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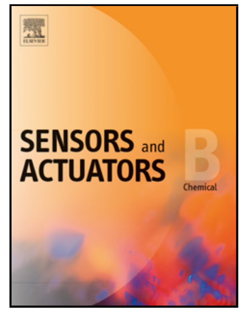

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Author: T.A. Vincent J.W. Gardner

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A low cost MEMS based NDIR system for the monitoring of carbon dioxide in breath analysis at ppm levels

T.A. Vincent, J.W. Gardner*

School of Engineering, University of Warwick, Coventry, UK.

*Corresponding author: Julian Gardner, J.W.Gardner@warwick.ac.uk, School of Engineering, University of Warwick, Library Road, Coventry, CV4 7AL, UK.

Highlights

- MEMS based NDIR system for ppm CO_2 detection with lock-in amplifier.
- Fast 1.3 s response time for breath-by-breath analysis.
- Portable breath analyser designed for measuring metabolic rate of subjects.
- Effect of path length on NDIR system investigated with novel sensor housing.
- Silicon on insulator IR emitter used for low power, low cost gas detection.

Abstract

The molecules in our breath can provide a wealth of information about the health and well-being of a person. The level of carbon dioxide (CO_2) is not only a sign of life but also when combined with the level of exhaled oxygen provides valuable health information in the form of our metabolic rate. We report upon the development of a MEMS-based non-dispersive infrared CO_2 sensor for inclusion in a hand held portable breath analyser. Our novel sensor system comprises a thermopile detector and low power MEMS silicon on insulator (SOI) wideband infrared (IR) emitter. A lock-in amplifier design permits a CO_2 concentration of 50 ppm to be detected on gas bench rig. Different IR path lengths were studied with gases in dry and humid (25 % and 50 % RH) in order to design a sensor suitable for detecting CO_2 in breath with concentrations in the range of 4 to 5 %. A breath analyser was constructed from acetal and in part 3D printed with a side-stream sampling mechanism and tested on a range of subjects with two data-sets presented here. The performance of the novel MEMS based sensor was validated using a reference commercial breath-by-breath sensor and produced comparable results and gave a response time of 1.3 s. Further work involves the detection of other compounds on breath for further metabolic analysis and reducing the overall resolution of our MEMS sensor system from *ca*. 250 ppm to 10 ppm.

Keywords

CO_2 ppm detection, breath analysis, metabolic rate, NDIR, SOI, thermopile detector.

1. Introduction

The carbon dioxide concentration of exhaled breath is commonly monitored for patients in intensive care units (ICUs). Often the measurement simply informs health carers that the patient is maintaining a steady breathing rate though, when paired with exhaled oxygen content, it can also provide metabolic information and guide the nutritional intake necessary for the optimal recovery [1]. Similarly, the measurement of exhaled breath outside hospital life support units is usually limited to those who volunteer for prolonged research studies or professional athletes [2]. Target metabolic rate advice would benefit the general population, not just select groups. The rollout of breath analysis units is limited by both the high equipment cost and lack of accurate sensors [3]. In this paper we present a low-cost, low power consumption and low response time sensor system, which is suitable for use in breath analysis. The device has an operating range from 5% CO_2 in air down to about 50 ppm depending on the IR path length configuration and sampling methodology. For example, the limit of detection of the system is around 250 ppm with a 10 mm optical path length in breath.

1.1. Breath Analysis

It has been reported a 43 % of ICU patients are malnourished, and often their dietary requirements are calculated from basic estimation equations, unsuited for such situations. Calculation errors have been reported in the range of 7-55 % [4]. The lack of accurate metabolic rate determination threatens their recovery process, where patients need varying calorific intake dependent on their recovery phase. Correct nutrition can preserve bodily functionality and hasten recovery, saving hospital space and money. Beyond the ICU, in the UK is it is estimated the National Health Service (NHS) will have spent £15.4 billion in 2015 on treatments related to obesity and overweight [5]. The level of morbid obesity has doubled over the past 20 years [6]. Prevention of further obesity cases and promotion of a healthy lifestyle would be beneficial to the community. Breath analysis offers a means of providing this guidance, specifically CO_2 analysis can contribute towards metabolic rate analysis; we are developing a portable device for such purposes, and report upon our success developing a breath CO_2 monitor. Our goal is to calculate daily energy expenditure (EE) of a subject to an accuracy of 1%, based on metabolic rate measurements over a 24 hour period. Data recorded from prior experiments in respiratory chambers demonstrated the concentration of CO_2 exhaled must be measured to within an accuracy of 1.25 %. Exhaled CO_2 is usually in the range of 4-5 % [7].

The current generation of commercial breath analysers are restricted to applications in a clinical practice, where clinicians are required to interpret raw data. We are working towards a user friendly measurement system for CO_2 content of breath, where results are displayed on an easy to read format on, for example, a smart phone or laptop computer. To compensate for sensor drift and varying environmental ambient conditions, accurate determination of energy expenditure therefore requires measurements of both inhaled and exhaled gas. The contrasting conditions of inhaled gas and exhaled breath prove challenging to develop a breath-by-breath monitoring system. Inhaled gas, from the ambient environment where the subject is located will vary, but usually, taking the UK as an example, the temperature of inhaled air will range from 4 to 22 °c and the humidity from 40 to 85 %. These parameters fall into the standard operating range for many gas sensors. However, exhaled breath is nearly completed saturated with water vapour (90 % RH expected or higher[8]) and at a higher than ambient temperature (36 °c [9]) it is prone to condense inside sensors, if colder and if the water vapour is not removed. Thus the type of sensors have to be selected as to be resilient to considerable humidity variance.

1.2. NDIR sensors

NDIR sensors operate on the principle of IR radiation being absorbed by a target gas. Utilisation of different absorption bands in the IR spectra allows the sensor to be specific to a desired gas. CO_2 strongly absorbs IR radiation at a wavelength of 4.3 μm, fortunately avoiding the water vapour absorption bands at 2.5 to 2.9 and 5.2 to 7.5 μm [10]. Exhaled breath can contain other gases and volatile organic compounds (VOCs), such as carbon monoxide, although these other components are usually in the ppm to ppb range [11].

The IR path length between the source and detector dictates the gas concentration the sensor is able to detect; the relationship is described by Beer's Law, $I(c) = I_0 \exp(-k_g c\, l)$ [12]. The law can be used to calculate the reduction in intensity of IR radiation from initial value (without any target gas present) I_0 to the intensity *I(c)* received by the thermopile, due to the concentration *(c)* of a particular gas [13]. The absorption index of CO_2 at a given wavelength (4.3 µm) is denoted by k_g and the IR optical path length given by *l*. The received signal, the intensity of IR radiation, decreases exponentially with increase in path length. A shorter path yields a higher signal output from the IR detector, however sensitivity is decreased, as a shorter optical path decreases the distance for the gas to be absorbed by the radiation. Consequently, a longer path produces a larger differential response for a chance in gas concentration, thus lower concentrations can be distinguished.

NDIR sensors traditionally use micro bulbs as sources of IR. Although readily available, bulbs provide a cumbersome radiation source, compared to newer technology such as micro hotplates. In particular, SOI IR emitters have greater reliability, faster response and smaller physical dimensions [14,15]. Exhaled gas is difficult to store and transport [16], thus sampling breath-by-breath is our preferred method. This presents an additional demand for the device, as a short exhale of approximately 3 seconds needs to be recorded and analysed [17]. In terms of affordability, MEMs based IR emitters cannot compete with a simple IR micro bulbs [18] but affordable CMOS based *systems* (with detectors) are possible but not yet produced for the mainstream market. The state of the art CO_2 detection system is yet to be well defined. The field of NDIR detection is still open to development [19], in that there is a need for low cost, low power and precise instruments (under €25) and this has yet to be realised.

Silicon CO_2 sensors have been studied for over a decade, however in recent years the focus of development has been on improving sensor design to enable greater sensitivity, faster response with lower power consumption [20]. More recently, plasmonic CMOS devices, such as the emitter reported here, offer enhanced emissivity at a desired IR wavelength. It has been reported an almost four fold improvement can be achieved when comparing the emission intensity of a plasmonic device to a non-plasmonic counterpart [21]. Small CO_2 sensors are commonplace in air quality monitoring. An array of thermopile sensors can be used to detect multiple gases (e.g. three on a 2×2 array, with one reference channel) [22]. CO_2 sensors based on the principle of radiometric thermal conductivity offer lower power consummation than traditional NDIR systems, however at the expense of resolution and complexity. Sensor systems designed to operate in ambient air avoid some of the challenges faced by systems required to operate in environments high in humidity or temperature. Medical CO_2 sensors must operate in the conditions experienced on exhaled breath. Harsh environment CO_2 sensors have also been reported [23], but their robust and costly design is not appropriate for breath sensing.

