Recent advances in quartz-enhanced photoacoustic sensing

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SPIE. PHOTONICS WEST 2018

OUTLINE

- Quartz Enhanced Photo-Acoustic Spectroscopy (QEPAS): basics and merits
- 2nd generation custom QTFs
  a) QTFs 1st overtone flexural mode
  b) Dual-gas QEPAS sensor
- 3rd generation of custom QTFs
  - Preliminary results
- PolySense Joint-research Lab.

Future Directions and Conclusions

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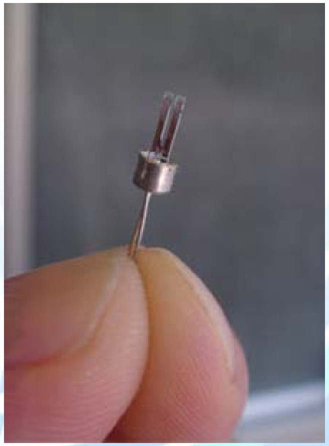
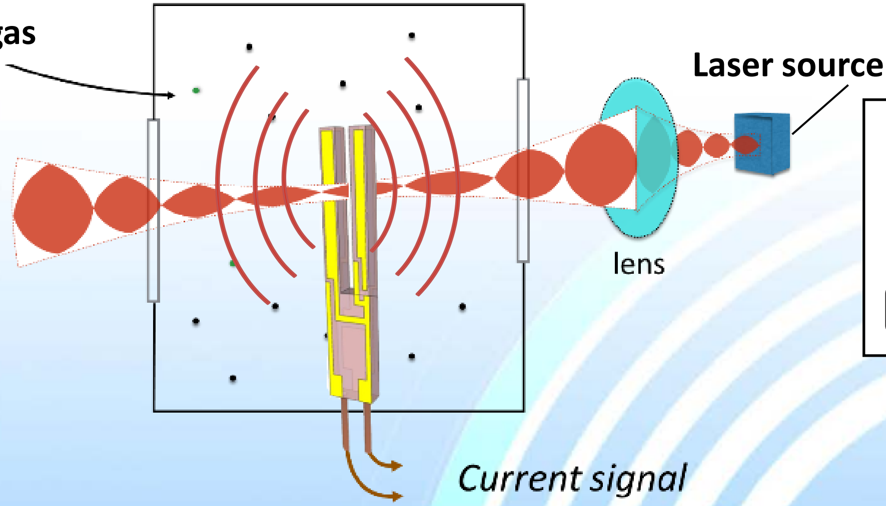
Quartz-Enhanced Photoacoustic Spectroscopy
Introduction and Basic Operation

- Optical radiation is focused between the prongs of a quartz tuning fork
- Trace gases absorb optical energy at characteristic frequencies
- A pressure (sound) wave is generated by modulating the laser power
- Resonant mechanical vibration is excited by the sound waves
- The mechanical vibration is converted to an electrical signal via the piezoelectric effect
- The trace gas concentration is proportional to the electrical signal





P. Patimisco A. Sampaolo, L. Dong, F.K. Tittel, V. Spagnolo, Applied Physics Review, in press, **2018**.

P. Patimisco A. Sampaolo, H. Zheng, L. Dong, F.K. Tittel, V. Spagnolo, Advances in Physics X 2, 169-187, **2016**.

P. Patimisco, G. Scamarcio, F.K. Tittel and V. Spagnolo,, Sensors 14, 6165-6206, **2014**.

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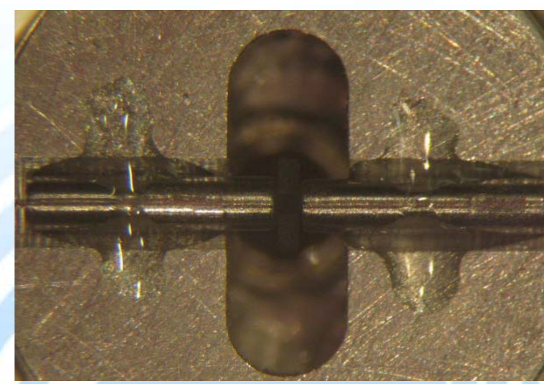
Quartz-Enhanced Photoacoustic Spectroscopy

Merits and main characteristics

- Small sensing module and sample volume (a few cm^3)
- Wavelength independent
- ***Optical detector is not required***
- Wide dynamic range (from % to ppt)
- Immune to environmental acoustic noise
- Acoustic micro-resonator(s) to enhance the QEPAS signal

- Sensitivity scales with laser power
- Cross sensitivity issues
- Alignment (no light must hit the QTF or micro-resonators)
- Responsivity depends on the molecular energy transfer processes



Record sensitivity: **50 part-per-trillion**

$\lambda = 10.54\ \mu m$ (mid – IR), SF_6

P. Patimisco, et al., Applied Physics Review, in press, **2018**.

P. Patimisco et al., Sensors 14, 6165, **2014**

V. Spagnolo et al., Optics Letters, 37, 4461–4463, **2012**.

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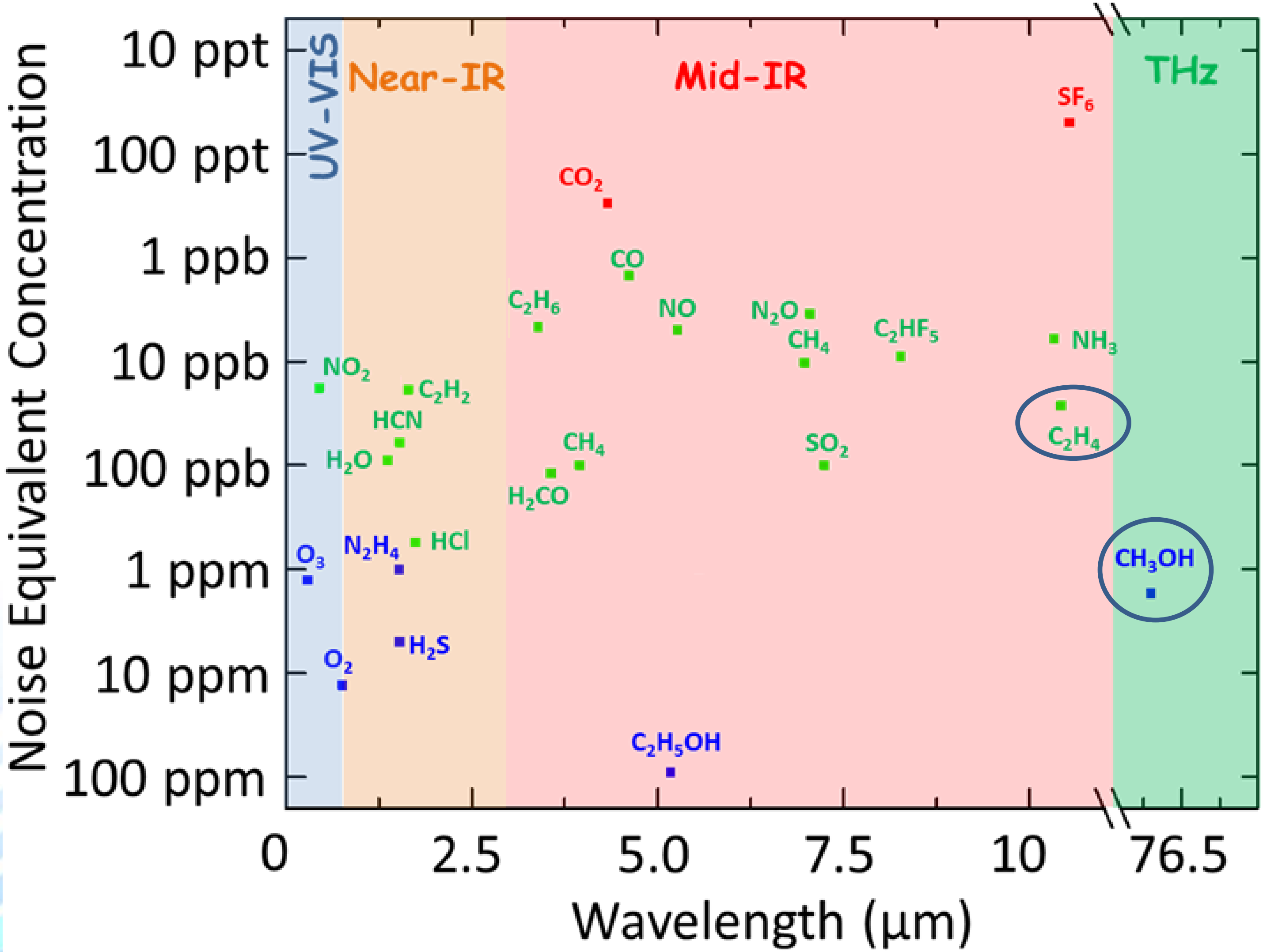
QEPAS gas sensing performance



