

Performance Analysis of a Novel Pyroelectric Device for Non-Dispersive Infra-Red CO₂ Detection

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Abstract—We present the performance characteristics of a digital output thin-film pyroelectric sensor for use in non-dispersive infra-red detection. The single channel pyroelectric sensor device was fitted with band-pass optical filter with a central wavelength of 4.26 μ m for the detection of carbon dioxide. The classification reported here is concerned with the stability of the device for long-term measurements, with systematic drift not dominating measurement error even after 10 hours operation. Comparative NDIR measurements were made for this novel pyroelectric device using two different optical sources, a standard filament lamp and a black body radiation source, operated at 5 Hz repetition rates. The overall limit of detection for the NDIR sensor was calculated to be 6 ppm and 3 ppm for CO₂ for the filament and blackbody source respectively, when the sensor was temperature stabilised at 35 °C and data was averaged over 110 minutes. The single shot measurement error was calculated to be 48 ppm and 22 ppm for the lamp and blackbody respectively, when the sensor temperature was stabilised at 35 °C. The response of the detector to increasing temperature was found to exhibit a trend in the signal output directly proportional to the pyroelectric coefficient, this was also evident during flow tests when the flowing gas cooled the device. The result of changing both the concentration of analyte and flow rate is also covered. This is the first time the performance of a fully digital output pyroelectric detector has been reported.

Index Terms—Pyroelectric,

I. INTRODUCTION

NON-DISPERSIVE infra-red (NDIR) technologies have become a commonly used method for optical gas detection, where the quantity of optical absorption by a target species provides a measurement of its concentration. Many different types of NDIR sensors are available [1], and they all have the same basic components; a broadband optical source, a sample chamber and an optical detection system. Unlike high resolution laser based gas sensors, which target a single narrow line-width absorption feature of the target gas, the broadband emission sources used for NDIR target the integrated absorption of a whole spectral band. The wavelength selectivity is then achieved using an optical band pass filter with a line-width of 10's of nanometres. This allows NDIR sensors to be manufactured at a significantly reduced cost to high resolution gas sensors, but can lead to cross-talk due to the reduced species selectivity as the broadband filter

allows light at wavelengths from other absorbing species to be detected.

Standard light generation sources for NDIR range from a tungsten filament lamp, a broadband light emitting diode and a true black-body source. For detection of infrared active chemical species thermal detectors are often used, for example thermocouples, thermopiles or bolometers [6], [7], all of which are known to have slow response times or high power consumption. Alternatively, pyroelectric materials can be utilised, where changes in temperature of a crystalline substance results in an electric charge. In this case, much higher response times can be achieved due to the response times of pyroelectric devices, in the order of microseconds to picoseconds [3]. However, this also requires the use of an optical source capable of light modulation at Hz - kHz frequencies. Pyroelectric devices have also been increasingly used in sensors for motion, fire and gas detection systems and for early thermal imaging [3]–[5]. For gas detection, the combination of an incandescent lamp and pyroelectric detector has proven effective with sensors being powered by battery or even a solar cell. Using 3 1.5V batteries (AAA size) a sensor can last for up to 10 years with the possibility of a longer lifetime under solar power, although this is still to be reported [8].

In this paper, we present performance analysis of a pyroelectric detector developed by Pyreos Ltd for NDIR gas detection. More specifically, their newly developed ezPyro™ surface mounted device (SMD), which offers both a very small package (5.65mm x 3.7mm x 1.55mm), microsecond response times and a fully digital output. This performance analysis has been carried out for CO₂ using an optical filter centred around 4.26 μ m, and a bandwidth of 180nm, ePY12231. The intensity of light incident upon the detector is proportional to the concentration of CO₂ and optical pathlength through the Beer-Lambert law,

$$I = I_0 \times e^{-(\alpha Cl)} \quad (1)$$

where α is the absorption coefficient of the target species, C is the species concentration and l is the light-gas interaction path length.

