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Bridging chromatographic and photoacoustic domains: A hybrid system for compositional and isotopic analysis of natural gas

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ABSTRACT

Natural gas (NG) analysis plays a pivotal role in the global energy landscape, and accurate quantification of its molecular and isotopic composition is essential for extraction process optimization, emission control, and environmental monitoring. Conventional detection methods based on gas chromatography (GC) coupled with flame ionization detector (FID) or mass spectrometry (MS) offer high sensitivity but either lack of specificity or are bulky and costly. This work presents a hybrid sensing platform that integrates GC with Quartz-Enhanced Photoacoustic Spectroscopy (QEPAS) combining the temporal separation capabilities of GC with the high sensitivity and molecular selectivity of mid-infrared laser spectroscopy. This dual-domain approach enables: (i) selective identification and quantification of light alkanes in complex natural gas mixtures; (ii) sub-ppm detection of propane using a single interband cascade laser emitting at 3.367 μm; and (iii) isotopic discrimination between methane isotopologues ($^{12}CH_4$ and $^{13}CH_4$) employing a quantum cascade laser operating near 7.81 μm. By overcoming spectral overlap through chromatographic separation and leveraging QEPAS's intrinsic sensitivity, the GC–QEPAS configuration offers a high-performance alternative to GC–FID/MS for in situ compositional and isotopic gas analysis.

1. Introduction

Trace gas sensing systems play a crucial role across a wide range of fields, including petroleum exploration, petrochemical processing, environmental monitoring, and biomedical diagnostics. Over the past decades, numerous chemical and optical detection methods have been developed, with the choice of the most suitable technique determined by factors such as the target gas type, concentration range, spectral resolution, sensitivity, selectivity, response time, and environmental conditions, as well as practical constraints related to system size and cost [1–3]. In recent years, the deployment of these systems for NG detection has gained increasing relevance within the oil and gas industry.

Among fossil fuels, natural gas is the cleanest and most versatile, serving as a major source for electricity generation, industrial processes, and residential energy supply, while emitting significantly fewer pollutants per unit of energy produced [4,5]. Accurate determination of its composition is therefore critical not only to ensure compliance with industrial quality standards and optimize reservoir exploitation, but also for evaluating its environmental impact. Of particular importance is the discrimination of light alkanes such as methane (CH_4), ethane (C_2H_6), propane (C_3H_8), and butane (C_4H_{10}) whose unambiguous identification in the mid-infrared spectral region (3000–4000 nm) and real-time analysis can substantially improve the accuracy of reservoir characterization and production forecasting, ultimately reducing the environmental footprint of petroleum exploration [6,7].

Furthermore, a deeper understanding of the role of light alkanes in global warming is urgently needed. Methane, ethane, and propane are potent greenhouse gases, and their monitoring is directly linked to climate modelling and mitigation strategies [8]. Particular attention has been devoted to the stability of NG hydrates, which are vast reservoirs of

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hydrocarbons trapped in oceanic sediments and permafrost regions. As ocean temperatures rise, the destabilization of these hydrates may lead to substantial methane release, amplifying greenhouse effects and triggering feedback mechanisms with far-reaching environmental consequences [9–11].

Beyond compositional analysis, isotopic ratio is a valuable indicator of processes involving isotopic fractionation. In particular, a measurement of the $^{13}CH_4/^{12}CH_4$ isotopic ratio and of its deviation from a standard reference defines the $\delta^{13}C$ parameter, which enables the identification of methane's biogenic, thermogenic, or pyrogenic origin. Biogenic methane, typically produced through microbial methanogenesis, exhibits a lower $\delta^{13}C$ value, whereas thermogenic methane, generated by thermal degradation of organic matter at depth, displays higher $\delta^{13}C$ enrichment [12–14]. Accurate isotopic discrimination therefore enables source apportionment of methane emissions, improving greenhouse gas inventories and supporting more effective mitigation strategies. Additionally, isotope-resolved detection offers insights into subsurface geochemical processes and gas migration pathways, contributing to a deeper understanding of reservoir dynamics in petroleum exploration and production [15,16].

Over the past decades, gas chromatography (GC) has become the benchmark technique for analyzing natural gas composition, owing to its ability to separate complex mixtures of volatile compounds prior to detection. In GC, a gaseous sample is carried by an inert gas through a column where interactions with the stationary phase determine distinct retention times. Upon elution, the analytes are detected through the generation of electrical signals proportional to their concentration [17,18]. Among the available detectors, the flame ionization detector (FID) remains the most widely employed one due to its high sensitivity, robustness, and linear response with respect to the number of carbon atoms, making it particularly suitable for hydrocarbon quantification in natural gas [19,20]. The FID is a destructive detector quantifying hydrocarbons by measuring the ion current generated during combustion in a hydrogen–air flame, providing a rapid and proportional response to the total carbon content of the analytes. Also, it lacks structural and isotopic selectivity, as its signal primarily depends on the overall carbon content rather than on subtle variations in molecular composition. To overcome this limitation, GC is often coupled with mass spectrometry (GC–MS), which represents the gold standard for volatile organic compound analysis. GC–MS combines the separation capability of GC with the molecular specificity of MS, enabling simultaneous qualitative and quantitative characterization of complex mixtures and trace components [21,22]. Despite its analytical superiority, GC–MS remains expensive and technically demanding, requiring skilled operation, regular maintenance of its vacuum and ionization systems, and occupying considerable space, factors that limit its suitability for compact or field-deployable platforms [23–25].