P. Patimisco, et al., Applied Physics Review, in press, **2018**.

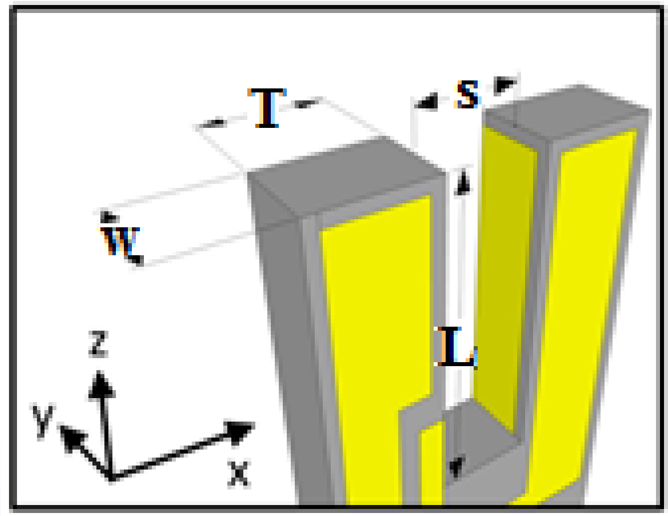
P. Patimisco et al., "Sensors, 14, 6165–6206, **2014**.

Quartz tuning fork Physics

Free motion conditions: Euler-Bernoulli equation

$$EI\frac{\partial^4 y(x,t)}{\partial x^4} + \rho A\frac{\partial^4 y(x,t)}{\partial t^4} = 0$$

Resonance frequencies ⇨ $f_n = \frac{\pi}{8\sqrt{12}} \cdot \frac{T}{L^2} \cdot n^2 \sqrt{\frac{E}{\rho}}$



QEPAS signal: $S \propto P\alpha Q\varepsilon$

Quality factor: $Q = f_n / \Delta f_{n\,FWHM}$

Piezoelectric signal: $I = a\frac{dx}{dt} = \frac{V}{R}$

Fork constant: $a = 3d_{11}E \cdot \frac{Tw}{L}$

Objective: Design of a tuning fork optimized for QEPAS sensing applications

P. Patimisco, et al., Applied Physics Review, in press, **2018**.

P. Patimisco et al., Sensors and Actuators B Chemical, 227, 539–546, **2016**.

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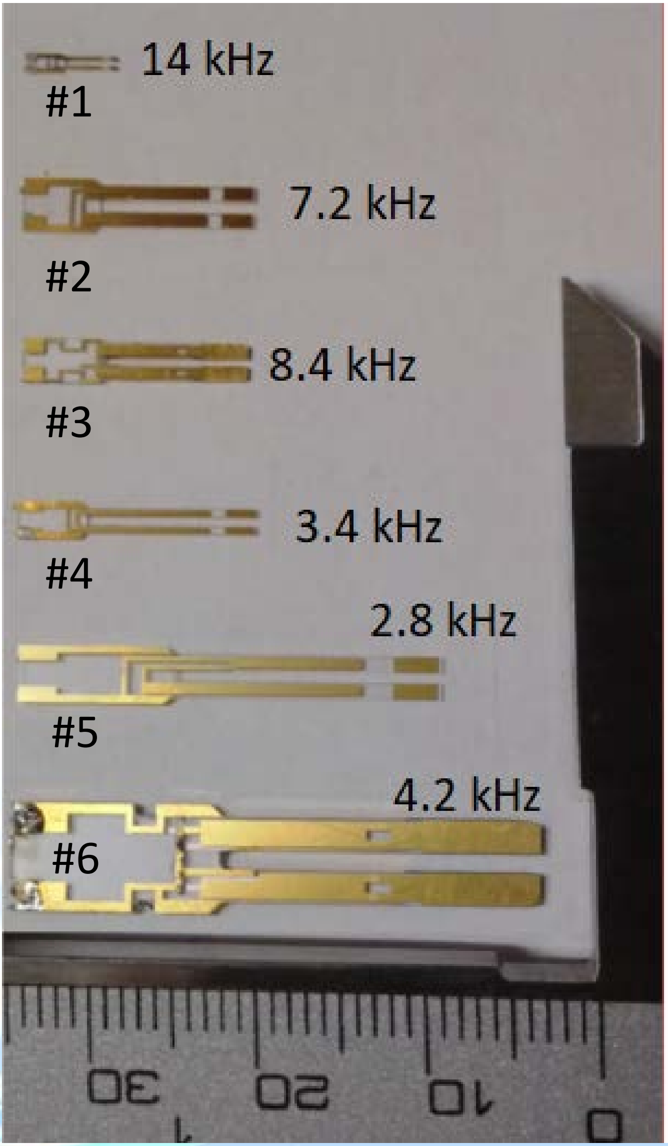
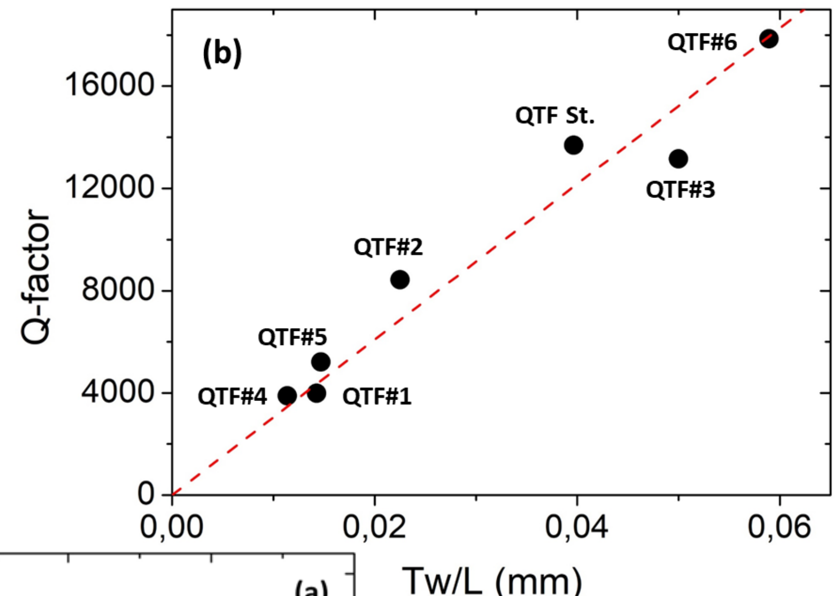
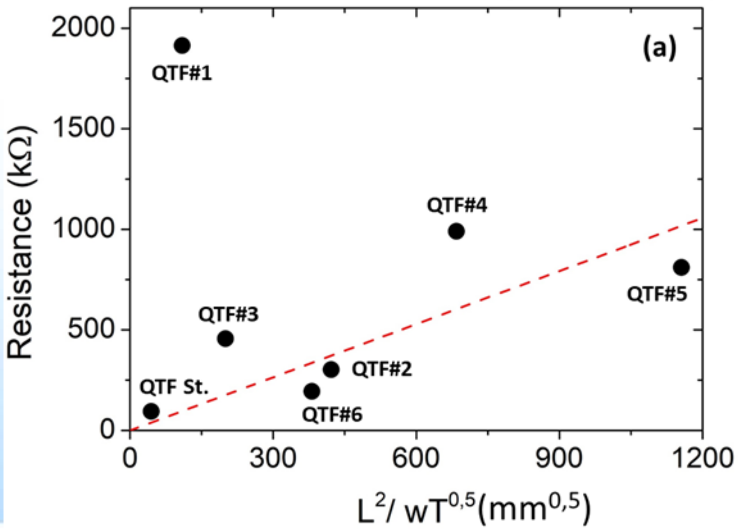
Custom QTF 2nd generation