Firstly, the ezPyro™ response as a function of CO₂ concentration is shown, with particular focus on parts-per-million (ppm) concentration levels. Data is then shown for the sensors

long term stability/drift, providing detailed information on the overall limit of detection. This includes a comparison between two optical emission sources; a standard incandescent bulb (Oshino) and a micro-electro-mechanical based broadband thermal infrared source (Axetris AG). Data is also presented to show the variability of the ezPyro™ response as a function of temperature and as a function of gas flow rate.

II. EXPERIMENTAL SET-UP

A small environmental chamber was constructed to provide controlled gas concentration and pressure for the testing of the ezPyro™ modules. The chamber took the form of a section of vacuum tubing with Con-Flat knife edge seals of 100mm diameter (CF100) at each end, allowing the attachment of sealed end pieces with gas and electrical feed-throughs. One end piece was a custom made blanking section (LewVac) with two 9-pin miniature d-sub electronic feed-throughs and two 1/4 inch compression fittings (Swagelok) supplying electrical and gas/vacuum connections respectively. The opposite end-piece was a quick-access door, rated to 1×10^{-8} Torr, to allow access to the sensor module within the chamber. Both end pieces were mechanically attached and sealed to the vacuum tubing section using copper gaskets.



Fig. 1. ezPyro™ pyroelectric sensor mounted onto break-out board.

The ezPyro™ device was supplied on a small circuit board, referred to as the break-out board (see figure 1). This break-out board is connected via a ribbon cable to a Pyreos designed printed circuit board, known as the back-plane board. The back-plane board can control and record data for up to 4 ezPyro™ sensors using a micro-controller (ST Nucleo-F303K8) and USB communications. Software developed by Pyreos permits the user to observe the output signal from each sensor and save these signals either continuously or in user definable blocks at regular time intervals. The ezPyro™ break-out board was mounted onto a small reflector/gas module designed to correctly locate the optical source in place and allow a 2cm path for light-gas interaction. The entire assembly, including the backplane board, was enclosed inside the environmental chamber. Connections for the USB communications for the backplane board, the optical source power supply and a thermocouple were made through the 9-pin d-sub feed-through. Two optical sources were used in this work; a

tungsten filament lamp and a source designed to provide true black-body emission from $2 \rightarrow 12\mu\text{m}$ (Axetris EMIRS50 AS01T). Both sources were operated with a 5 Hz square wave current pulse, with a 50% duty cycle, provided by an Axetris LabKit IRS G1 drive board, with peak currents of 85 and 67 mA for the filament and black-body sources respectively.

The gas concentration within the environmental chamber was controlled using two 10 to 1000 mL/min Bronkhorst mass flow controllers, specified for CO₂ and N₂. Calibrated gas mixtures of CO₂ were supplied by BOC with certified concentrations of 1.01 % and 518ppm, both with a nitrogen balance. Gas concentrations of lower levels were obtained by mixing appropriate quantities from the supplied BOC sample with nitrogen using the mass flow controllers prior to entering the environmental chamber. For flow measurements the environmental chamber was filled with 100 % nitrogen and controlled analyte concentrations were passed directly from the mass flow controllers through the sensor, via a gas fitting located on the optical reflector. For all flow measurements the total flow rate remained at a constant level of 600 mL/min, whilst the required concentration values were set by varying the flow rate ratio between the CO₂ and N₂, with the exception of during the flow switching simulation measurements. During these measurements the total flow was 600 ml/min during the CO₂ flow measurements and 300 ml/min for the nitrogen only periods to simulate changes in Heating, Ventilation and Air-Conditioning (HVAC) systems.

III. RESULTS

A. Static CO₂ Measurements

The performance of the ezPyro™ detector was initially characterised for CO₂ using a tungsten filament optical source. The source was driven with a 5 Hz square wave current pulse with a peak current of 86 mA. However, the lamp output took the form of a triangle wave-form, as measured by the ezPyro™. 5 Hz was chosen as the drive frequency due to responsivity of the source, as it provided the maximum illumination at the fastest repetition rate. At higher frequencies the average optical power of the lamp decreases, this can be seen as a reduced signal amplitude as shown in figure 2.