A promising alternative to the conventional physical or chemical detectors coupled with GC is represented by sensors based on Quartz-Enhanced Photoacoustic Spectroscopy (QEPAS), a non-destructive optical gas sensing technique that exploits the photoacoustic effect and employs quartz tuning forks (QTFs) as high-quality factor acoustic transducers [26–28]. In QEPAS, an infrared laser beam excites the target molecule to a vibrationally excited (rovibrational) level. The laser is modulated at one of the resonance frequencies of the QTF, which detects the pressure waves generated within the gas sample by the non-radiative relaxation processes following periodic optical absorption. Owing to its piezoelectric properties, the QTF converts these acoustic excitations into electrical signals proportional to the target gas concentration. The addition of acoustic micro-resonator tubes further enhances signal amplitude, enabling the achievement of detection limits down to the few parts-per-billion range for various hydrocarbons. Compared with conventional GC-based analytical configurations, a standalone QEPAS sensor offers several advantages, including compactness, portability, low operating cost, and the capability for in situ, real-time monitoring. Moreover, QEPAS, like other laser-based spectroscopic methods, is particularly suited for isotopic analysis, as it exploits the subtle shifts in vibrational transitions caused by the different mass of isotopologues [29]. Nevertheless, optical spectroscopic techniques can suffer from spectral interference arising from overlapping absorption features of multiple analytes within a complex gas mixture. In the mid-infrared region, for instance, the absorption bands of hydrocarbons often overlap, complicating selective detection and quantitative analysis. Additional interference from atmospheric constituents (e.g., H_2O, CO_2, etc.) can contribute to background absorption and baseline distortions, further compromising measurement specificity [30–32]. Moreover, as with other indirect spectroscopic techniques, QEPAS is susceptible to non-spectral cross-sensitivities, as the efficiency of the radiation-to-sound conversion depends on variations in the host gas matrix. In particular, the effects of oxygen and water vapor on QEPAS methane detection have been widely studied in terms of photoacoustic generation [29,33]. Nevertheless, when exploring GC-QEPAS hybrid systems, the aforementioned limitations of QEPAS can be effectively compensated by the principal advantage of GC of temporally separating the constituents of complex mixtures. The integration of these two analytical domains thus combines the chromatographic time separation provided by the column with the high sensitivity and intrinsic molecular selectivity of infrared spectroscopic detection. The first GC-QEPAS system has been demonstrated by Zampolli et al. for the detection of hazardous chemicals in mixtures containing propanol or gasoline [34] and subsequently refined into a more compact configuration employing a standard 32 kHz QTF within a 10 μL volume detection cell [35]. In this configuration, QEPAS served as an alternative to conventional physical detectors, offering enhanced molecular identification capabilities. The proposed approach relied on acquiring a complete photoacoustic spectrum approximately every 3 s throughout the analytes elution and employing the Pearson correlation coefficient to compare each measured QEPAS spectrum with reference absorption spectra from a database. However, a potential limitation of a broadband spectral correlation approach is that averaging over the whole spectral range may mask narrow spectral features not associated with the target molecule. In contrast, in this work a different spectroscopic methodology has been employed, targeting a specific absorption feature rather than relying on the correlation between broadband QEPAS spectra and reference spectra. This different methodology reduces both QEPAS measurement acquisition and analysis time.

In this work, the spectral fingerprinting capability of QEPAS and the temporal separation offered by GC are synergistically combined, allowing each technique to compensate for the limitations of the other in two key analytical scenarios. In the first one, the selectivity of QEPAS detection for alkanes, whose absorption features strongly overlap in the 3 μm – 4 μm spectral region, is enhanced through chromatographic time separation. In the second one, the intrinsic selectivity of a 7.8 μm quantum cascade laser (QCL) emission has been leveraged to excite two closely spaced optical transitions of $^{12}CH_4$ and $^{13}CH_4$, thereby overcoming the inability of conventional GC-coupled detectors to discriminate between methane isotopologues. The GC-QEPAS prototype developed and presented in this study employs a low-resonance frequency QTF to account for the relaxation dynamics of slow relaxing molecules [29]. Within this configuration, the proposed approach extends the application of GC–QEPAS systems to simultaneous compositional analysis of light hydrocarbons and isotopic discrimination of methane with high precision, providing a powerful alternative to conventional chromatographic detection.

2. Experimental setup

The core of the experimental apparatus employed in this work is shown in Fig. 1.

