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Title Page

Title:

A Novel, Low-Cost, Portable PID Sensor for the Detection of Volatile Organic Compounds

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Highlights:

- Design of PID sensor with compositional output for the test sample

- Successfully discriminate between very low concentrations of VOC samples

- Detectable concentration down to 2 ppm isobutylene

- Leak proof sensor design

Abstract:

We report on the design, fabrication and verification of a portable, low cost PID. Unlike commercial PID sensors, ours provides two outputs. One output correlates to the total chemical components and a second that provides some level of compositional information. We believe that this makes this sensor system more useful than a standard commercial PID, at a similar cost point. Our PID sensor was tested with gas concentrations down to 2 ppm isobutylene. The results presented indicate that the limit of detection will be well below 1 ppm. Compositional analysis was also carried out and the results

presented shows our sensor can successfully discriminate between low concentrations of 2-hexanone, isobutylene, propanol, 2-pentanone, 2-octanone and 2-heptanone.

Keywords:PID sensor, Photo ionization, VOC detector, Environmental monitoring

1   Introduction

There has been an increased need to detect and identify volatile organic compounds (VOCs) in a range of industries and environmental applications [1]. VOCs are ubiquitous, and their presence signify a range of physical or chemical processes that may be desired or harmful. The presence of some VOCs in air has been linked to a variety of serious health symptoms including allergic reactions, asthma, increased bronchial responsiveness [2,3]. Studies have reported a strong relationship between total exposure to VOCs and health problems, such as mucus membrane irritation and central nervous system damage [3]. The risk associated with hazardous VOCs has led to increased VOC monitoring by several governmental agencies including the United States Environmental Protection Agency (USEPA)[4]. In Europe, the European Parliament has introduced Air Quality Directive specifying mandatory monitoring of VOCs and other harmful gasses in ambient air [5]. VOC detection has also been applied in monitoring of agricultural produce [6-9], environment [10], food and beverages [11-13], pharmaceuticals and health [14-21]. Presence of VOCs such as those used in this paper from human waste have been investigated as biomarkers for lung cancer and IBD [22,23].   Consequently, there is a high demand for VOC detection instruments.

Currently, instruments used for VOC monitoring include gas chromatography-mass spectrometer (GC-MS), gas chromatography-ion mobility spectrometer (GC-IMS), photoionization detectors (PID), metal oxide sensors (MOX) and optical sensors to name a few. GC-MS is often stated as the gold standard for VOC analysis because of its high sensitivity, accuracy, reproducibility and overall robustness. However, wide adoption of this technologies is limited by several factors including cost of purchase, complexity of operation and long analysis time. Its size and weight reduce their portability and restrict their use to mostly laboratory analysis. MOX and PID instruments are smaller in size, significantly

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cheaper and more portable in comparison. Some commercial PIDs offer analysis times within a second [24]. MOX sensors are the cheapest but tend to suffer from cross sensitivity to inorganic gases and can drift over time with low accuracy outputs. PID sensors are still relatively low cost and are highly sensitive chemical sensors that are used extensively for the detection of a broad range of VOCs [25]. They can provide a linear output to a single chemical or mix of chemicals, in real-time, and have a sensitivity in the ppb (parts per billion) range [26,27]. Furthermore, as they are undertaking a physical measurement, they are less likely to drift over time. PIDs have also been used to detect effluent from gas chromatographs, liquid chromatographs and coupled with ion mobility spectrometers (IMS) and mass spectrometers [28]. Several PID sensors are commercially available from manufacturers including Alphasense and Mocon and cost under $500.

Although these attributes make the PID a suitable sensor for many VOC analysis, PID sensors only provides a concentration value of the sample being tested, without any additional information of the chemical composition. In this paper, we report on a novel PID design that offers additional compositional information about the sample under test.

1.1 Theory

The operation of commercial PIDs can be divided into three steps: supply of analytes in gas phase, formation of photoions (by interaction between analytes and high energy photons emitted from a photodischarge source such as an ultraviolet (UV) lamp), and finally detection [28,29]. The ionisation reaction mechanism leading to the formation of photoions is given by *Equation 1*.

$$hv + M \rightarrow M^*$$ Eq. 1

Photons (*hv*) from the photodischarge source are absorbed by a molecule of the analyte (*M*) leading to the formation of an excited molecule (*M**). When *hv* is greater than the ionisation energy (IE) of the molecule, the molecule releases an electron leading to the formation of a cation as described in *Equation 2*.

$$M^* \rightarrow M^+ + e^-$$ Eq. 2

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The cations are driven to an electrode and the resulting current is an indication of the quantity of M^+ present hence providing information on the concentration of the analytes.