Figure 3 shows the averaged peak to peak signal output from the ezPyro™ for CO₂ concentrations from 0-10,000 ppm. A total of 3000 waveforms are recorded for each corresponding analyte concentration. The exponential decay of the signal as the analyte concentration increases shows a similar trend to larger analogue pyroelectric devices, such as the Pyreos PY-ITV-Single/Dual detector and the Perkin-Elmer PYS3228 detector used by Hodgkinson *et.al.* [10]. Figure 4 shows data obtained at CO₂ concentrations of 10 - 10,000 ppm that has been converted from measured transmission to total absorption, and an exponential fit of the form $y = y_0 + e^x$. The transmission to absorption conversion is obtained by subtracting the acquired peak to peak data for the specific analyte concentration with the peak to peak value for 100% N₂. Finally, figure 5 shows absorption data for CO₂ gas concentrations ranging from 0 ppm CO₂ (nitrogen only) to 500 ppm with a corresponding linear fit. In figures 3, 4 and 5

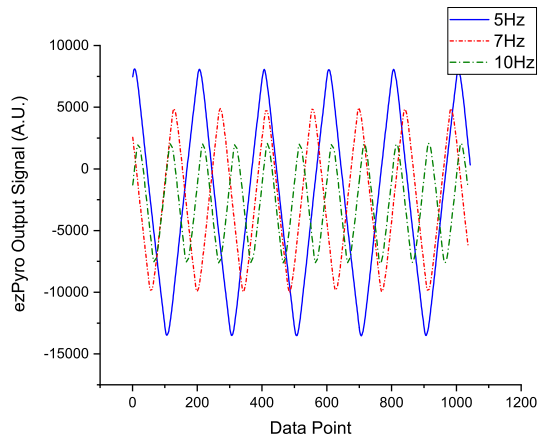


Fig. 2. Typical A.C. output signal of the ezPyro™ when using a standard tungsten filament lamp, recorded over 1 second, at 3 differing frequencies.

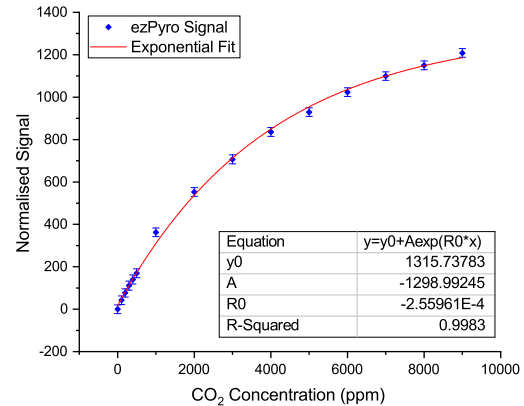


Fig. 4. Data for measurements up to 10,000ppm CO₂ rationalised against value for nitrogen only measurement.

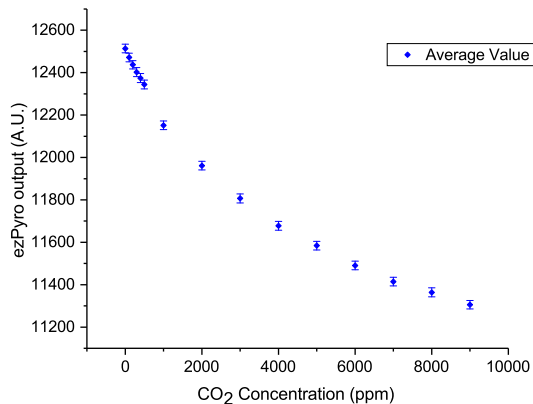


Fig. 3. Averaged response of ezPyro™ plotted with maximum and minimum values for each data set.