$Q \propto wT/L$





$R \propto L^2/w\sqrt{T}$

QTF DESIGN GUIDELINES

P. Patimisco, et al., Applied Physics Review, in press, **2018**.

P. Patimisco et al., Sensors and Actuators B Chemical, 227, 539–546,

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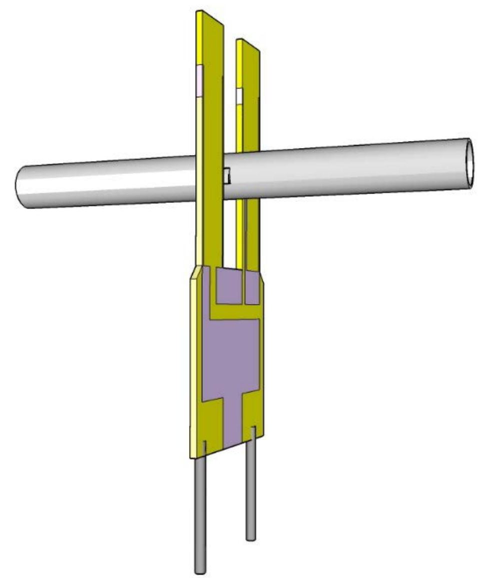
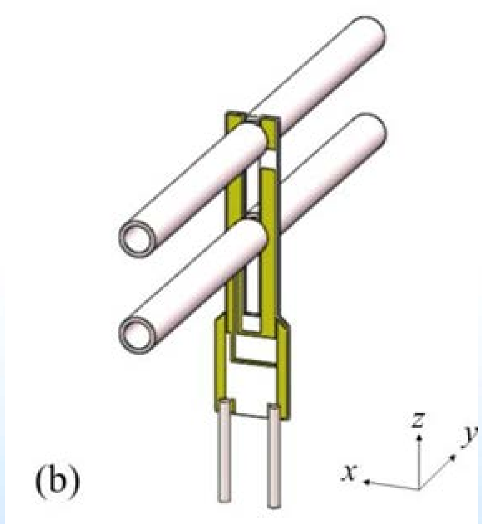
2nd generation main results

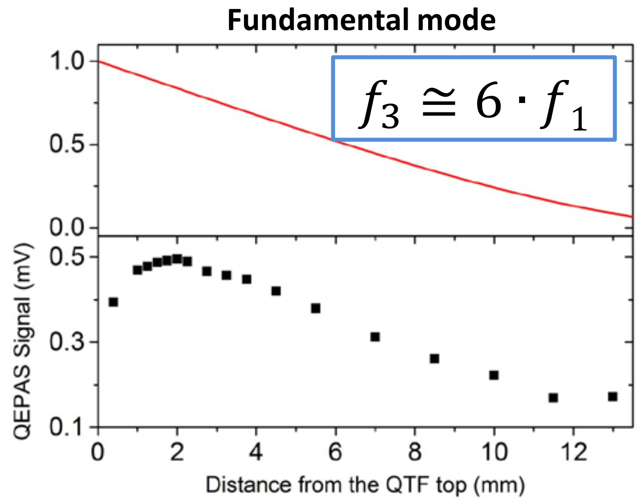
Single–tube microresonator

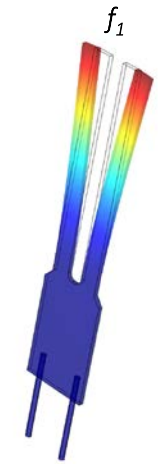


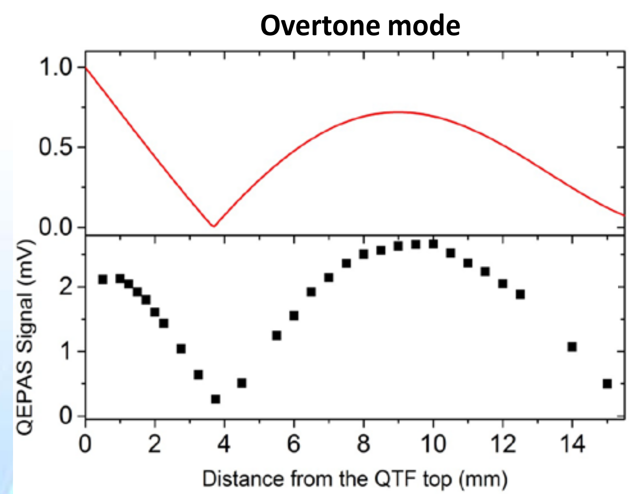
Double antinode excited SO-QEPAS

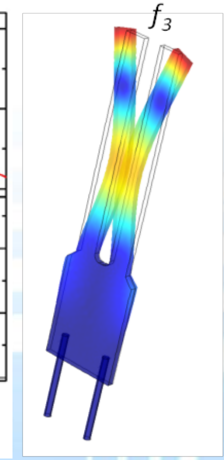


1st Overtone modes









A SNR gain factor of 500 with respect to the bare QTF operating in the fundamental mode

P. Patimisco, et al., Applied Physics Review, in press, **2018**.

H. Zengh, L. Dong, P. Patimisco et al, Applied Physics Letters 110, 021110, **2017**

H. Zengh, L. Dong, A. Sampaolo et al, Applied Physics Letters, *109, 111103,* **2016**

F. K. Tittel , A. Sampaolo, P. Patimisco et al., Optics Express 24, A682, **2016**.

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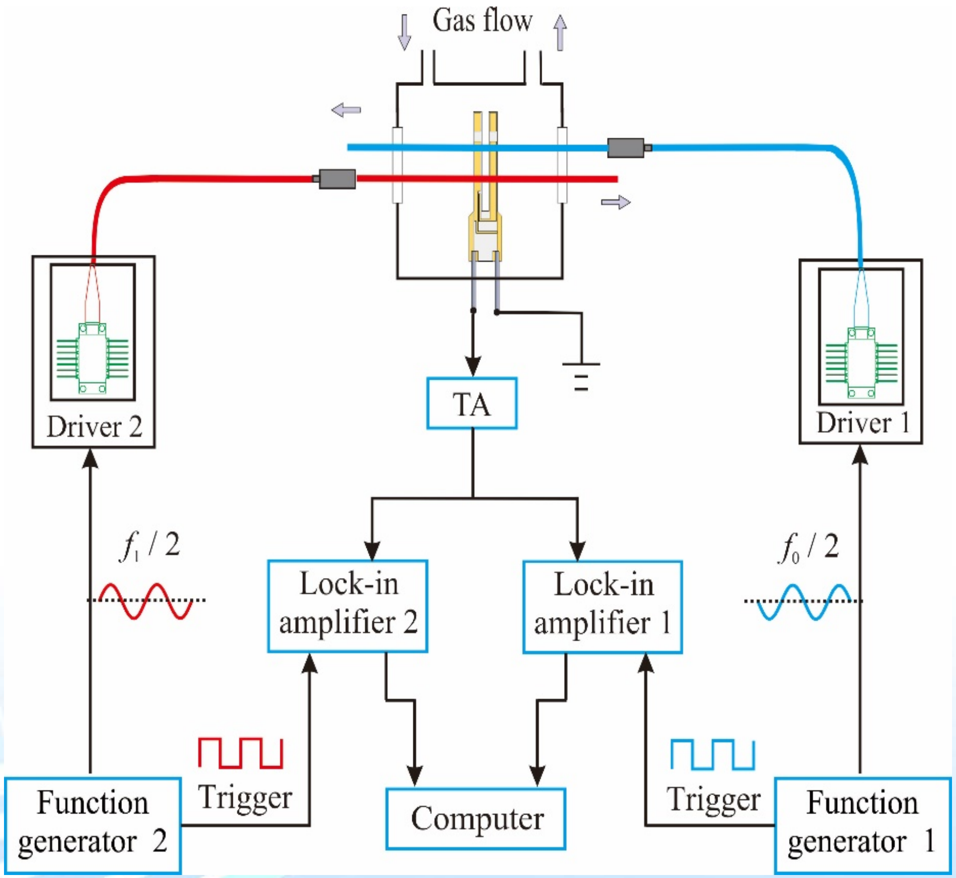
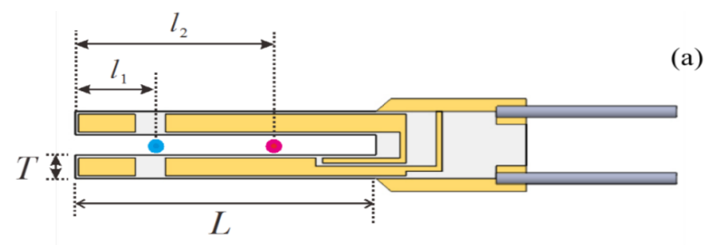
Dual-gas QEPAS operating at both the QTF fundamental and 1st overtone