The experimental setup was built around an Agilent 8890 gas chromatograph equipped with an HP-PLOT/Q capillary column (30 m length, 0.53 mm inner diameter, 3 μm film thickness), featuring a non-

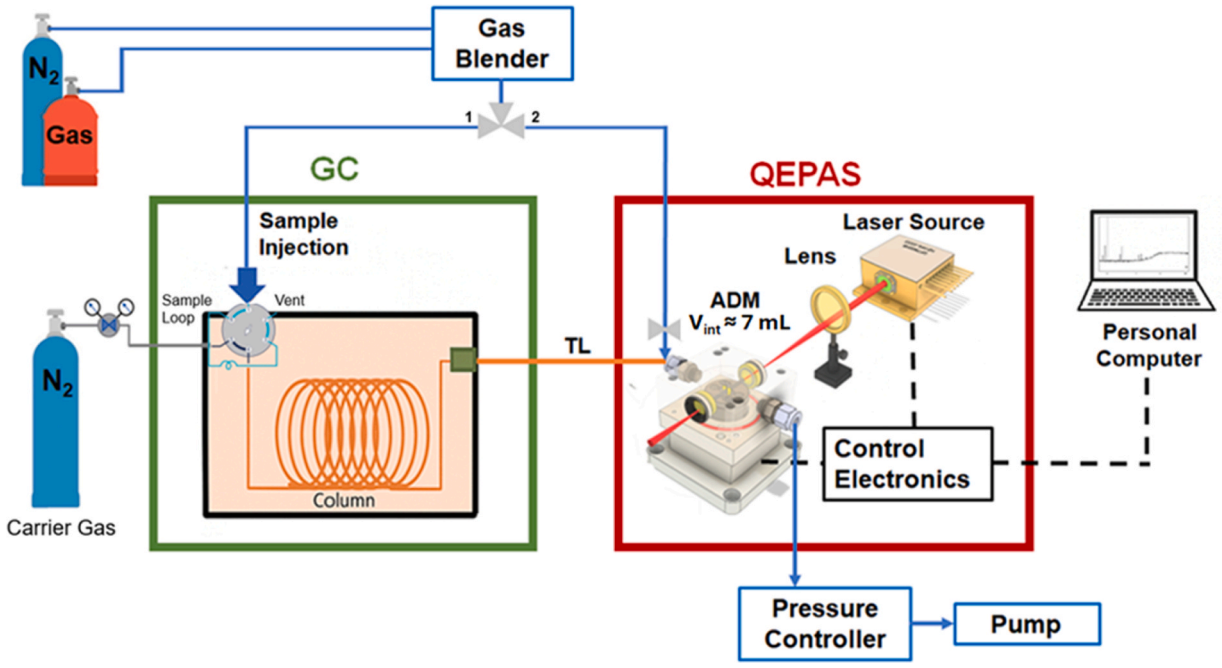

Fig. 1. Schematic of the core of the experimental apparatus. TL – Transfer Line; ADM- Acoustic Detection Module; ADM V_{int} – Acoustic Detection Module Internal Volume.

polar dimethylpolysiloxane stationary phase with an operational temperature range of – 60 °C to 260 °C. Nitrogen served as the carrier gas, with flow rates ranging from 5 to 15 standard cubic centimeters per minute (sccm). An auxiliary nitrogen makeup flow of 20 sccm was optionally applied to optimize chromatographic performance and maintain consistent analyte transfer to the detector. The oven temperature program was set to an initial temperature of 40 °C held for 4 min, followed by a linear ramp of 30 °C/min up to 180 °C, maintained for 8 min. Gas mixtures were prepared using a six-channel MCQ gas blender (MCQ Gas Blender 6000), which provides precise control over both the composition (number and type of hydrocarbons) and the concentration of each analyte. The gas blender was connected to the GC or the QEPAS inlet through the valves labelled as 1 or 2 in Fig. 1. The gas mixtures were introduced into the GC system using a gas sampling valve, with a sample loop of 250 µL, continuously delivered to the inlet. After chromatographic separation, analytes were transferred to the QEPAS detection module through a transfer line consisting in a 50 cm-long fused silica capillary (0.53 mm inner diameter) thermally insulated with a protective sleeve to minimize heat losses and prevent condensation, thus ensuring stable transfer conditions and preserving analyte integrity for subsequent photoacoustic detection. The separated species entered an acoustic detection module (ADM) with an internal volume of ~7 mL, equipped with two ZnSe windows coated with a 3–12 µm antireflection layer. The ADM housed a T-shaped quartz tuning fork with a resonance frequency f_o = 12454.4 Hz and a quality factor of 9000 at atmospheric pressure, acoustically coupled to a pair of resonator tubes (4.0 mm length, 0.6 mm inner diameter, 0.8 mm outer diameter) designed to enhance the acoustic response. For natural gas analysis, and propane detection in particular, photoacoustic excitation was provided by an interband cascade laser (ICL) emitting at 3367 nm, focused between the QTF prongs using a ZnSe lens with a 50 mm focal length. For methane isotopologues analysis, the ICL was replaced by a QCL emitting at 7.81 µm, selected to target strong absorption features of $^{12}CH_4$ at 1275.39 cm^{-1} and $^{13}CH_4$ at 1275.78 cm^{-1}, while maintaining sufficient spectral separation for accurate isotopic discrimination.