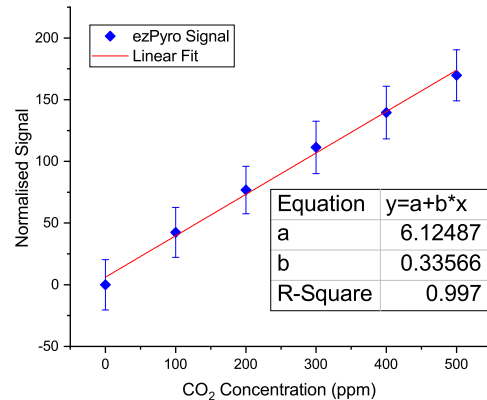


Fig. 5. Concentration measurements to a maximum of 500ppm CO₂.

the error in the measured concentration has been assumed to be the standard deviation of the 3000 measurements.

Through linear approximation, for $R_0x < 0.12$, of the exponential fit of the data shown in figure 4, $y = y_0 + Ae^{R_0x}$ we get,

$$y = y_0 + A(1 + R_0x) \quad (2)$$

This induces an error in the measurement of less than 6% for concentration measurements less than 500 ppm.

As the the aim of this research was to measure the performance properties of the ezPyro™ device over time, rather than developing a finalised product, Allan-Werle Variance [11] measurements were also taken to determine the long term sensor stability for CO₂. This was carried out for both the tungsten filament incandescent lamp, and also for a source designed to provide true blackbody emission from $2 \rightarrow 12\mu\text{m}$ (Axetris EMIRS50 AS01T). The latter device has been designed specifically to provide a more stable output, without exhibiting significant performance degradation as the device ages.

During these measurements the test chamber was filled with 500ppm CO₂, in balance nitrogen. Unlike sensors using dispersive infra-red spectroscopic techniques, pyroelectric sensors are regarded as more stable and less prone to systematic drift. For example the sensor described by Hodgkinson *et al.* in [10] displays an averaging time of approx. 4 hours before systematic drift begins to dominate.

Figure 6 shows the Allan-Werle plot for the incandescent lamp source. In this case, a 1 s data grab was taken every minute for 87.5 hours. As 1 second of data produced 5 peak-to-peak measurements, it was assumed that their average constituted one single measurement with a 1 second integration time. In this case, as data is not being recorded continuously, we cannot state an absolute minimum detection limit, as further averaging may improve the minimum limit of detection prior to the influence of drift. However, the Allan-Werle plot does give an accurate indication of the overall system drift as a function of time. The typical response of CO₂ NDIR sensors on the market show detection limits of $\pm 50\text{ppm}$, with integration times of the order of a 30 seconds. As can be seen from figure 6, the response of the ezPyro™ displays a similar

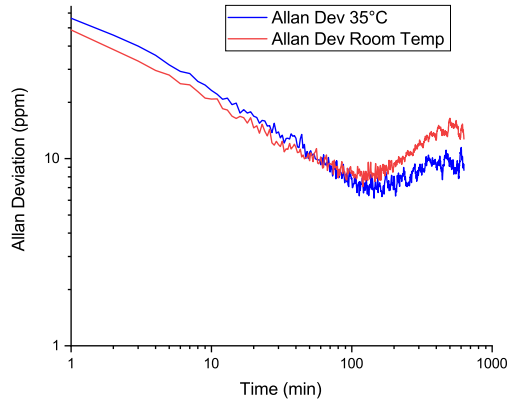


Fig. 6. Allan Variance plots for 500ppm CO₂ with the sensor heated to 35°C and at room temperature showing a lower detection limit (1σ) of 6ppm when heated and 8ppm at room temperature.

detection limit for an integration time of 1 minute. However, it must be noted that in this one minute integration time, the detector is only acquiring data for 1 s. The sensor minimum detection limit decreases linearly until approximately 110 minutes of operation, showing a final minimum detection limit of 6ppm. Systematic drift then begins to affect the confidence in the signal. However, even after approximately 10 hours of operation the minimum detection limit is still below that for 1 s of operation. It must also be noted that this data was taken without a reference channel, and the factors affecting the drift may be due to the performance of the optical emitter, the ezPyro™ or even variation in atmospheric temperature. The difference in the overall sensitivities can be attributed to variations between day and night temperatures for the room temperature data whereas the active heating of the system to 35°C created a more stable environment.