Two beams from two independently modulated lasers are focused between the prongs of a quartz tuning fork at two different positions to excite both the fundamental and first overtone flexural modes simultaneously

Dual-gas quartz-enhanced photoacoustic spectroscopy (QEPAS) sensor system based on a frequency division multiplexing technique

H. Wu, X, Yin, L. Dong, K. Pei, A. Sampaolo, P. Patimisco et al, Appl. Phys. Lett., *110, 121104,* **2017**

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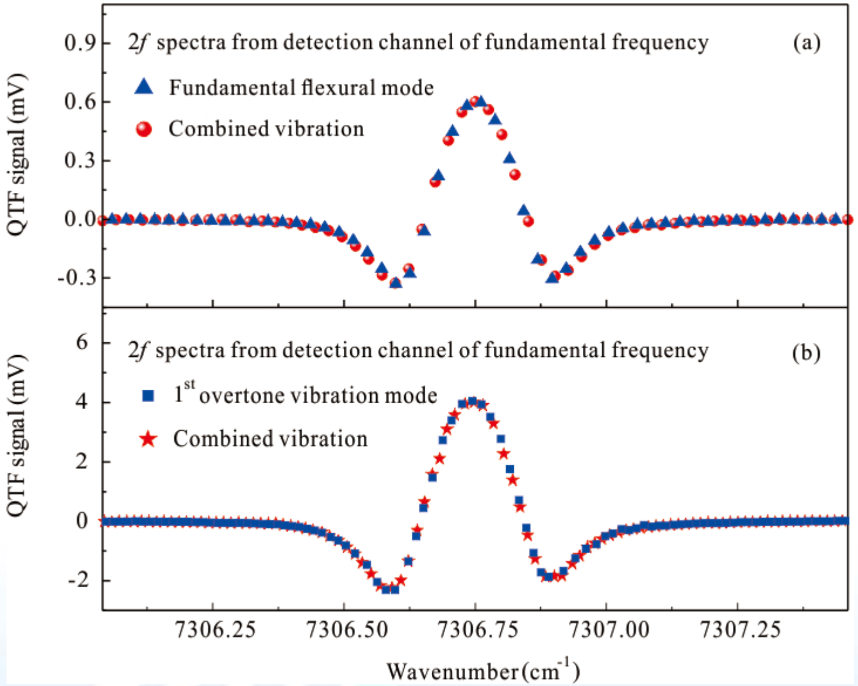
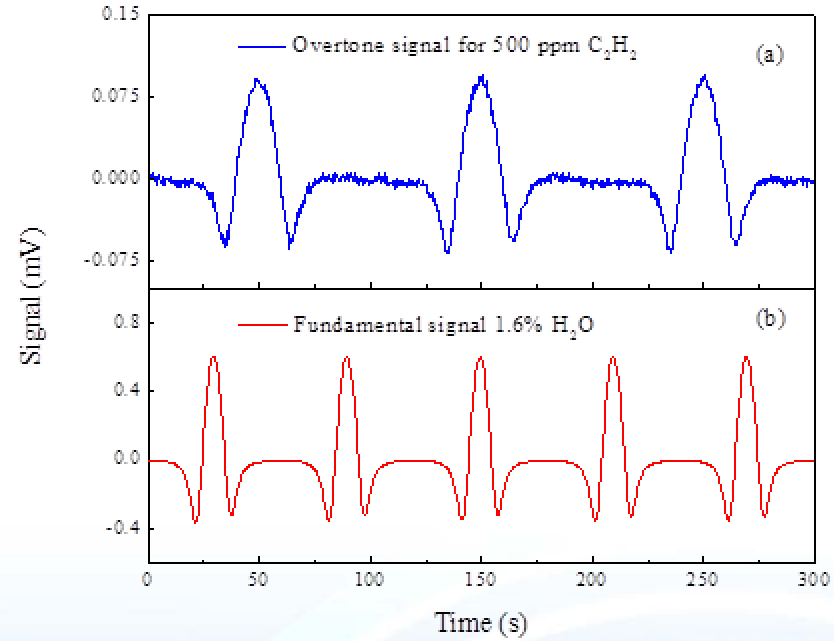
Dual-gas QEPAS operating at both the QTF fundamental and 1st overtone





- No cross-talk between fundamental and 1st overtone
- Simultaneously dual-gas detection (eg. C_2H_2 and H_2O)
- Future improvements using single-tube resonators
- Applications include: industrial process control, isotope ratio measurements, breath analysis

H. Wu, X, Yin, L. Dong, K. Pei, A. Sampaolo, P. Patimisco et al, Appl. Phys. Lett., *110, 121104,* **2017**

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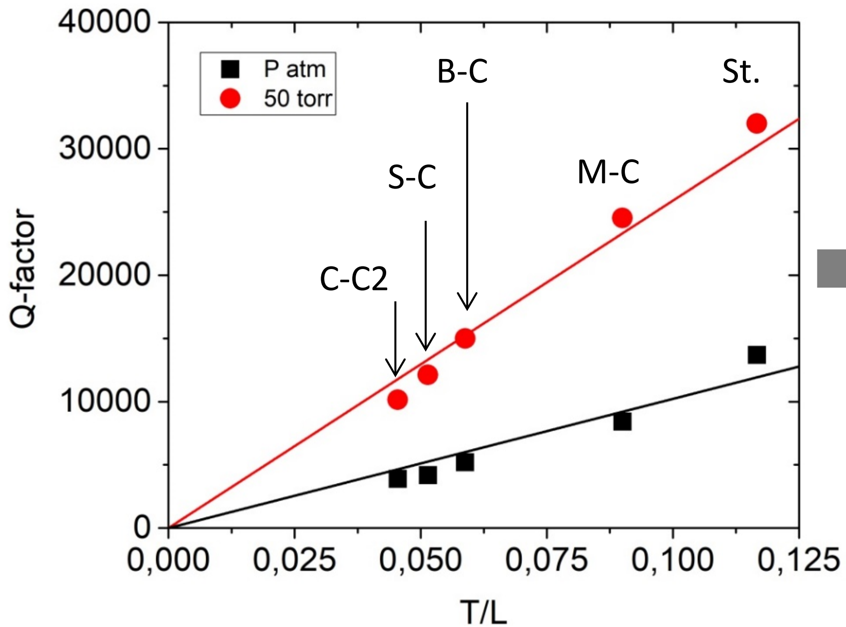
3rd generation of custom QTFs