Inherent in this process are two advantages of PIDs. One advantage is that some level of selectivity can be achieved due to the difference in hv and IE of the analytes. Depending on the choice of UV lamp used for ionisation, detection could be targeted at a mixture of compounds below the ionisation potential of the UV lamp. However, the system will still detect all molecules below the ionisation potential and so specificity is limited. This advantage enables analysis in air since the components of air have higher IE than most UV lamps. Frequently used UV lamps include argon, krypton and xenon lamps. Their ionisation energies in comparison to some VOCs are shown in Table 1 [28,29]. A second advantage in using PID sensors is their ability to function in ambient pressure requiring less auxiliary equipment to operate. This facilitates portability of PID sensors and makes them suitable for testing in a range of environments and has resulted in their increased use for *in situ* analysis.

2   Materials and methods

2.1   Overview

In our work, we developed a full custom PID 'like' sensor. The sensor was designed with a single PCB at its core to simplify manufacture. The detection electrodes were housed on one side of the PCB and the electonics to process the signal on the other side. An ionisation chamber and fluidics are fitted over the detectors. The whole device weighs approximately 200g and measures 7 cm x 5 cm x 4 cm. During operation, the sample is introduced into the sensor through the inlet. The sample arrives the ionisation chamber where they are ionized by photons emitted from a 10.6 eV krypton UV lamp obtained from Alphasense Ltd, UK. An illustration of our system is shown in Figure 1. The stream of ions reach the electric field region where a high electric field is applied perpendicular to direction of ion travel. This results in a deflection of ions with some ions reaching the detection electrode. A current signal is generated and measured at the current electrode and is related to the ion species reaching the electrode for a given electric field.

2.2 Sensor Design

2.2.1 Ionisation Chamber

The ionisation chamber (Figure 2a) was designed from a single block of aluminium and measures 46 x 18 x 10 mm. Two 10-32 UNF thread was machined on opposite sides of the block's length. Two 10-32 UNF to 1/8 inch tubing fittings were screwed into the threads to serve as inlet and outlet for the sensor. A track path was machined across the under surface of the block to allow passage for the sample. A 6.1 mm bore is machined above this path to suspend the UV lamp above the sample path. The sample path underneath the bore is constricted to ensure the whole sample is exposed to the high energy photons travelling in a perpendicular direction to the sample flow. The chamber extends to cover the electrode, guiding the stream of ions over the electrode surface to the outlet. To ensure real time analysis, the sample stream in our sensor is not pulsed, allowing a constant strream of samples through the sensor at any given time. An o-ring is inserted around the sample path and 12 M2 screws are used to compress the o-ring beween the PCB and the chamber. This design approach ensures a leak free sensor for testing hazardous VOCs.

2.2.2 Electric field and electrodes

The generated ions are propelled over an array of electrodes for detection. This array consists of electrodes supplying high electric field used to deflect the stream of ions towards other electrodes serving as detectors. Two of this array configuration was implemented in the design.

In one array, the electric field was held constant and the output from this configuration is an indication of the total sample concentration (similar to commercial PID sensors). In the second array, the electric field could be varied between -10kV/m to +10kV/m. This output provides some composition information on the analytes. The arrays are separated by a grounded plate to ensure there is no interference between the electrode configurations. Additionally, each configuration sample path is pneumatically isolated, therefore, ions from one configuration cannot reach the detectors of the other configuration.

2.2.3 Ion detector electronics

A two-stage amplification process was used to amplify ion current signal from both detection electrodes. In the first stage, an AD823AR operational amplifier (opamp) was implemented as a transimpedance amplifier converting the measured detector current (in picoamperes) into voltage. A schematic of this circuit is shown in Figure 2(b).

The second stage utilizes the AD823 opamp as a differential amplifier. This stage amplifies the difference between the bias amplified voltage in the first stage. Next the amplified signal is passed through a first order low pass filter to reduce noise before the signal is read by a 12-bit analogue to digital converter. The circuit is capable of detecting signals down to 1 pico-amperes. The sensor is controlled using an ATSAMD21G18 ARM Cortex M0 processor. This processor is connected wirelessly to a computer via Bluetooth LE protocol with the Nordic nRF51822 chipset.

2.2.4 Software

A Universal Windows Platform (UWP) app was designed in-house to drive the electronics, electric field, flowrate, and record the amplified signals from the detection electrodes for concentration and composition over time. The software user interface is shown in Figure 3.