A comparative Allan-Werle measurement using the blackbody source is shown in figure 7, where the measurement procedure was kept to be the same as above. The 1 minute integration time shows a non-averaged minimum detection limit of 32ppm compared to 48ppm, showing that the pulse to pulse variation for the blackbody source is lower than the incandescent lamp. However, the overall influence of drift on the minimum detection limit begins after approximately 100 minutes. One reason for this increase in drift is that the increased optical power in the infra-red for the blackbody emitter has resulted in secondary heating of the ezPyro™, causing thermal drift. In this configuration when the sensor undergoes active heating to 35°C the non-averaged minimum detection limit is 22ppm, the point where drift begins to dominate the measurements returns to 110 minutes and a final detection limit of 3ppm is achieved.

For thermal detectors, without a thermal compensation pixel, signal stability is better when heated since it is held at a more constantly and accurately controlled temperature which improves the accuracy of the measurement. The tests reported in this section were performed under controlled conditions, the following sections will deal with introducing factors that could

affect the performance of the pyroelectric detector namely increasing temperature and air-flow.

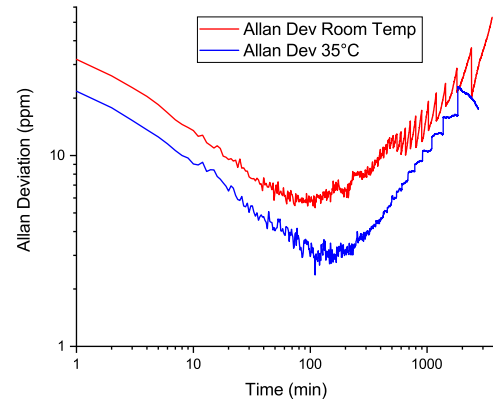


Fig. 7. Allan variance measurements with the blackbody source replacing the incandescent lamp.

B. Temperature Response

The ezPyro™ device will be incorporated into systems where the ambient temperature is not controlled, and may even be located in areas where the temperature will exceed the normal operational temperature of the sensor. The measurements in section III-A were carried out at a temperature of 24 °C, which is slightly above the measured room temperature due to the thermal heating from the tungsten lamp. To investigate the response of the ezPyro™ and tungsten source to increasing temperature a flexible heating element (Thorlabs HT10K) was wrapped around the assembled ezPyro™ device and reflector module. The heating element was controlled using a Thorlabs TC200-EC temperature controller, with feedback provided by a 10kΩ NTC thermistor located within the heating element. The ezPyro™ temperature was also measured independently using a type-K thermocouple, located on the break-out board behind the position of the ezPyro™. This also allowed the temperature stability as a function of time to be measured, by logging the temperature at 1 s intervals for 15 minutes at each temperature increment. The typical standard deviation of the measured temperature was measured to be 0.2 % of the average.

Initially, the ezPyro™ response as a function of temperature was measured using 100 % nitrogen for temperatures ranging from 25 °C to 60 °C at increments of 5 °C, as shown in figure 8. It is clear that the signal from the ezPyro™ begins to drop as a function of temperature. This may be a result of the type of pyroelectric crystal used in the manufacture of the ezPyro™ as shown by Whatmore [3]. This is further evidenced through the response of the ezPyro™ above 65°C, where the signals become distorted, similar to that of some TGS (triglycene sulphate) materials tested by Whatmore. However, this could also be a result of the reduced performance of the tungsten filament at higher temperatures, or overheating of the backplane electronic components. It should also be noted that

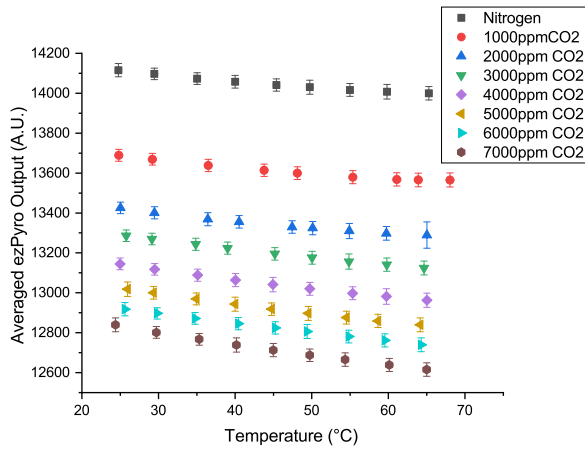


Fig. 8. Plot of averaged signal against increasing temperature for several different CO₂ concentrations.

an additional reference channel would compensate for effects of temperature variations on the detector element.