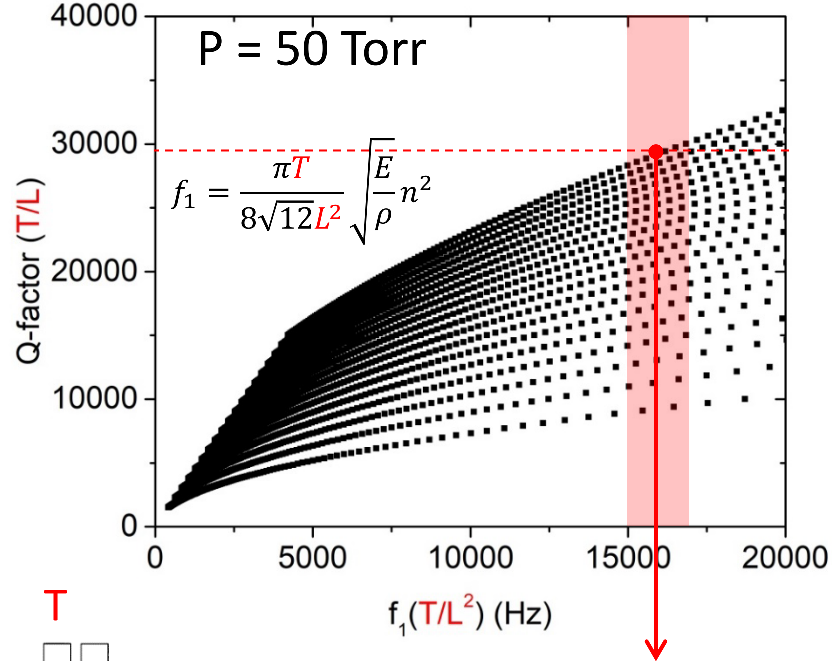
Objective: Design of QTFs with a **high Q-factor** and resonant frequency in the range 15-17 kHz

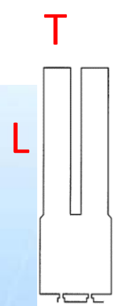
Starting from QTFs 2nd generation



Air damping dominates $Q \propto \frac{T}{L}$

Simulation





Selected design:

L = 9.4 mm

T = 2 mm

Q ~ 30000 @ 50 Torr

Q ~ 10000 @ Atm P.

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3rd generation of custom QTFs

Goal: Realize custom quartz tuning forks, targeting: i) reduction of the resonance frequency; 2) maintenance of a high the Q-factor; 3) optimized electrode layout for overtone flexural mode

5 NEW DESIGNS

QTF S08
(prongs spacing 0.8 mm)

<u>Reducing air damping</u> while keeping f_0 15-17 kHz

QTF S08 - G
(prongs spacing 0.8 mm)

<u>Grooves engraved</u> on both sides of the QTF prongs

QTF S08 – TOP
(prongs spacing 0.8 mm)

<u>Wider top end</u> on both prongs

QTF S15
(prongs spacing 1.5 mm)

<u>Increased prong spacing</u> of 1.5 mm to <u>facilitate optical alignment</u> of the focused laser beam

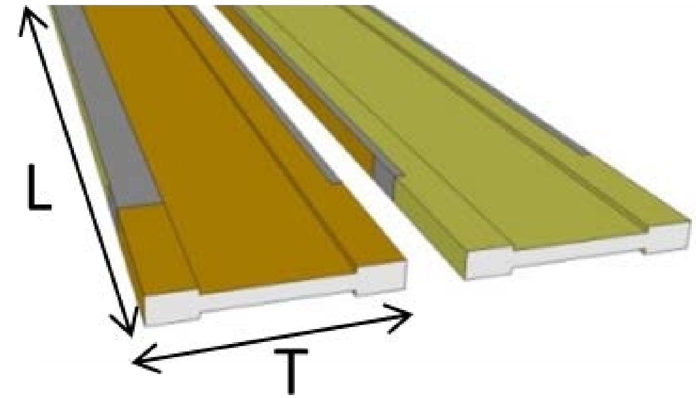
QTF-Overtone
(prongs spacing 0.7 mm)

Enhanced <u>overtone mode</u> operation @ 17 kHz with an optimized <u>electrode layout</u> design

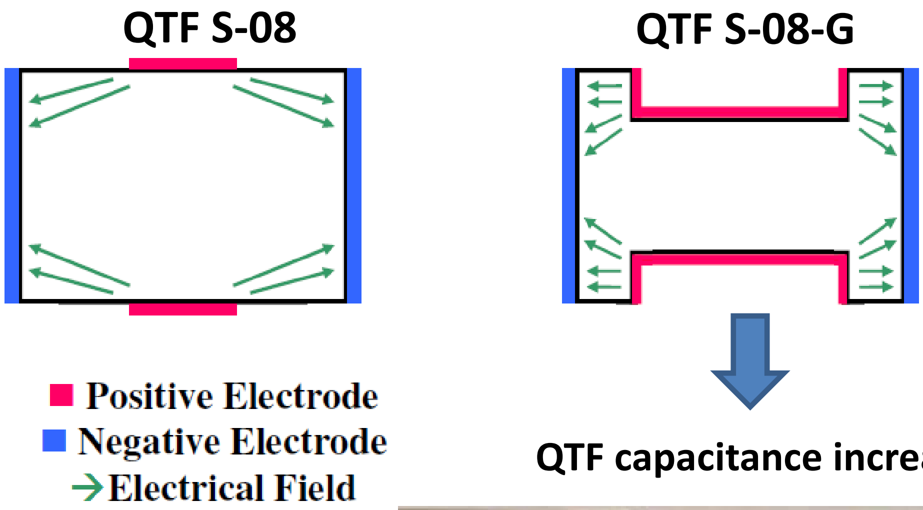
All these QTFs have the same prong length and thickness

Design considerations for QTF S-08-G

Same geometry of QTF S-08, but **with grooves** engraved on both surfaces of **the prongs** to reduce the electrical resistance



Top view of one prong



$$C = \frac{1}{2\pi Q R f}$$

Q and **f** depend on *T* and *L*

Not affected by grooves

R should be reduced

QTF capacitance increases

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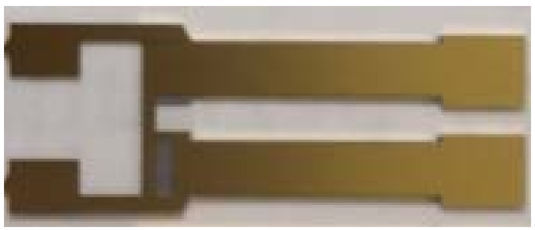
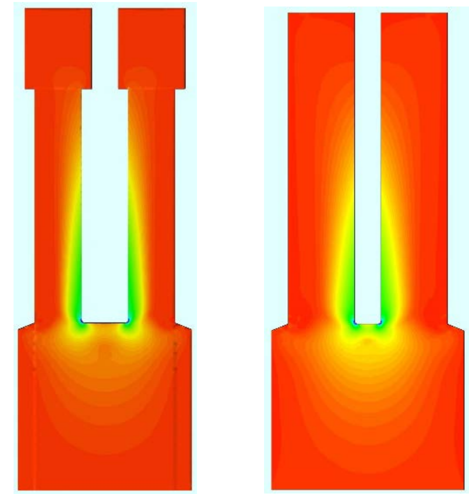
Design considerations for:

QTF-S08-TOP

QTF S08 CAPU

Same geometry as QTF S08 but with a **wider top end** of on both prongs to **better distribute the stress** field along the prongs and increase the **generated piezo-charges**

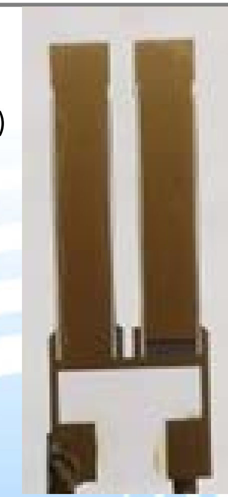




QTF S08

QTF-S15

QTF S15
(prongs spacing 1.5 mm)



Same geometry as QTF S08

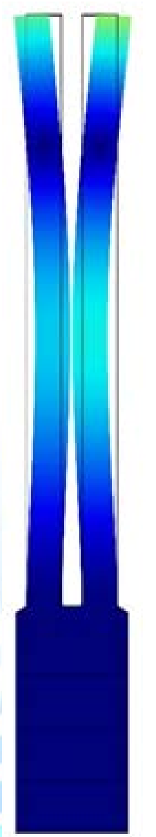
Increased prong spacing up to 1.5 mm to:

