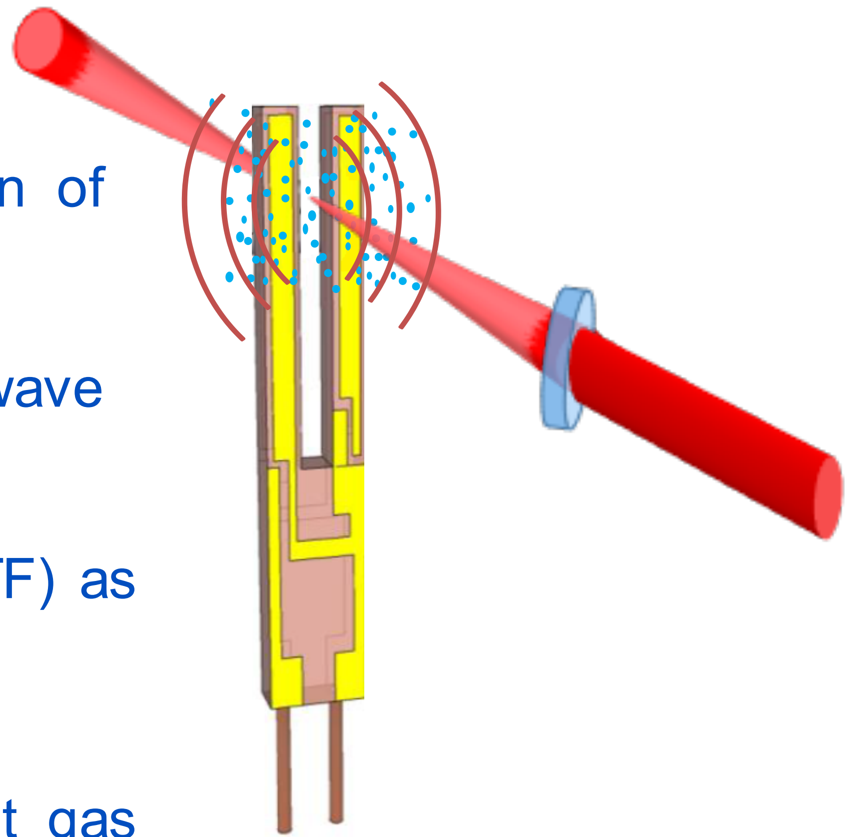
PENDAR TECHNOLOGIES

OptoKnowledge

Quartz Enhanced PhotoAcoustic Spectroscopy (QEPAS)

- Target gas excited by laser source
- Photoacoustic effect: heat conversion of light absorbed by target

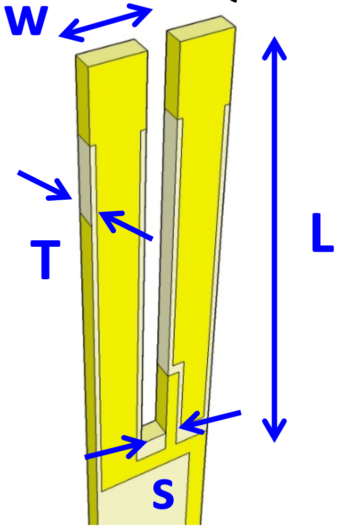
  laser modulation → pressure wave generation



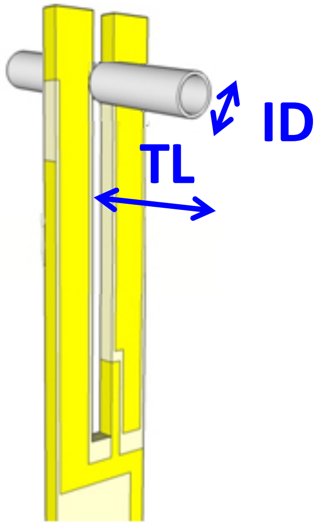
- Piezoelectric Quartz Tuning Fork (QTF) as acousto-electrical transducer
- Signal intensity proportional to target gas concentration

QEPAS spectrophones

Bare QTF



Dual-tube



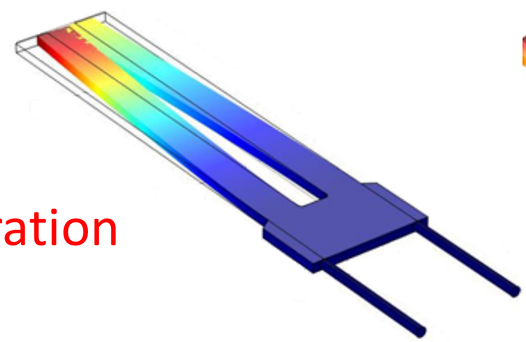
Standard QTF: L = 3 mm; w = 0.35 mm; T = 0.34 mm; s = 0.3 mm.

