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# Artificial olfaction system for on-site odour measurement

by Michael Atzeni, Craig Lobsey, David Mayer
and Gavin Parcsi

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**Researcher Contact Details**

Mr Michael Atzeni
Department of Agriculture and Fisheries
PO Box 102
Toowoomba QLD 4350

Phone: 07 4529 4188
Email: michael.atzeni@daf.qld.gov.au

In submitting this report, the researcher has agreed to RIRDC publishing this material in its edited form.

# Foreword

Odour nuisance arising from intensive livestock production facilities is an ongoing problem, which is complicated by the subjectivity of odour perception. To tackle this problem, production industries need tools and technologies to reliably demonstrate the efficacy of odour reduction strategies. Similarly, regulatory authorities require tools to evaluate and validate negative impacts arising from odour nuisance in order to investigate odour complaints. However, a lack of convenient, affordable and objective odour measurement tools makes this extremely difficult in practice.

An artificial olfaction system (AOS) is a chemical sensing instrument that can target specific smells such as aromas and malodours. These so-called 'electronic noses' have been commercially available since the mid-1990s. However, their application has been largely limited to laboratory-based, quality control in the food and beverage industries.

Adapting AOS technology to environmental applications has presented new challenges for developers. Although researchers have demonstrated AOS data can be used to predict meat chicken odour concentrations in-shed, they are yet to establish whether an AOS can provide reliable odour concentration measurements beyond the shed.

This project aimed to develop an AOS for field assessment of chicken farm odour emissions. Successful development of a portable AOS will benefit those involved in environmental odour assessments including farm managers, integrators, their consultants, regulators and researchers.

This report is an addition to RIRDC's diverse range of over 2000 research publications and it forms part of our Chicken Meat R&D program, which aims to stimulate and promote R&D that will deliver a profitable, productive and sustainable Australian chicken meat industry that provides quality wholesome food to the nation.

Most of RIRDC's publications are available for viewing, free downloading or purchasing online at www.rirdc.gov.au. Purchases can also be made by phoning 1300 634 313.


**John Harvey**
Managing Director
Rural Industries Research and Development Corporation

# Acknowledgments

# Abbreviations

| | |
|---|---|
| ABARES | Australian Bureau of Agricultural and Resource Economics and Sciences |
| AOS | artificial olfaction system |
| CPAP | continuous positive airway pressure |
| DAFF | Department of Agriculture, Fisheries and Forestry, Queensland. |
| DO | dynamic olfactometry |
| enose | electronic nose |
| FO | field olfactometer |
| GLM | general linear model |
| MFC | mass flow controller |
| MOS | metal oxide semiconductor |
| NMVOC | non-methane volatile organic compound |
| ou, OU | odour unit. (1 ou = dilution threshold at which 50% of panel can detect an odour.) |
| PCA | principal components analysis |
| PID, pid | photo-ionisation detector |
| PLS | partial least squares |
| PTR-MS | proton transfer reaction - mass spectrometry |
| RH | relative humidity |
| SIFT-MS | selected ion flow tube - mass spectrometry |
| TD-GC-MS | thermal desorption - gas chromatography - mass spectrometry (may be referred to using the abbreviation GC-MS) |
| TGS, tgs | a brand of MOS sensor |
| TVOCs | total volatile organic compounds |
| VOC | volatile organic compound |

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# Executive Summary

## What the report is about

Odour impacts and concerns are an impediment to the growth of the Australian chicken meat industry. To manage these, the industry has to be able to demonstrate the efficacy of its odour reduction strategies scientifically and defensibly; however, it currently lacks reliable, cost effective and objective tools to do so. This report describes the development of an artificial olfaction system (AOS) to measure meat chicken farm odour. This report describes the market research undertaken to determine the demand for such a tool, the development and evaluation of three AOS prototypes, data analysis and odour prediction modelling, and the development of two complementary odour measurement tools, namely, a volatile organic compound (VOC) pre-concentrator and a field olfactometer.

## Who is the report targeted at?

This report is aimed at investors in poultry odour research and those charged with, or interested in, assessment of odour on chicken farms, including farm managers, integrators, their consultants, regulators and researchers. The findings will influence the focus of future environmental odour measurement research.

## Where are the relevant industries located in Australia?

Australia wide; the chicken meat industry is comprised of some 700 growers and 40,000 employees. Chicken meat production occurs in all Australian states, and is usually situated or located in close proximity to major metropolitan centres. The projected gross value for 2013-14 of all Australian poultry meat production, of which chicken meat comprises about 95%, is $2.291 billion according, to Australian Bureau of Agricultural and Resource Economics (ABARES).

## Background

The chicken meat industry requires an objective means to measure odour on-site so that more informed steps can be taken to minimise odour impacts. Currently, no such tools are commercially available. In recent years, the focus has turned to adapting electronic nose technology (a traditionally qualitative technique) to poultry odour measurement. Initial research was promising but limited to in-shed assessment at a single shed (Sohn *et al*., 2008; Dunlop *et al*., 2011). Due to the unpredictability of odour emission rates, the greater need is to be able to measure odour downwind, preferably at the odour receptor, or at the farm boundary. This current study is a comprehensive investigation to develop an AOS for both in-shed and downwind assessment of meat chicken farm odours.

## Aims/objectives

The aim was to develop a tool or proxy measurement to quantify the intensity of meat chicken odour. This tool would provide a measure of odour that is consistent and less subjective than current techniques relying on human panels (such as dynamic olfactometry). It would also enable on-site odour measurement and thereby address problems with transporting samples and lagged olfactometry analyses, for example, the issue of odour decay during transportation of samples. Finally, it would reduce the cost of odour quantification to enable on-line monitoring of odour change, for example, for research purposes.

## Methods used

The project methodology was as follows:
- Establish the market needs and expectations for a commercial AOS solution.

- Develop gas-sensing instrumentations utilising an array of sensors (that is, AOS instruments) for VOC measurement.

- Collect a range of representative poultry odour samples from within chicken sheds and downwind (from different stages of a batch, and at multiple farms).

- Analyse these samples using (a) the developed AOS (on-site) and (b) dynamic olfactometry (at the odour laboratory), and build a database of sensor and odour unit (ou) measurements.

- Develop the calibration models to directly relate the sensor measurements to ou measurements and then estimate the ou concentration of future unknown samples.

## Results/key findings

- The research undertaken demonstrated that AOS can only provide 'ball park' predictions of meat chicken farm odour concentrations, in and around chicken sheds.

- AOS predictions are subjective and unreliable due to the inadequate (too insensitive) sensors available and the inherent ambiguity of non-specific sensor data.

- Current sensor technology limits AOS development; any improvement will be dependent on better, more specific and sensitive sensors becoming available.

- Demand for affordable AOS solutions is high.

- At this stage, AOS is not commercially viable for poultry odour measurement.

- SIFT-MS (and similar analytical tools; for example, PTR-MS) provide new opportunities to assess and quantify odour (using odorant data) that may facilitate and complement AOS development in future.

## Implications for relevant stakeholders

Quantification of odours in the field will remain subjective and problematic in the foreseeable future. Initial results using AOS at meat chicken sheds proved promising, but the real-life challenges associated with measuring odours in real time from chicken sheds became apparent as the project progressed. AOS was found to have limited value for measuring odour from meat chicken sheds due to the insensitivity of the electronic sensors to key odorants and interferences by ammonia and humidity.

In limited circumstances, AOS technology may prove useful in odour measurement (for example, for research applications with local calibration), however, it is unlikely that any AOS utilising low-cost sensors could provide a reliable on-site odour measurement tool for monitoring poultry odours, or any other intensive livestock odours for that matter, in the foreseeable future.

## Recommendations

Future odour measurement research should concentrate on measurement of odorants and on developing odour prediction models from odorant data. It is recommended that SIFT-MS or PTR-MS be evaluated for this purpose.

Any further efforts with AOS should be contingent on (a) identifying the key odorants responsible for odour nuisance, then (b) developing or sourcing sensors that can detect the target odorants at the concentrations they occur.

# Introduction

> "Measurement is the first step that leads to control and eventually to improvement. If you can't measure something, you can't understand it. If you can't understand it, you can't control it. If you can't control it, you can't improve it."
>
> — H. James Harrington

The intensive livestock industries are required to minimise odour impacts on neighbours and surrounding communities. Despite the implementation of odour abatement and control measures, public concerns and complaints about nuisance odour continue to hamper the expansion of these industries.