For each analysis, the software is designed to setup and control parameters such as time, electric field used to deflect streams of ion, and flowrate based on inputs entered by a user. At the start of an experiment, an instruction packet is sent to the ATSAMD21G18 ARM Cortex M0 processor requesting a byte stream of the instrument status. Next, another instruction is sent to specify the electric field and flowrate through the sensor before starting analysis and powering on the UV source. Since UV bulb life is limited, the software ensures the bulb is only powered during analysis to preserve its lifetime. Next the software creates a dispatcher to run the analysis task on a new thread in the host computer or tablet. This ensures accurate timing of the experiment since the process is not interrupted or delayed by other activities in the host computer or tablet. Timing is critical since sequence of inputs and results are required to be the same across sample test for accurate data processing. The software then requests a stream of sensor variables including output from both electrodes, flowrate, and electric field. With each packet of variables received, a signal processing using moving variable filter is applied to the electrode

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outputs. The raw and filtered variables are streamed to a .csv file for future data analysis. The test process is synchronised to the host device processor clock to achieve accurate and repeatable timing. Using this timing, a high priority instruction is sent to the ATSAMD21G18 ARM Cortex M0 processor to change the electric field and/or flowrate as dictated by the user settings every 100ms.

2.3   Results and discussion

2.3.1   Chemical concentration information

Performance of the PID sensors were characterised using isobutylene (ISB). This is a common test gas for characterising PIDs and is used extensively by industry. The standard test procedure involves supplying a known concentration of ISB gas into the sensor and measuring the current generated at the detection electrode. This output is related to the concentration of ISB gas. An ISB gas bottle was purchased (BOC, UK) at 100ppm concentration in air. It was diluted with zero air gas using an API Model T700 Dynamic Calibrator to generate low concentration ISB gas at 9mL/min. The diluted gas was supplied into the PID sensor and the current generated at the detector electrode was measured using our UWP app. To characterise the dynamic response time and repeatability of the sensor, 10 ppm ISB was supplied into the sensor inlet for 10s followed by a supply of zero air for 10s. This sequence was repeated 4 times. Figure 4(a) shows the amplified signal response. Once the system has stabilised in clean air, our sensor shows a fast response time of less than 2 seconds (defined as time to achieve 90% of final value). This response time can be adjusted in our software by increasing/reducing the cycle time and noise reducing algorithm effect but is ultimately limited by the characteristics of our test station. However, we observed better stability between pulses using 100ms cycling time. The result also shows repeatability of the sensor to a fixed concentration. The voltage output was similar across all four tests shown.

One of the advantages of PID sensors is fast response to varying concentration. This property of the sensor was tested by delivering increasing concentrations of ISB gas into the sensor. ISB was delivered into the sensor with concentration increasing from 0ppm to 18ppm in steps of 2ppm. The sensor

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concentration output shown in Figure 4(b) shows that the limit of detection is likely to be in the ppb region for ISB.

2.3.2   Chemical composition information

Our PID sensor provides a second output with information on the chemical composition of the analytes, a feature not available in commercial PID sensors. This feature was investigated by analysing various VOCs on our sensor and comparing the sensor response. During analysis, a supply of test VOC with fixed concentration was delivered to the sensor. The electric field, which results in a deflection of cations, was varied from -7kV/m to +7kV/m in steps of 500V/m. At zero electric field strength there is no deflection of the flow of ions and all of the ions contact the detector plate and giving the maximum response. Sweeping the electric field in the positive or negative direction results in reduced current (and thus signal) due to loss in charge when ions hit the walls of the chamber and thus do not reach the detection electrode. The deflection observed could be due to the interaction between the charge and/or weight of the ions in the stream and the intensity of the electric field. Between each step change in electric field, the gas supply was turned off and resumed after changing the electric field. Signal response due to the varying amount of ions reaching the compositional electrode is measured over the period of test. This analysis was repeated for 10 ppm isobutylene and 2-pentanone. To remove any bias associated with concentration, the compositional sensor response was normalised. This is shown in Figure 5.

The results show significant difference in sensor output for ISB and 2-pentanone over the range of applied electric fields. An electric field of 1kV/m on the composition electrode results in a 0.67 V separation between the signal obtained for both compounds. This information can be used to train the sensor to target the identification of certain compounds. To achieve this, the electric field could be set to a value associated with a known response for the target compound. The response from the concentration electrode array could also be added providing increased dimensionality for statistical methods such as linear discriminate analysis.