1.3. Signal Acquisition

Thermopile detectors are susceptible to high levels of noise, due to three types of noise: thermal, shot and signal noise [20]. In the case of an NDIR system the thermopile receives IR radiation transmitted by an emitter. Fitted with a 4.26 µm filter, the radiation received by the thermopile can be related to the concentration of CO_2 present in the test chamber. However, the relatively small, desired response to a change in concentration cannot always be extracted from the high noise level. Thermal noise has been reported as the predominant noise source, being at least five times greater than radiation background noise [20]. The noise is related to the mean-square noise voltage (V_{rms}) over a conductor with resistance R by $V_{rms} = \sqrt{4\, k\, T\, R\, \Delta v}$. The measurement frequency bandwidth is denoted by Δv, the temperature by T and Boltzmann's constant by k. The resistance of the thermopile (in the range of 60-70 kΩ) can therefore be seen to affect the level of thermal noise, which also depends on the signal bandwidth. This type of noise usually requires high order filters to remove. In this paper we report a lock-in amplifier system with low bandwidth and filters to monitor accurately the change in received IR radiation caused by the concentration of CO_2 in a test chamber.

A lock-in amplifier compares a received signal, plagued with noise, and compares it with a given reference signal. Signals with the frequency of the reference wave are recovered from the combination of all other components of the input noisy signal [24]. For this technique to be effective, the operation frequency of the NDIR system must avoid bands of noise, for instance 50 Hz noise from laboratory equipment or DC low frequency noise. It is possible to implement the device using a microcontroller, however for our work, we wish to minimise the computational load. Thus a dedicated IC chip was used for extracting the desired signal. Lock in amplifiers are commercially available; previous reports of using lock-in amplifiers in CO_2 detection commonly use laboratory based equipment, unsuitable for a portable instrument [12,25]. Affordable commercial CO_2 sensors often report response times in the range of 4-30s [26]. The optical absorption process occurs almost instantaneously, thus the processing of the signal must lengthen the response time. Breath-by-breath sensors require the highest dynamic response times with perhaps a resting respiratory rate of 10-12 cycles per minute [27]. Use of a lock-in amplifier aids quick extraction of the desired response signal, mitigating the need for high order filter, with corresponding high time constants.

2. Experimental Setup

Our NDIR system was designed for inclusion in a mobile device, thus size and power consumption are critical factors. The system comprises a wideband IR micro-hotplate source (CCS112, Cambridge CMOS Sensors Ltd, UK) with a silicon thermopile detector (HMS-J21, Heimann Sensor, Germany). The device has an active area of 1.2 mm x 1.2 mm and a noise level of 37 nV/ $Hz^{1/2}$. The detectivity of the thermopile is 1.4×10^8 cm $Hz^{1/2}$ /W. The theoretical limit of detection of our system was calculated as 12 ppm, using a formula suggested by Sklorz [28] and parameters reported for the device [29,30], given a temperature of 500 °c. The gas specific absorption coefficient was calculated as 241.9 cm^{-1}. The components are shown in Figure 1, including a MEMS based IR emitter. The emitter has been developed as to optimise IR emissivity in the region desired for CO_2 detection. A 4.26 µm IR filter (silicone coated with 0.18 µm bandwidth) was employed to detect CO_2 but minimise the cross-sensitivity to other gases such as CO and water vapour. Within the bandwidth of the filter, the absorbance of water has been reported to be in the range of 0.02 to 0.03 [31]. A single channel detector was used. A dual channel device was trialled in preliminary measurements. It reduces base line drift but does increase the instantaneous level of voltage noise in the system - the noise present in the reference and sensing channels are not in phase. When the outputs are processed, the noise became additive, thus the signal to noise ratio decreased by a factor of $\sqrt{2}$. A single channel setup was preferred to provide an improved signal to noise ratio, however at the cost of possible drift without a reference channel.

(Figure 1 near here)

The lock-in amplifier system was developed on a gas bench test rig, where precision mixtures of gases can be created. The bench top system comprises four mass flow controllers (MFCs), enabling the level of CO_2 to be varied, with a balance of synthetic air. The level of water vapour can be varied, created by passing synthetic air through a hydration unit. The station is controlled via a LabVIEW interface (National Instruments, v2014). A block diagram of the system is shown in Figure 2.

(Figure 2 near here)

2.1. Sensor Chamber Design

Component size is a major factor for the selection of sensors to include in a portable breath analyser. In the case of NDIR, the distance between the detector and emitter (which determines the concentration range the sensor can detect) is often the component that dictates the dimensions of the sensor. Fortunately, for CO_2 breath analysis, the level of CO_2 is in the range of 4 to 5 %, thus the CO_2 concentration can be measured with relative short paths lengths. It has previously been reported path lengths of 10 mm or lower and up to 10 cm have been used for CO_2 detection in the concentrations expected on breath [32,33]. Preliminary experiments, with a stable 80 mm path length (in an aluminium chamber shown in Figure 3) were performed to provide a stable benchmark standard.

(Figure 3 near here)

In order to determine the optimal path length for breath analysis, a prototype measurement system was developed (Figure 4) which allowed the path length to be varied. Aluminium tubing created both an easy mechanism in order to adjust path length and critically a sealed chamber for testing a known concentration of gas. The path length was trialled at 10, 20 and 40 mm with concentrations of CO_2 varied between 0.5 % and 5 %. The aluminium tubing is commercially available (Albon Alloys, UK), specified as 45 mm x 0.45 mm.

(Figure 4 near here)

The data obtained at the three path lengths aided the development of a portable breath analyser Figure 5. The body of the analyser is manufactured from the photo-curable plastic acetal, providing a lightweight, but robust construction. The holder for the IR emitter and detector was 3D printed, to provide an exact fit for the manufactured sensor housing. In previous work [34] we have discussed development of a main stream breath sampling system, where the sensors are directly exposed to the breath sample. The revised prototype utilises a side-stream sampling mechanism, where a sample of the exhalation is extracted and passed through a sensor chamber. The side-stream approach permits slower flow rate and smaller dead volume inside the sensor chamber. Gas is continuously pumped through the side-stream system at a rate of 0.15 l/min, compared to the mainstream flow, which can reach 10 l/min during an average exhalation [35,36]. Furthermore, the design of the side-stream tubing can prevent the build-up of humidity. An exhalation is almost completed saturated, with RH levels exceeding 90 %. In our design the sampled gas is extracted vertically upwards, thus preventing water droplets from condensing inside the tubing. The completed unit contained sensors for oxygen, flow, temperature, humidity and carbon dioxide, although here we will focus on the CO_2 measurement system. The CO_2 sensor was calibrated against a reference commercial breath-by-breath sensor (Hummingbird IR3107). The IR3107 is a commercially available ($1500) breath by breath NDIR CO_2 sensor, offering a <100 ms response time. It uses single beam single wavelength technology. However the device is bulky (65 x 30 x 40 mm) and requires a peak current of >2 A, thus although it provides a serial readout, it cannot be powered from a computer.

(Figure 5 near here)

2.2. Signal Processing

The lock-in amplifier component forms the base of our measurement system. The received signal from the thermopile circuitry is compared to the emitter drive signal and the original sinusoid waveform recovered. A simplified circuit schematic is shown in Figure 6, where the voltage regulator circuit and variable resistors that adjust the cut-off frequency are omitted.

(Figure 6 near here)

The operation of the lock-in amplifier is demonstrated in Figure 7. The heater drive signal (which serves also as the reference signal to the lock-in chip) is generated by a microcontroller (Arduino Pro Mini) paired with an AD9837 (Analog Devices) programmable function generator. The system is currently limited to a frequency drive of 5 Hz, where the size of the emitter dictates the operating speed. The function generator IC chip permits drive frequencies of up to 5 MHz with an accuracy of 0.02Hz. Future generations of our NDIR system aim to increase the excitation frequency to 100 Hz. The heater is pulsed between 150 °c and 500 °c, controlled by a current drive circuit. The peak power consumption (at 500 °c) is 120 mW. The device has an emissivity of 0.79 at 4.26 µm. The aluminium tubing is essential for the detector to receive the IR signal. Without the tubing the voltage received was found to drop from ~0.9 V (sinusoid with amplitude of 0.3 V) to the mV range (amplitude 60 mV). The limit of detection of the system is related to the signal to noise ratio of the system, which in turn is limited by the intensity of IR emitted from the source. The IR source was pulsed with a 5 Hz sinusoid between 200 °c and the maximum recommended temperature for long term use (500 °c). The device is capable of temperatures exceeding 550 °c, but with reduced reliability and life time.

(Figure 7 near here)

The output from the thermopile detector is in the micro-volt range. For the signal to be recorded on a USB data acquisition module the raw output is amplified using two gain stages (non-inverting amplifiers), by a factor of ~3000. Following the gain stages, an active low pass filter removes the remnants of noise on the 5 Hz signal, and further amplifies the signal (factor 5x) suitable for acquisition via a USB DAQ (National Instruments 6343 module). The change in amplitude of the sinusoid waveform recorded corresponds to the concentration of CO_2 to which the sensor is exposed. The results presented below show the amplitude of the sinusoid, normalised to a baseline of one. As the CO_2 absorbs the IR radiation, the signal decreases with an increase in the concentration of CO_2. The amplitude of the sinusoid is taken to help mitigate drift (for example due to environmental temperature variance). The heater is pulsed between two temperatures, therefore the amplitude of the signal varies less with environmental temperature than a constant drive temperature. However, this technique cannot completely remove drift. Current work involves adding a reference thermopile channel, for temperature compensation. Normalisation of the signal to a fixed baseline of ~1 (when no CO_2 is present) allowed for easy comparison between experiments, where path length, and thus received IR intensity, vary.