- Dual-tube spectrophone configuration ID = 0.6 mm; TL = 4.4 mm; Tube-QTF distance = 30 µm

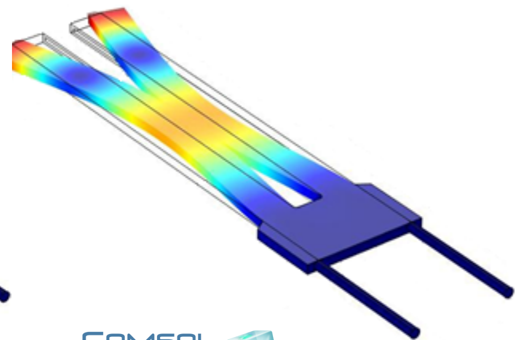
Custom QTFs:

- Lower resonance frequency
- Higher Quality factor
- Single-tube spectrophone configuration
- Enhance the first overtone mode

Fundamental mode



Overtone mode

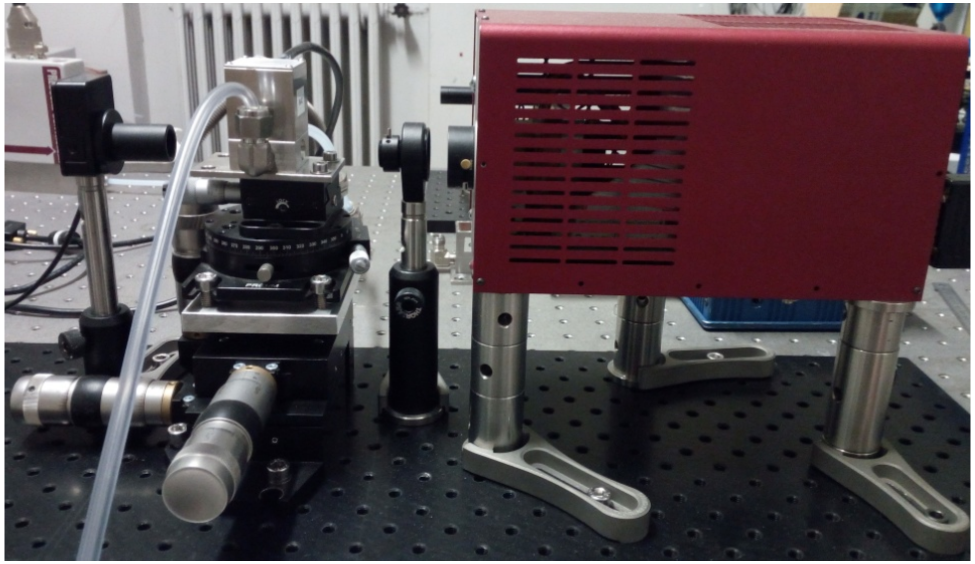


Dong, Lei, et al *Applied Physics B: Lasers and Optics* 100.3 (2010): 627-635.

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

Ethylene (C_2H_4): one of the most basic hydrocarbon chemical building blocks. Processing chemical plants turn it into polyethylene, polyester, PVC, polystyrene, ethyline glycole. Ethylene detection is fundamental for even rising demand of this gas.

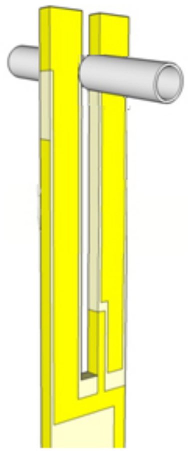
THORLABS QLC
Emission @ 10,33µm
Optical Power = 68 mW



C_2H_4 absorption line
Wavenumber: 966.38 cm^{-1}
Linestrength : 2.2 10^{-20} cm/mol

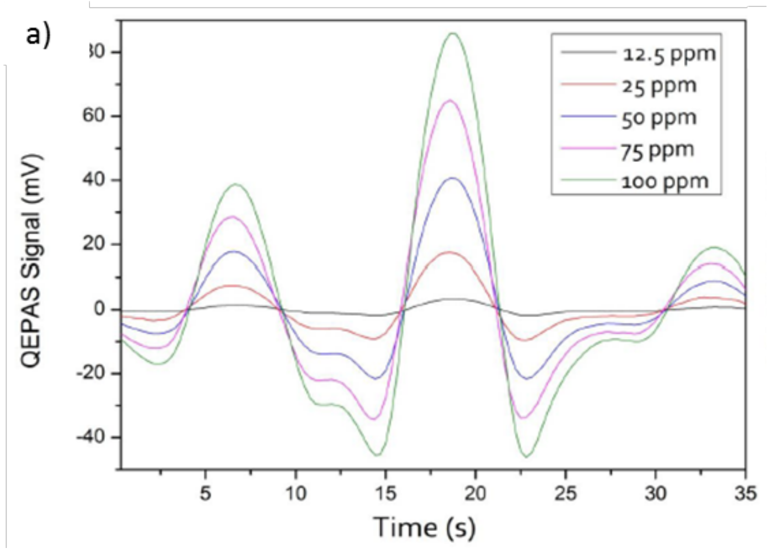
Sensor uses a2nd gen. QTF coupled with 2 micro-resonator tubes