Odour complaints have escalated in some areas due to intensification of production facilities and urban and industrial encroachment into traditional farming areas. Commonly with odour complaints, questions are raised about the odour source, odour strength, and the veracity of the complaint.

With proposed new developments, or proposals for expansion of existing facilities, questions invariably arise about the accuracy of odour dispersion modelling and whether the proposed separation distances between the farm and neighbouring receptors have been either underestimated or overestimated. Evidence to prove that proposed odour control strategies will work is often requested.

In the case of new proposals, these questions could be better resolved if there were an objective, convenient tool for measuring and monitoring odour on-site, or at similar establishments (preferably in the same area). However, there is currently no such instrument commercially available.

Advances in computing and sensor technologies, and demand for cost-effective, robust, portable odour assessment instruments, continue to fuel interest in the development of sensor array systems for odour monitoring and other environmental applications (Brattoli *et al*. 2011; Wilson 2013; Gutiérrez and Horrillo 2014). The current trend is to develop more specialised systems with narrow-spectrum sensor arrays for specific applications, because such instruments are more affordable and aligned to industry requirements (Wilson 2013). This is especially applicable for environmental odour assessment given the time- and site-specific limitations that apply (Stuetz *et al*. 1999; Bourgeois *et al*. 2003).

The Australian chicken meat and egg industries have invested heavily in gathering odour data to better understand odour generation and dispersion, and address odour impacts (e.g. Jiang and Sands 2001; Dunlop *et al*. 2011). Following promising results using a prototype AOS for in-shed odour monitoring at meat chicken farms (Sohn *et al*. 2008, 2010), the chicken meat industry funded further AOS research through this project in the hope that a prototype with wider utility could be developed for commercialisation.

This report covers the development and comprehensive evaluation of an AOS for on-site odour measurement at meat chicken farms and discusses the future prospects of AOS in the poultry industry. Research recommendations for future odour assessment purposes are made.

# Objectives

The broad aim of this research was to develop a prototype instrument for onsite meat chicken farm odour assessment. The prototype must meet specifications including robust and reliable portable odour assessment in utilising non-specific gas sensor array(s) combined with a signal processing engine, for on-site meat chicken farm odour assessment.

The specific objectives were to:

- undertake market research to identify potential market segments with an interest in accessing a poultry odour AOS and their needs with respect to features required;

- incorporate appropriate design features that will meet the needs of identified key market segments;

- develop a prototype AOS to meet the needs of identified key market segments;

- evaluate and demonstrate the sensitivity and discrimination capability of the AOS through intensive field trials in Australia;

- develop a VOC pre-concentration technique;

- develop a prototype field olfactometer to enable more cost-effective AOS training;

- validate the AOS prototype.

# Methodology

## Project implementation

The project was implemented as follows:

- Market research was conducted using an on-line survey to determine the market requirements and expectations of AOS solutions for odour assessment (see Chapter 1).

- Based on outcome of the market research, a prototype handheld AOS was designed, developed and evaluated (see Chapter 2).

- Based on identified shortcomings of the handheld AOS, the first of two research-grade systems (AOS MK1) was built and evaluated in the laboratory (see Chapter 2), then trialled for three months on a southeast Queensland chicken farm (farm A) to test sensor array performance and continuous monitoring capability (see Chapter 3).

- Based on the above trial, AOS MK2 was built and evaluated in the laboratory (see Chapter 4), then trialled at two southeast Queensland chicken farms (farms B and C). This entailed conducting AOS odour measurements of a sufficient number of olfactometry samples to develop an *odour prediction model* to predict odour strength in odour units (ou) (see Chapter 5).

- Bagged samples for olfactometry were assessed on-site with the AOS MK2, then analysed in the odour laboratory at Toowoomba by dynamic olfactometry within six hours of collection. Before each AOS odour assessment, a baseline measurement using clean air was conducted to account for sensor drift over time. After returning from the field, sensors were calibrated using isobutylene and ammonia reference gases to correct for changes in sensor response over time. The sensors were also calibrated for relative humidity (see Chapter 5).

- Odour samples were collected onto Tenax tubes for analysis by thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS) using a method developed for poultry odour. From the GC-MS analyses a database of the non-methane volatile organic compounds (NMVOCs, that is, odorants) detected was compiled (see Chapter 6 and Appendix B).

- AOS MK2 sensor data was analysed using a number of multivariate analysis techniques (see Chapter 7).

- An odour prediction model for AOS MK2 was developed and tested during a validation trial at an independent farm (farm A) on odour samples collected there for olfactometry analysis (see Chapter 7).

- Odour samples were also collected and analysed using a selected ion flow tube mass spectrometer (SIFT-MS) during the above validation trial. This helped in evaluating the AOS sensor data responses more objectively and confirmed the presence and concentration of key odorants (NMVOCs and sulphide compounds) (see Chapter 6).

- A field olfactometer was developed to augment laboratory-based dynamic olfactometry (see Chapter 8).

- A VOC pre-concentrator was built to enable collection of concentrated odour samples (on Tenax tubes) for odour assessment (see Chapter 9).

## Olfactometry analysis

Analyses were conducted according to the Australian/New Zealand Standard for Dynamic Olfactometry (AS4323.3) (Standards Australia/Standards New Zealand, 2001) using an eight-panellist forced choice dynamic olfactometer as shown in Figure 1. The development of this olfactometer is described by Zeller *et al.* (2002).

To reduce error in olfactometry results, panels were sourced from a pool of 12 experienced panellists. To improve the accuracy of the measurement, two extra rounds (dilution series) were conducted on each sample, provided there was enough sample to do so.

The panellists were screened using *n*-butanol to ensure their individual detection threshold was in the range of 20–80 ppb (v/v). The procedures are described in Sohn *et al.* (2008).



**Figure 1.    Panellists evaluating odour at the dynamic olfactometry laboratory**

## TD-GC-MS sampling and analysis

TD-GC-MS was used to identify non-methane volatile organic compounds (NMVOCs, otherwise referred to as odorants) in the odour samples collected for this purpose.

### Sample collection

Tenax sorbent tubes were used to trap odorants for TD-GC-MS analysis. Samples were collected in duplicate via a common manifold connected to the sample line. A vacuum pump was used to draw sample through the tubes at 100 mL/min (at standard conditions 0°C, 101.325 kPa). The sample period was 30 minutes, unless otherwise indicated in the results. The detailed methodology is described in Dunlop *et al.* (2011).

At the end of each sample period, the pump was turned off and the tubes were immediately capped at both ends. They were then stored in a cool area away from direct sunlight. Details of the samples were recorded on field sheets.

### Analysis of NMVOCs

The following desorption and analysis parameter settings program were used:

- Thermal desorption: Tube desorbed for five minutes at 275°C, held by a cold trap at -10°C before the trap was heated at a rate of 40°C per minute to a final temperature of 290°C. It was then held for five minutes.

- Gas chromatograph: Oven program started by holding the sample for two minutes at 50°C before increasing the temperature by 10°C per minute until it reached 175°C. Then the temperature was increased at a rate of 25°C per minute until 225°C was reached. The sample was held for two minutes for a total run time of 18.5 minutes. The GC column used was J&W Scientific DB-VRX 30 m x 0.250 mm x 1.4μm.

- Mass spectrometer: Operated using constant scan 35-335 m/z, 1.25 minute solvent delay.

The amount of each analyte was reported as nanograms (ng) on the tube to two significant figures.

# 1. Market research

Market research was conducted in 2009 to determine user requirements for a commercial AOS. Atzeni *et al*. (2010) summarised this market research. The complete survey results, survey questionnaire and additional comments of respondents are contained in Appendix A. The following is a brief overview.

## Online survey

The market research was conducted using an online survey sent to 230 prospective users of AOS worldwide.

## Results

The survey results revealed there was a general lack of confidence in the reliability of current field odour assessment methods, and dissatisfaction with the cost of both laboratory and field odour assessments. Recognition of enose technology is high, and there are strong demands for reliable, portable odour sensing devices.

Concerning the important features for design and development of the AOS, accuracy and reproducibility/precision were considered the two most important factors, followed by sensitivity, portability and cost. Survey results showed that potential users were anticipating a portable field device, retailing for $10,000–$15,000 and weighing no more than 10 kg.

