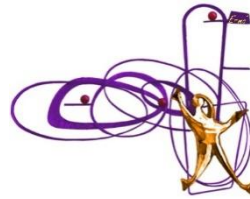




UNIVERSITÀ
DEGLI STUDI DI BARI
ALDO MORO



DIPARTIMENTO INTERATENEO DI FISICA "M. MERLIN"

SCUOLA DI DOTTORATO DI RICERCA IN FISICA XXXI CICLO

Presentazione di dottorato

INNOVATIVE QUARTZ-ENHANCED PHOTOACOUSTIC SENSORS FOR TRACE GAS DETECTION

Dottoranda: **MARILENA GIGLIO**

Relatore: Prof. Vincenzo Spagnolo

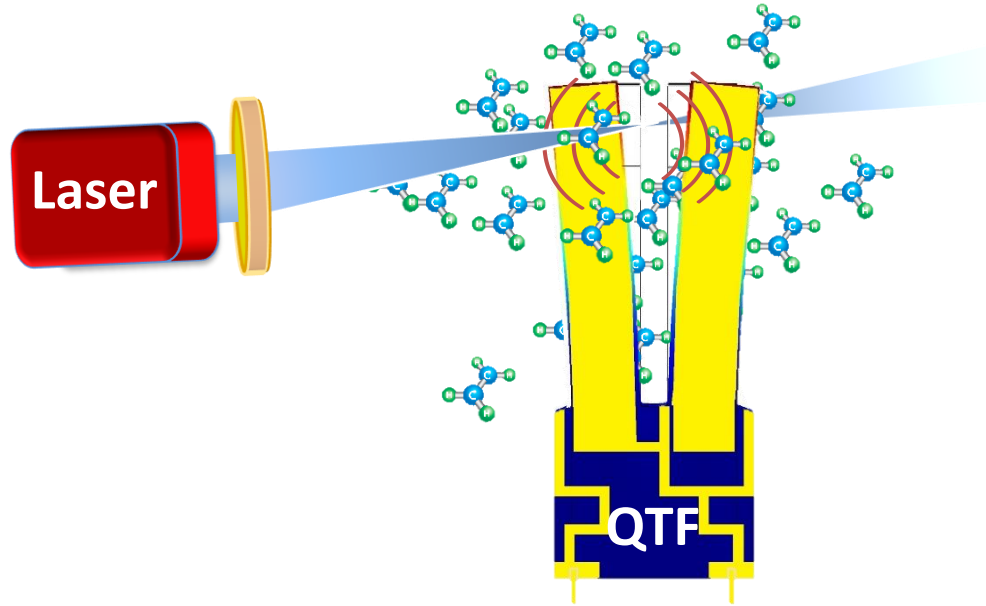
PolySenSe



Outline

- Introduction to **QEPAS**
- **Hollow-core waveguides** as spatial beam filters
- **Custom quartz tuning forks** design and implementation in **QEPAS sensors**
- **Broadband gases** detection
- **Conclusions**
- List of **courses, awards, publications, proceedings, conference talks, teaching activities**

Quartz-Enhanced Photoacoustic Spectroscopy



- Target gas excitation by a laser source
- Laser modulation to generate pressure waves
- Sound waves detection by a resonant quartz tuning fork
- Charge distribution generation via piezoelectric effect
- Charge collection by gold electrodes

- *QEPAS Signal* $\propto P \propto Q$
- No optical detector required
- Small detection module volume
- High selectivity and sensitivity
- Wide pressure and temperature dynamic range
- Detector insensitive to the excitation wavelength

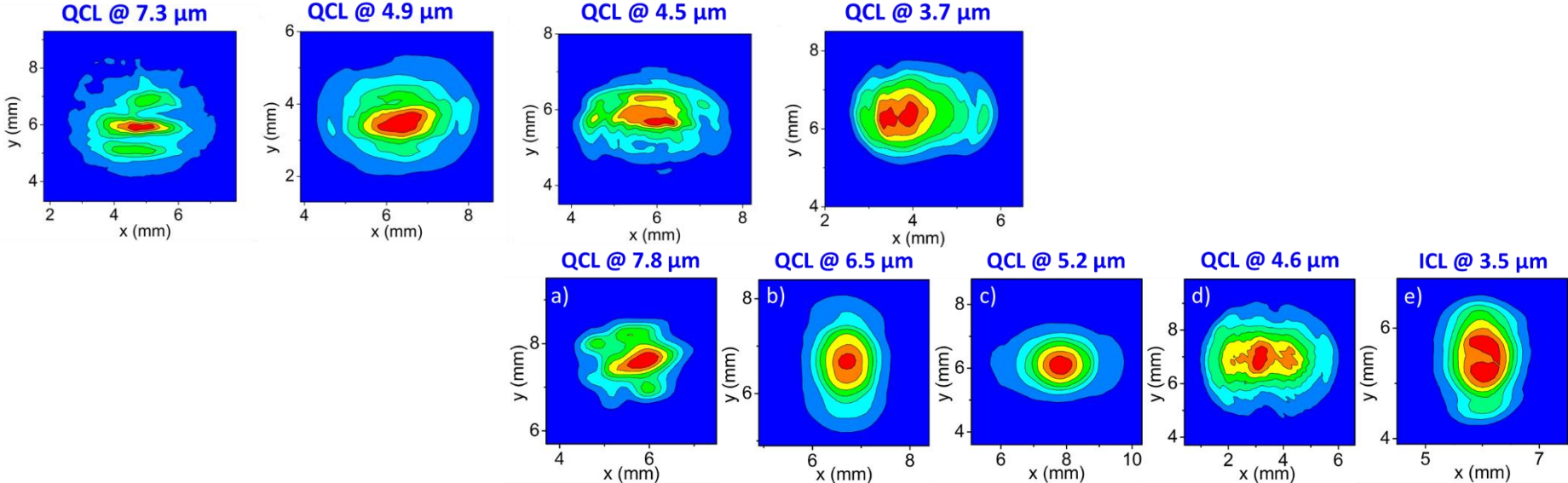
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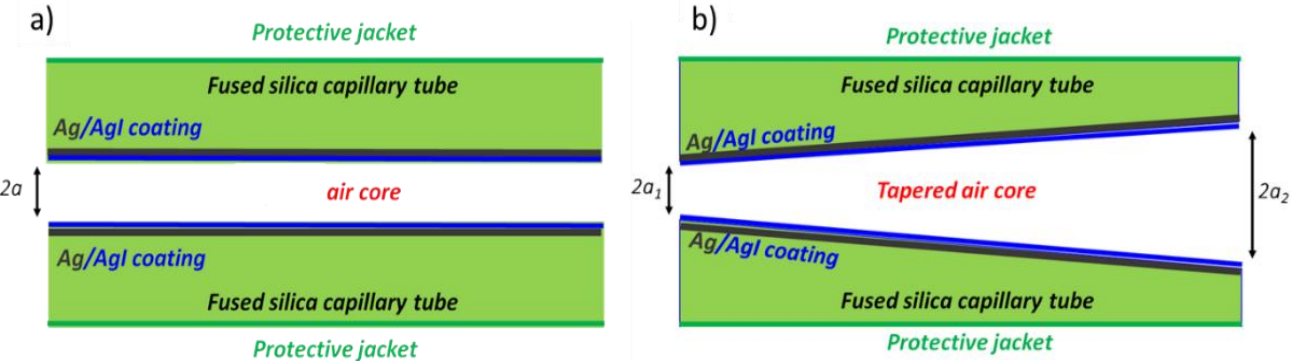
Hollow-core waveguides as spatial beam filters

Motivations

Low spatial quality of laser beams can affect the ultimate performance of gas sensors