3. Results and Discussion

The lock-in amplifier NDIR system was trialled with three different sensor configurations: First an aluminium chamber (80 mm IR path length), secondly a variable path length (10, 20, 40 mm) bench top prototype unit and finally a portable breath analyser (10 mm path). The peak power consumption of the system was ~350 mW, independent of chamber design. The majority of the power required was associated with the pump (~150 mW) and about one third attributed to the IR emitter. The low-power consumption is ideal for a portable device, compared to the reference breath-by-breath IR3107 sensor, which consumed on average 3 W while acquiring measurements, and peaked at 6 W during warm-up. In all, the system has been tested on gas concentrations from 50 ppm to 5 %, in conditions of dry gases and 25 % RH.

3.1. Low ppm concentration – 80 mm path (dry and 25 % RH conditions)

The first prototype bench-top unit was formed of an aluminium chamber (Figure 3) with an IR path length of 80 mm between the IR emitter and thermopile detector. The sensor was exposed to CO_2 concentrations from 50 ppm to 2.5 %, generated on a gas test bench, with a constant flow rate of 0.5 SLPM. In preliminary measurements, the limit of detection of a shorter path length, such as 10 mm was found to be 250 ppm. Figure 8 a) and (b) demonstrates the resilience of the system to humidity, where the system was tested in dry gases and 25 % RH. The sensor outputs displayed excellent stability. Concentrations from 100 ppm to 2.5 % were repeated twice during the experiment, with average variations of ~0.23 % and 0.10 % between the repetitions for dry and 25 % RH respectively. The addition of humidity increased the stability of the sensor output, but decreased the normalised reading by an average of 2.3 %. The normalised values were seen to decrease by 5.4 % on average in 2.5 % CO_2 but by only 0.4 % in 50 and 100 ppm experiments.

(Figure 8 near here)

The lock-in amplifier maintains a frequency lock throughout the experiments, where no spurious spikes from other frequencies are generated. An average time of 2.4 s for the sensor to reach 90 % of final output (t_{90}) was calculated. The response time of the sensors was in part related to the gas concentration. The shortest response time (1.5 s) was found for the 2 % CO_2 concentration step and the longest response time (6.1 s) was found for the 50 ppm step. A possible explanation for the wide range of response times is the gas measurement rig. A single cylinder of CO_2 was used throughout the measurements. A low flow rate was required for the low ppm concentration gases ($\leq$ 100 ppm) of perhaps just 5 ml/min and lower. For MFCs designed to operate with a maximum flow of 500 ml/min a finite period of time will be needed (for stabilisation) when low flow rates are requested. Although low ppm measurements of CO_2 are not necessary for breath analysis, other compounds, such as acetone, can be present on breath in such concentrations. Diabetic subjects can exhibit a strong level of acetone [8,37], which would be of interest for our breath analysis studies. Future work will involve trailing filter caps on the detector with bandwidth centred around perhaps 3.39 µm [38] in order to detect acetone on breath. In the case of our CO_2 sensor, an 80 mm path was found to be unsuitable for CO_2 breath analysis, where the sensor saturated with ~ 2.5 % CO_2. The lower limit of 50 ppm would be useful for air quality monitoring, but is not required for breath analysis. Our next stage of work was to identify the best suited path length for our portable analyser. The limit of detection was found to be ~ 50 ppm. Lower ppm concentrations (.e.g 10 ppm) were found to be difficult to distinguish from the baseline output, in synthetic air.

3.2. Optimum IR path length for breath CO_2 measurement (dry, 25 % and 50 % RH conditions)

Our preliminary experiments proved that that using our system with an 80 mm path length CO_2 concentrations of interest in breath analysis (e.g. between 4 and 5 %) could not be distinguished. A prototype test unit was developed, including rapid prototyped parts, in order to trial path lengths. The device, shown in Figure 4, allowed large variation in path length, only restricted by the length of aluminium tube. Furthermore, the identical aluminium tubing could be used in our prototype portable breath analyser, thus removing anomalies in tubing specification between our bench test rig and prototype analyser. Our previous work in NDIR sensing suggested the finish of the tubing is of paramount importance to the strength of IR radiation transmitted between the emitter and detector, where a given gold plated tube has a far greater reflectivity than an otherwise identical aluminium counterpart. In this current work only aluminium tubing was trialled.

The length of tubing (optical path distance) in our NDIR assembly was varied between 10, 20 and 40 mm. The concentration of CO_2 was varied between 0.5, 1, 2, 3, 4 and 5 %. Each gas concentration was trialled for 1 minute, returning to a baseline of synthetic air with no CO_2 present between each. The gas flow rate was set at a constant 0.5 SLPM throughout the experiments. The processed time series data for a 40 mm path length is shown in Figure 9. To ensure the reliability of our measurements, each path length was tested five for repetitions.

(Figure 9 near here)

The ability to adjust the length of the tube consequently entails the possibly of introducing alignment errors between the IR source and detector. This was mitigated by a visual inspection of the setup between repetitions, verification of the output voltage level of the thermopile detector on the gas test rig and constant monitoring of the gas flow rate out of the sensor (i.e. in a system with no leaks, the gas input will equal the gas output). The data recorded over the five repetitions for path length the average value for each gas concentration (with dry gases) is summarised in Figure 10 and compared with each of the three selected path lengths.

(Figure 10 near here)

Accurate detection of CO_2 concentrations in the region of 4 to 5 % is important for breath analysis. The response curves for 20 mm and 40 mm path lengths exhibit a similar trend to the higher concentrations shown on the 80 mm path length initial experiment, where the sensor output is becoming saturated. The error bars on the graph represent the variation in average recorded value between repetitions. The error bars plotted for the 40 mm 4 % and 5 % measurement values are separated only by 0.007 (fractional change), thus the output response for 4 and 5 % cannot conclusively be determined. The 20 mm result demonstrates an improved result, where including the error approximation, the 4 % and 5 % readings are separated by twice the fractional change of the 40 mm result (0.014). However this result is still barely a 1.4 % response change and therefore determining the accurate gas concentration is likely to become difficult, especially when measurements are taken with non-synthetic gases and with water vapour.

Although the sensor response is lower for a given concentration, the 10 mm path length results demonstrate promising responses to 4 and 5 % CO_2. The output response varies the least, ~25 % given a 5 % change in CO_2 concentration, out of the three tube sizes tested. Comparatively, a ~29 % change in output response is observed with the 20 mm path configuration and a ~30 % change for the 40 mm path length. However for the same change in CO_2 concentration, the output response varies by ~2.5 %, accounting for measurement errors. The dry results indicate the 10 mm path length is most suitable for measuring CO_2 concentrations in the range of 0 to 5 %, with synthetic gases. The high water content of breath can has previously been reported to have an effect on NDIR systems, where the absorption of water occurs at certain wavelengths [10].

The system with 10, 20 and 40 mm path lengths was tested at RH levels of 25 % and 50 %, over the same CO_2 concentrations (0, 0.5, 1, 2, 3, 4 and 5 %). A time series plot for a 40 mm path length experiment is shown in Figure 11. The experimental results are summarised in Figure 12, at the same scale as the corresponding dry plot. Overall the response has been muted, regardless of path length. In dry conditions, 2% CO_2 produced output responses of ~16 %, ~18 % and ~22 % intensity change for 10, 20 and 40 mm path length respectively. With a gas mixture containing 25 % RH, the responses have been decreased on average to 12 %, ~14% and ~15 % respectively for a 2 % step in CO_2 concentration. The presence of the water also affected the measurement error, where additional noise was introduced into the system. For dry gases the standard deviation between all measurements for the 10, 20 and 40 mm path lengths were recorded as 0.1%, 0.1 % and 0.5 % respectively. In conditions of 25 % RH these errors were calculated as 0.4 %, 0.1 % and 0.1%. The 10 mm path length demonstrates a quadrupling of error, however the 40 mm path length demonstrates a fifth of the error when comparing dry to 25 % humid conditions.

(Figure 11 and 12 near here)

The most notable change from a dry to 25 % RH environment is the reduced separation between the 20 and 40 mm path length responses. In dry conditions the 40 mm path produced on a higher response by on average 1.5 % (across all concentrations), but at 25 % RH the response was only 0.6 % greater. The 10 mm path length produces a lower response, but still maintains easy distinction between 4 % CO_2 and 5%. The 50 % RH condition response plot (Figure 13) demonstrate an even smaller difference in intensity measured for the 20 mm and 40 mm path lengths, with just a 0.5 % greater response for the longer path. The results demonstrate humidity decreases the response to CO_2, particularly with the smaller diameter tube (3.1 mm internal for the portable breath analyser tubing, compared to 10 mm for the aluminium chamber). It is a possibility water droplets condensed inside the tube, and effected the IR radiation in a greater manner for the smaller diameter tubing.