Portability was considered secondary to reliability of the measurements, provided the instrument can also be left *in situ.* Incorporation of global positioning system (GPS) and meteorological data collection functionality was considered desirable but not essential. Incorporation of additional specific sensors for measuring certain odorants (for example, ammonia) in tandem with odour was considered highly desirable, if cost-effective.

A retail market exists for AOS, as well as a rental market. Renting would be useful in a situation where multiple units are required for monitoring.

## Key findings

From the collective responses of those involved in odour assessment services, odour regulation, odour research and funding of that research, it can be concluded that:

- there is a ready market for AOS devices

- there is a general lack of confidence in current field odour measurements

- laboratory and field odour assessments are generally considered too costly

- there is significant interest in the development of reliable, portable AOS devices for on-site measurement for various odour emissions

- there are industry needs for reliable odour measurement devices tailored to specific odours.

In developing any commercial AOS solution, the following identified beliefs and expectations should be considered:

- accuracy and reproducibility/precision are the two most important factors, followed by sensitivity, portability and least of all, cost

- a retail price of $10,000 – $15,000 for a portable AOS is considered acceptable

- a portable AOS should weigh no more than 10 kg, but this is of secondary importance provided the AOS is reliable and can be left operating *in situ*

- incorporation of additional sensors for measuring specific key odorants in tandem with odour is highly desirable.

# 2. AOS hardware

Three AOS prototypes were developed during this project. The first was a customised handheld device designed according to market beliefs and expectations about cost, portability and what odorants should be measured in addition to odour. The next two prototypes were research-grade instruments (hereon referred to as AOS MK1 and AOS MK2) built with the ultimate aim of developing a system that could reliably quantify chicken shed odours in the field.

## Sensors

Five different metal oxide semiconductors (MOS sensors) were used. These sensors have a sensing element comprised of a metal oxide semiconductor layer formed on an alumina substrate of a sensing chip together with an integrated heater. They function by absorbing vapours into the semiconductor surface, which changes the electrical conductivity of the sensor.

Three different photo-ionisation detectors (PID) were used. This type of sensor is widely used in portable instruments for detecting a variety of VOCs and some inorganic gases in ambient air. They operate on the principle that a percentage of molecules are ionised according to their ionisation potential during exposure to high-energy vacuum ultra violet (UV) radiation generated by a gas discharge lamp, resulting in a measurable response.

In addition, sensors for ammonia ($NH_3$), temperature and relative humidity (RH) were added to the sensor array. A suitable cost-effective sensor for detecting hydrogen sulphide ($H_2S$) at the concentrations present in poultry odour was not found. Those trialled were abandoned.

## Sensor arrays

The sensor array configuration was different for each AOS prototype as shown in Table 1.

For the handheld AOS (Figure 2), the sensors were mounted inside the casing and directly exposed to the airstream. The sensors were powered by a rechargeable lithium battery. The airstream was generated by a battery-charged pump.

For AOS MK1 and AOS MK2, sensors were housed in stainless steel chambers based on the design used by Sohn *et al*. (2008). AOS MK 1 was solar powered for remote deployment; AOS MK2 was battery powered for portability but could also be run off mains power. Both used embedded computing systems for data acquisition and control.

**Table 1.     Sensors used for development of three AOS sensor arrays.**

| Sensor | Type | Usage | Handheld AOS | AOS MK1 | AOS MK2 |
|--------|------|-------|--------------|---------|---------|
| Figaro TGS 2600 | MOS | Detection of air contaminants | Y | Y | Y |
| Figaro TGS 2602 | MOS | Detection of air contaminants | | Y | Y |
| Figaro TGS 2610 | MOS | Detection of LP gas | | | Y |
| Figaro TGS 2611 | MOS | Detection of methane | | | Y |
| Figaro TGS 2620 | MOS | Detection of solvent vapours | Y | Y | Y |
| piD-TECH®*plus*10.6 | PID | Detection of VOCs and other gases with Ionisation Potential < 10.6 eV | Y | Y | |
| Alphasense piD-AH 10.6 eV | PID | Detection of VOCs and other gases with Ionisation Potential < 10.6 eV | | | Y |
| Alphasense piD-AH 9.6 eV | PID | Detection of VOCs and other gases with Ionisation Potential < 9.6 eV | | | Y |
| Sensoric NH$_3$ 3E 100 SE | Chemo-electrical | Ammonia monitoring (0-100 ppm) | | Y | Y |
| Sensoric H$_2$S 3E 30 | Chemo-electrical | Hydrogen sulphide monitoring (0-30 ppm) | | Y | |
| Honeywell HIH-4000 series RH sensor | Humidity Sensor | Humidity monitoring | | Y | Y |
| Texas instruments LM35 | Temperature sensor | Temperature monitoring | | Y | |
| PT100 | Temperature sensor | Temperature monitoring | | | Y |
| Sensirion SHT71 | Temperature and humidity sensor | Temperature and humidity monitoring | Y | | |

# Customised handheld AOS

## Description

A portable handheld AOS was designed and developed in line with the market survey findings. This conceptual 'commercialisable prototype' is shown in Figure 2. The sensor array and AOS operating system were incorporated into the housing from another commercially available gas sensing device, adopted for cosmetic and functional purposes (existing sampling pump, recharging cradle, and power control). Sensor data was accessible via Bluetooth for further analysis.

## Evaluation

This AOS met market expectations about design, portability, affordability and functionality; that is, hybrid sensor array for odour and ammonia assessment. However, the sensor data was considered to be unreliable for odour prediction purposes after comparative tests revealed the AOS temperature readings were unacceptably higher and the relative humidity readings were lower than the ambient air (sample) being measured.

It was concluded that the heat generated by the unshielded MOS sensors was enough to maintain a higher internal temperature inside the casing (and hence lower relative humidity)

than that of the incoming sample being measured. The unacceptable temperature difference was demonstrated by running the AOS in a refrigerator to exaggerate the effect (see Figure 3). At room temperature, the AOS temperature reading was around 17% higher than the ambient temperature.

It follows that the MOS and ammonia sensor responses would be compromised, given the significant influence temperature and particularly relative humidity of the sample has on the sensor responses. As these factors needed to be accurately measured and compensated for in an AOS, the handheld system, which was too restrictive to modify, was abandoned. Instead, the aims of the project were reassessed and revised to what was realistically achievable. Commercialisation plans (for example, applying for a provisional patent) were considered premature and put on hold while priority shifted to developing a research-grade AOS to investigate and resolve the odour measurement problem.



**Figure 2.    Prototype handheld portable odour sensing system.**
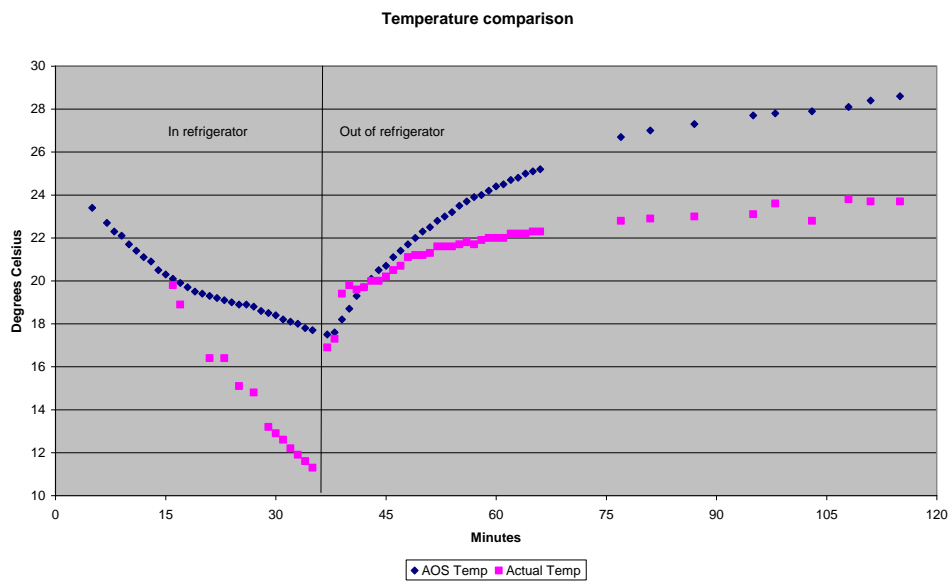


**Figure 3.    Comparison of AOS temperature and ambient temperature data.**

# AOS MK1

## Description

The chemical sensor array in AOS MK1 was a hybrid array consisting of three non-specific MOS sensors, one PID sensor, two electrochemical sensors (for ammonia and hydrogen sulphide) and temperature and humidity sensors. Specific sensor details are given in Table 1.