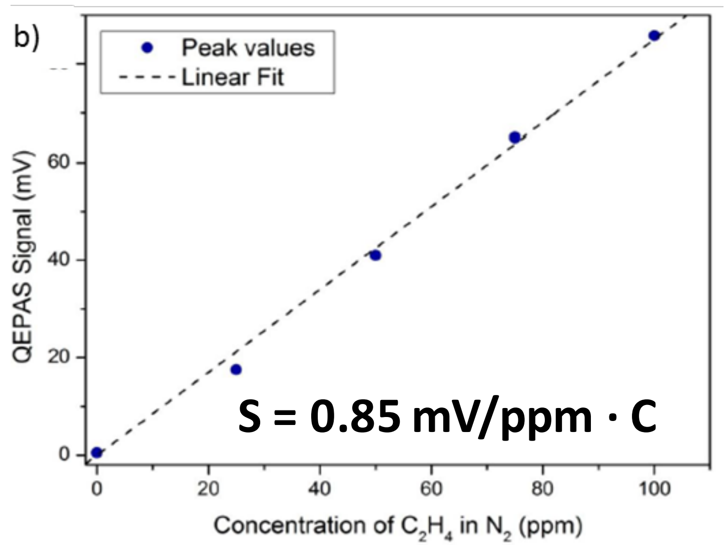
f: 21503 Hz
Q factor: 23900



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

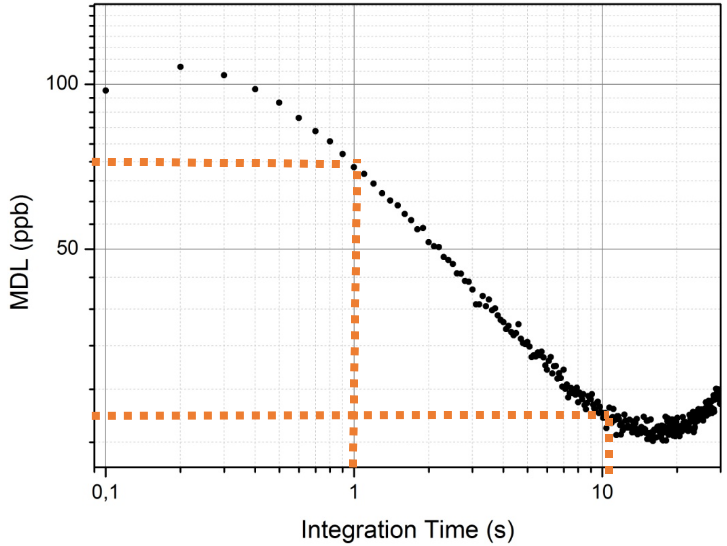
Sensor calibration





Minimum Detection Limit

@1 s Integration time
Noise level: 50 µV
Minimum Detection Level: 70 ppb in 1 sec
(State-of-Art 1.7ppm @ 1.6 µm)
NNEA: 2.36×10^{-8} $cm^{-1}W/\sqrt{Hz}$
(State-of-Art 5.4×10^{-9} $cm^{-1}W/\sqrt{Hz}$ @ 1.6 µm)



Quartz-enhanced photoacoustic gas sensor employing a broad-band laser source in pulsed operation

32 DFB QCLs array

Emission range: 1200-1310 cm^{-1}

Linewidth of each QCL emission: ~ 1.5 cm^{-1}

Frequency spacing between consecutive QCL emitters: ~ 4 cm^{-1}

Custom QTF @750 Torr

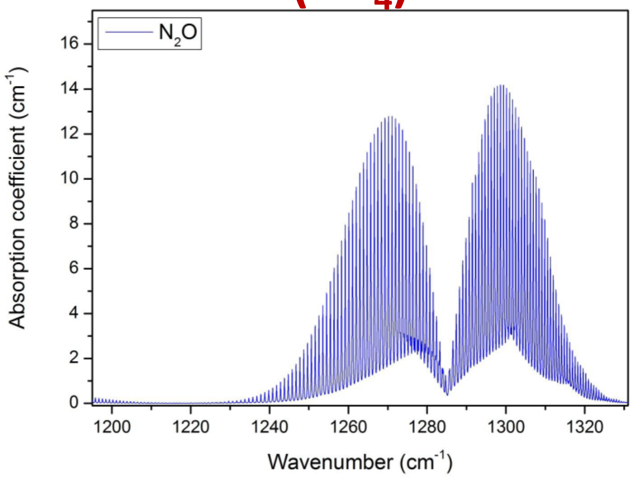
F: 25400 Hz (first overtone mode)

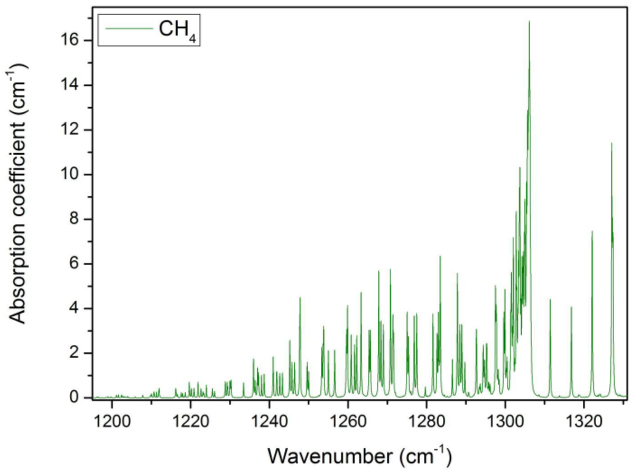
Q factor: 28940

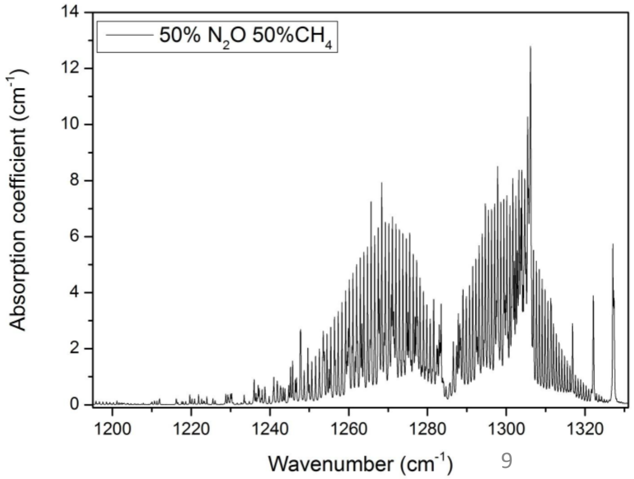
Gas target

Nitrous Oxide (N_2O)