C. Flowing Gas

The performance of the ezPyro™ and tungsten filament sensor was also measured as a function of gas flow rate. In this case, a length of thin walled plastic tubing, with a 3.5 mm internal diameter, was connected to the gas inlet of the test chamber feeding directly into the sensor module.

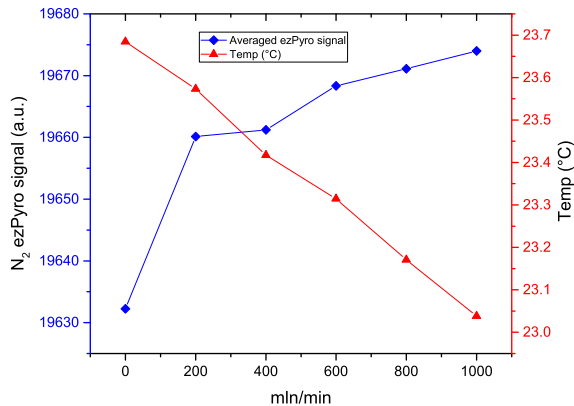


Fig. 9. Averaged peak-to-peak values for increasing flow rates of nitrogen (Blue \blacklozenge) with corresponding sensor temperature measurements (Red \blacktriangle)

Initially, 100 % nitrogen was flowed through the cell from flow rates of 200 to 1000 ml/min at increments of 200 ml/min, as shown in figure 9 (blue \blacklozenge , left hand y-axis). The data shows a significant increase in the averaged peak-to-peak signal between the static gas sample and the first flow measurement of 200 ml/min. This response variation is also seen to be inversely proportional to the measured ezPyro™ temperature figure 9 (red \blacktriangle and right-hand y-axis). This response is a result of the cooling of the device since the signal is directly proportional to the pyroelectric coefficient, p , where $p = \frac{\delta P}{\delta T}$,

here δP is the change in polarisation in relation to the material dipole moment and δT is change in temperature. So if it cools, δT is smaller (relative to RT , where R is responsivity) and therefore the pyroelectric coefficient and the signal increases.

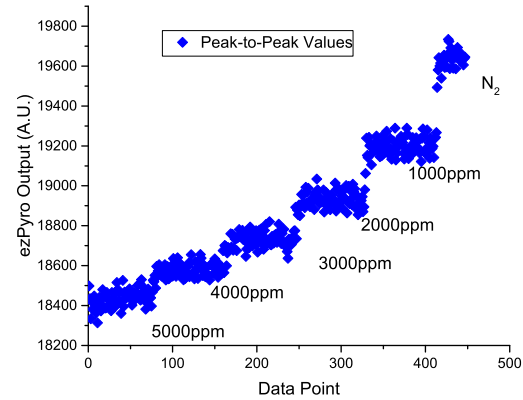


Fig. 10. ezPyro™ response to reducing concentration at a constant flow rate of 600mln/min.

Further flow measurements were taken for a single flow rate of 600 ml/min, with varying CO₂ concentrations from 0 to 5000 ppm, as shown in figure 10. For these measurements, peak-to-peak values from the ezPyro™ were recorded for a total of 15 minutes, where each concentration of gas was flowed for 2.5 minutes. The changes in concentration are clearly visible, with evidence of measurements made during the transition between concentrations as the gases passed through the tubing, showing the response of the ezPyro™ is fast enough for most gas sensing applications.