An embedded computer system was used for data acquisition and control. Metal oxide sensors are sensitive to air flow across the sensor surface and consequently require consistent flow conditions. Therefore, the system was designed to include sensing of the sample flow rate using a flow sensor and feedback control of the sample pump to ensure a stable sample flow rate of 500 mL /min.

Sensor baseline was performed using a clean air reference generated using a separate pump to deliver filtered air (using moisture and carbon filters) at a rate of 1000 mL/min to the sample inlet. At programmed intervals during field deployment the reference pump is started and the inlet valve opened to generate purified air and baseline the AOS sensors. The baseline referencing system was designed to ensure pressure in the sensing chamber is the same under measurement and referencing scenarios.

The AOS was designed to operate from 12 volt power and was solar-powered for extended field deployment. Components in contact with the sample gas were constructed from stainless steel and Teflon where possible (Figure 4). A schematic diagram of the hardware is shown in Figure 5.

## Evaluation

The field evaluation of AOS MK1 is described in Chapter 3.
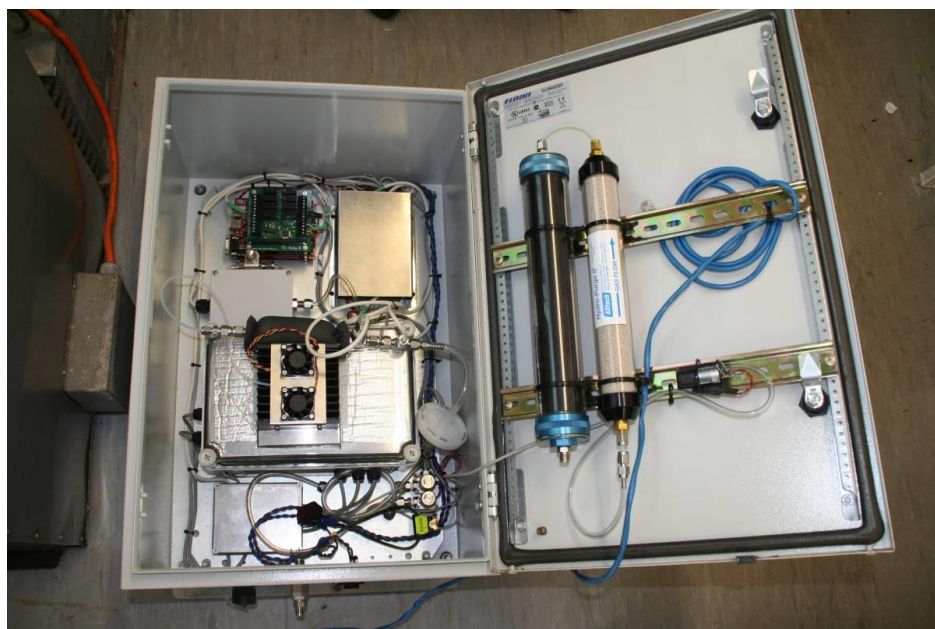


**Figure 4.    Internal view of AOS MK1.**
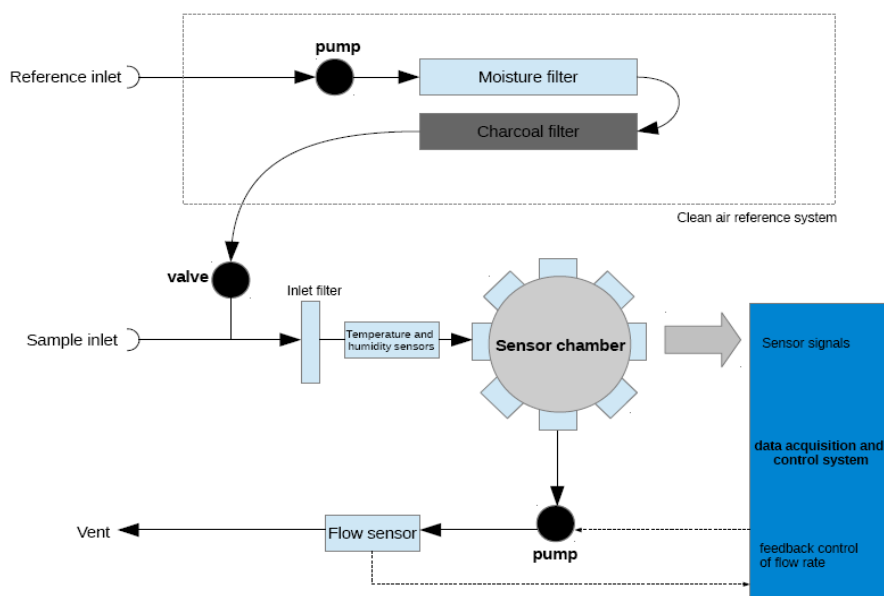
**Figure 5.  Schematic of AOS MK1.**


# AOS MK2

## Description

The AOS MK2 chemical sensor array was a hybrid array consisting of five non-specific MOS sensors, two PIDs, an electrochemical ammonia sensor, and temperature and humidity sensors as indicated in Table 1.

The 9.6 eV PID was added to the sensor array in an effort to provide a more selective measure of VOC quantities in the presence of ammonia. The sensor will only respond to the presence of compounds with an ionisation energy less than 9.6 eV and will therefore not respond to ammonia.

Again, all components in contact with the sample gas were constructed from stainless steel and Teflon where possible.

### Controlling and compensating for temperature and humidity

The sensors used in the AOS are sensitive to the temperature and humidity of the analysed gas. The AOS MK2 was designed to minimise the influence of these variables.

The effects of temperature variation were avoided by controlling and maintaining sample temperatures and the sensor chambers at a consistent temperature. The sensor chambers were contained in an insulated controlled temperature environment, which is maintained by the AOS control system at 30°C. The incoming sample temperature is also monitored by temperature sensors and the sample line is heated with feedback control to ensure samples are consistently analysed at this temperature. This also prevents condensation in the sample chambers (observed during field trials of the AOS MK1) by ensuring the chambers' and sample's temperatures are above dew point.

Correction for variation in relative humidity was attempted in two ways: (a) by using an RH correction model which is applied to the sensor outputs; and (b) by conducting a baseline reference in purified air at the same relative humidity as measured during sample analysis. To achieve this, the baseline referencing system was improved to enable clean air referencing at a selectable humidity. This system consisted of two mass flow controllers (MFCs) which control variable flow of purified dry air through a heated bubble humidifier. The data acquisition and control system in the AOS monitors the sample RH and continually adjusts the MFC flow rate to maintain a stable and specified RH during baseline.