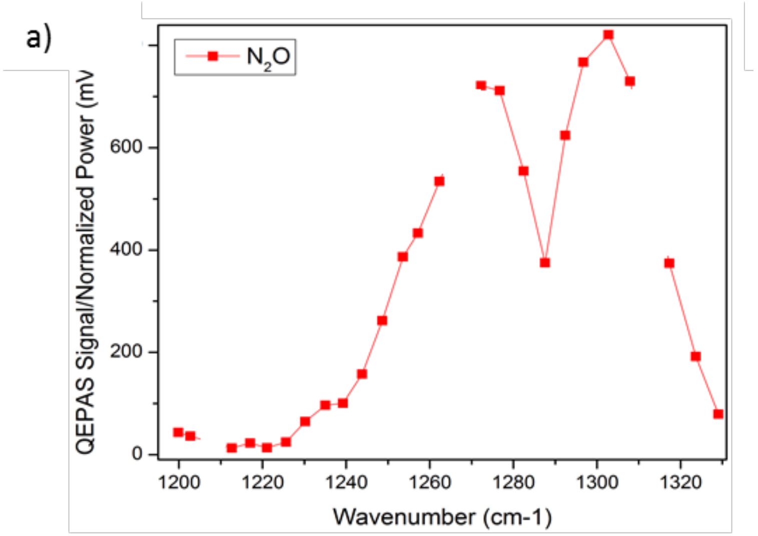
Methane (CH_4)



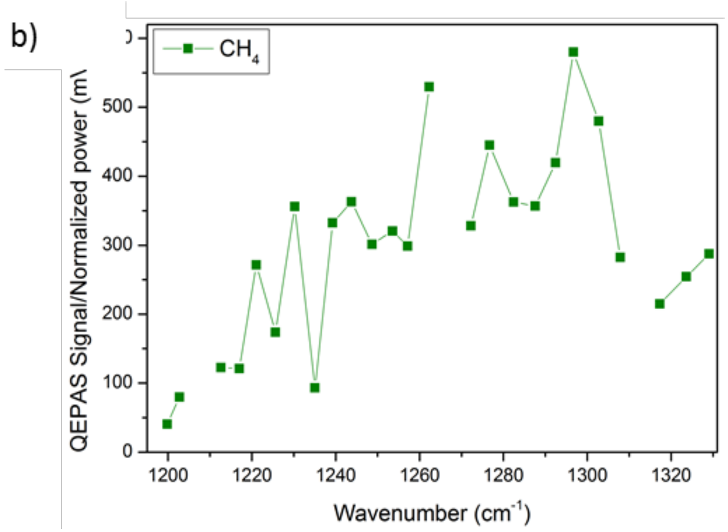




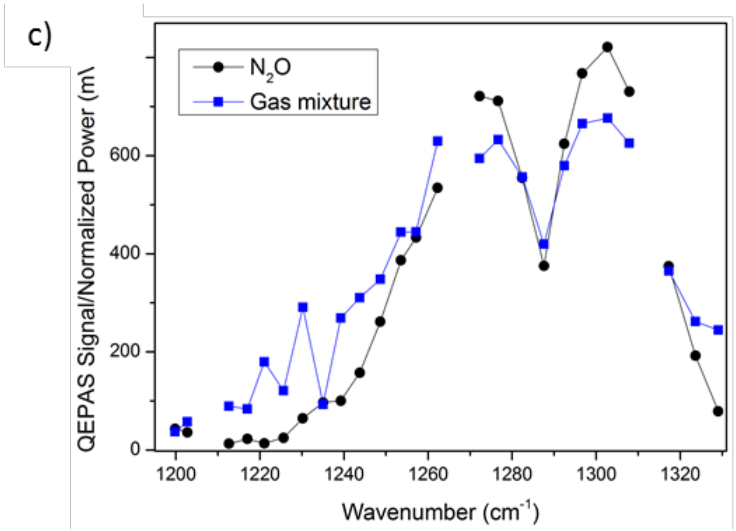
Quartz-enhanced photoacoustic gas sensor employing a broad-band laser source in pulsed operation