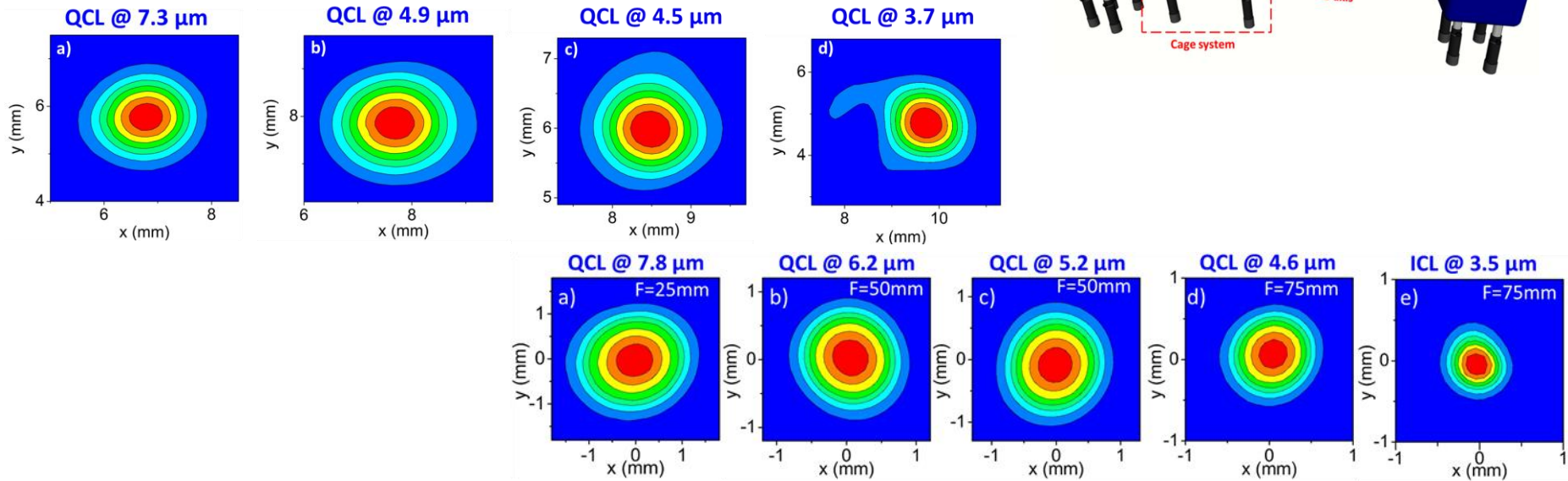
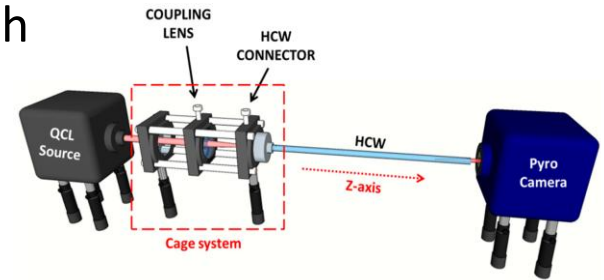


Optical coupling with HCWs acting as spatial filters

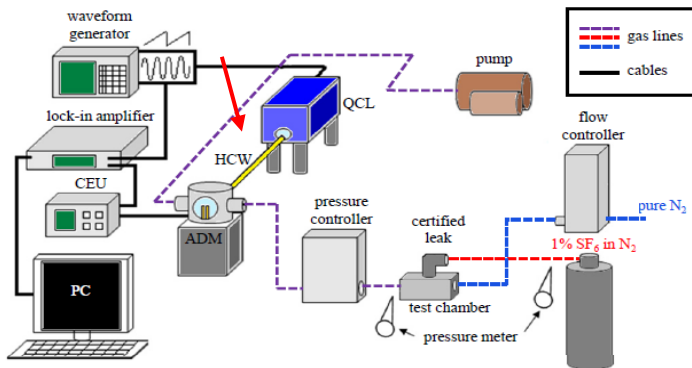


Hollow-core waveguides as spatial beam filters

Theoretical model to select the coupling lens focal length for best optical coupling conditions (low-losses, single mode delivery)



HCW-coupled QEPAS sensor for SF₆ leaks detection



Detection limit: **2.7 ppb** @ integration time 1 s



Leak flows sensitivity of **10⁻⁹ mbar·l/s**

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Custom quartz tuning forks design

Motivations

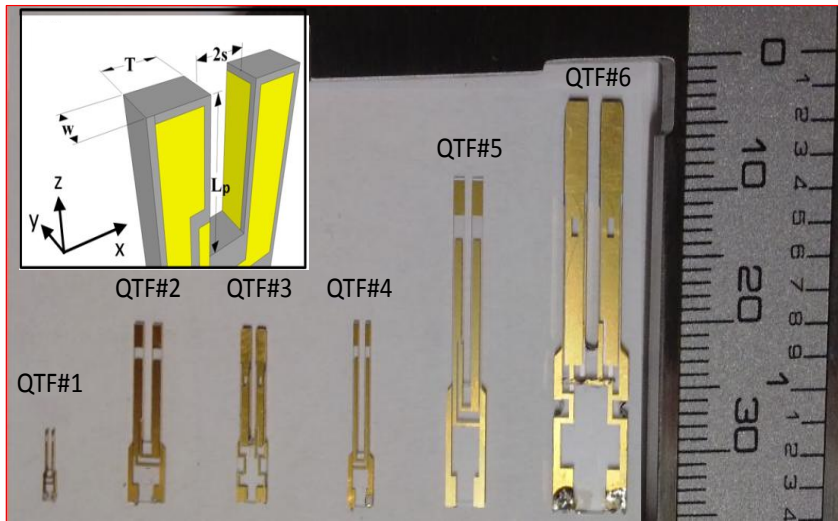


Commercial 32.78 kHz QTF optimized for timing purposes



A new design for spectroscopic applications is needed!

Influence of geometrical parameters on QTF f , Q-factor, R

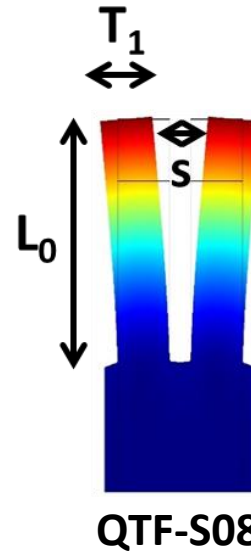
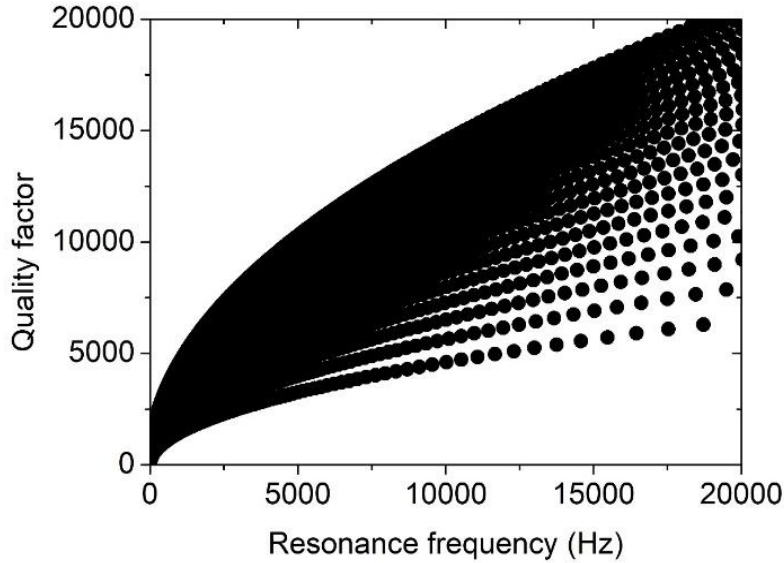


Resonance frequency $< 1/2\pi\tau$ $f_0 \propto \frac{T}{L_p^2}$

High quality factor (Q-factor) $Q \propto \frac{Tw}{L_p}$

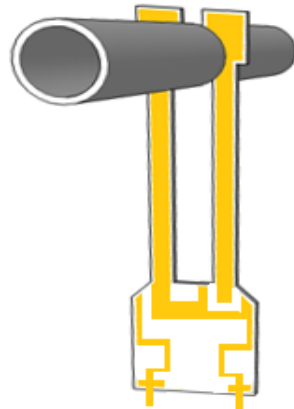
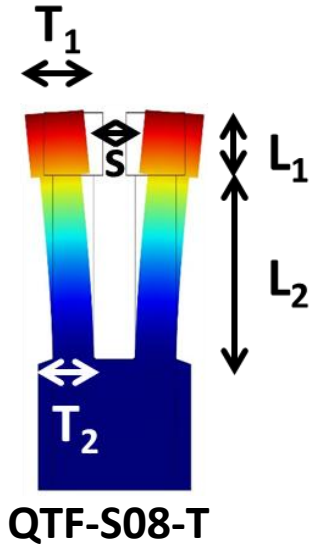
Low electrical resistance $R \propto \frac{L_p^2}{w\sqrt{T}}$