A schematic diagram of the AOS MK 2 hardware is shown in Figure 6.
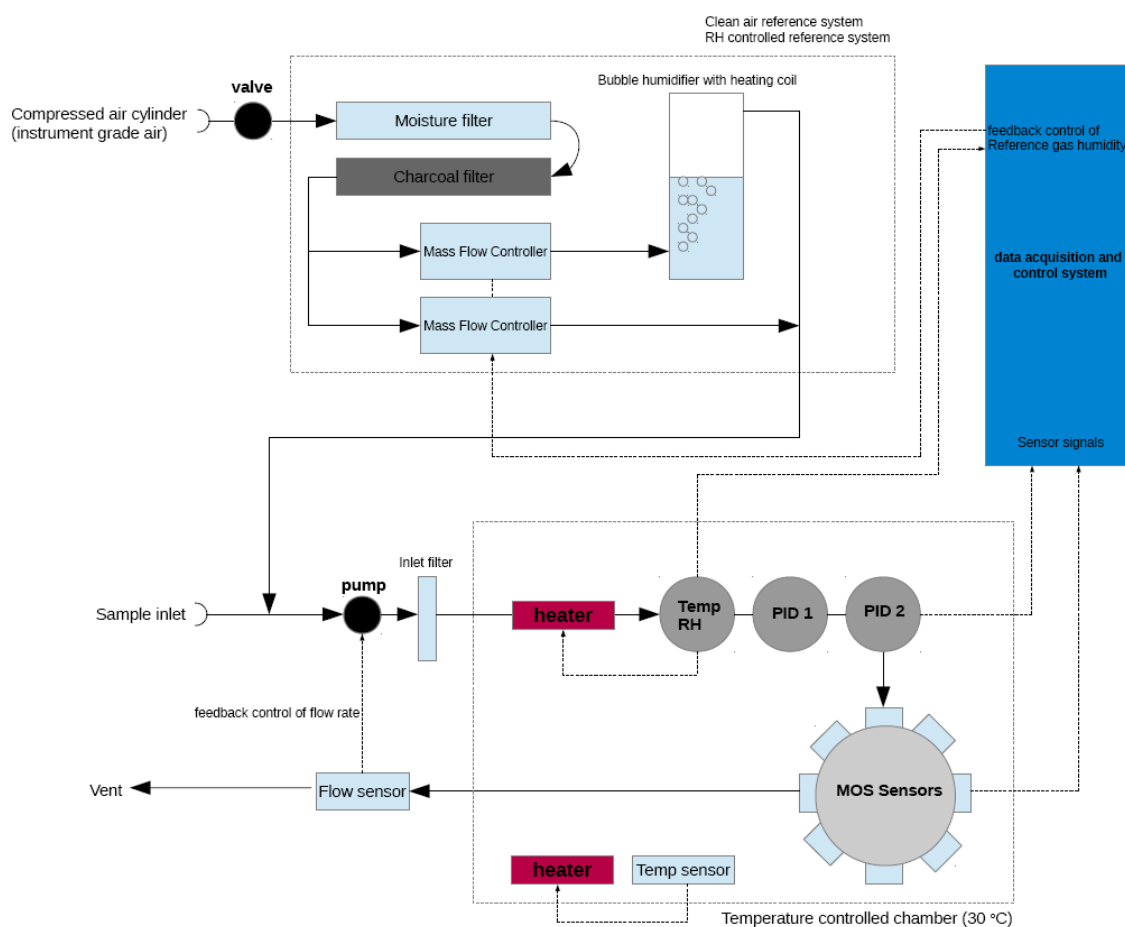


**Figure 6.     Schematic of AOS MK2.**

## Evaluation

The laboratory and field evaluations of AOS MK2 are described in Chapter 4 and Chapter 5, respectively.

# 3. Field trial one (AOS MK1)

## Background

This field trial evaluated the sensing hardware and software to demonstrate the ability of the AOS to continuously monitor meat chicken shed emissions on-site throughout a batch. The strategy adopted was to monitor the odour plume shifts caused by wind direction changes, as reflected by changes in the sensor responses. It was also necessary to determine whether the AOS could be operated continuously as a solar-powered unit.

## Study site

The study site used was farm A, which is situated in south-east Queensland, a subtropical region with summer-dominant rainfall. An aerial view of the study site is shown in Figure 7. There are other production facilities immediately to the west and south of this site but their influence, if any, was considered negligible for the purposes of this AOS trial.

The sheds are tunnel ventilated with a duty fan at the eastern end and a row of five exhaust fans on each side near the western end (back of the shed). The shed dimensions are approximately 124 m long by 14 m wide and they were stocked with approximately 31,800 chickens during each batch. Duration of each batch was seven to eight weeks.
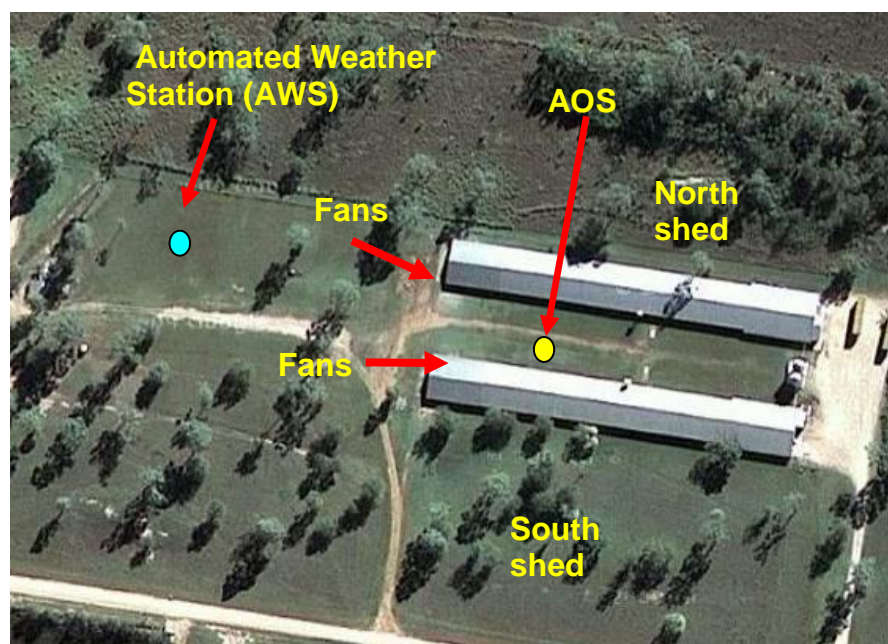


**Figure 7.    Aerial photograph of farm A showing the locations of the AOS MK1 and weather station relative to the monitored chicken sheds.**

## Methodology

The AOS MK1 was mounted on a purpose-built tripod and positioned midway between the two chicken sheds pictured, just east of the ventilation fans (see Figure 7 and Figure 8). This position was just out of the direct influence of the fans so that odour fluctuations due to change in wind direction could be detected. The AOS was left running continuously for three months over winter/spring 2011.

A 10 m weather station was installed on-site at the start of the project, approximately 100 m west of the two monitored sheds in a relatively open area to minimise influence of nearby trees and buildings on the readings. Data was regularly downloaded, checked for consistency and cross-referenced against Bureau of Meteorology data from a nearby station. The weather data was used to check the wind's influence on the sensor array response, and to compile a site-specific meteorological data file for odour dispersion modelling, if required.



**Figure 8.    AOS MK1 positioned near ventilation fans between two meat chicken sheds.**

Both sheds were instrumented to enable recording of fan activity, internal shed pressure and temperature. Shed data was downloaded to a computer each field visit and visually inspected to ensure it was sensible.

# Results

Figure 9 and Figure 10 illustrate the PID sensor response in relation to wind changes. Peaks occurred in the morning after tunnel ventilation resumed. They were greatest when conditions were stable (very low wind speeds) and the breeze is from the west, forcing the odour plume towards the AOS. Conversely, with an easterly wind, the plume was directed away and the PID response dropped to its baseline value. At times the PID sensor reached its upper detection limit and it was surmised that this response was due to ammonia, but it could not rule out that VOCs were also contributing to the response.
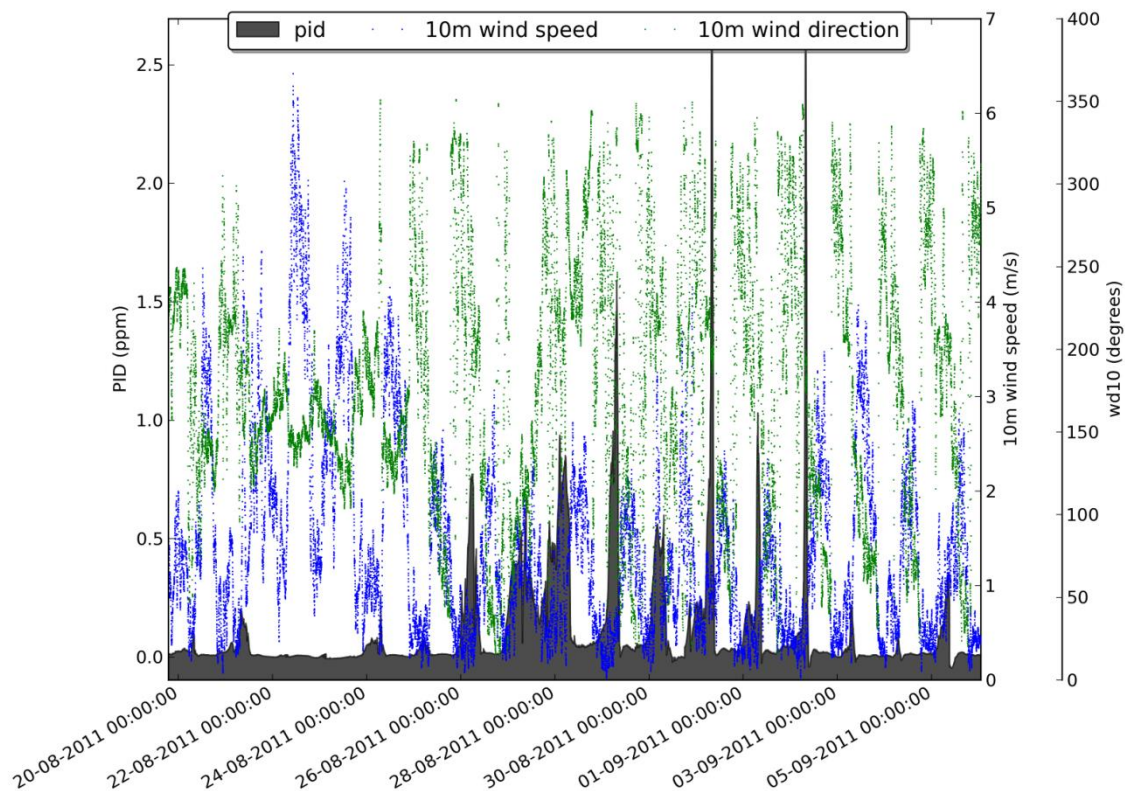
**Figure 9.    PID sensor response in relation to wind speed (blue dots) and wind direction (green dots) over an 18-day period.**