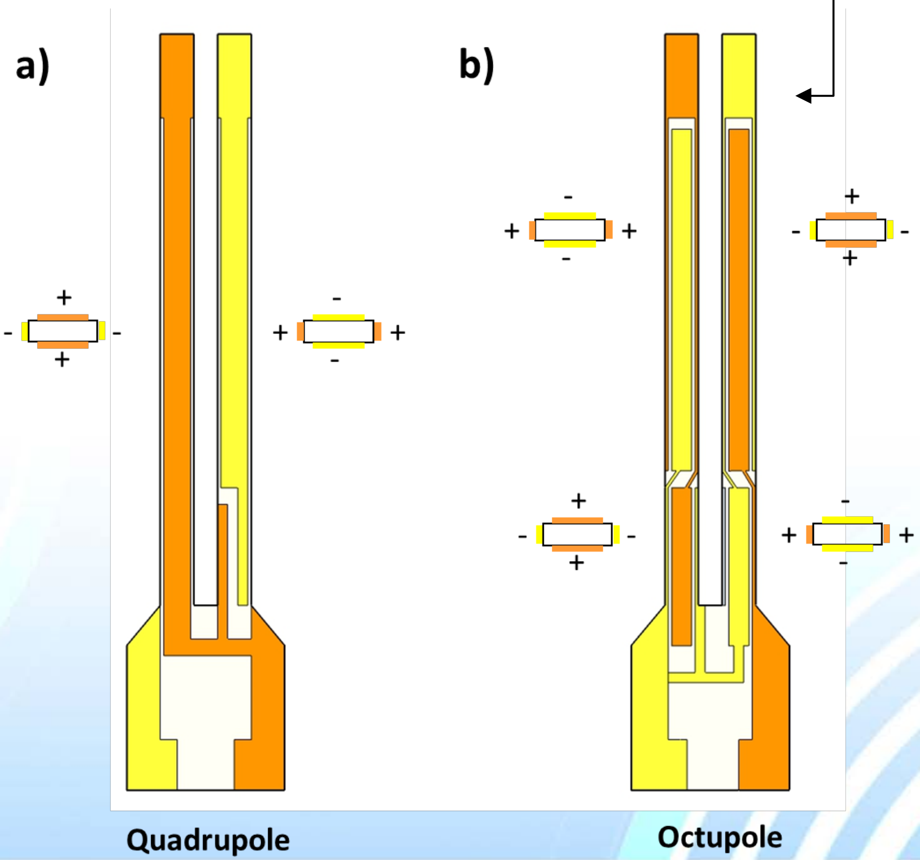
- **Facilitate the optical alignment** of the focused laser beam
- Employ laser source with **poor spatial beam** quality or emitting in the **THz range**
- Implement micro-resonator tubes with **large inner diameters**
- **Investigate** of the influence of the **prongs spacing** on the QEPAS signal

Design considerations of QTF-overtone

QTF design optimized for **overtone mode** operation @ 17 kHz

Electrode layout designed to efficiently collect the **generated piezo-charges** (octupole configuration)





QTF-overtone

(prongs spacing 0.7 mm)

P. Patimisco et al, Optics Letters, *in press,* **2018**

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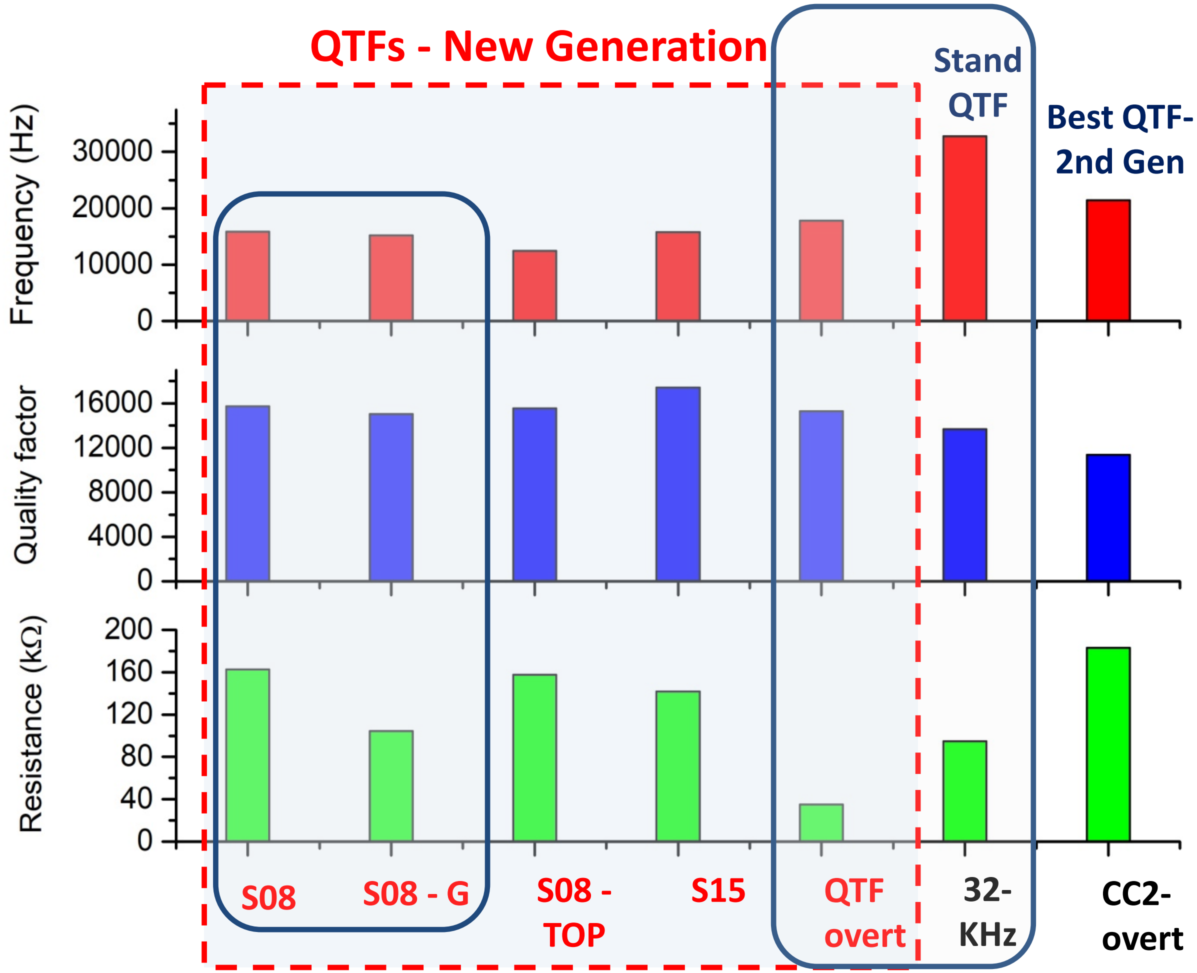
3rd gen. QTFs Electrical Characterization