Custom quartz tuning forks design



**Standard geometry-
QTF
(Rectangular
parallelepiped,
I-shaped prong)**

T-shaped prongs QTF

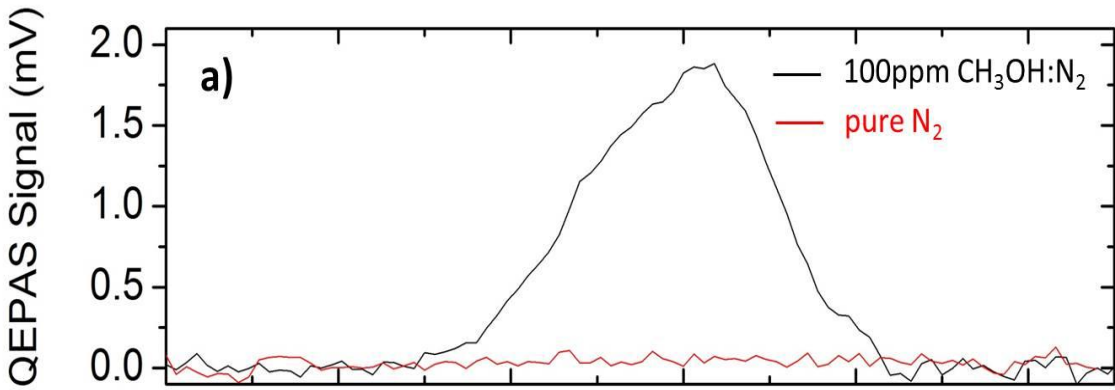
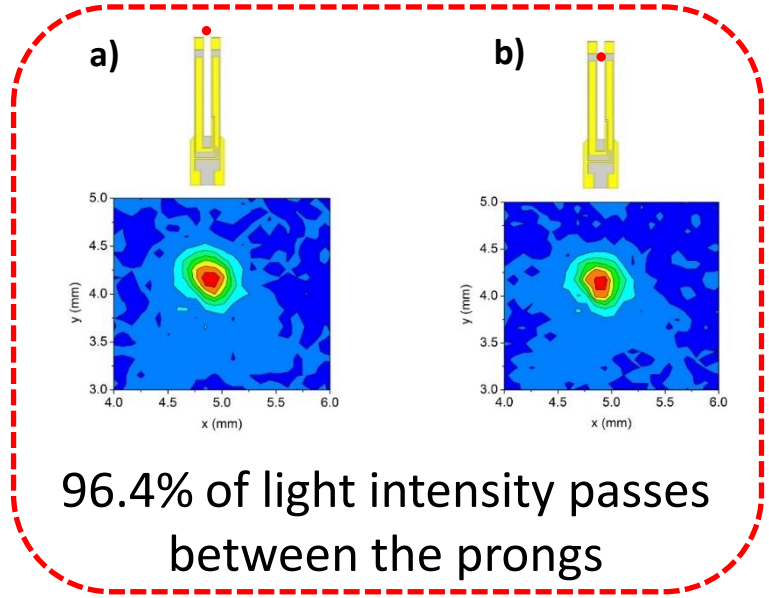
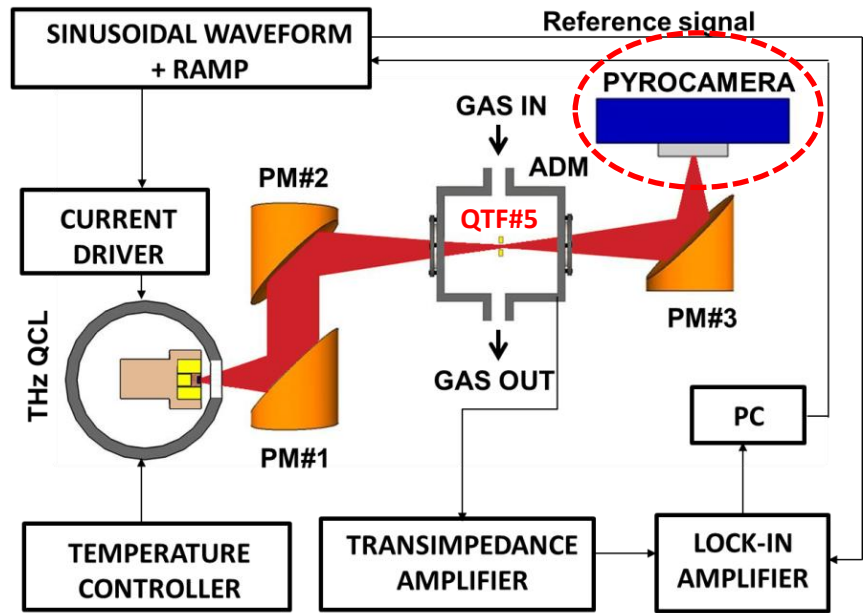


	SNR enhancement
I-shaped+AMR tubes	x 28
T-shaped+AMR tubes	x 60

Record!

Custom QTFs implementation in QEPAS sensors

Methanol detection in THz spectral range (3.93 THz-QCL)

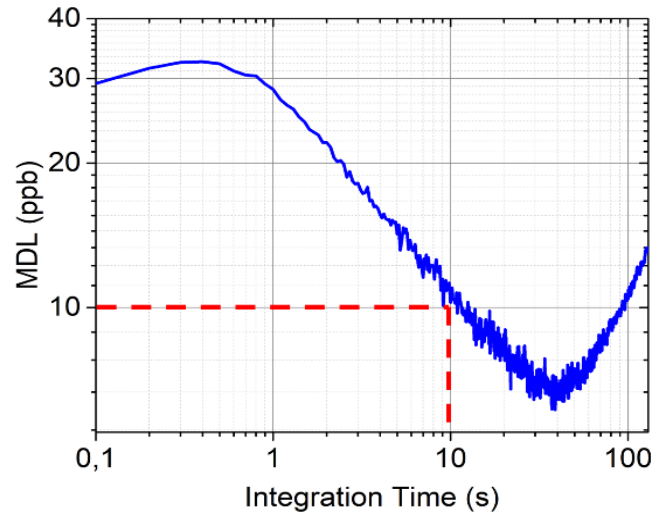
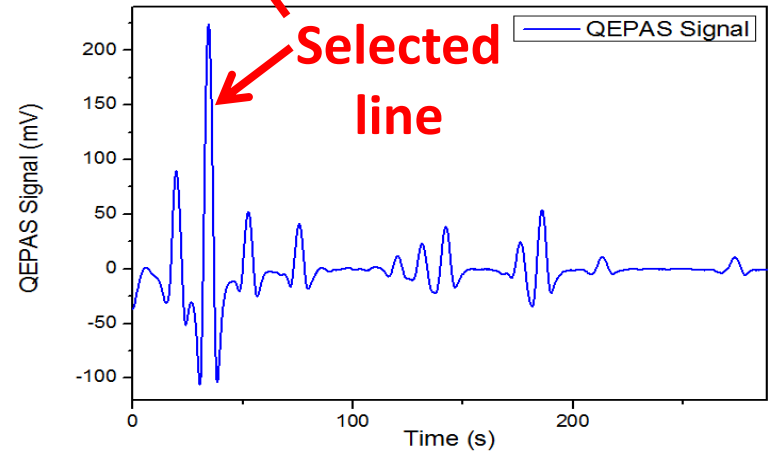
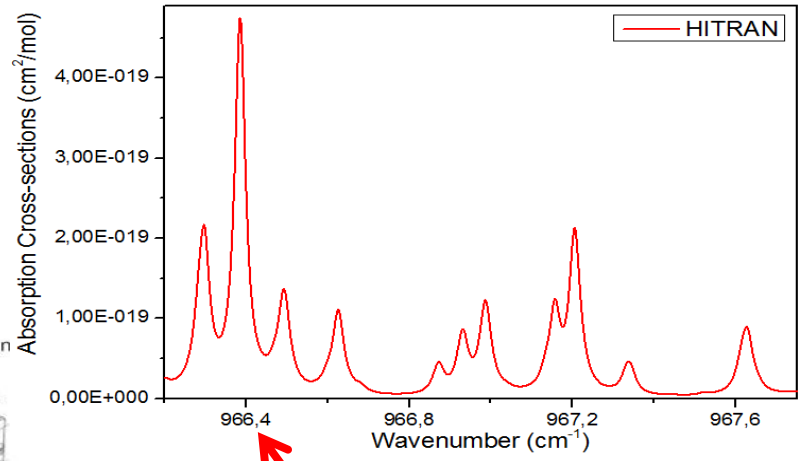
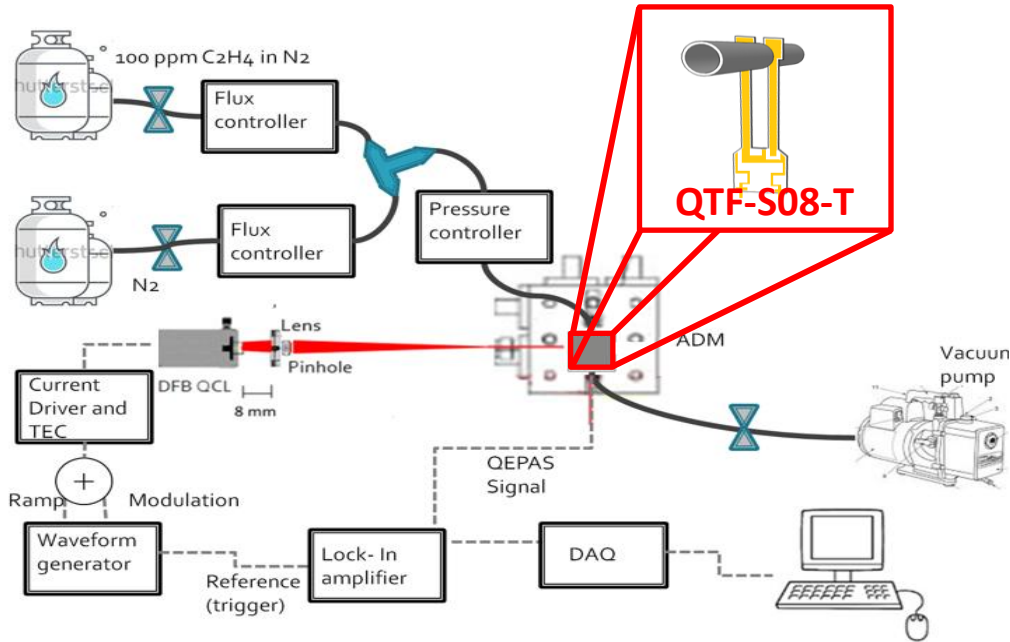