Occasionally, the sensors yielded unusual responses that were attributed to condensation in the sensor chamber. The AOS MK1's design allowed the sensor chamber to cool overnight when exposed to low ambient temperatures. Due to the high thermal mass of the sensor chamber, its temperature remained below the dew point of the shed environment in the early hours of the morning. When the exhaust fan on the shed opened, warm humid air from the shed environment was sampled by the system and likely condensed within the chamber. This could occur whenever sufficient temperature differential and humidity exists.

To ensure the temperature of the sensor chamber remained above the dew point of the sample being analysed, we designed the AOS MK2 with a controlled temperature (heated) sensing environment. A heated sensor chamber would also remove temperature effects on the sensors; that is, remove temperature variability.

Other outputs were indicative of increased flock activity, perhaps due to human disturbance, causing an increase in odour concentration, but such events were not recorded so this could not be verified.

The hydrogen sulphide ($H_2S$) sensor was non-responsive. This is attributed to the very low $H_2S$ concentrations found in meat chicken shed emissions (< 100 ppb). These concentrations would be difficult to measure using available sensors, with the electrochemical $H_2S$ sensor used having a detection range to 30 ppm.

AOS MK1 operated without problems throughout the trial indicating the design and software were robust.

16
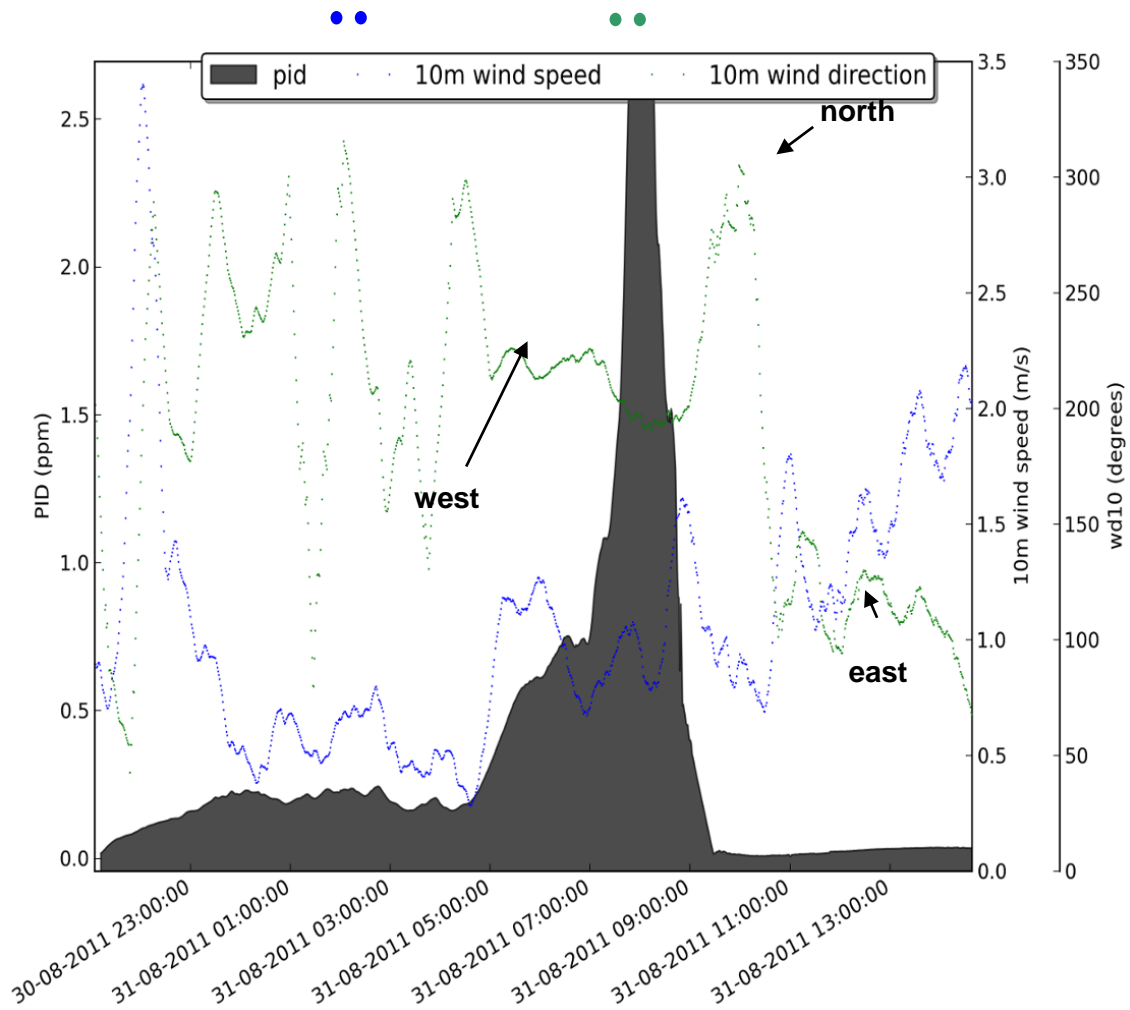
**Figure 10. PID sensor response in relation to wind speed and wind direction over a 14-hr period.**

## Key findings

The AOS MK1 field evaluation provided the following information:

- the PID was too sensitive (upper limits were reached on occasions)

- the $H_2S$ sensor was not sensitive enough (in-shed $H_2S$ concentrations are very low)

- unusual sensor readings were attributed to water condensation in the sensor chamber

- some fluctuations could probably be attributed to internal shed activity or bird disturbance

- measurements were correlated with fan activity, wind and shed conditions; however, it is difficult to model these relationships

- the PID sensor was particularly responsive, raising suspicions the compound causing this was ammonia, and concerns that ammonia was masking the total VOCs response for odour prediction purposes

- the instrument and sensors are robust, operating continuously for three months without malfunctioning.

From this trial the following was concluded:

- sensor selection should be modified with a less sensitive PID and the electrochemical $H_2S$ should be replaced

- thermal control of the sensor chamber is required to prevent condensation

- video and audio monitoring capabilities could be added to the AOS for in-shed monitoring of flock activity levels and disturbances

- more than one AOS would be beneficial and enable:

  - simultaneous in-shed and downwind monitoring

  - determination of odour emission rates from multiple sheds on the same farm.

# 4. Laboratory evaluation (AOS MK2)

## Background

The AOS MK1 design was revised based on the findings of the field trial. An experiment was then designed to control external factors, minimise sample delay and maximise sampling rate. The purpose of this laboratory evaluation was to ensure the revised AOS (MK2) worked before deployment in the field.