Envelope of N_2O spectral lines well reproduced



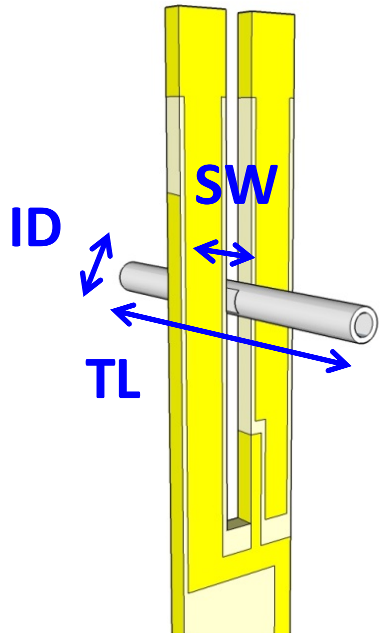
Peaks related to CH_4 absorption lines



Some CH_4 peaks clearly appear over the N_2O band

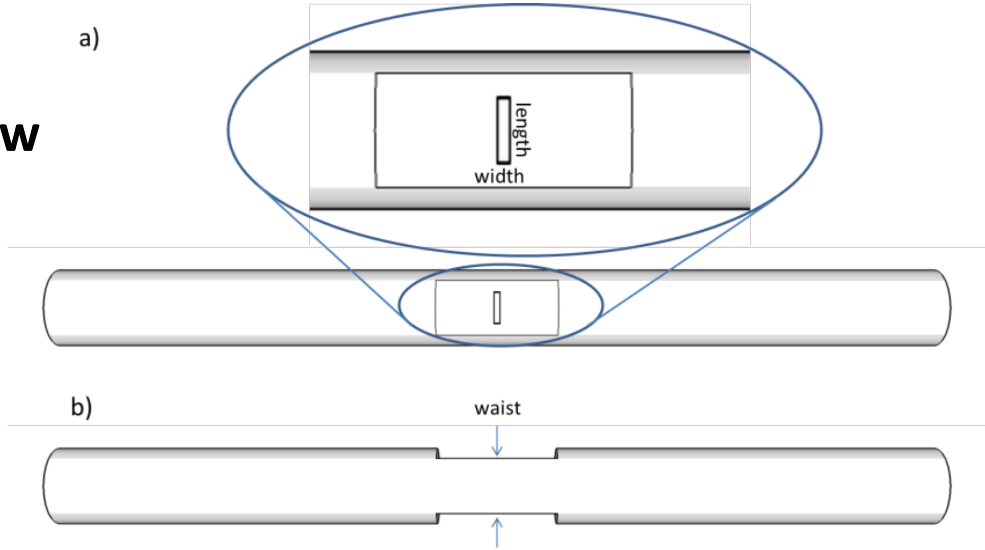
Single-tube on beam quartz-enhanced photoacoustic spectrophones exploiting a custom QTF operating in the overtone mode

QEPAS measurements with QTF operating with the First Overtone mode (F: 25400 Hz, Q factor: 28940) and a Single-Tube set at 12 mm from the top in On-Beam configuration (SO-QEPAS)



Front view

Top view

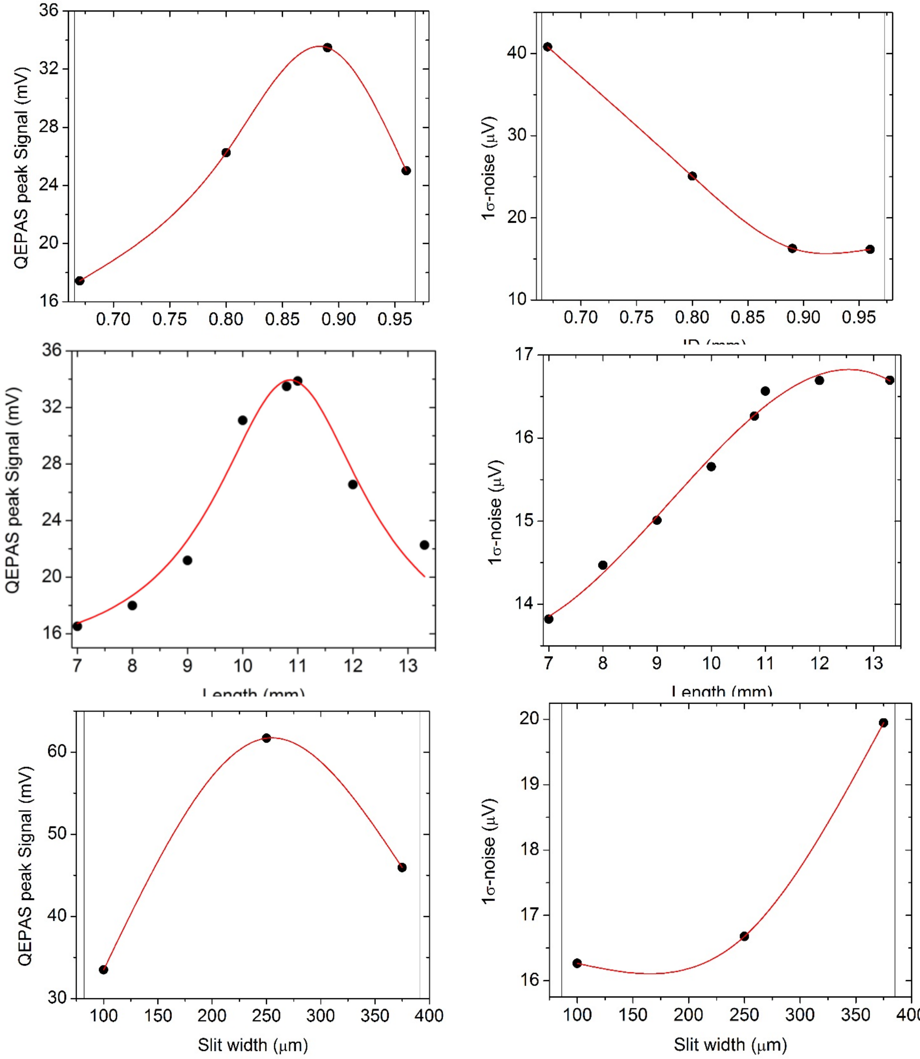


Polished waist for outer diameter larger than the prong spacing

Patimisco, P, et al., *Advances in Physics: X* 2(1), pagg 169-187 (2017)

Single-tube on beam quartz-enhanced photoacoustic spectrophones exploiting a custom QTF operating in the overtone mode

Investigation on the influence of the tube Internal Diameter, Length and Slit with on the QEPAS signal