MDL = 160ppb
NNEA = $3.75 \times 10^{-11} \text{ cm}^{-1} \text{ W/VHz}$

2016 QEPAS RECORD!

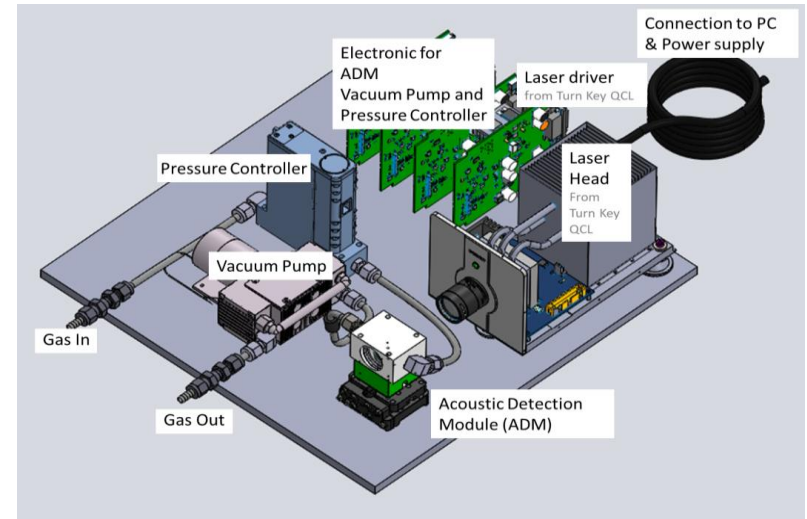
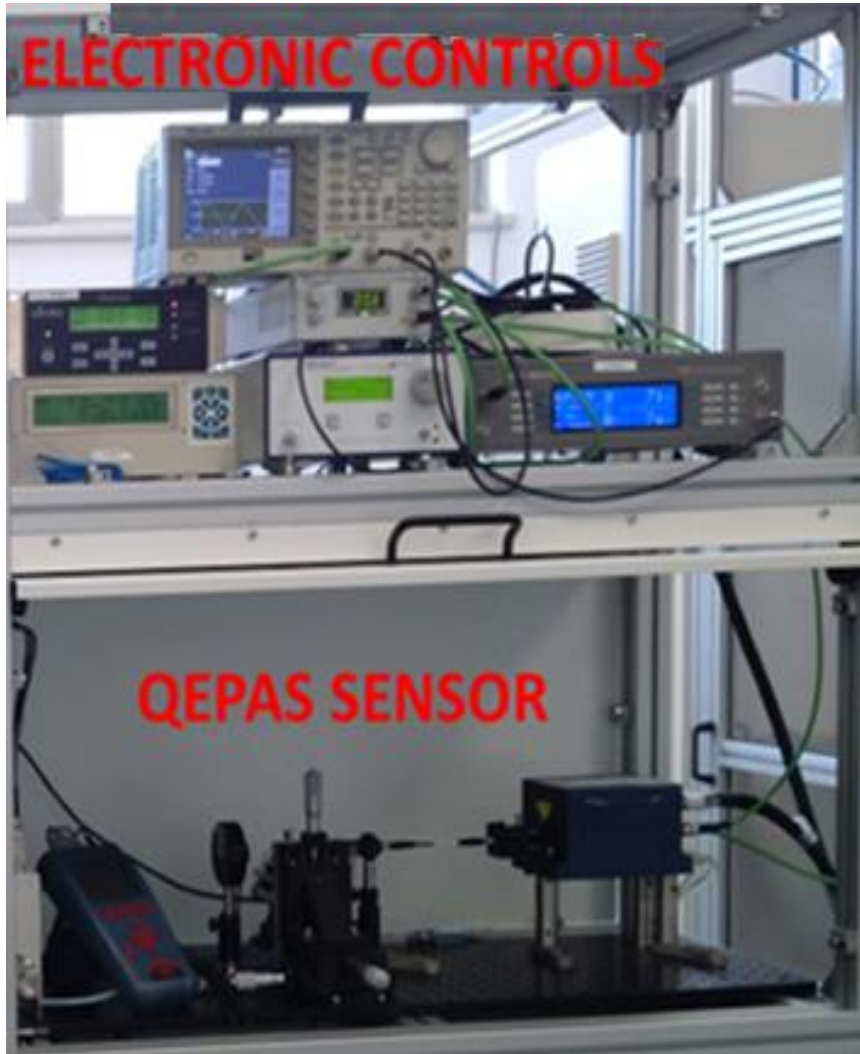
Custom QTFs implementation in QEPAS sensors

Ethylene detection in mid-IR spectral range (10.3 μm -QCL)