## Methodology

Representative odours were generated using shed litter samples collected from four farms (supplying to two different processing companies). The farms included Farm A and Farm BOdour samples were generated and analysed on the day of litter collection. For each litter sample, a series of litter headspace samples was collected using a flux hood. The flux hood was merely to facilitate odour collection and was not operated to any Standard. The quantity of litter within the flux hood was varied for each sample collected to achieve different odorant concentrations. The headspace samples were immediately analysed by AOS followed by forced-choice dynamic olfactometry soon after (commencing within 10 minutes of completion of the AOS analysis) ensuring odorant changes were minimised. The raw sensor data were processed and compared to the dynamic olfactometry results.

## Results

As expected, the ou increased with increasing litter used under the flux hood. The relationship between the response of the 10.6eV photo-ionisation detector and the measured odour concentration was also highly linear and consistent between the different litter samples
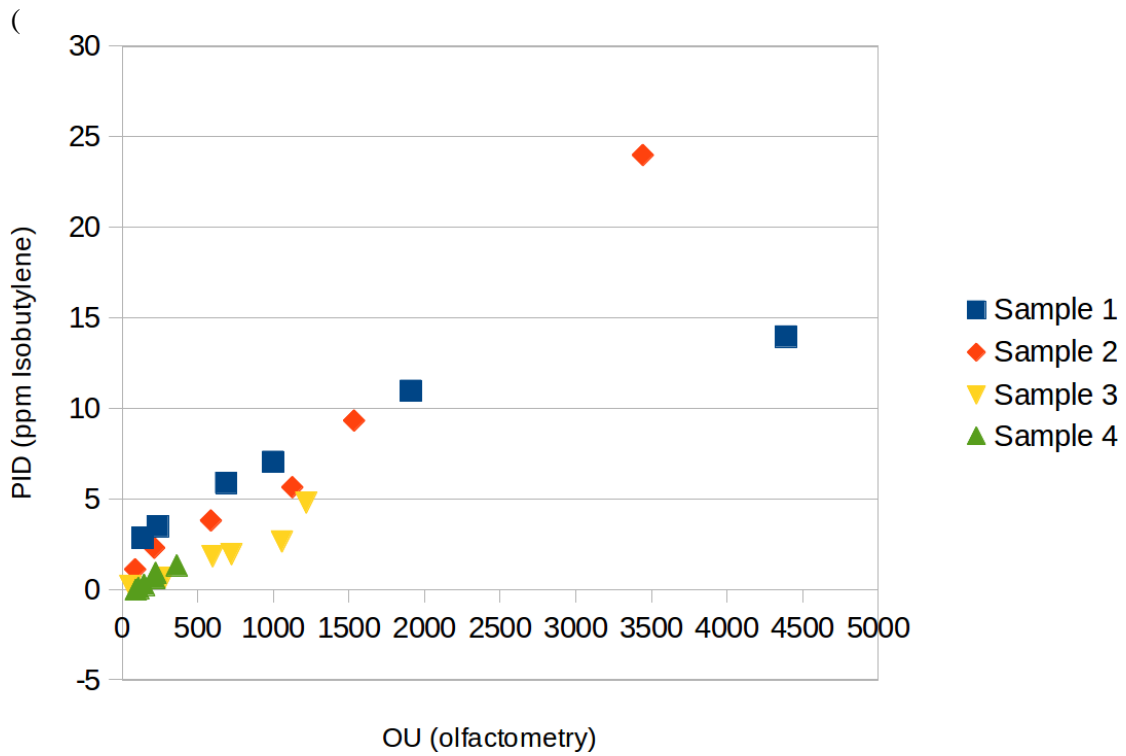
Figure 11).

This was initially a very positive result (in regards to sensor measurement of odour concentration); however these results were attributed to the high concentrations of ammonia generated by the disturbed litter samples (Figure 12). Figure 13 shows the measured ammonia concentration using the AOS electrochemical ammonia sensor compared to the PID response is also a highly linear relationship.
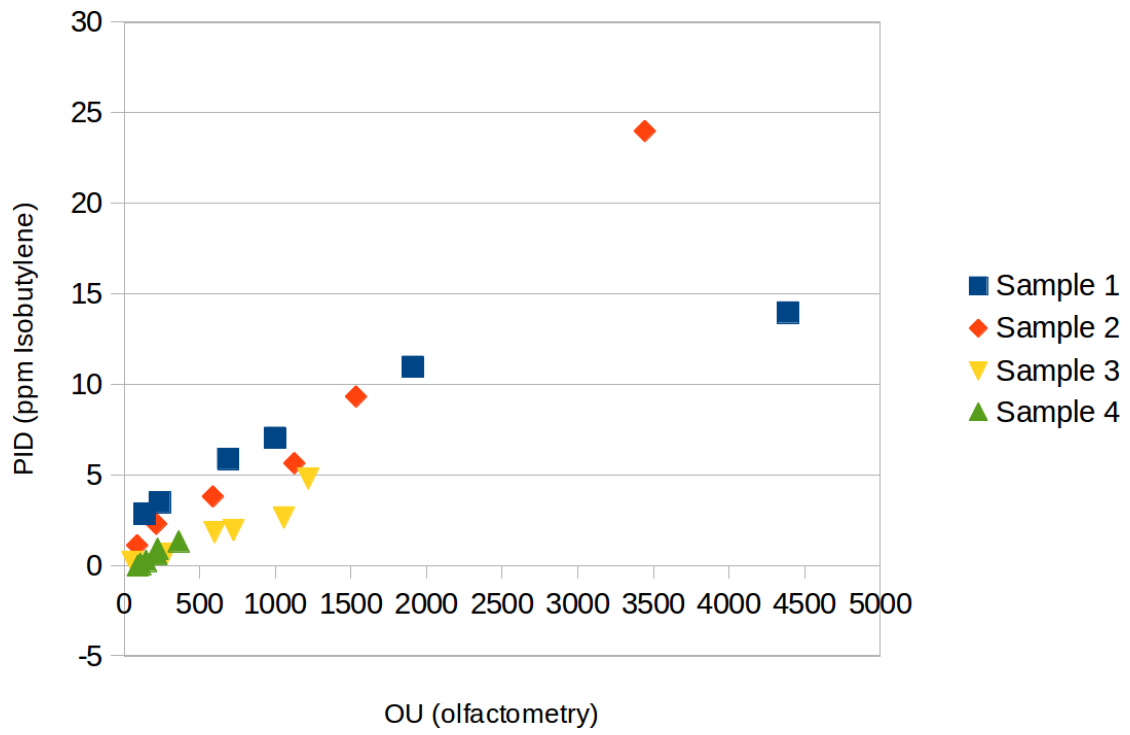
**Figure 11.  Litter headspace PID response compared to odour concentration.**
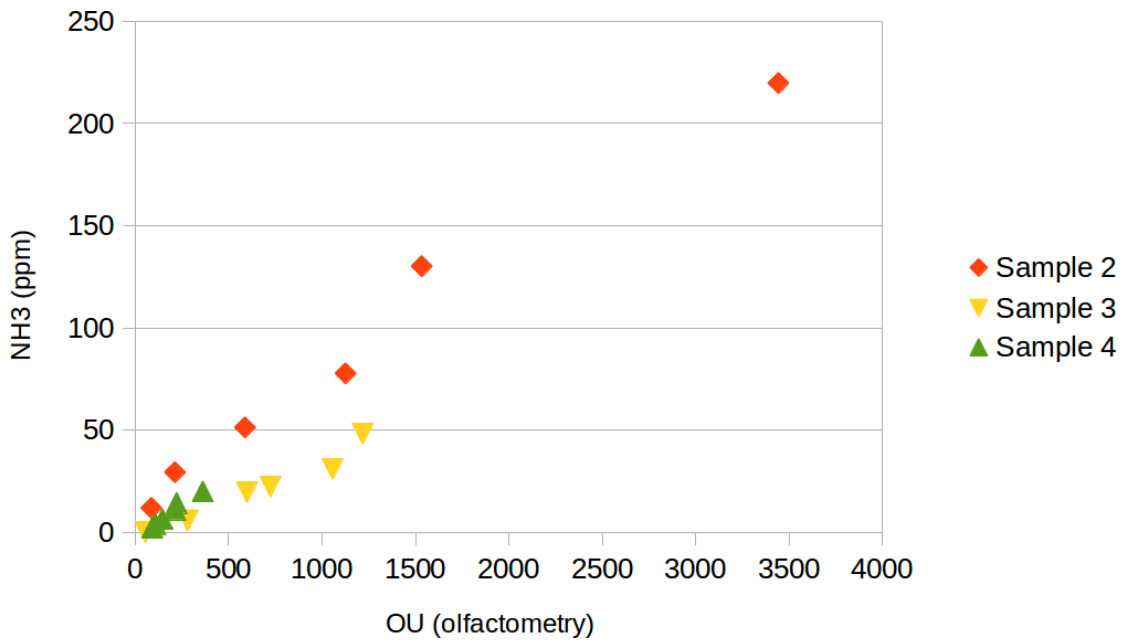
**Figure 12. Litter headspace ammonia concentration compared to odour concentration.**
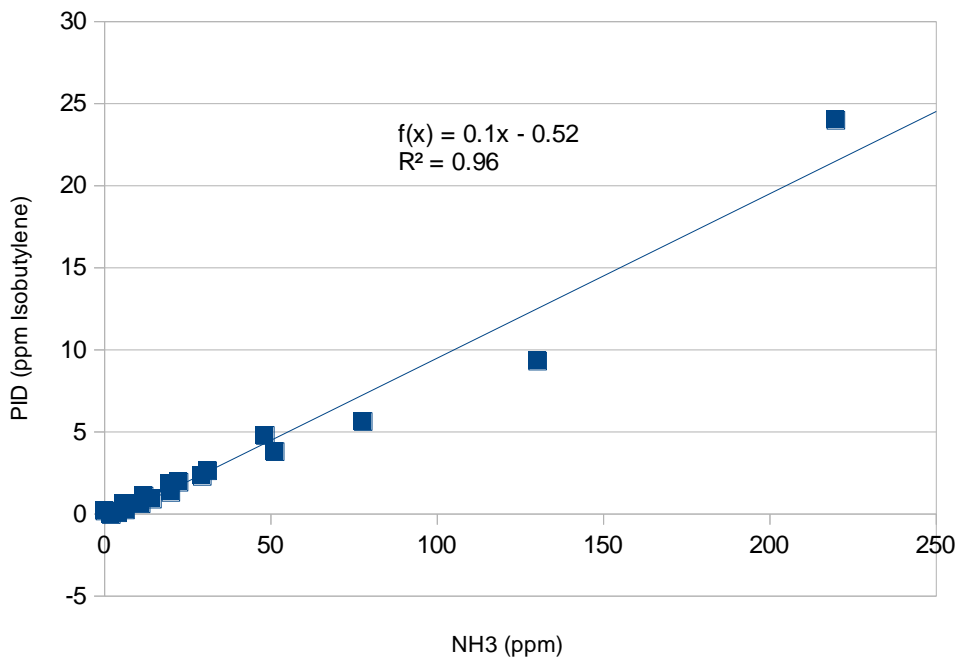