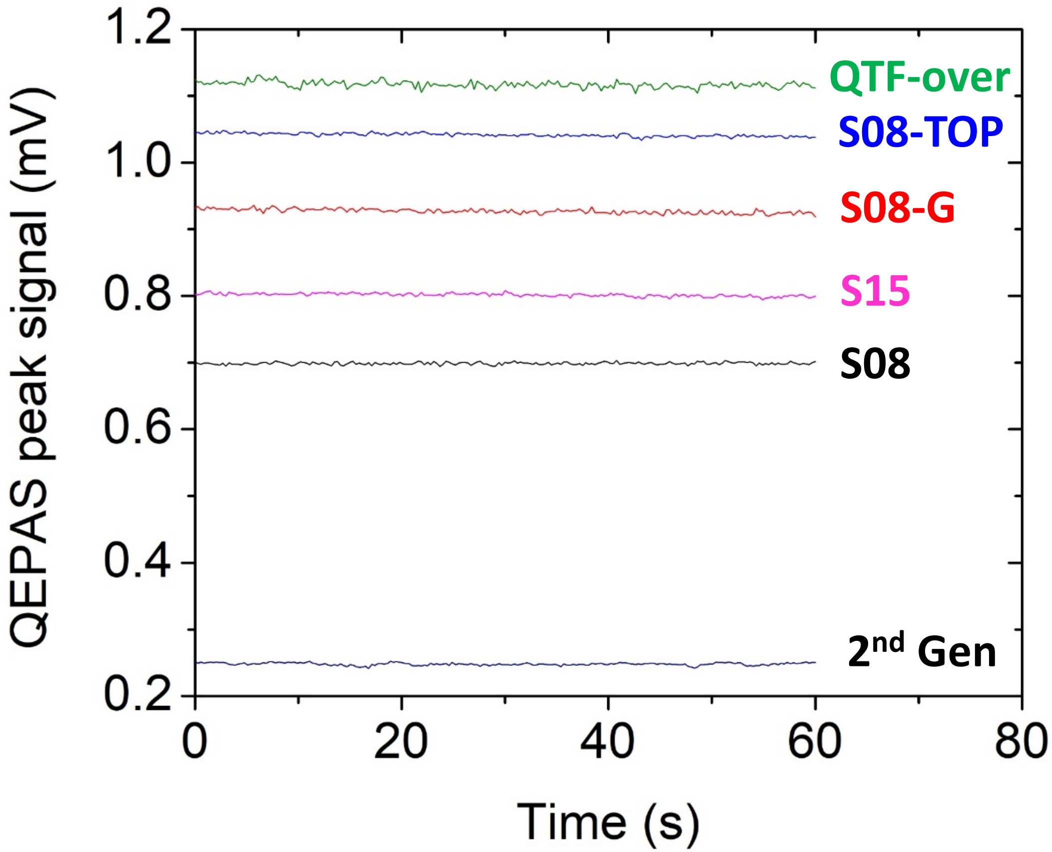
Results are compared with a *standard 32 kHz-Q*TF and the best QTF of the 2nd generation (*C-C2, overtone*)

- The **frequency was decreased by a factor of 2** with respect to the 32 KHz-QTF, thus achieving **higher quality Q-factors.**
- Engraving **grooves** on the prongs' surface **decreased the QTF electrical resistance by a factor of 2** (see **S08-G vs S08)**.
- The **QTF operating in the overtone mode** (**QTF-overt**) exhibits a **lower electrical resistance** than a 32KHz-QTF.

3rd gen. QTFs –Photoacoustic performances

Detection of a water line @7.7 µm, atm pressure

QEPAS line-locking measurements



All new QTFs show **higher QEPAS signals** than the 2nd generation.

The **noise level is nearly the same** for all QTFs except for QTF-over, due to its narrower prongs spacing (700 µm).

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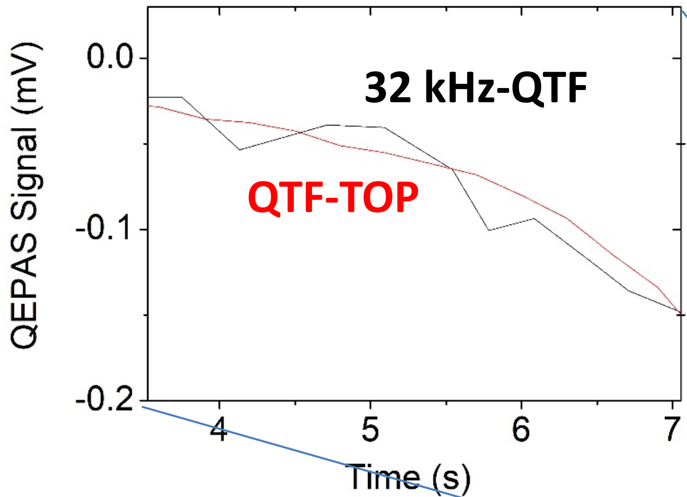
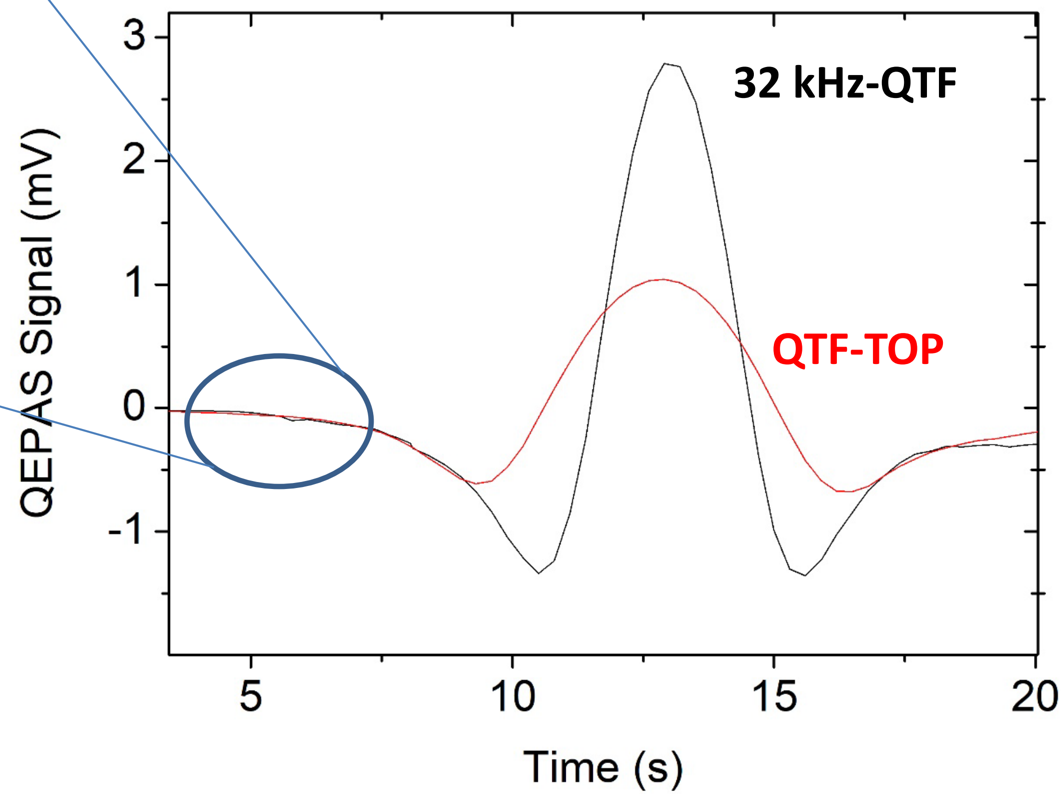
3rd gen. QTFs – Comparison with 32 kHz-QTF