(Figure 13 near here)

The measurements taken in 50 % RH demonstrate the system is still able to function with humid gases and the sensor remains stable, despite a decrease in response. The system is overall more stable with the higher level of humidity, where similar standard deviations were calculated as 0.1 %, 0.1 % and 0.08% for the 10, 20 and 40 mm experiments at 50 % RH. The response of the system becomes closer to a linear plot, with increasing levels of RH. The received signal strength drops with increasing humidity, but stability increases. Perhaps this phenomenon is due to the change in thermal conductivity (and heat capacity for transients) of air, which decreases (increases) with increasing moisture content. The overall effect is not linear with increasing humidity. For a CO_2 NDIR system to be suitable use in a metabolism analyser the sensor must be able to discriminate between 4 and 5 % concentrations. The 10 mm path length was selected for the portable breath analyser, based on its physically compact design and ability to provide an adequate response as to separate 4 % and 5 % concentrations of CO_2, in both dry and wet conditions.

The raw output voltage of the thermopile decreased in a non-linear form. The mean values of the thermopile circuitry raw output voltage (before normalisation) was compared (Figure 14) for all the CO_2 concentrations tested. The values shown are for an environment of 50 % RH.

(Figure 14 near here)

3.3. Comparison with simulated values

A modified version of Beer's law was determined, equation (1), to simulate the effect of a reflecting tube, accounting for the highest intensity reflections. The version of Beer's law shown in section 1.1 was found to demonstrate a poor fit (R^2=0.64). The 10 mm path in 50 % RH conditions was compared to the modelled output, as shown in Figure 15. The modified version of the Beer-Lambert equation includes a term for the intensity lost due to reflections inside the tube and emitter itself (I_{int}), which do not reach the detector. Also, the modified equation includes two path lengths l_1 (direct path length) and l_2 reflected path length (the highest intensity reflection, with only one bounce). l_2 is related to l_1 by $l_2 = \sqrt{l_1^2 + 4r^2}$, where r is the radius of the tube, and g denotes the reflections in all angles around the tube. The value for the constant k_g was determined as 241.9 cm^{-1}

$$I(c) = I_{int} + I_0\left[e^{-k_g\, l_1 c} + g\, e^{-k_g\, l_2 c}\right] \quad (1)$$

(Figure 15 near here)

The modified equation was used to model the 50 % RH data with a 10 mm path length. The modelling provided an excellent fit (R^2=0.999). The model demonstrates the reflections contribute a large proportion of the light received by the detector. The values for I_{int}, I_0 and g were found to be 0.72, 0.26 and 0.059 respectively

3.4. NDIR system for CO_2 measurement on exhaled breath

A portable breath analyser was designed based on the 10 mm path layout. One reason for the reduced intensity measurements in the bench top trials was possibly the condensation of water droplets. The bench top system did not allow, any filter nor trap to capture any condensing water, where the humid gas was passed directly between the IR emitter and detector. In the design of the breath analyser, the sample extraction mechanism was designed (section 2.1) as to limit the collection of water droplets. A major advantage of using a side-stream sampling system, opposed to main stream, is the possibility to include filters and flow stabilisation components, without disturbing the exhalation path. For metabolic rate measurements it is important to have a low resistance breath analyser, where the subject can breathe normally through the device.

The prototype measurement unit (Figure 5) was calibrated against a reference commercial breath-by-breath sensor during an initial breathing sequence. Subject A was requested to exhale and inhale at a reduced rate of 10 s per cycle (6 breaths per minute, compared to an average human breathing rate of 10 breaths per minute [34]). An exponential curve fit, of the shape presented in Figure 13 was fitted to the concentrations of CO_2 measured in the breathing sequence shown in Figure 16.

(Figure 16 near here)

Data from subject A and B are presented in Figure 17 a) and b), calibrated using the same values calculated from the initial data. For these measurements the subjects were asked to exhale at a rate of 10 breaths per minute, with a 1:1 ratio between inhalation and exhalation. The data from both devices is filtered using a median filter of 15 samples. The sensor required calibrating to a breath exhalation, rather than laboratory gases, due to the distinctive characteristics of breath gases, which are difficult to replicate. The elevated temperature and humidity are challenging to accurately reproduce. Furthermore the side-stream sampling system extracts samples of exhaled and inhaled air at a slower rate (0.15 SLPM) than the experiments performed on the gas rig (0.5 SLPM).

(Figure 17 near here)

The prototype breath analyser demonstrates similar performance, in terms of response time, to the expensive commercial counterpart. Its power consumption is sufficiently low as to be capable of being powered from a smartphone alone (~ 350 mW). The response time (t_{90}) is similar (1.3 s), compared to 0.9 s for the commercial reference. The response time is a great improvement on affordable commercial CO_2 sensors (~4s) and is sufficient to capture a 3 s exhale. Breathing rate will vary between subjects, perhaps up to 15 to 21 breaths per minute for obese patients [27]. Therefore a sensor with a faster response would be required. Future work involves driving the system at a higher frequency, to improve response time and reduce noise. The system is currently limited in operation frequency due to the response time of the MEMS IR emitter. Fast emitters are available, but with reduce emissivity. With a lower intensity emitter the system would not be able to measure the CO_2 concentrations as accurately.

The 3 s exhales began to plateau for both the commercial reference sensor and the novel prototype analyser. The results were consistent between exhalations, where there is little variation between the reference sensor and prototype device. To meet our EE measurement goal the concentration of CO_2 on breath needs to be determined to an accuracy of 1.25 %. The system currently has an accuracy of 2.9 %.

3.5. Development of system for portable smartphone data logging

Any sensor system which requires a computer is somewhat limited in portability, when a bulky instrument is needed to log data. Although even a modern smartphone lacks the processing capabilities of a basic laptop computer, smart mobile devices still offer the ability to log data and send results to another device for further processing. Here we present only the capability for a mobile device logger. Figure 18 shows a prototype breath analyser, connected to an Android smartphone. The final device will contain sensors for O_2, CO_2, temperature/humidity, flow rate and a VOC metal oxide device. The android application shown on the screen of the smartphone (Figure 18) displays raw data, logged from a serial interface. A new application is in development to log data from the sensors in the unit and display the output results in an easy to read format. The details of which will be presented in a separate article.

(Figure 18 near here)

4. Conclusions

CO_2 concentrations in the range of 50 ppm and 2.5 % have been measured with a bench top NDIR detection system, using a lock-in amplifier. An 80 mm path length provided sufficient resolution to identify low ppm levels of CO_2 with low power consumption (0.35 W) and could be run from a standard

USB port. The lock-in amplifier system, used a dedicated IC component, reduced the processing load of the data logging system. The 80 mm path system was trialled with 25 % humidity, which decreased the normalised reading by on average 2.3 %, however increased stability by 0.13%. During the first stages of developing a portable breath analyser, the IR path length required to measure CO_2 on breath was investigated. Experiments with varying path length (10, 20 and 40 mm) demonstrated 10 mm path length permitted concentrations up to 5 % to be detected. Besides the decreased size of a 10 mm system compared to a 40 mm system the shorter path length better suited 4 to 5 % CO_2 detection, where the system became saturated at longer path lengths. To verify the functionality of the system when the test gas contained water vapour, the system was exposed to dry, 25 % RH and 50 % RH conditions. The 10 mm path length was adversely affected, demonstrating an increase in noise. However, the longer path lengths performance, although marginally less affected by the presence of humidity, were not sufficiently sensitive to breath levels of CO_2; therefore the breath analyser was designed with a 10 mm IR path length.

A portable breath CO_2 analyser has been developed. It was tested on subjects with real breath, and verified to match a commercial breath-by-breath sensor. The device was constructed by 3D printing mainly from acetal, providing a light weight but robust structure. The device produced comparable results to the commercial counterpart. Response time was 1.3 s, compared to the research device of 0.9 s. The device demonstrated excellent stability between subjects, where one set of calibration data remained accurate between subjects. The device was tested to fixed inhalation and exhalation cycles, lasting 6 s and 10 s per cycle.

Future work involves completing the development of our portable breath analyser. In order to measure metabolic rate we have included sensors for O_2 and flow rate. We have reported upon the novel CO_2 sensor, and are currently developing additional NDIR sensors to monitor other compounds on breath. The performance of the CO_2 sensor will further be improved with a higher drive frequency. The current generation is limited in frequency drive to < 10 Hz, where future generations aim to operation up to 100 Hz. With these improvements, and further signal processing we aim to detect down to 10 ppm in our bench top system, and aim for faster detection for use in breath analysis, with reduced noise.