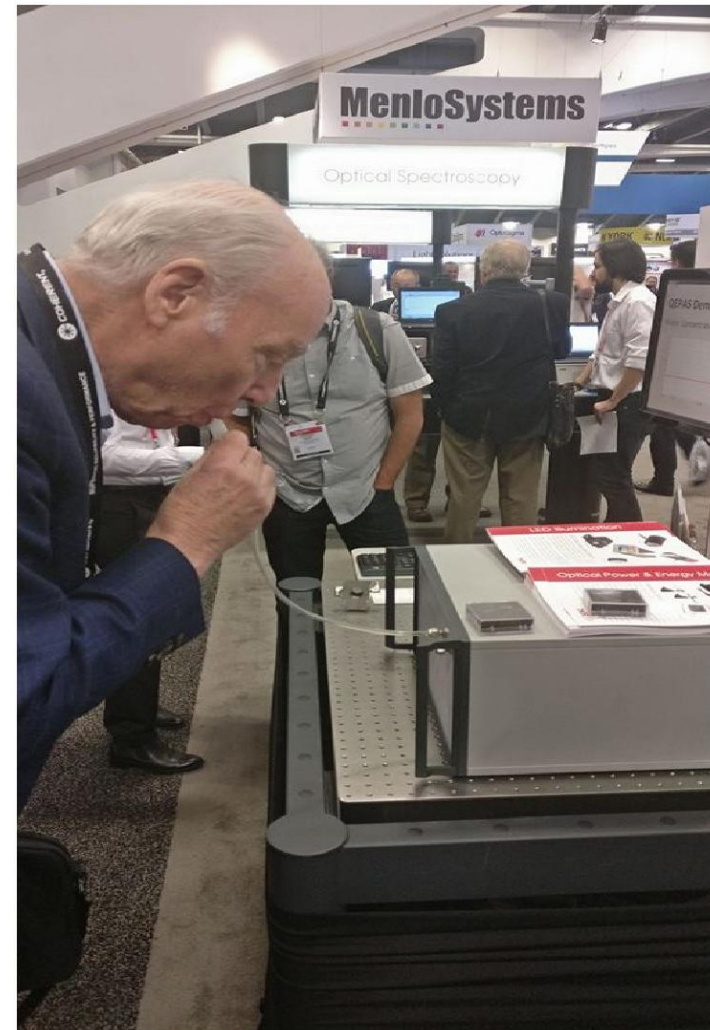
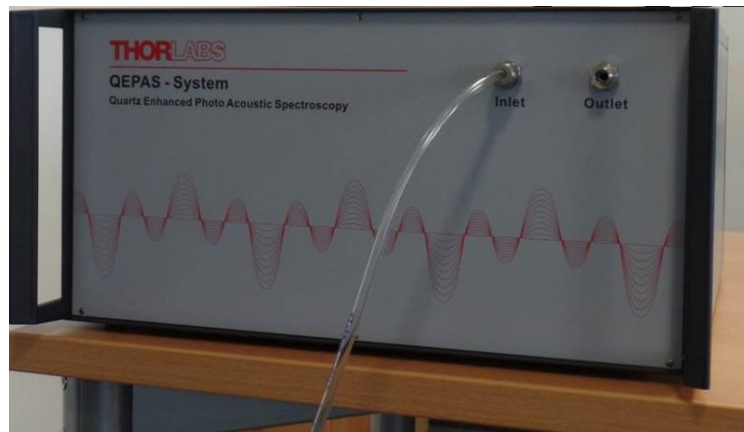
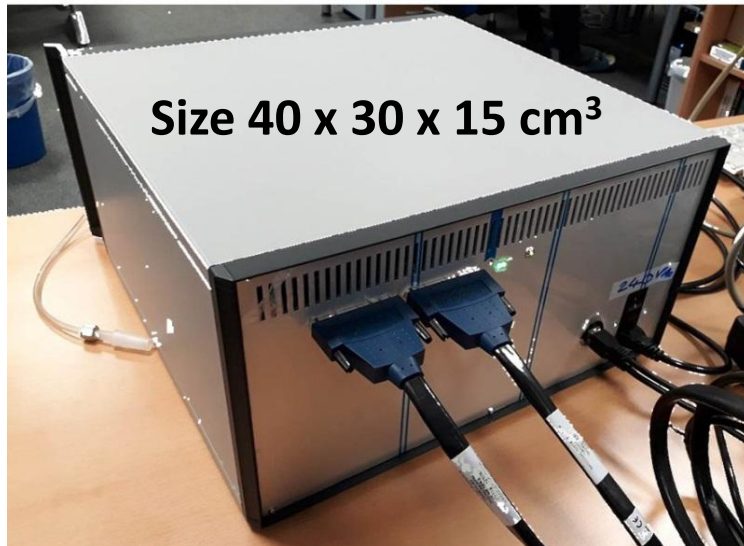
@10 s integration time
MDL: 10ppb

Compact QEPAS Sensor



**SF₆ leak Sensor
@Masmec S.p.A.**

Compact QEPAS Sensor



Photonics West 2018 – San Francisco
QEPAS Sensor @Thorlabs Booths

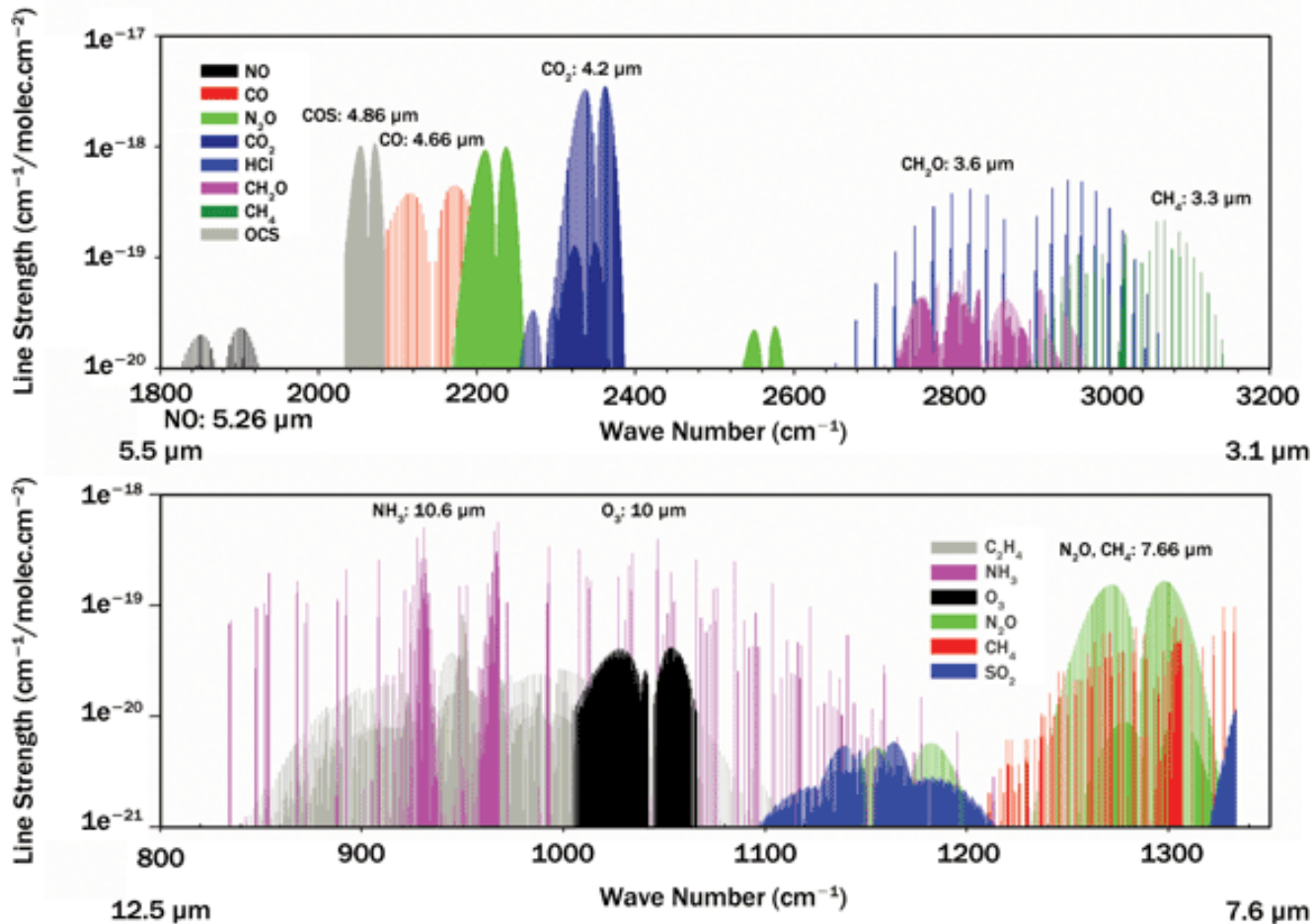
Outline

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Broadband absorbers detection