Detection of a water line @7.7 µm, atm pressure



QEPAS spectral scan measurements



32 kHz-QTF exhibits a **higher signal**

but also a **higher noise background**

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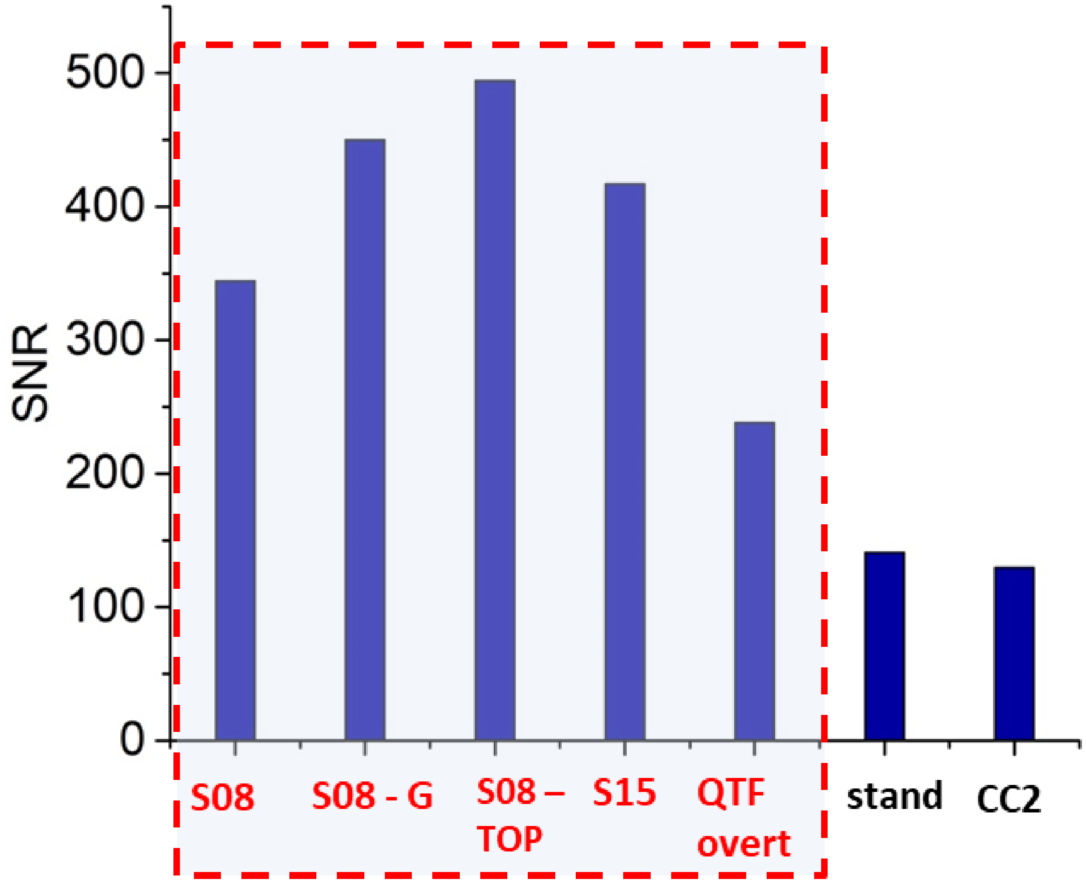
3rd gen. QTFs –SNR performances



All 3rd gen. QTFs exhibit **higher performance (SNR)** with respect to the 2nd gen. QTFs and the standard 32kHz QTF.

Future goals:

- New designs combining both prong surface engraving and top-end layer thickening.
- Testing of QTFs with dual and single-tube acoustic micro-resonators.

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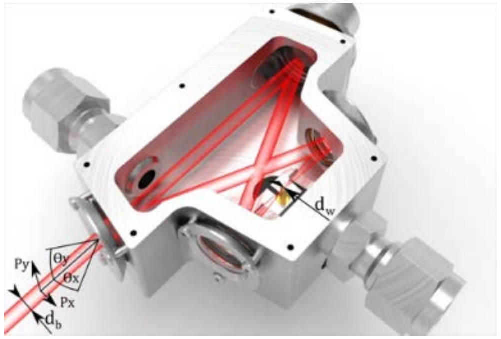
Joint Research Lab PolySenSe

RESEARCH ACTIVITIES

- **Novel gas sensing techniques development and implementation**
- **Highly sensitive trace gas sensors portable for in-situ & real time detection**



- **Potential applications:**
  - Breath analysis
  - Hydrocarbon gas monitoring
  - Environmental monitoring
  - Leak detection
  - Monitoring of hotspot areas (toxic gases,explosive precursors)




- **On-demand chemical sensing**
- **Third party consulting**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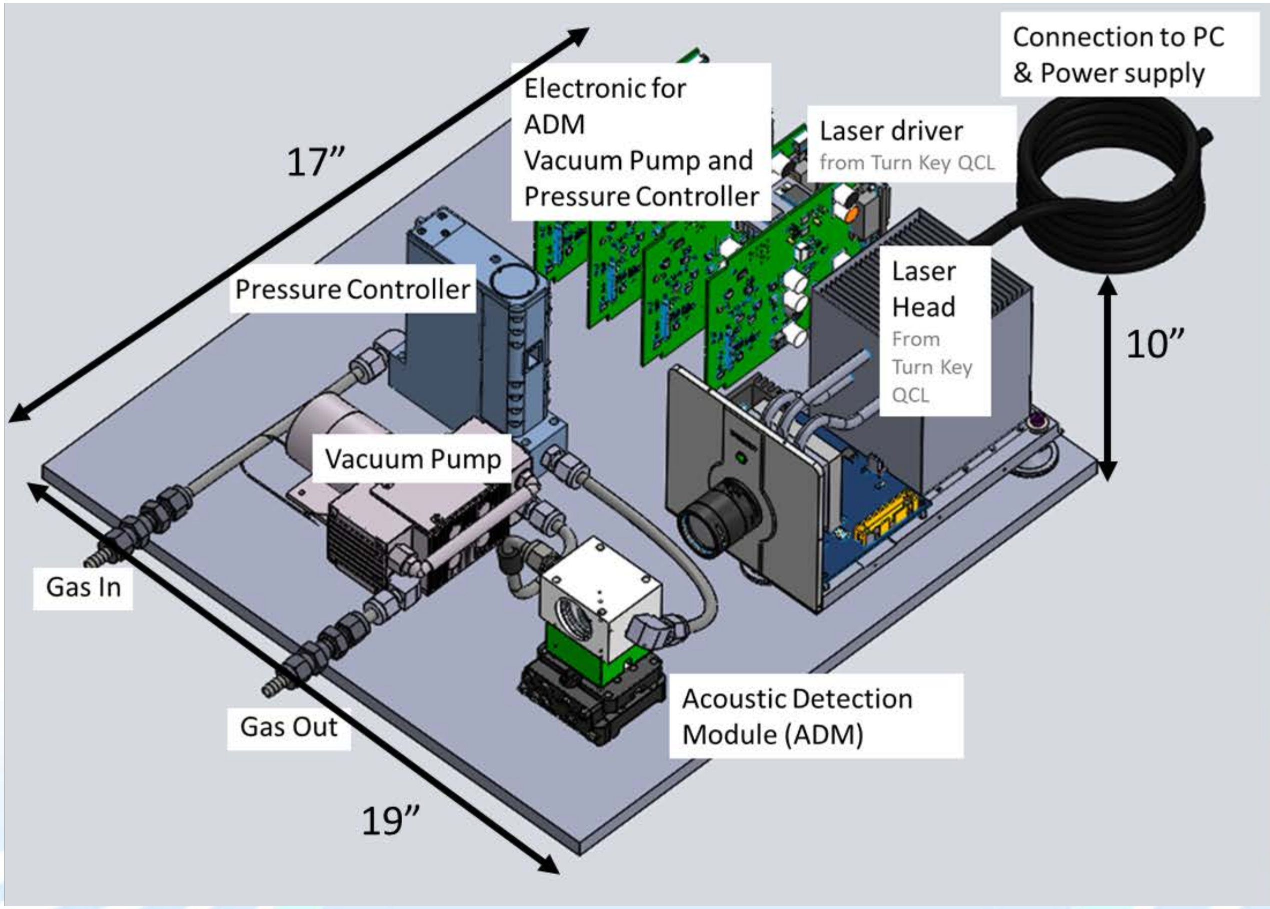
Proposed new QEPAS acoustic sensor system



NEW ADM implementing 3° Gen QTF

A compact QEPAS sensor for ethylene detection implementing the new ADM and 3rd gen. QTF wa first shown at the THORLABS booth, PW2018

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Conclusions and Future Perspectives

- Demonstration of ***QEPAS sensors*** employing custom ***QTFs with new geometry*** and gold contact pattern with improved sensitivity.
- ***First demonstration*** of ***QEPAS dual-gas detection*** with QTF simultaneously operate at fundamental and 1st overtone flexural mode
- Realization of ***QTFs 3rd generation*** exploiting top-end thickening, surface grooves and octupole contact patterns approaches, ***all showing improved performances*** with respect to standard and 2nd gen. QTFs.

✓ Develop QEPAS-based breath sensing systems

✓ Develop a QEPAS sensor for ethane-methane-propane detection

✓ Push QEPAS sensor systems towards commercialization level

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Thank You