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Biographies

Timothy Vincent graduated from the University of Warwick (UK) in 2013 with a first class degree in Electronic Engineering with Computer Engineering. He continued his work at the university, where he is currently a PhD candidate. His research interests include breath analysis and the development of fast response microsensors for portable gas sensing. His work focuses on the development of microcontroller based sensor systems and their application in healthcare towards point of care monitoring of well-being.

Julian Gardner BSc PhD DSc FIET FREng obtained a first in physics from Birmingham University, followed by a PhD in physical electronics from Cambridge University and a DSc in Electronic Engineering from Warwick University. He has 5 years' experience in industry as an R & D Engineer and 25 years working in the field of chemical microsensors at Warwick University, and is currently Professor of Electronic Engineering. He co-founded Cambridge CMOS Sensors Ltd in 2008 and was its first CTO; in 2013 he became its Chief Scientist. His expertise is in the design of MEMS based chemical sensors, and the signal processing/data analysis of sensor arrays, including electronic noses and electronic tongues. He has published over 500 papers in the field of micro sensors and authored 10 books. He has won a number of engineering award and is a Fellow of both the UK Institution of Engineering and Technology and the Royal Academy of Engineering.

Figures

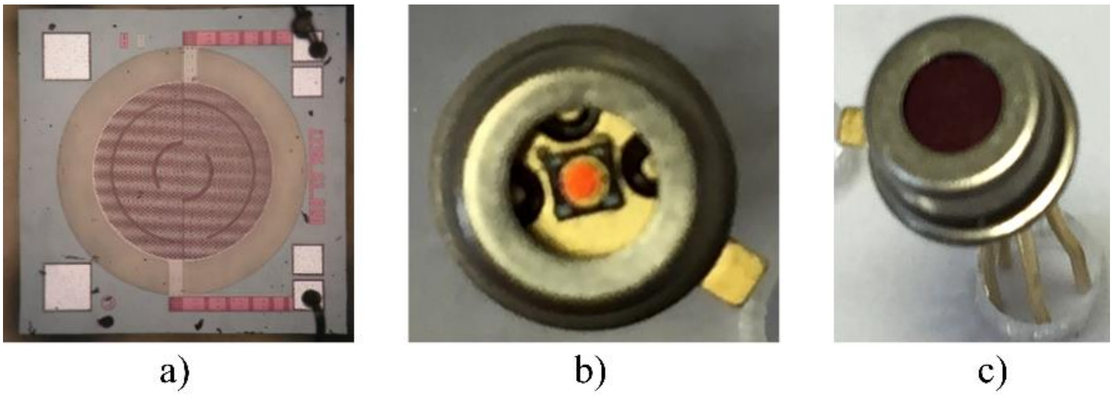


Figure 1 a) SOI emitter, provided by Cambridge CMOS Sensors, with 0.28 mm^2 heating area, b) Emitter mounted on TO46 header for fitting into NDIR system, c) Heimann commercial thermopile also mounted on TO46 header.

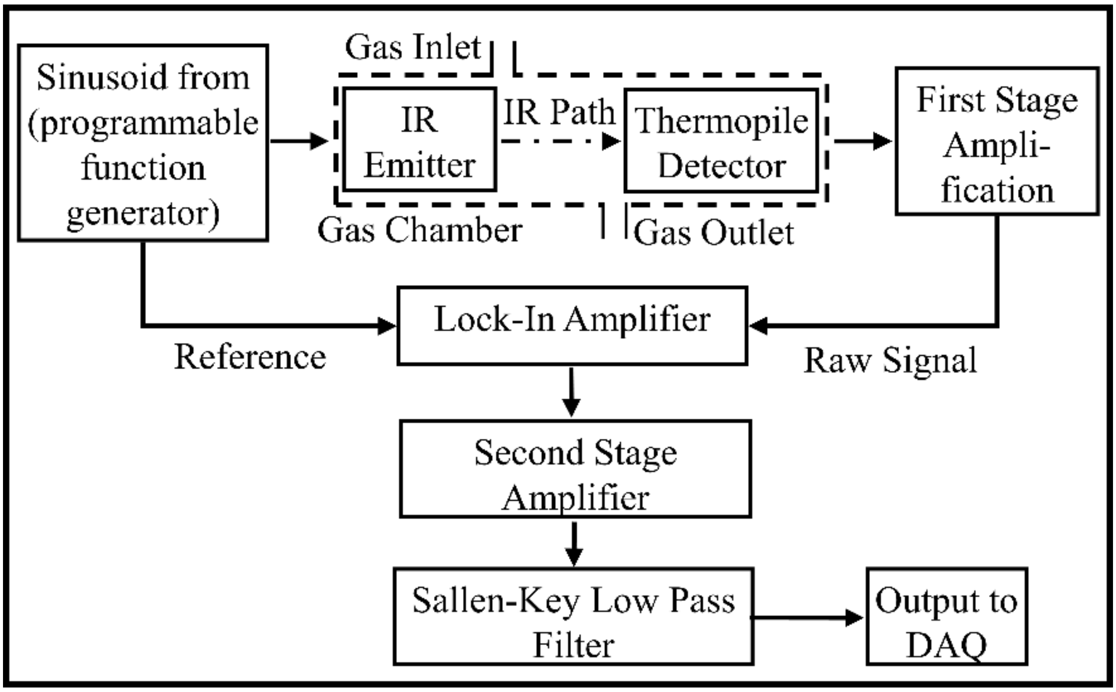


Figure 2 – Functional block diagram of NDIR sensor system with lock-in amplifier.

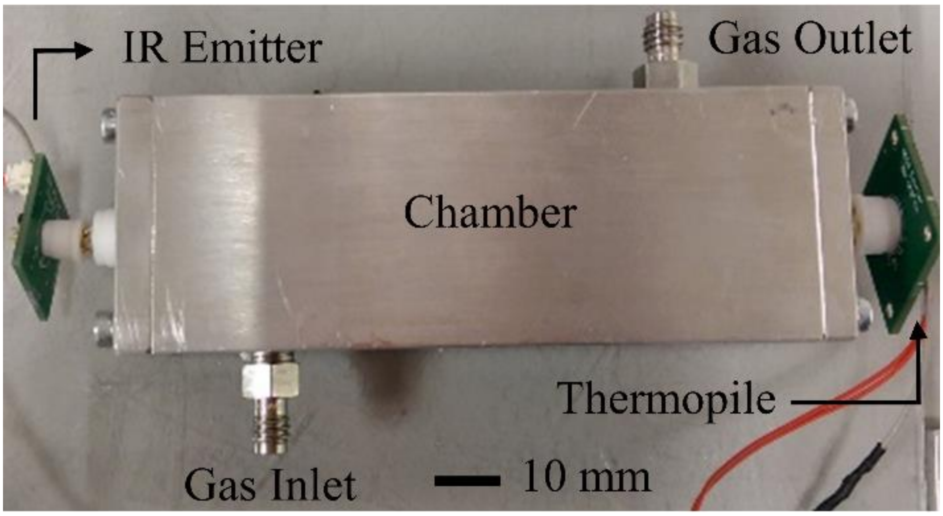


Figure 3 – Aluminium bench top chamber showing PCBs for IR emitter and thermopile detector.

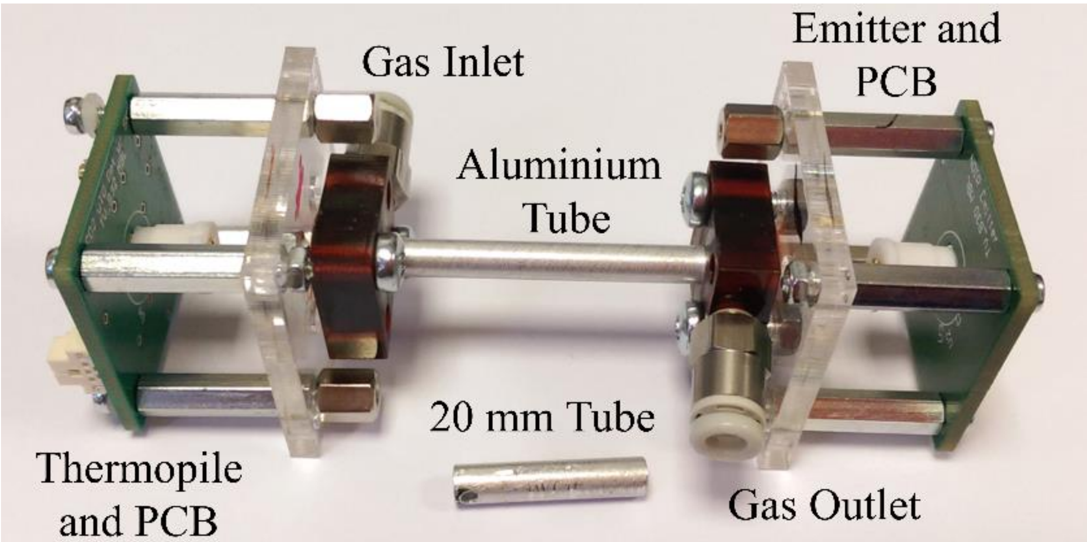


Figure 4 – Adjustable path length design of IR emitter and detector housing, fitted with 40 mm aluminium tube and with 20 mm tube.