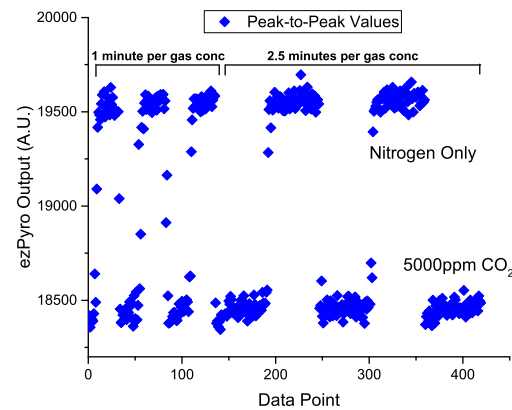


Fig. 11. ezPyro™ output of concentration switching to simulate effect of HVAC system.

In figure 11 a combined flow rate of 600 mln/min consisting of 300 mln/min each of the 1% CO₂ and N₂ was introduced directly into the sensor. At certain times the CO₂ flow was manually switched on and off using a feed valve connected before the gas mixing zone. This not only periodically removed the CO₂ from the sample but halved the flow rate during

those periods. These statistics are indicative for different levels of resolution of room temperature measurements of CO₂ concentrations in Smart buildings/offices to control HVAC systems, windows, etc [12].

IV. CONCLUSION

Although further testing will be required to fully characterise the ezPyro™ these preliminary tests have shown it to be capable of long term stability of measurement and resolution equivalent to, or better than, the currently available commercial sensors. Where the ezPyro™ exceeds the current format for NDIR sensors is in its compact size, being a surface mount device measuring $5.65 \times 3.7 \times 1.55$ mm, and in that the output is purely digital.

From the data shown in figures 3 4 and 5, it may be the case that any gas measurement system incorporating the ezPyro™ would benefit from the addition of a reference channel to normalise the output for intensity fluctuations from the optical source.

With single measurement deviations of ± 48 ppm and ± 22 ppm (lamp and blackbody respectively) for the two sources used and a noise equivalent detection (1σ) of 3ppm for the CO₂ sensor displays the potential of the ezPyro™. The response to changes in operational temperature showed a consistent trend which could easily be compensated for during measurements either by improved thermal management or the additional reference channel. Flow measurements with changing analyte concentration displayed the feasibility for the ezPyro™ to be incorporated into process control and air quality monitoring systems.

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Dr. John W. Phair, CTO leads the development team at Pyreos Ltd, a company which develops unique thin-film pyroelectric infrared detectors. Since graduating from the University of Melbourne with a Doctorate in Chemical Engineering with a focus on Geopolymers in 2001, Dr. Phair has worked on fundamental microstructure characterization of cements as a Research Associate at the National Research Council in Washington, DC, on hydrogen separation membranes at the Commonwealth Scientific and Industrial Research Organisation (CSIRO) in Australia, and on solid-oxide fuel cell fabrication and characterization at the Technical University of Denmark in collaboration with Topsoe Fuel Cell. Prior to joining Pyreos, he worked at poLight AS, a Norwegian company developing a MEMS autofocus lens component based on piezo technology for mobile applications. He has over ten years of experience in optical MEMS and functional thin-film materials and over 20 years in materials science.

Dr. Michael Lengden received the M.Phys. degree in physics and the Ph.D. degree from the University of Manchester, Manchester, U.K., in 2001 and 2006, respectively, where he was engaged in stepwise excitation of atomic and molecular metastable states. He was an Applications Engineer at the Laboratory Impex Systems, Ltd., from 2006 to 2007 and a Research Fellow at the Centre for Microsystems and Photonics from 2007 to 2010. He is currently a Senior Lecturer in the Electronics and Electrical Engineering Department, University of Strathclyde, Glasgow, U.K., where he is engaged in high-temperature and high-pressure gas composition measurements using tunable diode laser spectroscopy. His current research interests include applied tunable diode laser spectroscopy techniques in harsh environments and miniaturized photoacoustic sensor development. Dr. Lengden is a member of the Institute of Physics.