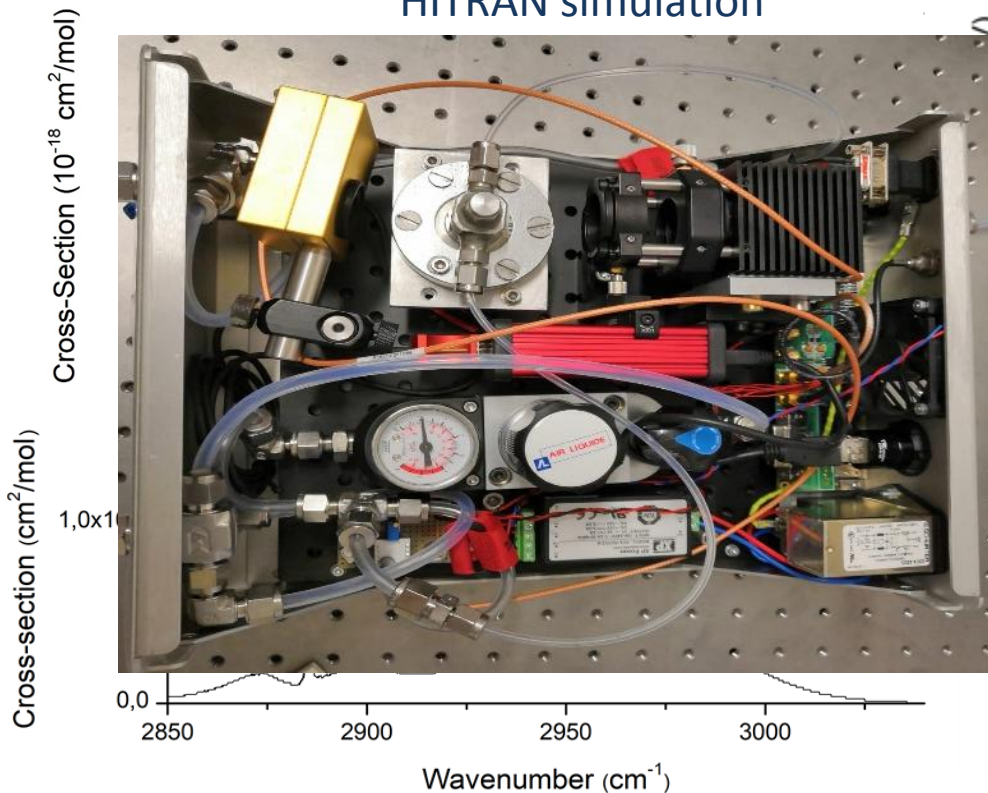
Motivations

Many harmful, greenhouse, biomarker gases belong to the class of broadband absorbers. For example, hydrocarbons and nitrous oxide

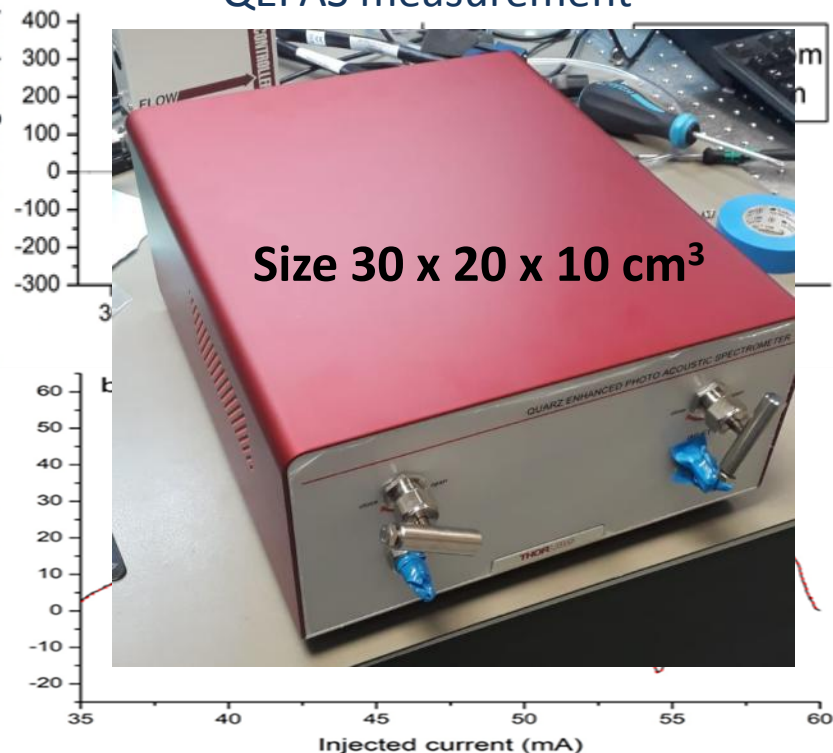


Methane (C1), ethane (C2) and propane(C3) detection employing a single ICL (@ 3345 nm)

HITRAN simulation



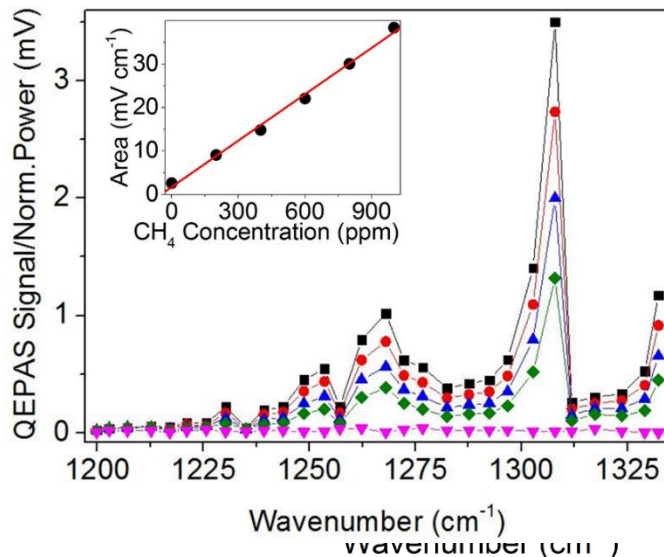
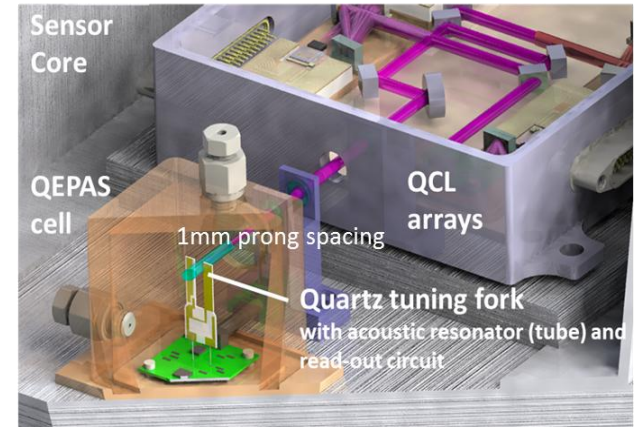
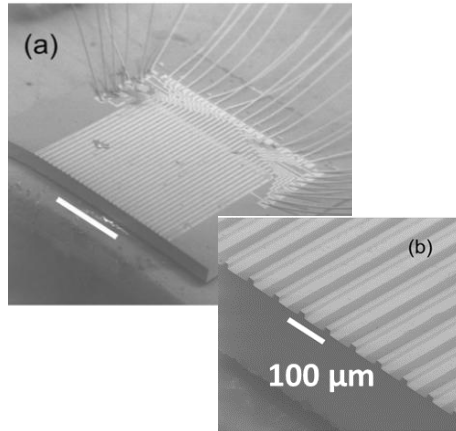
QEPAS measurement



- Detection limit C1: **90 ppb** @ integration time 1 s
- Detection limit C2: **7 ppb** @ integration time 1 s
- Detection limit C3: **3 ppm** @ integration time 1 s

RECORD for QEPAS technique!