In another test, 2 ppm of 2-hexanone was fed into the sensor at 7ml/min. In this test, the supply of analytes remained constant for the duration of the analysis. The electric field voltage was varied from -3 kV/cm to 3 kV/cm in steps of 200 V/cm. This procedure was repeated for 10 ppm propanol. Testing compounds of different concentration was carried out to represent real life test scenarios where test compounds will not always have a similar concentration, but the instrument is expected to provide responses not biased towards concentration. A comparison of the normalised compositional signal response for both compounds sensor is shown in Figure 6.

We observe 0.063 V separation in signal response when the electric field is set to 1000 V/cm, which was repeatable over 5 different experiments. This shows that by sweeping through a range of electric field, we can distinguish between VOCs. Although the compositional output at a fixed electric field is different, we believe the shape of the curve and area under the curve for the period of analysis provides more information for pattern recognition analysis.

The repeatability of results obtained during analysis of a VOC and our sensors potential to distinguish between VOC compounds were further verified using 10 ppm 2-hexanone, 2-octanone, propanol and 2-heptanone, in dry zero air. In this analysis, a fixed concentration of VOC was supplied in to the sensor inlet. The electric field was varied from -7 kV/m to +7 kV/m while the voltage response from the sensor was measured and recorded. After sweeping through the voltages, the supply of VOC was shut off. This procedure was repeated 20 times for 2-hexanone, 2-octanone and propanol. Principal Component Analysis (PCA) was utilized to reduce the dimensionality of the recorded data. The first and second principal component accounted for 96 % of data variance in the original dataset and a scaled plot of these components is shown in Figure 7. The results from our sensor shows discrimination between responses obtained for 2-hexanone, 2-octanone and propanol.

Further tests were carried out using 10 ppm 2-heptanone and 2-octanone as test VOC samples. Fixed concentration of VOC was flowed into the sensor during the analysis. The electric field was varied from -7 kV/m to +7 kV/m. This procedure was repeated 20 times for both VOCs. The compositional responses measured during the tests were recorded and analysed using PCA. Figure 8 shows a scaled plot of the first and second principal components.

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The first and second components accounted for 97.8 % variance in the original data set. As depicted in Figure 8, there is also clear discrimination between compositional information obtained for 2-heptanone and 2-octanone.

These results demonstrate that our sensors can be used to detect low concentration VOCs. They also show that our sensor can distinguish between VOCs. In the current sensor design, the high voltage electric field required for VOC identification results in considerable interference and signal loss at lower concentration. Our future efforts will focus on improving the electric field generator to reduce interference and improve signal response when testing low concentration VOCs. Additionally, we will also investigate our sensor electric field potential to separate complex mixtures of VOCs during analysis.

3   Conclusion

In this paper, we report on the design of a novel PID sensor with added sample composition output that has been designed, manufactured and tested. The sensor provides two voltage outputs proportional to the concentration and composition of the test sample. Concentration tests were carried out with low concentration isobutylene and the concentration response was found to increase linearly with concentration. An increase in concentration in from 0 – 18 ppm resulted in an increase in output voltage of 2.3V, with an estimated sensitivity of below 1 ppm. The composition response shows a significant distinction between 2-pentanone and isobutylene when 1kV/m was applied in the chamber. A 0.063 V difference was observed between 2-heptanone and propanol when 1000 V/cm electric field was applied. Our sensor compositional response also discriminated between 10 ppm 2-hexanone, 2-octanone and propanol when the electric field was varied from -7 kV/m to +7 kV/m. We also reported discrimination between 2-heptanone and 2-octanone over the same electric field range. The added composition information, leak proof feature and low cost makes this unit potentially provide more information at a lower price point when compared with existing commercial sensors.

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Biographies

Samuel O. Agbroko is a PhD student at the School of Engineering, University of Warwick. He received his bachelor's degree in Mechanical Engineering from University of Benin, Nigeria in 2010 and his MSc in Mechanical Systems from the University of Warwick in 2013. His areas of interest include mechatronics, digital and analogue electronics, embedded systems, software development, and emerging gas sensor technologies. His current research focuses on developing novel gas chromatography ion mobility spectrometry devices and software for biomedical applications.