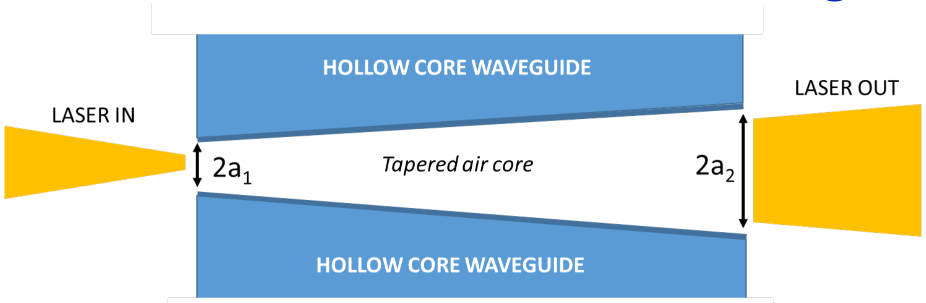
ID = 0.88 mm
TL = 11 mm
SW = 250 μm

SNR enhancement with respect to the bare QTF: x32

Low-loss and single-mode tapered hollow-core waveguide optically coupled with interband and quantum cascade lasers

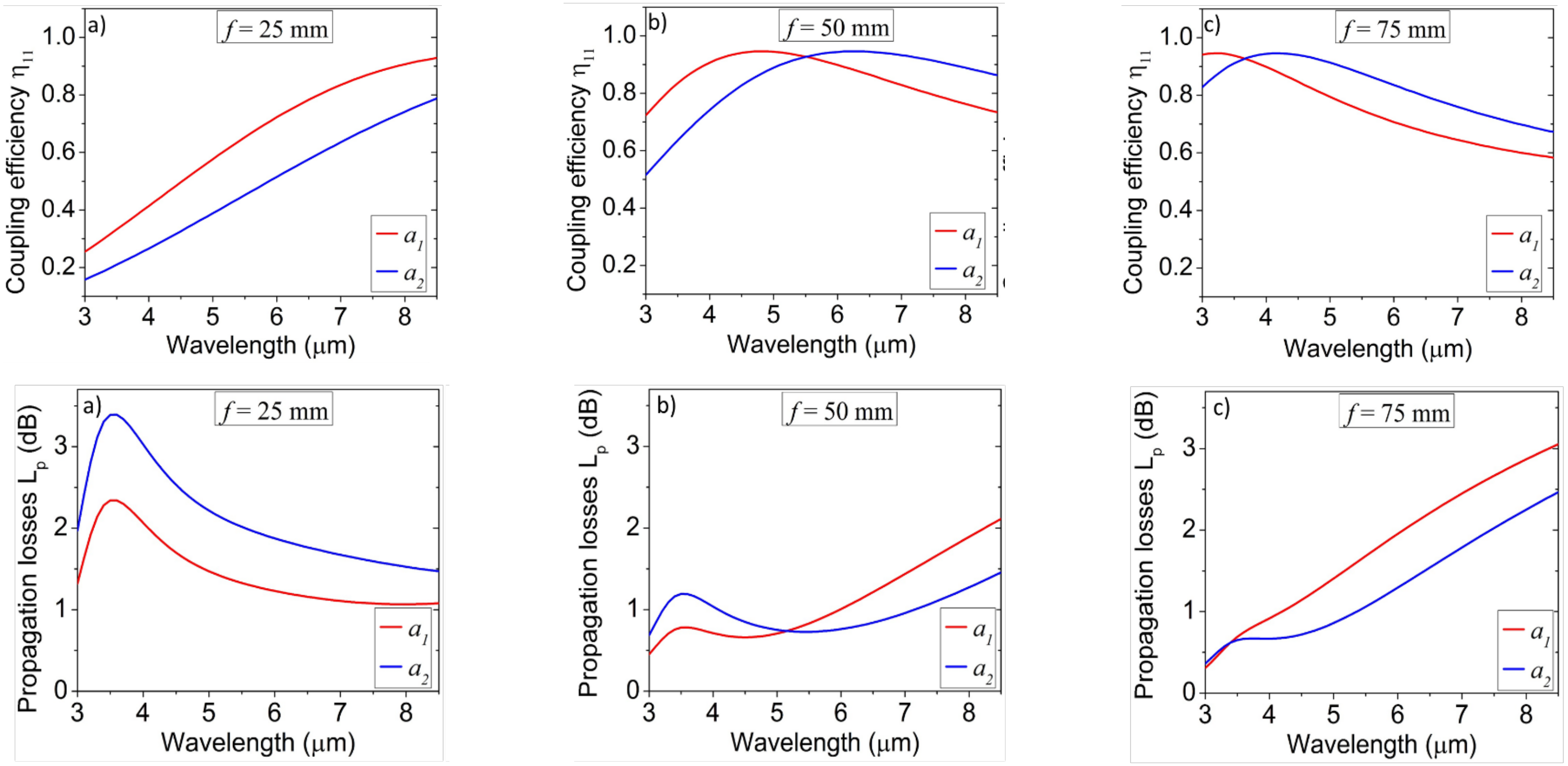
Theoretical modeling for best coupling conditions

$$\eta_{11} = \frac{\left| \int_0^a G(r) J\left(u_{11}\frac{r}{a}\right) r dr \right|^2}{\int_0^a G^2(r) r dr \int_0^a J^2\left(u_{11}\frac{r}{a}\right) r dr}$$



$$L_p(dB) = -10 Log_{10}\left(\sum_m \eta_{1m} \exp\left(-2\int_0^L \alpha_{1m}(z)dz\right)\right)$$