The laser source was driven by a Thorlabs ITC4002 current and temperature controller. In both configurations, the measurement was performed in wavelength modulation (WM) mode by applying a sinusoidal dither at $f_o/2$ to the laser current, whose setpoint was kept fixed in correspondence of a specific emission wavelength, using a Keysight EDU33212A waveform generator, while the quartz tuning fork signal was demodulated at f_o by a Zurich MFLI lock-in amplifier, with the integration time fixed at 100 ms.

The operating pressure inside the ADM was regulated by an Alicat MC3S-D pressure controller placed downstream of the gas cell and connected to a diaphragm pump, ensuring stable and reproducible operating conditions.

3. Natural gas analysis

To provide a clear and representative demonstration of the effectiveness of the proposed hybrid approach, and to compare the performance of a conventional detector with that of the photoacoustic module, chromatograms of a certified natural gas-like mixture were acquired and analyzed as a case study. An NG-like gas cylinder composed of 85% CH_4, 5% C_2H_6, 3% C_3H_8, 2% C_4H_{10}, and 5% CO_2 was diluted to 20 vol% NG in N_2. The gas chromatograph was first coupled with an FID and subsequently with the QEPAS module. In both configurations, the valve 1 was open and the valve 2 was closed, the GC column flow was set at 15 sccm and the same temperature ramp program was employed, selected to optimize the hydrocarbon separation. The FID was operated at 300 °C, while the QEPAS detector functioned at ambient temperature and atmospheric pressure, with an additional make-up flow of 20 sccm to partially compensate for the 7 mL ADM volume and enhance the hydrocarbons separation throughout the module. The ICL current was fixed at 72 mA, corresponding to a wavelength of 2967.4 cm^{-1}, which coincides with an absorption peak of propane, a choice further discussed in the following section. At this fixed wavelength, absorption features of methane, ethane, and butane are known to partially overlap [30], due to the common C−H bond stretching mode, resulting in signals whose amplitude depend on both the absorption band intensity strength and the concentration of each of the first four alkanes in the mixture. The chromatograms acquired by coupling the GC with the FID and the QEPAS detector are shown in Fig. 2.

Coupling a QEPAS system with a chromatographic column enables sequential photoacoustic detection of individual hydrocarbons within a

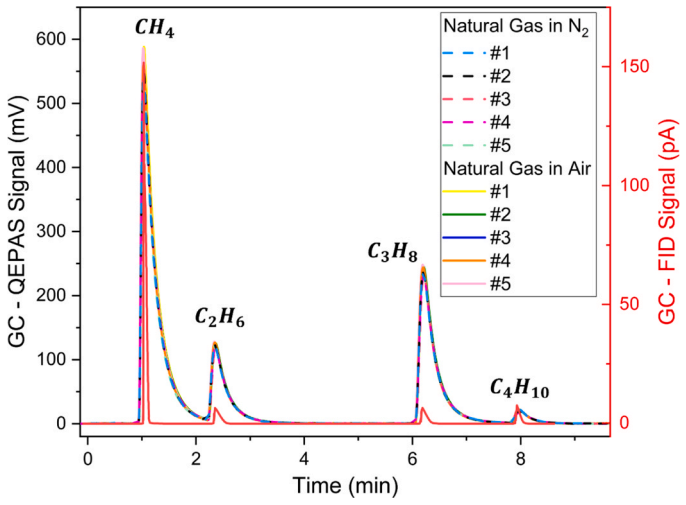

Fig. 2. Left black y-axis: chromatograms of 20% of NG (85% methane, 5% ethane, 3% propane, and 2% butane) diluted in N_2 (cyan, black, red, magenta, and light green dashed lines) or in wet air (yellow, green, blue, orange, and pink solid lines) obtained using the GC-QEPAS system. Right red y-axis: chromatogram of 20% of NG diluted in 80% N_2 obtained using the GC-FID (red solid line). Peaks corresponding to CH_4, C_2H_6, C_3H_8, and C_4H_{10} are highlighted.

mixture, effectively overcoming the spectral interference issues encountered in standalone QEPAS experiments. As expected, the retention times were identical for both detectors, as they depend solely on the GC column characteristics and oven temperature ramp. Specifically, CH_4, C_2H_6, C_3H_8, and C_4H_{10} were eluted at 0.9, 2.2, 6, and 7.85 min, at a temperature of 40, 40, 97, and 150 °C, respectively. However, while the FID produced sharp and well-defined chromatographic peaks, QEPAS detector yielded broader peaks with noticeable tailing and a reduced resolution. This broadening/tailing can be attributed to the larger internal volume of the ADM and to the additional connectors required to couple it to the transfer line, both of which contribute to band dispersion.