James A. Covington, received his BEng 1996 in Electronic Engineering at Warwick University and remained their receiving his PhD 2000. His PhD was on the development of CMOS and SOI CMOS gas sensors for room temperature and high temperature operation. He worked as a research fellow for both Warwick University and Cambridge University on the development of gas and chemical sensors. He was appointment as a lecturer in 2002 in the School of Engineering, University of Warwick, being promoted to Associate Professor in 2006 and is now a Professor in Electronic Engineering. He heads the BioMedical Sensors Laboratory, School of Engineering. He has authored or co-authored over 150 technical papers and patents. His current research interests focus on the development of micro-analysis systems, electronic noses and artificial olfaction, employing a range of novel sensing materials, device structures and micro-fabrication methods for applications with the environmental and medical application domains.
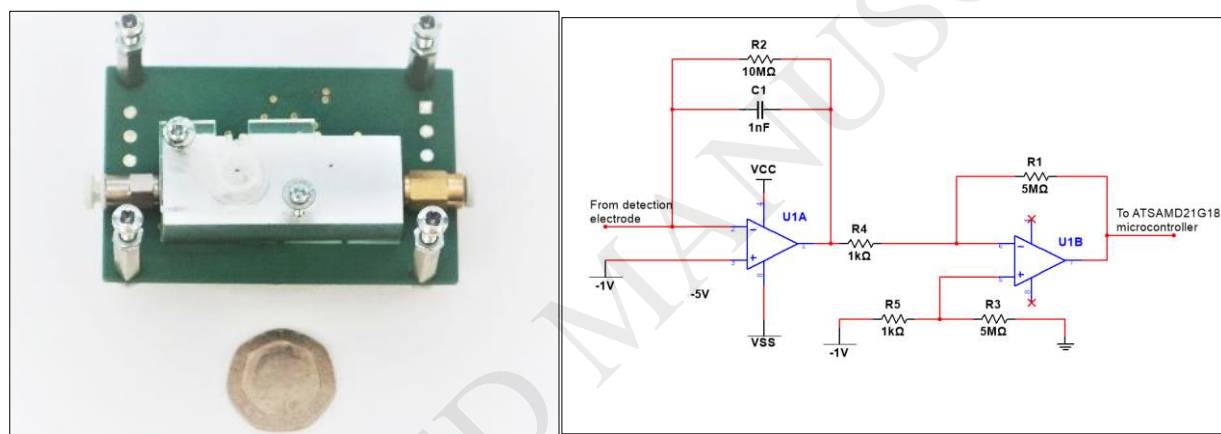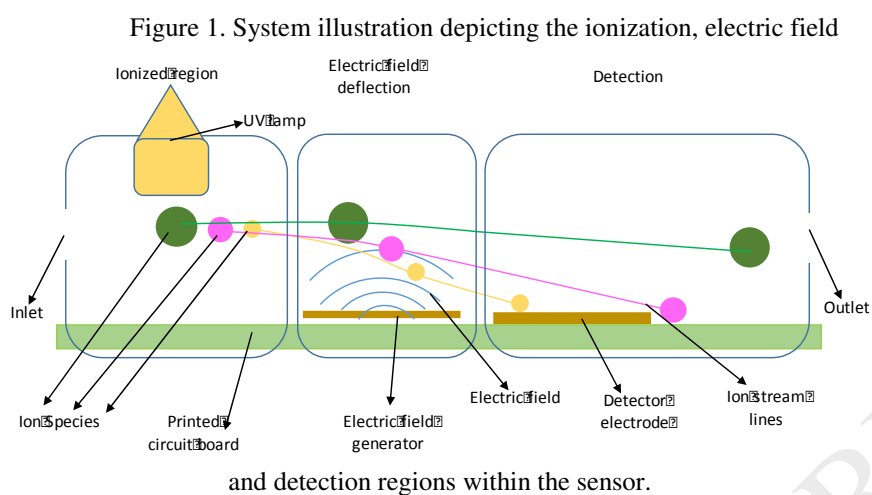
Figures

Figure 1. System illustration depicting the ionization, electric field

and detection regions within the sensor.

Figure 2. (**a**) Bottom view of the sensor showing the ionization chamber, UV lamp, inlet and outlet; (**b**)

Schematic representation of our sensor amplification electronics.