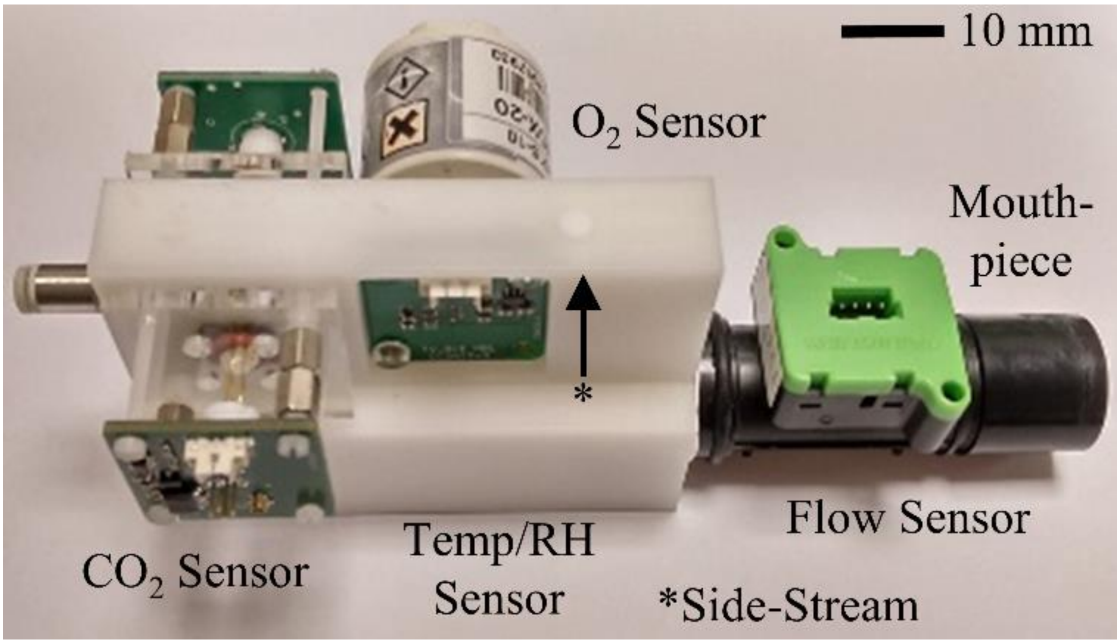


Figure 5 – Photo of prototype breath analyser, with CO_2 sensor mounted on left. Mainstream flow goes through flow sensor. Side-stream extracted vertically upwards.

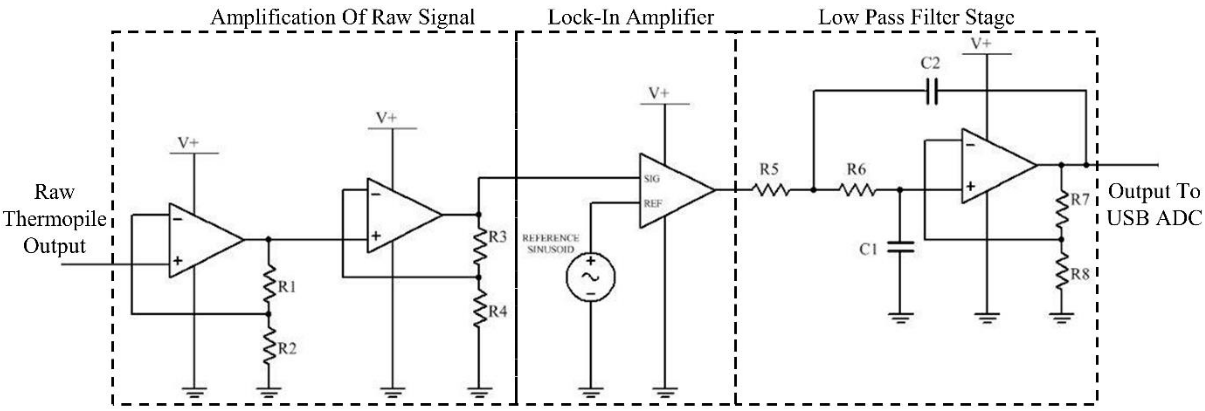


Figure 6 – Simplified circuit schematic of NDIR lock-in system, showing initial amplification stage, lock-in IC chip and final filtering process.

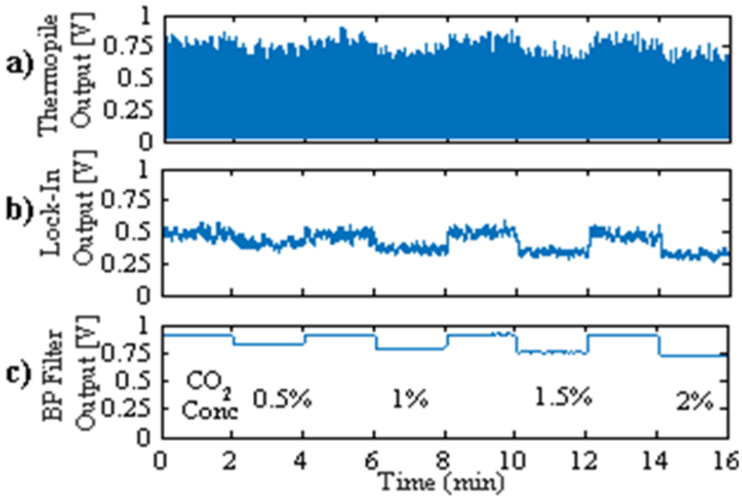
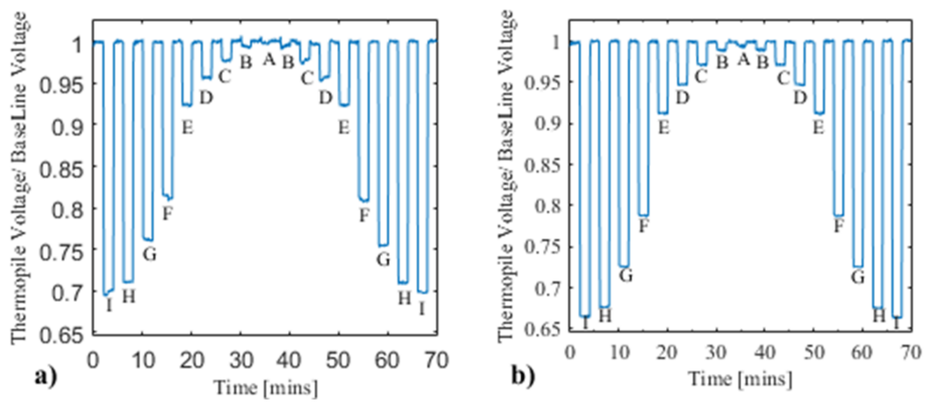


Figure 7 – Operation of lock-in amplifier. Raw thermopile output (a) is compared to a reference sinusoid with lock in amplifier (b) and then output is filtered (c).



Concentration	
A	50 ppm
B	100 ppm
C	250 ppm
D	500 ppm
E	0.1%
F	0.5 %
G	1 %
H	2 %
I	2.5 %

Figure 8 – Sensor outputs (relative to baseline of synthetic air with 80 mm path length showing detection of CO_2 in the range of 50 ppm to 2.5 %, (a) with dry gases and (b) in a constant environment of 25 % relative humidity.

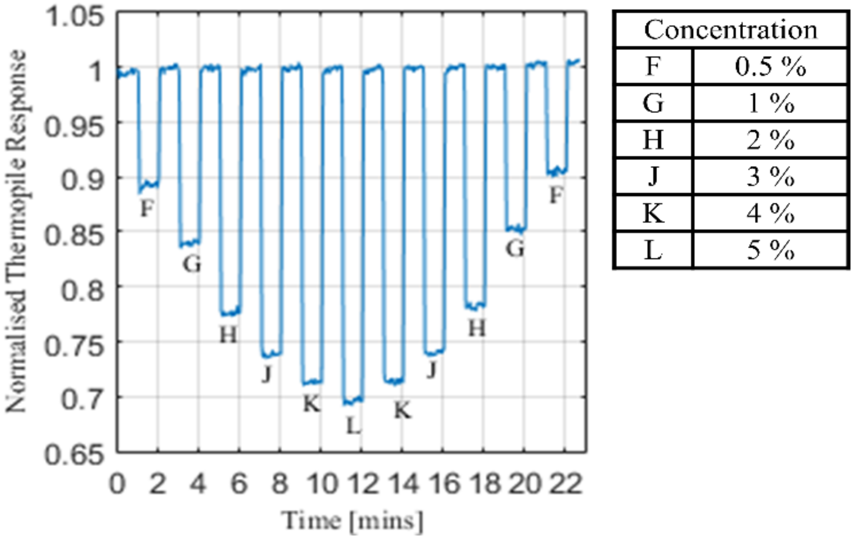


Figure 9 – Response of sensor system with 40 mm path length to CO_2 concentrations from 0.5 % to 5 % in dry conditions.