Regarding chromatogram intensities, the FID response is proportional to the analyte concentration and the number of carbon atoms, explaining the predominance of the methane signal in the chromatogram, followed by those of the heavier hydrocarbons. In contrast, the QEPAS signal depends not only on the analyte concentration but also on the absorption strength at the laser emission wavelength, which can enhance the response of a less abundant molecule. Indeed, with the laser tuned to the propane absorption peak, the propane signal is higher than the ethane one, despite its lower concentration.

To assess the GC-QEPAS measurement repeatability, a set of five subsequent measurements was performed. To further demonstrate the applicability of the proof-of-concept hybrid system in practical fields, such as detection of natural gas leaks dispersed in ambient air, the same set of five measurements was repeated by diluting NG-like mixture to 20 vol% NG in wet air, as shown in Fig. 2.

For each chromatogram, the analyte signal was evaluated by integrating the area under its corresponding chromatographic peak. It should be noted that in the GC–QEPAS configuration, the analyte interacts with the sensitive element as a temporally distributed packet. Therefore, with the laser wavelength fixed at the absorption peak of propane, the QEPAS signal directly reflects the instantaneous concentration as a function of time, whereas the total analyte amount corresponds to the integrated area under the chromatographic peaks.

For each chromatographic peak, raw GC–QEPAS traces were baseline-corrected by subtracting the signal recorded with pure nitrogen, in order to remove the instrumental offset. The integration window was defined based on the baseline noise level: the starting point was set at the first data point exceeding the baseline by more than 2σ (σ being the standard deviation of the baseline), while the endpoint was defined as the last data point returning within 1σ of the baseline.

The signal within this interval was integrated using a rectangular approximation method. The standard deviation σ_{Area} of the area underneath the GC–QEPAS spectrum can be obtained by using the propagation of uncertainty law:

$$\sigma_{Area} = \sqrt{\sum \left[\left(\frac{\partial A}{\partial x_i} \right)^2 \sigma_{x_i}^2 + \left(\frac{\partial A}{\partial S_i} \right)^2 \sigma_{S_i}^2 \right]} \quad (1)$$

where x_i and S_i are the i^{th} rectangle base and height, respectively. Here, σ_{x_i} accounts for the lock-in sampling time uncertainty (0.15 s), while σ_{S_i} corresponds to the 1σ noise of the QEPAS signal.

To evaluate the system stability, the mean integrated area was then calculated for each analyte within each set of measurements and has been reported in Table 1, together with the standard deviation of the mean and the relative standard deviation (RSD).

The relative standard deviation values were below 3.1% in all cases, demonstrating good repeatability and overall system stability. No significant differences were observed between measurements performed in nitrogen and wet air, indicating that environmental interferents, such as O_2, CO_2, and H_2O, do not introduce measurable effects. The consistency of the results across the two matrices therefore provides combined validation of the absence of interference and the robustness of the measurements.

The stability of the QTF resonance frequency throughout the chromatographic run was also evaluated. With this aim, the QTF response to a sinusoidal voltage excitation at the frequency f_0, exploiting the inverse piezoelectric effect, was acquired with the laser switched off. Variations of less than 1% of the QTF electrical signal were observed as each hydrocarbon packet passed through the ADM, indicating a slight resonance shift caused by changes in the gas matrix affecting QTF oscillation damping. However, these small perturbations were negligible and did not significantly impact photoacoustic detection performance.

4. Propane detection

The spectroscopic investigation of hydrocarbons conducted so far has shown that only methane and ethane can be readily detected with a very low cross-sensitivity, even at high concentrations, owing to their well-spaced, quasi-Lorentzian transitions distributed throughout the 3 – 4 μm spectral region. Within the same range, propane is the first of the heavier alkanes that requires extensive multivariate analysis for its discrimination from CH_4 and C_2H_6. This difficulty arises from its broader and less intense absorption features, which contribute primarily to a sort of diffuse absorption background.

To improve the discriminability of propane with respect to methane and ethane, and thus the overall selectivity of the analytical approach, previous purely spectroscopic studies adopted operating conditions that were dramatically unfavourable for propane itself, both in terms of pressure and modulation depth. Consequently, the detection limits for C_3H_8 in hydrocarbon mixtures remained above the ppm level [30]. For this reason, propane was selected in the present study as a benchmark analyte to demonstrate the advantages of coupling a QEPAS sensor with

Table 1
Mean integrated area, standard deviation of the mean (SD) and relative standard deviation (RSD) for each analyte peak calculated over the two sets of measurements in N_2 and in wet air.