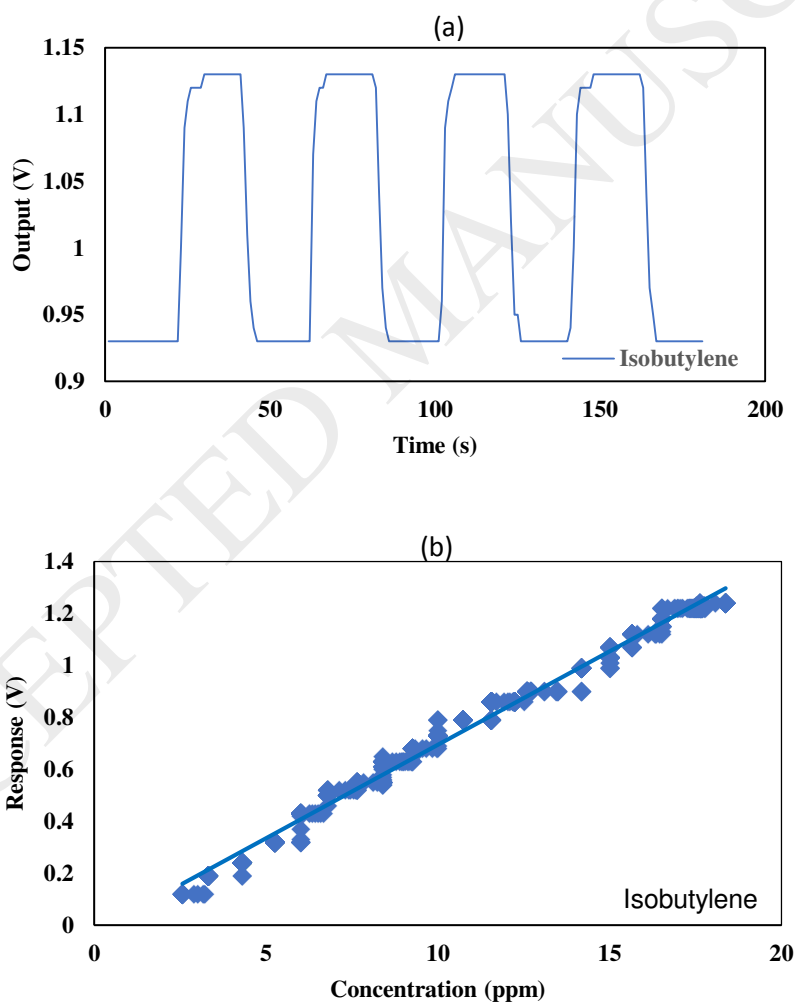Figure 3. The PID control software developed using Visual Studio 2017.

Figure 4. (**a**) Dynamic concentration response for isobutylene;
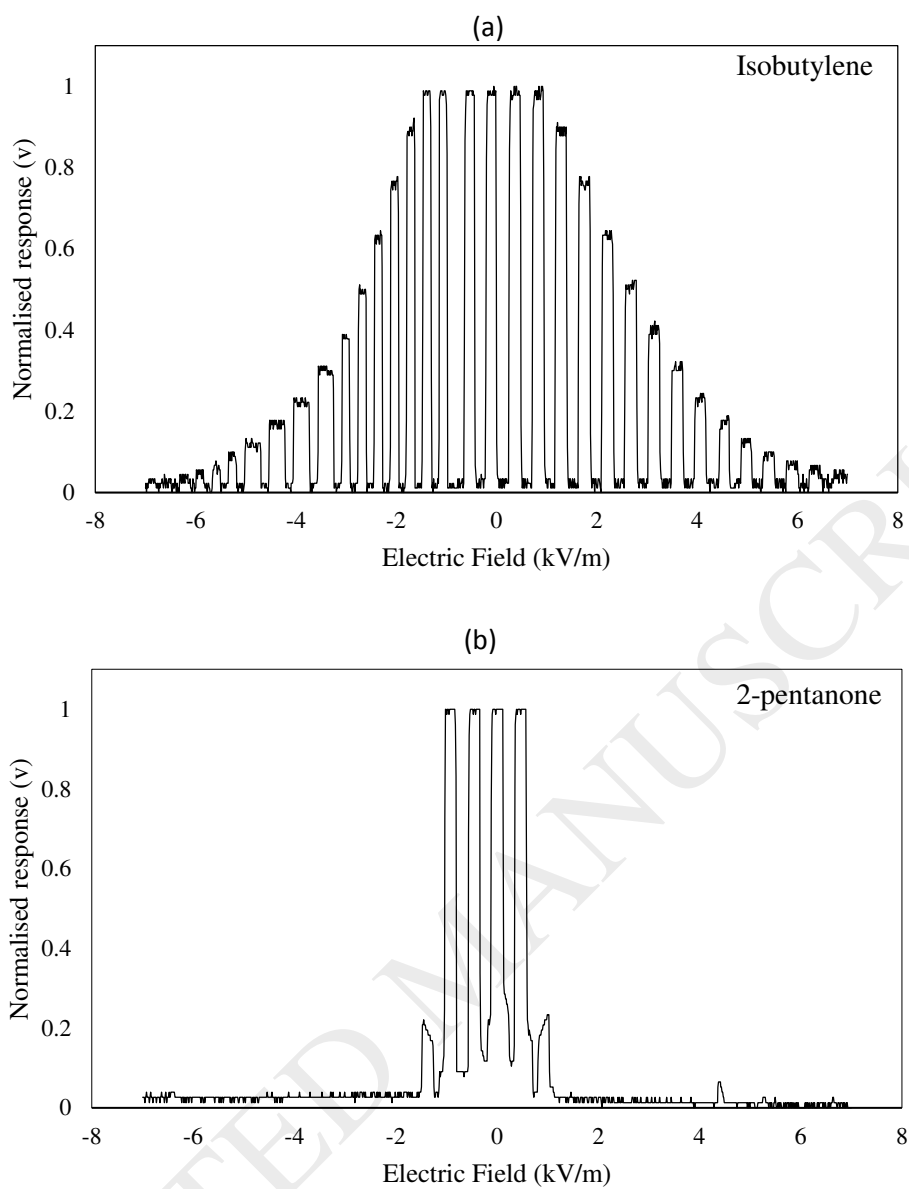(**b**) Dynamic response for 0-18ppm isobutylene at 10 seconds intervals.