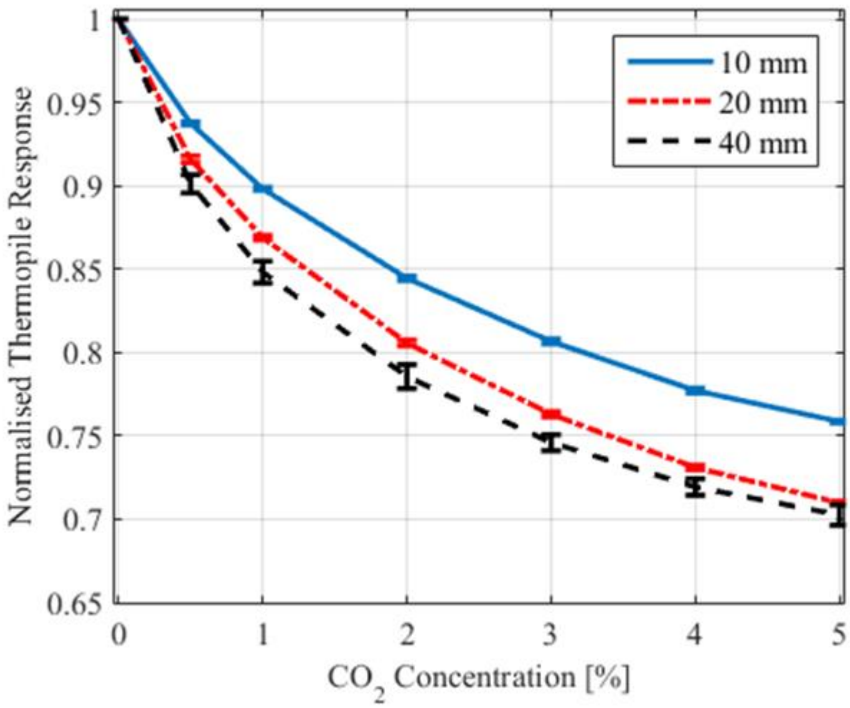


Figure 10 – Summarised response data for 10, 20 and 40 mm path lengths. Detection of CO_2 in the range of 0.5 to 5 % in dry conditions.

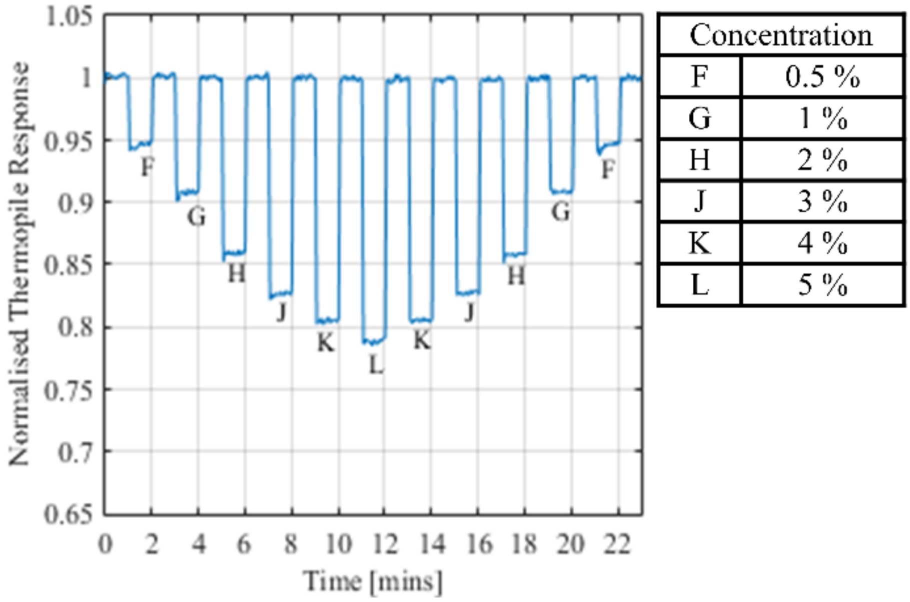


Figure 11 – Time series data for 40 mm path in 25 % RH conditions with CO_2 varied between 0.5 % and 5 % with constant 0.5 SLPM flow.

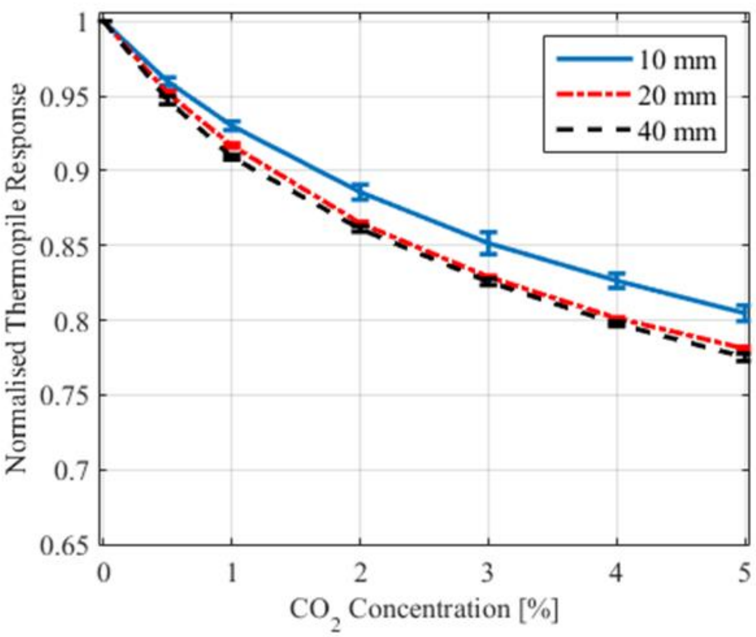


Figure 12 – Summary of data recorded in 25 % RH conditions with path lengths of 10, 20 and 40 mm.

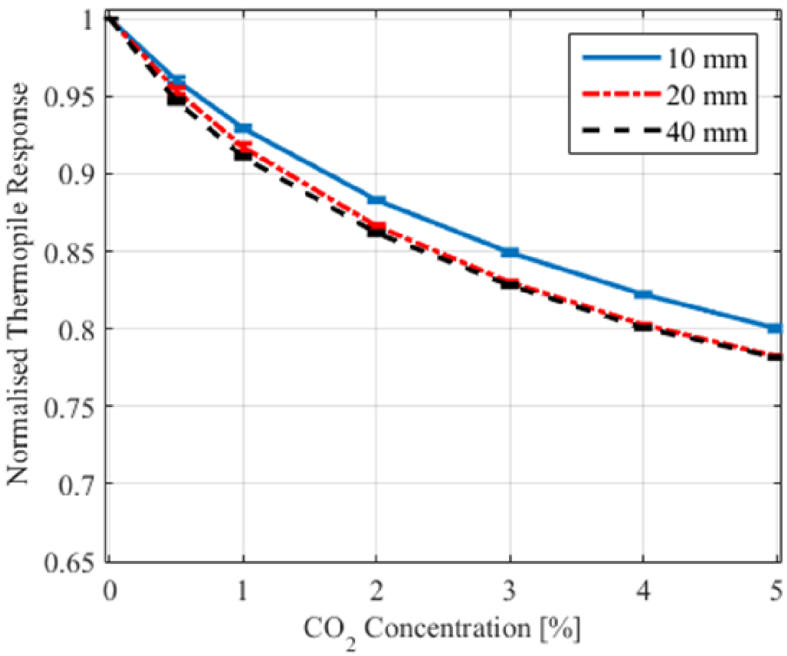


Figure 13 – Summary of data recorded in 50 % RH conditions with path lengths of 10, 20 and 40 mm.

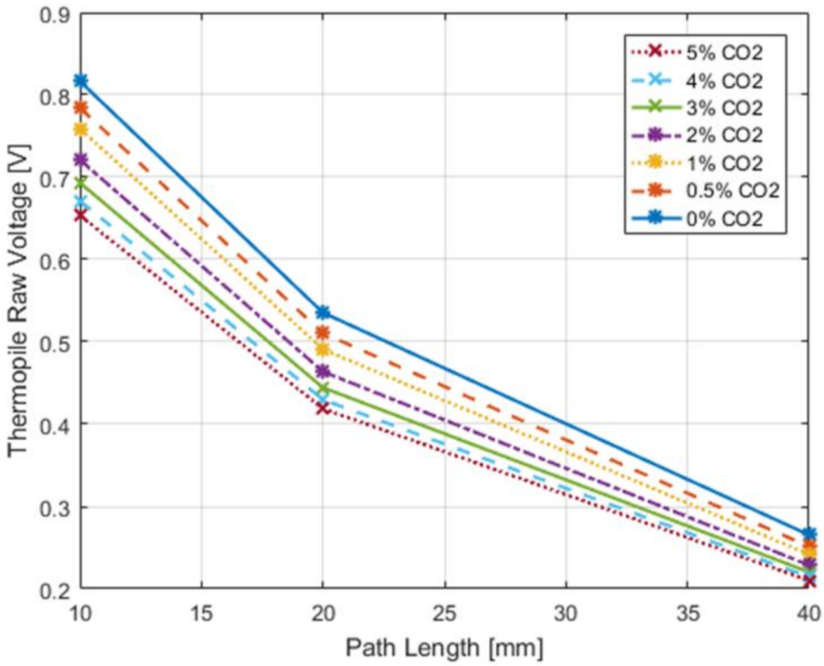


Figure 14 – Thermopile raw output voltage compared for path lengths of 10, 20 and 40 mm across CO_2 gas concentrations in the range of 0 to 5 %.