	Mean Integrated Area [mV*sec]		SD [mV*sec]		RSD [%]	
	N_2	Wet Air	N_2	Wet Air	N_2	Wet Air
CH_4	*10620.0*	*9925.0*	*168.0*	*193.0*	*1.6*	*2.0*
C_2H_6	*2349.4*	*2397.5*	*60.0*	*19.7*	*2.6*	*0.8*
C_3H_8	*4720.0*	*4905.4*	*122.6*	*82.2*	*2.6*	*1.7*
C_4H_{10}	*371.0*	*366.7*	*9.7*	*11.0*	*2.6*	*3.0*

a GC, thereby eliminating spectral interference from methane and ethane and enabling propane sub-ppm detection under optimized operating conditions, i.e., the laser modulation depth and the operating pressure.

For the quantitative evaluation of propane, the sensor was calibrated using a certified gas cylinder containing 0.1% of C_3H_8 in N_2 and the corresponding GC–QEPAS responses were recorded over a concentration range from 100 to 1000 ppm.

The same procedure used for the quantification of the NG-like sample, based on the integration of the chromatographic peak area, was also applied to the evaluation of the propane signal for sensitivity assessment. The resulting peak areas, together with the corresponding σ_{Area} defined in Eq. 1, were plotted against the known propane concentrations to construct the calibration curve shown in Fig. 3.

A linear regression of the calibration curve yielded a slope of 5.07 mV·sec·ppm^{-1} . The limit of detection (LoD) was defined as the propane concentration corresponding to an integrated area equal to σ_{area} and, for the GC–QEPAS method using the setup described above, this value was found to be 0.4 ppm. With this configuration, an order of magnitude improvement in sensitivity is achieved compared to the use of a standalone QEPAS sensor in [36], by benefiting from both the temporal compression of the discrete gas sample by the chromatographic process and the optimized pressure and modulation depth operating conditions. A more rigorous evaluation of the peak area would require fitting the chromatographic profiles with functions accounting for stream deformation and diffusion effects arising from the volume mismatch between the transfer line and the acoustic detection module. However, such modelling lies beyond the methodological scope of the present work and would, in any case, be specific to the internal volume and geometry of the ADM used in the presentsetup.

5. Methane isotopologues analysis

As discussed above, optical techniques, and QEPAS in particular, benefit significantly from coupling with a GC, as the temporal separation provided by the chromatographic process enhances selectivity and improves detection performance, especially for gaseous components exhibiting broad and weak absorption bands.

However, this advantage does not extend to isotopic analysis, since the isotopologues of the same molecule are eluted at the same time and are further detected within a single chromatographic peak when using conventional detectors such as an FID. In this context, infrared spectroscopy complements gas chromatography by exploiting the high spectral resolution of narrowband laser sources (in the order of MHz or below), which provide intrinsic selectivity for the optical transitions of individual isotopologues. Although these transitions are spectrally close when sharing the same ground state, they remain readily distinguishable. This capability is particularly evident for methane isotopologues $^{12}CH_4$ and $^{13}CH_4$, which co-elute chromatographically and remain indistinguishable by FID, but they can be clearly resolved by QEPAS due to their distinct absorption features [37].

To identify the most suitable absorption lines and optimal operating conditions for isotopologue discrimination, preliminary exploratory measurements were conducted using the standalone QEPAS configuration, by closing the valve 1 and opening the valve 2. By superimposing a slow triangular voltage ramp at 10 mHz onto the fast dither at $f_0/2$, the QCL current was swept in order to vary its emission wavelength and obtain a QEPAS spectral scan of the target absorption lines of the two methane isotopologues. The measurements were carried out using a certified gas cylinder containing 0.1% of CH_4 in N_2 at a working pressure of 200 Torr, chosen to ensure both adequate spectral resolution and high signal intensity. The optimal dither amplitudes maximizing the $^{12}CH_4$ and $^{13}CH_4$ signals were determined to be 12 mA and 20 mA, respectively. The spectral scan shown in Fig. 4 was obtained with a modulation amplitude of 20 mV, selected to maximize the weakest $^{13}CH_4$ signal.

GC–QEPAS measurements were subsequently performed using a gas cylinder containing 10% of methane in N_2, with a certified isotopic composition of 98.8% $^{12}CH_4$ and 1.12% $^{13}CH_4$. The valve 1 was opened and the valve 2 was closed. The column flow was kept at 15 sccm and no makeup gas was required. To selectively target each isotopologue, the QCL current was first set to 269 mA, corresponding to the absorption peak of $^{12}CH_4$, and then adjusted to 278 mA to address the $^{13}CH_4$ transition. Each measurement was performed using the previously optimized modulation amplitude. The resulting chromatograms, shown in Fig. 5, clearly demonstrate that the two isotopologues co-elute at the same retention time but are distinctly identified by the QEPAS detector through their specific absorption features.