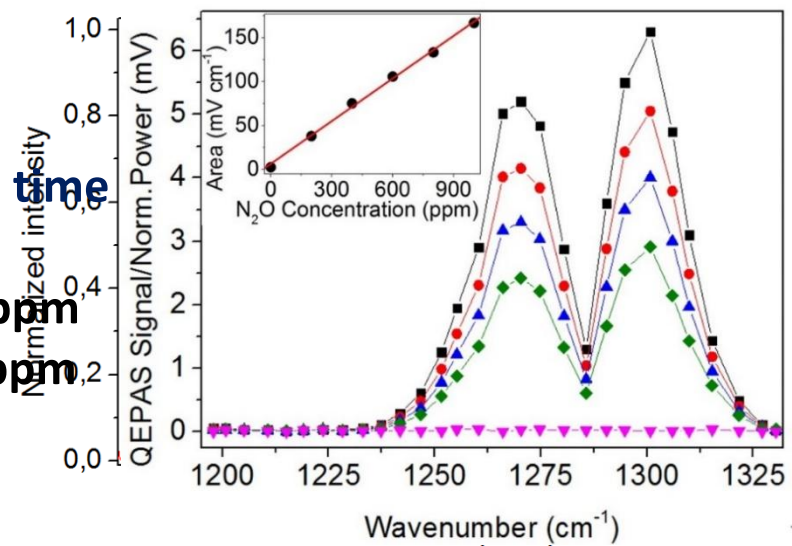
Methane and nitrous oxide detection employing a monolithic 32 DFB-QCLs array



CH₄:N₂

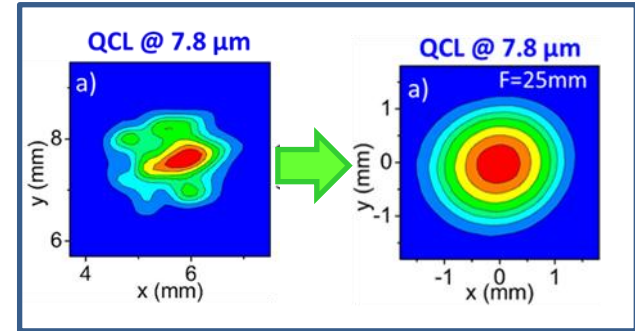
@10s integration time

MDL N₂O : 0.06 ppm
MDL CH₄ : 0.22 ppm



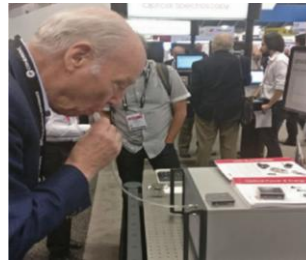
Conclusions

- HCWs for single mode delivery

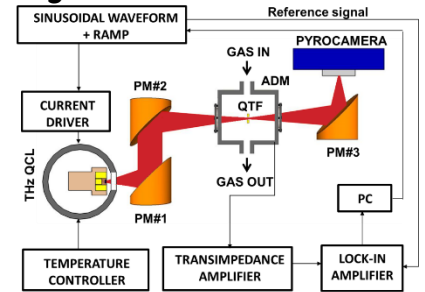


- QEPAS-based sensors employing new-design QTFs

C₂H₄ detector @PWEST



CH₃OH detector in THz

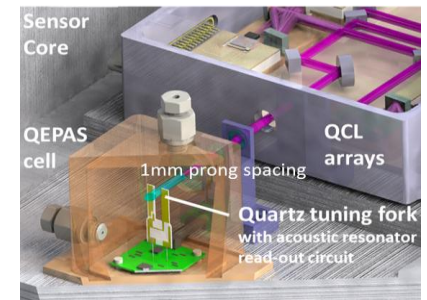


- Broadband absorbers detectors

**Ruggedized
CH₄, C₂H₆,
C₃H₈
detector**



CH₄, N₂O detector



Courses (16 CFU)

- 1) Management and knowledge of European research model and promotion of research results
- 2) How to prepare a technical speech in English
- 3) LabView introductory Course
- 4) Complex Systems
- 5) Optical sensors
- 6) Principles of optoelectronic and photonic sensors
- 7) Programming FPGA with LabView
- 8) Gaseous detectors

Teaching activities

- 1) Attività didattico-integrative per l'insegnamento "Fisica Generale" per la durata di **40 ore**, Corsi Comuni – Politecnico di Bari, responsabile Prof. V. Spagnolo. Marzo 2018-Giugno 2018
- 2) Attività didattica relativa alla disciplina di FISICA nei corsi di preparazione e approfondimento per lo studio delle materie di base e del CAD per la durata di **8 ore** – Politecnico di Bari. 13 settembre 2018 - 20 settembre 2018
- 3) Attività didattico-integrative per l'insegnamento "Fisica Generale A", Corso di Ingegneria Elettronica e delle Telecomunicazioni – Politecnico di Bari, per la durata di **40 ore** nel periodo Aprile 2016-Luglio 2016, responsabile Prof. V. Spagnolo.

Awards

- 1) Best presentation at the 70th Workshop: TeraHz physics, Erice, Sicily, 16-23 July 2016
- 2) Miglior progetto di ricerca nel contesto del corso "Management and knowledge of European research model and promotion of research results" A.A. 2015-2016

List of publications (1)

- 1) **M. Giglio**, P. Patimisco, A. Sampaolo, G. Scamarcio, F.K. Tittel, V. Spagnolo, *Allan deviation plot as a tool for quartz enhanced photoacoustic sensors noise analysis*, IEEE- Transactions on Ultrasonics, Ferroelectrics, and Frequency Control, vol. 63 (2016)
- 2) P. Patimisco, A. Sampaolo, **M. Giglio**, J.M. Kriesel, F.K. Tittel, V. Spagnolo, *Hollow Core Waveguide as Mid-IR Laser Modal Beam Filter*, Journal of Applied Physics, vol. 118, issue 12 (2015)
- 3) P. Patimisco, A. Sampaolo, L. Dong, **M. Giglio**, G. Scamarcio, F.K. Tittel, V. Spagnolo, *Analysis of the electro-elastic properties of custom quartz tuning forks for optoacoustic gas sensing*, Sensors and Actuators B: Chemical, vol. 227 (2016)
- 4) P. Patimisco, A. Sampaolo, L. Mihai , **M. Giglio**, J. Kriesel , D. Sporea, G. Scamarcio, F. K. Tittel, and V. Spagnolo, *Low-loss coupling of quantum cascade lasers into hollow core waveguides with single mode output in the 3.7-7.3 μm spectral range*, Sensors, vol. 16 (2016)
- 5) A. Sampaolo, P. Patimisco, **M. Giglio**, M. S. Vitiello, H. E. Beere, D. A. Ritchie, G. Scamarcio, F.K. Tittel, V. Spagnolo, *Improved Tuning Fork for Terahertz Quartz-Enhanced Photoacoustic Spectroscopy*, Sensors, vol. 16 (2016)
- 6) A. Sampaolo, P. Patimisco, **M. Giglio**, L. Chieco, G. Scamarcio, F.K. Tittel, V. Spagnolo, *Highly sensitive gas leak detector based on a quartz-enhanced photoacoustic SF6 sensor*, Optics Express, vol. 24 (2016)
- 7) **M. Giglio**, P. Patimisco, A. Sampaolo, J. Kriesel, F.K. Tittel, V. Spagnolo, *Low-loss and single-mode tapered hollow-core waveguides optically coupled with interband and quantum cascade lasers*, Optical Engineering, vol. 57, issue 1 (2017)
- 8) P. Patimisco, A. Sampaolo, **M. Giglio**, V. Mackowiak, H. Rossmadl, B. Gross, A. Cable, F.K. Tittel, V. Spagnolo, *Octupole electrode pattern for tuning forks vibrating at the first overtone mode in quartz-enhanced photoacoustic spectroscopy*, Optics Letters, vol.43, issue 15 (2018)
- 9) **M. Giglio**, P. Patimisco, A. Sampaolo, A. Zifarelli, R. Blanchard, C. Pfluegl, M. F. Witinski, D. Vakhshoori, F.K. Tittel, V. Spagnolo, *Nitrous oxide quartz-enhanced photoacoustic detection employing a broadband distributed-feedback quantum cascade laser array*, Applied Physics Letters, vol. 113, n° 17, 171101 (2018)

List of publications (2)

- 10) A. Sampaolo, S. Csutak, P. Patimisco, **M. Giglio**, G. Menduni, V. Passaro, F.K. Tittel, M. Deffenbaugh, V. Spagnolo, Interband Cascade Laser Based Quartz-enhanced Photoacoustic Sensor for Multiple Hydrocarbons Detection, *Sensors and Actuators: B Chemical*, *in press* (2018)
- 11) P. Patimisco, A. Sampaolo, **M. Giglio**, S. Dello Russo, V. Mackowiak, H. Rossmadl, A. Cable, F.K. Tittel, V. Spagnolo, Tuning forks with optimized geometries for quartz-enhanced photoacoustic spectroscopy, *Optics express*, *in press* (2018)
- 12) **M. Giglio**, G. Menduni, P. Patimisco, A. Sampaolo, A. Elefante, V. Passaro, V. Spagnolo, Damping mechanisms of piezoelectric quartz tuning forks employed in photoacoustic spectroscopy for trace gas sensing, *Physica status solidi*, *in press* (2018)