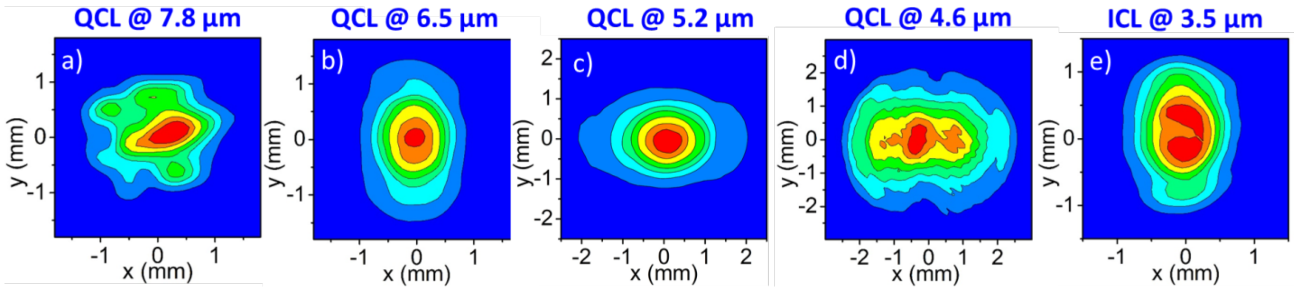
L = 50 cm; a_1 = 130µm; a_2 = 130µm.



Low-loss and single-mode tapered hollow-core waveguide optically coupled with interband and quantum cascade lasers

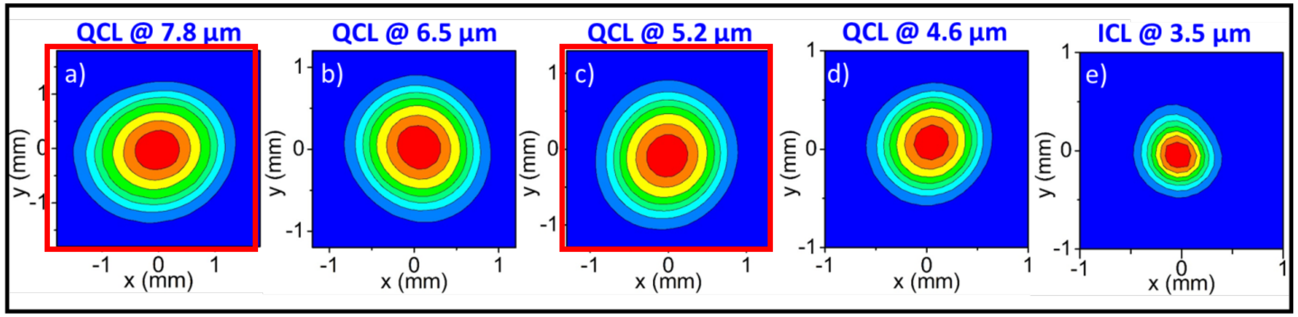
Experimental results

Beam profile at laser exit

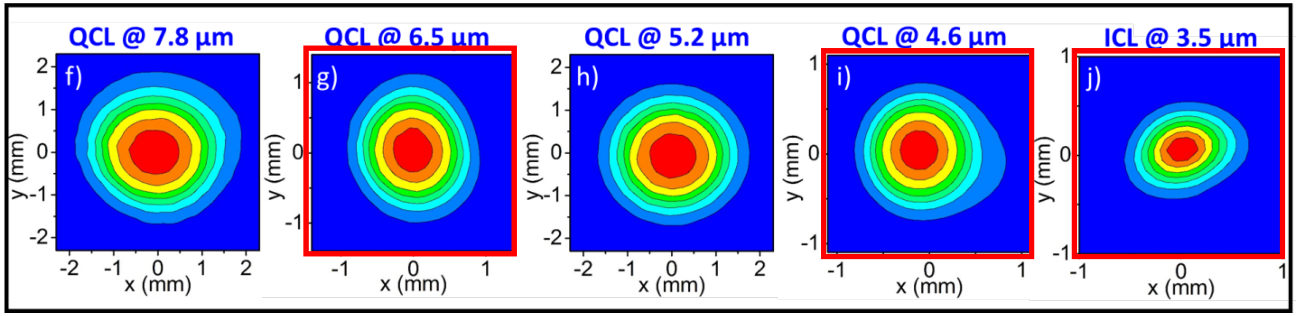


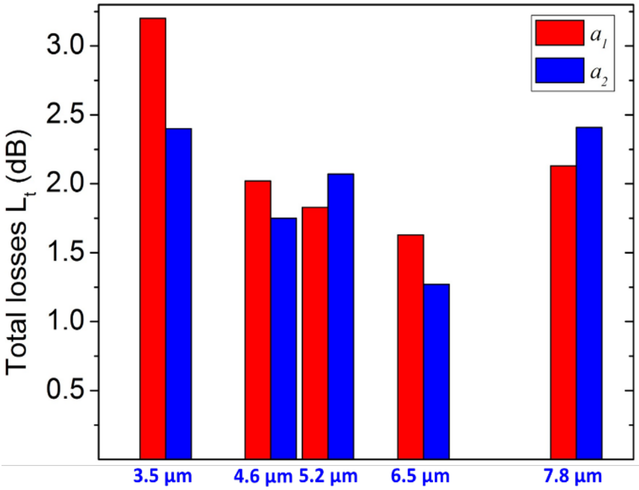
Beam profile at HCW exit and propagation losses