To quantify the system response, calibration experiments were performed by varying the methane concentration from 1% to 10%. As in the case of propane calibration, the integrated peak areas were plotted

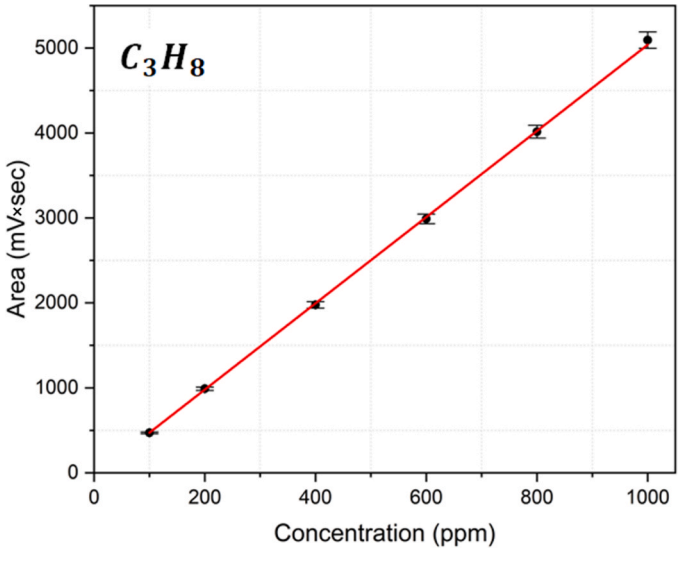

Fig. 3. Calibration curve for propane obtained with the GC–QEPAS system. The plot shows the integrated signal area (mV×sec) as a function of propane concentration (ppm) (black dots) and the corresponding best linear fit (red solid line).

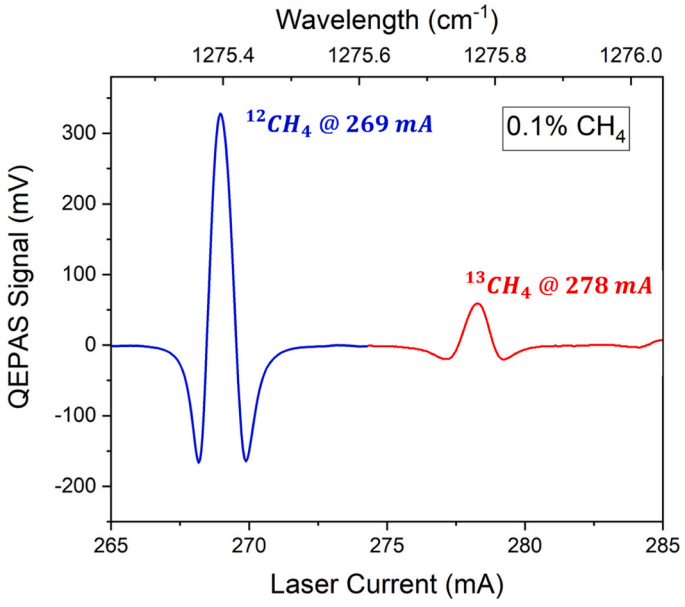

Fig. 4. Spectral scans of selected $^{12}CH_4$ absorption line (blue line) and $^{13}CH_4$ (red line) absorption line.

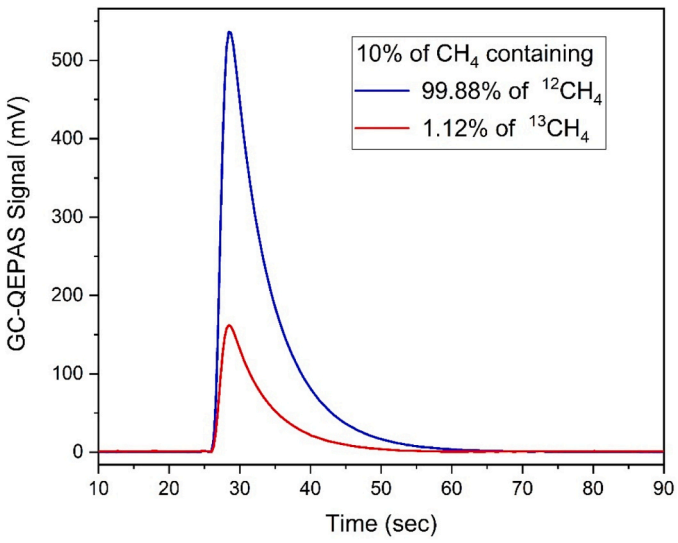

Fig. 5. Overlapped chromatograms of 10% of methane in N_2, with an isotopic composition of 98.8% $^{12}CH_4$ and 1.12% $^{13}CH_4$.

against the isotopologue concentrations to construct the calibration curves shown in Fig. 6.

In both cases, a linear response was observed, with slopes of 399.3 mV·sec·ppm^{-1} for $^{12}CH_4$ (Fig. 6(a)) and 1.0 mV·sec·ppm^{-1} for $^{13}CH_4$ (Fig. 6(b)). These calibration factors were subsequently used to determine the LoDs, which were found to be 15 ppm for $^{12}CH_4$ and 2.5 ppm for $^{13}CH_4$, with a lock-in integration time of 100 ms. Following the same procedure described in [37], these values yield an estimated sensitivity to δ^{13}_Cvariations of ~ 22‰, a precision value still acceptable for natural gas applications, such as source apportionment, considering that biogenic methane is often depleted in ^{13}C, with $\delta^{13}C$ values lower than 60‰, whereas thermogenic methane typically exhibits $\delta^{13}C$ values higher than 50‰ [37–40].