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Figure 5. Comparison of normalised chemical compositional signal response for (a) 10 ppm isobutylene; (b) 2-pentanone.
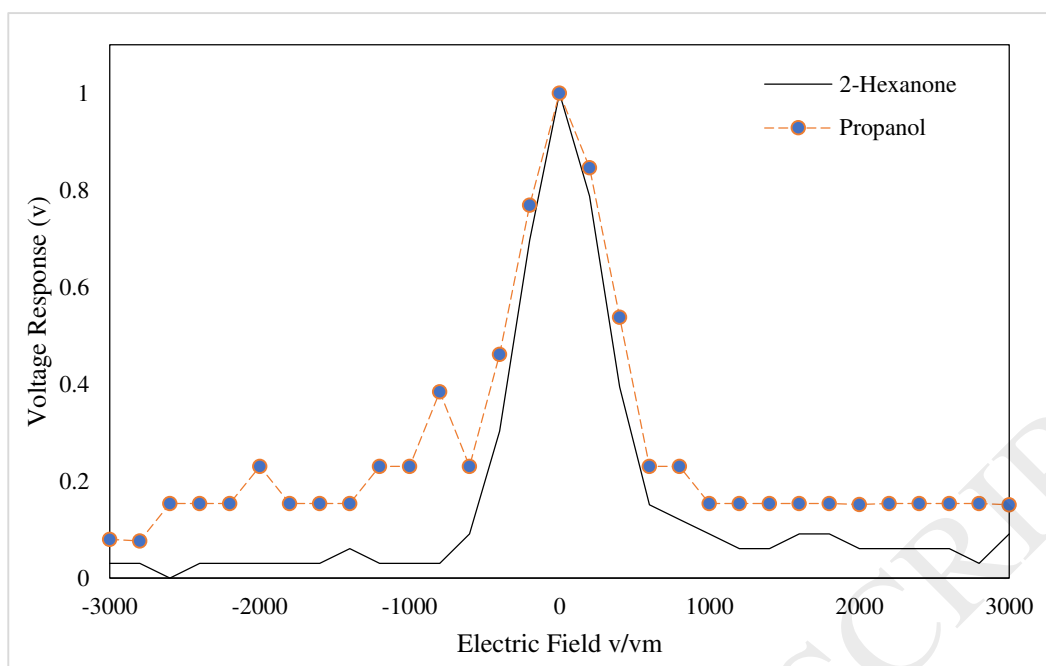
Figure 6. Comparison of chemical compositional signal response for 2-hexanone and propanol
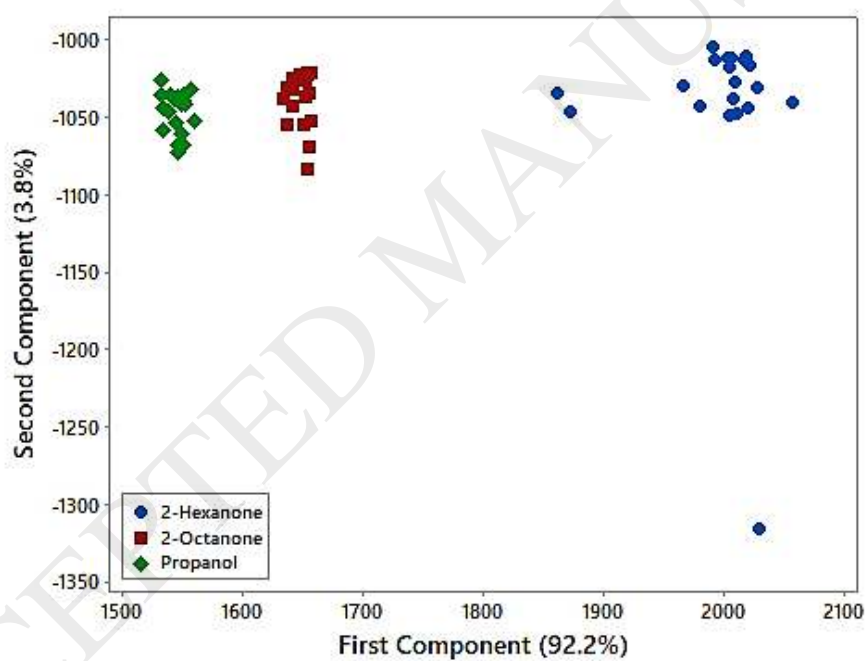