100-130



130-100



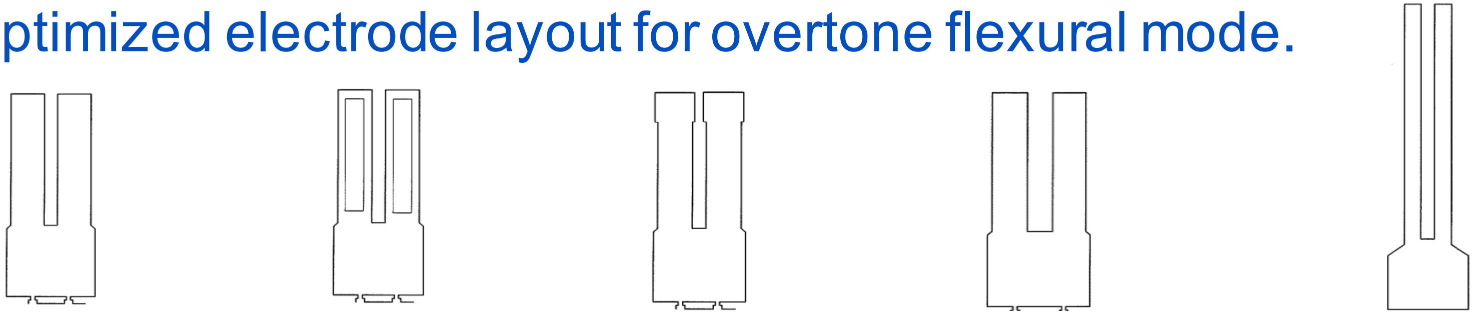


Single-mode output in the 3.5- .8 µm spectral range with minimum losses of 1.27 dB at 6.2 µm

Giglio, M. et al. Optical Engineering, 57(1), p.011004 (2017)

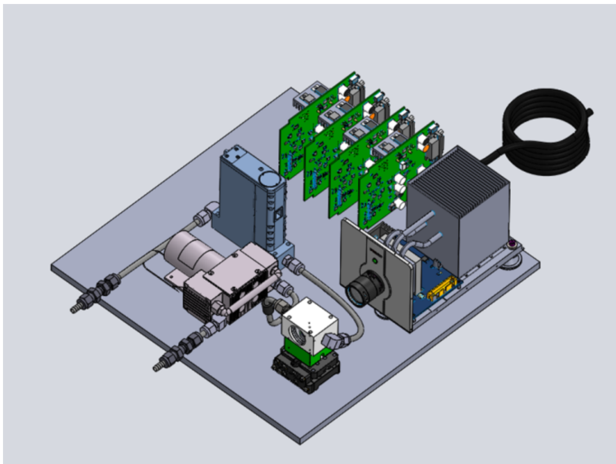
3° year PhD goals

- Design and realization of custom quartz tuning forks, aiming at:
  1) reduce the resonance frequency;
  2) keep high the Q-factor;
  3) optimized electrode layout for overtone flexural mode.



(prongs spacing 0,8 mm) (prongs spacing 0,8 mm, groove) (prongs spacing 0,8 mm, Top enlarged) (prongs spacing 1,5 mm) (prongs spacing 0,7 mm, octupole configuration)

- Development of a compact version of the Ethylene QEPAS sensor, implementing an ADM equipped with a new generation QTF and a single micro-resonator tube for PW2018.



List of publications

1) **Giglio, M.**, Patimisco, P., Sampaolo, A., Kriesel, J.M., Tittel, F.K. and Spagnolo, V. Low-loss and single-mode tapered hollow-core waveguides optically coupled with interband and quantum cascade lasers. Optical Engineering, 57(1), p.011004 (2017).

Conference proceedings

1) **Giglio, M.,** Sampaolo, A., Patimisco, P., Zheng, H., Wu, H., Dong, L., Tittel, F.K. and Spagnolo, V., Single-tube on beam quartz-enhanced photoacoustic spectrophones exploiting a custom quartz tuning fork operating in the overtone mode, SPIE OPTO, 2017
2) Sampaolo, A., Patimisco, P., Gluszek, G., Hudzikowski, A., **Giglio, M.**, Zheng, H., Tittel, F.K. and Spagnolo, V., Low power consumption quartz-enhanced photoacoustic gas sensor employing a quantum cascade laser in pulsed operation, SPIE OPTO, 2017

Conference talks

1) Tittel, F.K., Spagnolo, V., Patimisco, P., **Giglio, M.**, Sampaolo, A., Ye, W., He, Q., Zheng, H., and Lou, M., Recent Advances and Applications of Mid-infrared Cavity and Quartz Enhanced Photoacoustic Spectroscopy, Mirsens, 2017
2) Sampaolo, A., Patimisco, P., Zheng, H., **Giglio, M.**, Dong, L., Tittel, F.K. and Spagnolo, V., Recent Advances In Quartz-Enhanced Photoacoustic Sensors Employing Custom Tuning Fork Operating At The First Overtone Flexural Mode, CLEO Europe, 2017
3) Spagnolo, V., Sampaolo, A., Patimisco, P., Zheng, H., **Giglio, M.**, Dong, L., Tittel, F.K., New developments in quartz enhanced photoacoustic gas sensing, Freiburg Infrared Colloquium, 2017
4) Spagnolo, V., Sampaolo, A., Patimisco, H., **Giglio, M.**, Tittel, F.K., Quartz-enhanced photo-acoustic spectroscopy with QCLs, International Training School - Beyond Conventional Tissue Imaging, COST 2017