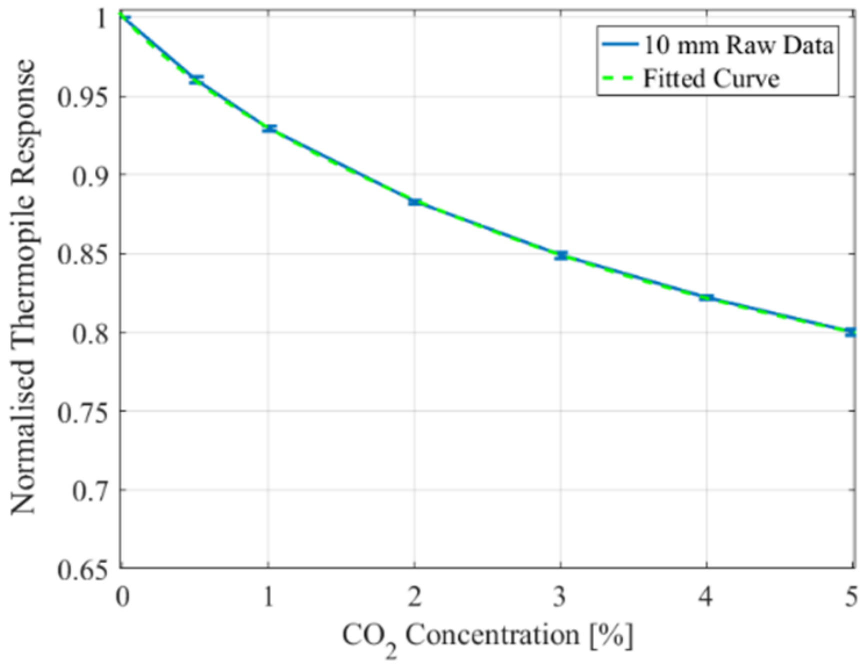


Figure 15 – Modified Beer's law equation fitted to data collected for 10 mm path length in 50 % RH conditions.

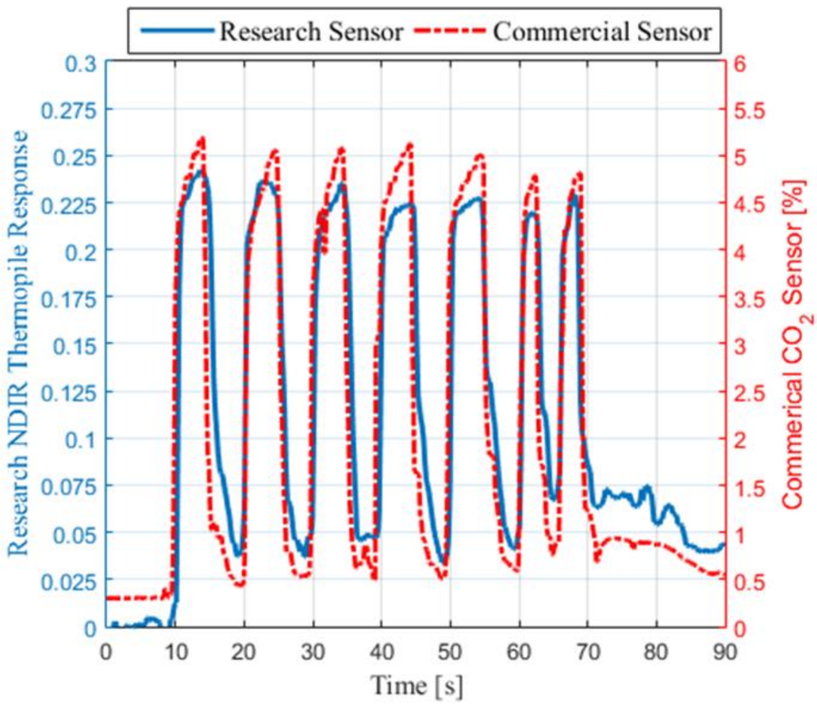


Figure 16 – Five second exhale and inhale sequence (Subject A, repeated over a 1 minute period) used to provide calibration data for the research device against the commercial counterpart.

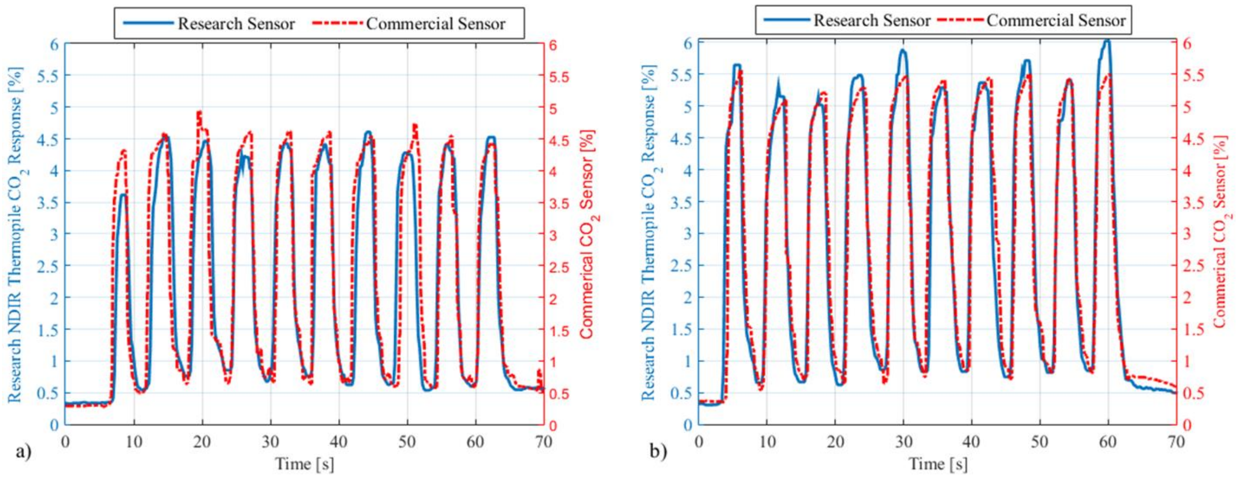


Figure 17 - exhalation data over a 1 minute period with fixed 6 second breathing cycle (a) for Subject A and (b) for Subject B.

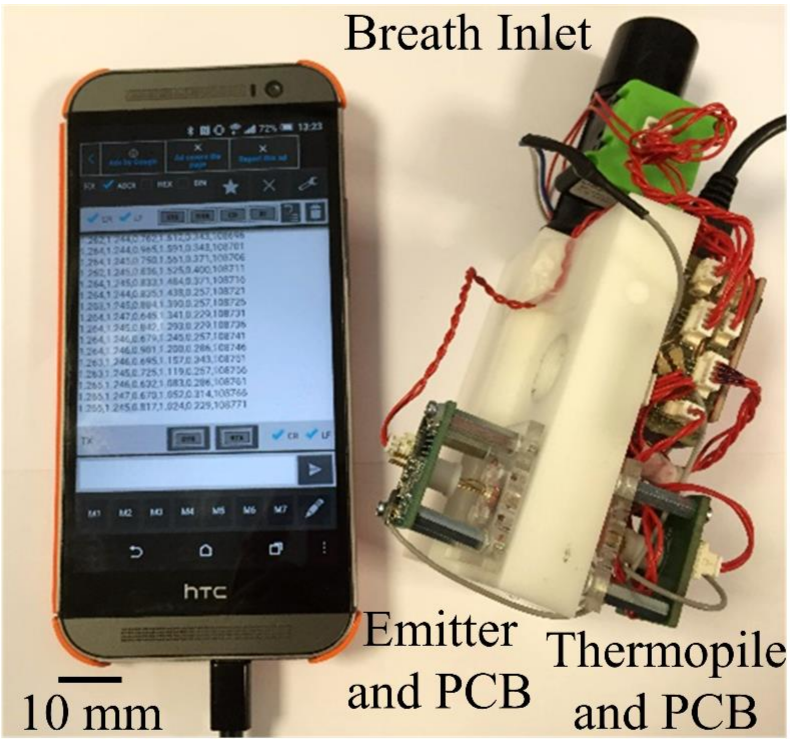


Figure 18 – Photograph of portable side-stream breath analyser. Smartphone screen shows raw data logged from breath analyser. The NDIR system is located at the end of the side-stream tubing.