Figure 7. Scaled PCA plot of our sensor response for 2-hexanone, 2-octanone and propanol
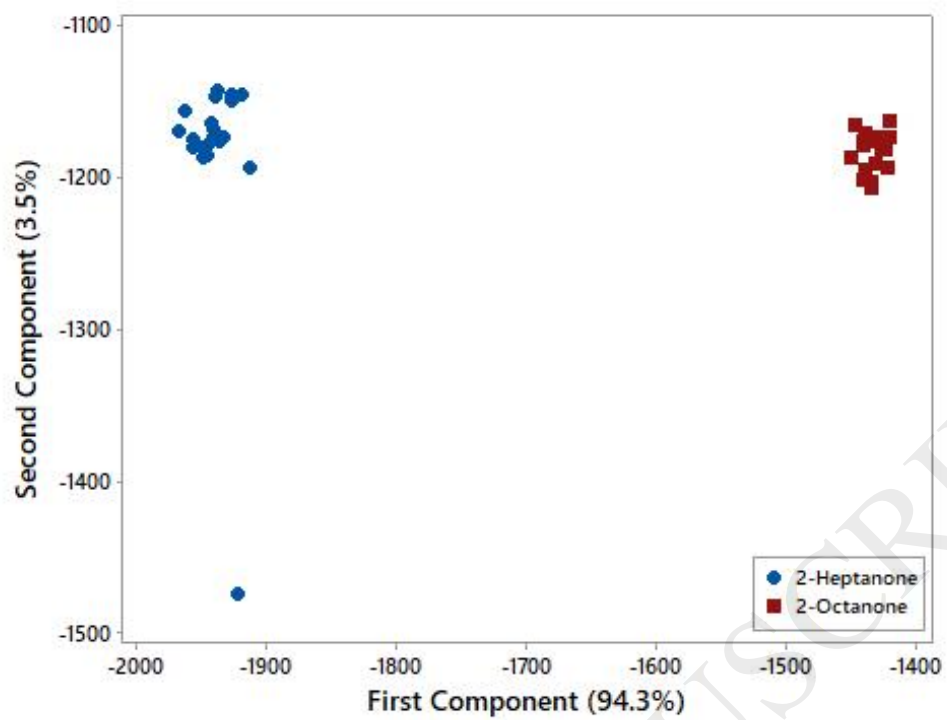
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Figure 8. Scaled PCA plot of our sensor response for 2-heptanone and 2-octanone

Table 1 Ionisation energy of common VOCs and UV lamps

UV lamp	Compound	IE
	Oxygen	12.07
	Acetonitrile	12.2
	Nitrogen	15.58
	Water	12.62
Ar: 11.7		
	Acetic acid	10.65
	Methanol	10.84
	Chloroform	11.37
Kr: 10.6		
	Isopropanol	10.17
	Ammonia	10.07
	Hexane	10.13
	Heptane	9.93
	Acetone	9.7
	Toluene	8.83
	Propanol	10.02
	Ethanol	10.48
	Isobutylene	9.22
	2-pentanone	9.381
	2-hexanone	9.35
Xe: 8.4		
	Benzyl radical	7.2
	Acridine	7.8
	Anisole	8.2
	Naphthalene	8.14