Conference Proceedings (1)

- 1) V. Spagnolo, P. Patimisco, A. Sampaolo, **M. Giglio**, L. Dong, G. Scamarcio, F.K. Tittel, Innovative quartz enhanced photoacoustic sensors for trace gas detection, *Proceedings of SPIE - The International Society for Optical Engineering* 9899,98990S (2016)
- 2) V. Spagnolo, P. Patimisco, A. Sampaolo, **M. Giglio**, F.K. Tittel, New developments in THz quartz enhanced photoacoustic spectroscopy, *Proceedings of SPIE - The International Society for Optical Engineering* 9934, 99340C (2016)
- 3) A. Sampaolo, P. Patimisco, **M. Giglio**, P.P. Calabrese, L. Chieco, G. Scamarcio, F.K. Tittel, V. Spagnolo, Quartz enhanced photoacoustic leak sensor for mechatronic components, *Proceedings of SPIE - The International Society for Optical Engineering* 9755, 97550D (2016)
- 4) P. Patimisco, L. Mihai, **M. Giglio**, A. Sampaolo, P.P. Calabrese, J. Kriesel, D. Sporea, G. Scamarcio, F. K. Tittel, and V. Spagnolo, Hollow-core waveguide for single-mode laser beam propagation in the spectral range of 3.7-7.3 μm , *Proceedings of SPIE - The International Society for Optical Engineering* 9755,97552M (2016)
- 5) V. Spagnolo, A. Sampaolo, P. Patimisco, L. Dong, Y. Gupta, Y. Yu, A. Geras, **M. Giglio**, P.P. Calabrese, T. Starecki, G. Scamarcio, F.K. Tittel, Quartz tuning forks with novel geometries for optoacoustic gas sensing, *Proceedings of SPIE - The International Society for Optical Engineering* 9755,97552L (2016)

Conference Proceedings (2)

- 6) A. Sampaolo, P. Patimisco, H. Zeng, **M. Giglio**, L. Dong, F. K. Tittel, and V. Spagnolo, Recent advances in quartz-enhanced photoacoustic sensors employing custom tuning fork operating at the first overtone flexural mode, Optics InfoBase Conference Papers Part F82-CLEO_Europe 2017 (2017)
- 7) V. Spagnolo, P. Patimisco, A. Sampaolo, **M. Giglio**, F.K. Tittel, Recent advances in quartz enhanced photoacoustic sensing, Optics InfoBase Conference Papers (2017)
- 8) F.K. Tittel, R.J. Griffin, N.P. Sanchez, W. Ye, Q. He, H. Zheng, **M. Giglio**, P. Patimisco, A. Sampaolo, V. Spagnolo, Trace gas spectroscopy using state-of-the-art mid-infrared semiconductor laser sources: Progress, status, and applications, Proceedings of SPIE - The International Society for Optical Engineering, 10403,1040307 (2017)
- 9) A. Sampaolo, P. Patimisco, A. Gluszek, A. Hudzikowski, **M. Giglio**, H. Zeng, F.K. Tittel, V. Spagnolo, Low power consumption quartz-enhanced photoacoustic gas sensor employing a quantum cascade laser in pulsed operation, Proceedings of SPIE - The International Society for Optical Engineering, 10111Y (2017)
- 10) **M. Giglio**, A. Sampaolo, P. Patimisco, H. Zheng, H. Wu, L. Dong, F.K. Tittel, V. Spagnolo, Single-tube on beam quartz-enhanced photoacoustic spectrophones exploiting a custom quartz tuning fork operating in the overtone mode, Proceedings of SPIE - The International Society for Optical Engineering, 10111 (2017)
- 11) V. Spagnolo, P. Patimisco, A. Sampaolo, **M. Giglio**, L. Dong, F.K. Tittel, Recent advances in quartz-enhanced photoacoustic sensing, Journal Proceedings of SPIE - The International Society for Optical Engineering 10540-105402O (2018)
- 12) A. Sampaolo, S. Csutak, P. Patimisco, **M. Giglio**, G. Menduni, V. Passaro, F.K. Tittel, M. Deffenbaugh, V. Spagnolo, Interband Cascade Laser Based Quartz-enhanced Photoacoustic Sensor for Multiple Hydrocarbons Detection, Proceedings of SPIE - The International Society for Optical Engineering 10540-105400C (2018)
- 13) P. Patimisco, A. Sampaolo, **M. Giglio**, F. Sgobba, H. Rossmadl, V. Mackowiak, B. Gross, A. Cable, F.K. Tittel, V. Spagnolo, Compact and Low-noise Quartz-enhanced Photoacoustic Sensor for Sub-ppm Ethylene Detection in Atmosphere, Proceedings of SPIE - The International Society for Optical Engineering 10540-105401Q (2018)
- 14) V. Spagnolo, P. Patimisco, A. Sampaolo, **M. Giglio**, V. Mackowiak, H. Rossmadl, B. Gross, A. Cable, F.K. Tittel, V. Spagnolo, New developments in quartz-enhanced photoacoustic spectroscopy for gas sensing applications, Optics InfoBase Conference Paper, Volume Part F110-Sensors 2018 (2018)

Conference Proceedings (3)

15) **M. Giglio**, P. Patimisco, A. Sampaolo, P.P. Calabrese, J.M. Kriesel, F.K. Tittel, V. Spagnolo, Tapered Hollow-Core Fibers Providing Single-mode Output in the 3.5-7.8 μm spectral range, Proceedings of SPIE - The International Society for Optical Engineering 10540-105402L (2018)

Conference Talks

- 1) **M. Giglio**, P. Patimisco, A. Sampaolo, F.K. Tittel, V. Spagnolo, Terahertz quartz-enhanced photoacoustic spectroscopy employing a tuning fork with enhanced sensing performance, Annual conference of COST Action MP 1204 & International Conference SMMO2016, Lisbon, Portugal
- 2) **M. Giglio**, P. Patimisco, A. Sampaolo, F.K. Tittel, V. Spagnolo, Methanol Quartz-Enhanced Photoacoustic Spectroscopy Sensor Employing a Terahertz Quantum Cascade Laser Source, 70th Workshop: TeraHz physics, Erice, Italy, 2016
- 3) **M. Giglio**, 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 Photonics West 2017, San Francisco, CA
- 4) **M. Giglio**, P. Patimisco, A. Sampaolo, P.P. Calabrese, J.M. Kriesel, F.K. Tittel, V. Spagnolo, Tapered Hollow-Core Fibers Providing Single-mode Output in the 3.5-7.8 μm spectral range, SPIE Photonics West 2018 San Francisco, CA
- 5) **M. Giglio**, P. Patimisco, A. Sampaolo, A. Elefante, F. Sgobba, F.K. Tittel, V. Spagnolo, Ethylene trace gas detection exploiting a compact quartz-enhanced photoacoustic spectroscopy-based sensor, PIERS, 2018, Toyama, Japan
- 6) **M. Giglio**, P. Patimisco, A. Sampaolo, J.M. Kriesel, F.K. Tittel, V. Spagnolo, Hollow-core waveguides for single mode delivery in the mid-infrared spectral range, (invited) D-Photon 2018, Bari, Italy
- 7) **M. Giglio**, P. Patimisco, A. Sampaolo, A. Elefante, G. Menduni, F.K. Tittel, V. Spagnolo, Recent avances in quartz-enhanced photoacoustic spectroscopy, IFN-day 2018, Lecco, Italy

Thank You

PolySense



**Polysense lab
Politecnico of Bari
University of Bari
Physics Department**

Vincenzo Spagnolo*
Pietro Patimisco*
Angelo Sampaolo*
Marilena Giglio*
Giansergio Menduni
Arianna Elefante
Stefano dello Russo
Fabrizio Sgobba
Andrea Zifarelli

*CNR-IFN Bari Unit



**Aramco Service and
Company**

Max Deffenbaugh
Sebastian Csutak



Pendar Technologies

Romain Blanchard
Christian Pfluegl
Daryoosh Vakhouri

THORLABS

Thorlabs, Inc.

Alex Cable
Bruno Gross
Verena Mackowiak
Hubert Rossmadl
Christian Kipplinger



Statek Corporation

Frank Fang



Rice University

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Opto Knowledge

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