6. Conclusions

In this work, we have presented a gas sensing system that integrates gas chromatography with quartz-enhanced photoacoustic spectroscopy, using natural gas as a representative case study. The combination of these two analytical domains enhances both the sensitivity and the selectivity of each technique when used independently. The proposed GC–QEPAS configuration demonstrated clear discrimination among light alkanes in complex gas mixtures, in nitrogen- as well as in air-based matrices, while the chromatographic separation enabled highly sensitive propane detection down to 400 ppb, an order of magnitude improvement over previous standalone QEPAS results, without requiring extensive data post-processing. Furthermore, the system successfully resolved methane isotopologues ($^{12}CH_4$ and $^{13}CH_4$), a task achievable by gas chromatography only when coupled with a mass spectrometer.

At its current stage of development, the system relies on laboratory-scale instrumentation and is not yet optimized for compactness or portability. However, compared to GC–MS approaches, the GC–QEPAS methodology offers important intrinsic advantages, including reduced maintenance requirements and enhanced portability. These features provide a strong foundation for the development of more portable and field-deployable platforms.

Future developments will therefore focus on system miniaturization and integration. In particular, efforts will be directed toward improving chromatographic resolution by minimizing internal volumes, especially through the design of an ADM with a total volume below 1 mL, potentially coupled with a micro-GC. For isotopic applications, the use of dual laser beams coupled to a single QTF in a frequency-multiplexing detection scheme will be investigated to enable simultaneous detection of methane isotopologues [37].

Thanks to its versatility and high sensitivity, the GC–QEPAS approach holds great promise for a wide range of analytical applications beyond hydrocarbon detection. Current research efforts are extending this method to food quality assessment, with ongoing studies focused on the spectroscopic characterization of volatile compounds in coffee and hazelnuts [41].

CRediT authorship contribution statement

Pietro Patimisco: Writing – review & editing, Investigation. **Vincenzo Spagnolo:** Writing – review & editing, Supervision, Conceptualization. **Jimmy Zanotto:** Writing – review & editing, Methodology. **Damien Fernandez:** Writing – review & editing, Methodology. **Liam O'Faolain:** Writing – review & editing, Resources, Funding acquisition. **Giansergio Menduni:** Writing – review & editing, Methodology, Data curation. **Arianna Elefante:** Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **Lavinia Anna Mongelli:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Angelo Sampaolo:** Writing – review & editing, Writing – original draft,

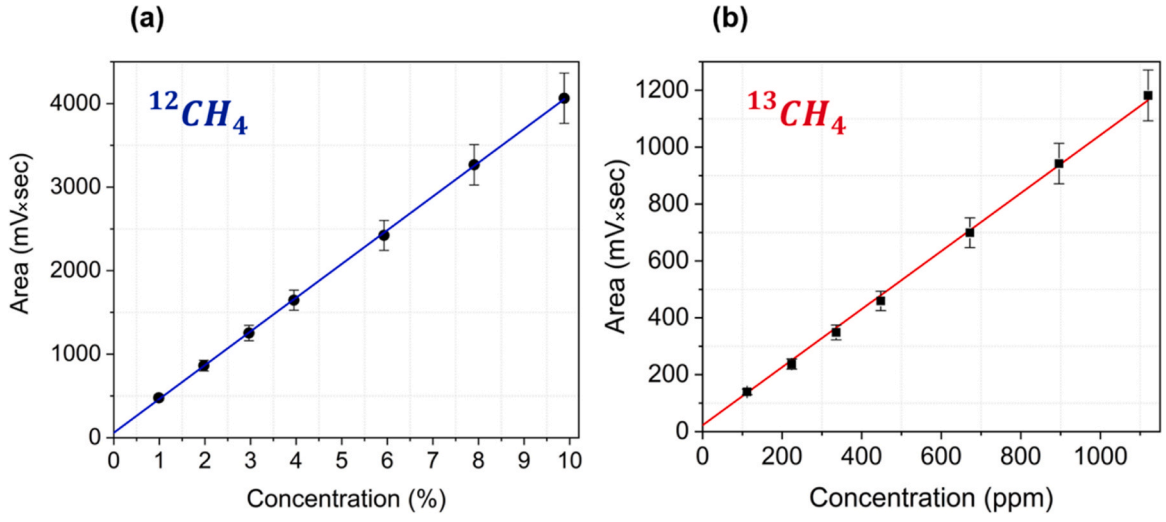

Fig. 6. Calibration curve for $^{12}CH_4$ (a) and for $^{13}CH_4$ (b) and the corresponding best linear fit.

Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Andrea Zifarelli:** Writing – review & editing, Investigation. **Marilena Giglio:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Formal analysis, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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