**Figure 13. Litter headspace PID response compared to ammonia concentration.**

The functional characteristics of the PID sensing technique should enable differentiation of the VOC and NH$_3$ components of its response when coupled with the measurements of the NH$_3$ sensor in the AOS.

The response of a PID sensor (calibrated to isobutylene reference) can be assumed to be additive such that the measured output of the sensor is a sum of all volatile compounds (organic and inorganic) detected by the sensor as shown in Equation 1:

$$PID_{response} = VC_1 \times CF_1 + VC_2 \times CF_2 + \ldots VC_n \times CF_n$$

**Equation 1**

where $VC_n$ is the concentration and $CF_n$ is the correction (response) factor for the PID sensor's response to volatile compound n. CFs are well defined and published by the manufacturer. For the 10.6eV sensor used in this study, ammonia has a CF of 8.5. Since ammonia is the only significant inorganic compound, the concentration of total VOCs (TVOCs) could be approximated by Equation 2 below:

$$[TVOCs] = PID_{response} - ([NH_3] \times CF_{NH3})$$

**Equation 2**

where [TVOCs] is the total concentration of VOCs as measured by a PID sensor (that is, only VOCs with appropriate ionisation energies and including individual response factors). Figure 14 shows the calculated TVOCs concentration using this approach. Due to the relative abundance of ammonia compared to TVOCs, ammonia dominates the PID sensor response and it is difficult (if not impossible) to quantify the TVOCs and, therefore odorants, since they are likely present at very low concentrations.

The calculation of negative TVOCs quantities can be attributed to the inherent measurement and calibration errors. The measured quantities are most likely due to sensor variability and calibration error. However, in some cases the ammonia concentrations are also well above what is expected in a real measurement scenario (due to the disturbed litter).
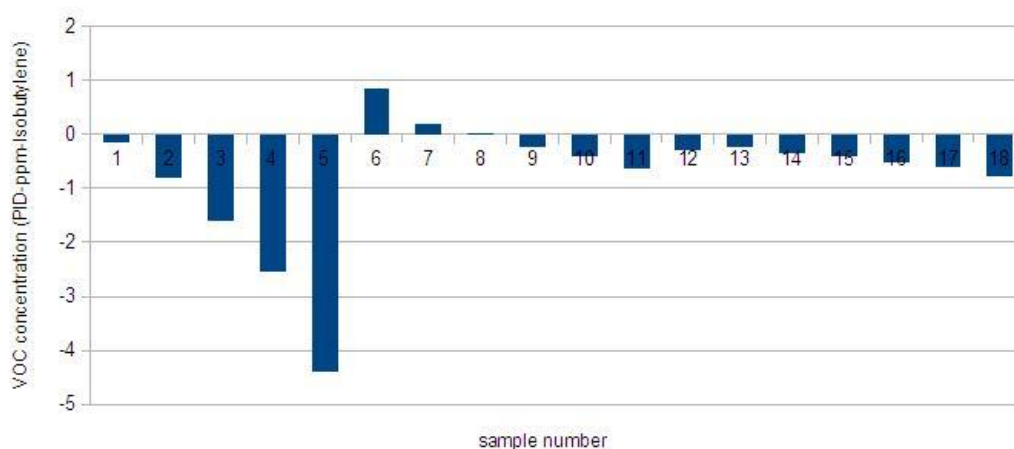


**Figure 14.  Measurable VOCs in 18 litter headspace samples calculated using a photo-ionisation detector and electrochemical ammonia sensor.**

## Key findings

- Ammonia ($NH_3$) masks the total VOC response of PID sensors and presumably, the MOS sensors.

- If $NH_3$ is highly variable and independent from odour it may negatively impact on the ability to model sensor response to odour concentrations.

- If an $NH_3$ sensor is relied upon to 'correct' for this interference in the odour modelling process, given the high magnitudes of this interference, even small variations in $NH_3$ sensor response characteristics (for example, drift or calibration error) may have large impacts on estimated odour concentration.

# 5. Field trial two (AOS MK2)

## Background

The purpose of this trial was to evaluate the revised AOS system and compile the database of odour measurements required to develop an odour prediction model. In a previous study (Dunlop *et al*., 2011, Sohn *et al*., 2008, 2010), odour prediction models were fitted to in-shed data from single sheds. The modelling was based on the training set only and could therefore be subject to 'overfitting' of the model and possibly poor prediction performance on new samples. The models were not tested against odour samples from downwind and other sheds on the farm. A retrospective attempt to apply the models from these earlier studies to another farm was unsuccessful.

For this trial, sampling was restricted to two farms supplying to the same processing company, with the aim of developing a generic model for both that could then be applied to a third farm. It was reasoned that if a robust odour prediction model could not be developed for two 'similar' farms (that is, same integrator) then it would be even more unlikely across three or more farms, regardless of the integrators involved.

## Study sites

Proximity to the olfactometry laboratory in Toowoomba was the main consideration in selecting farms for this trial, in order to minimise time between collection and analysis of odour samples. The two farms chosen (farms B and C) belong to the same integrator. Both sites had five tunnel ventilated sheds and stocking rates of approximately 19 birds/$m^2$. The batch grow-out period was usually 7-8 weeks.

Figure 15 shows the shed layout and approximate odour sampling points at both sites. At some sampling points, multiple samples were taken; for example, 'in-shed' samples were generally collected from in front of the same ventilation fan each time. The research team did not enter the sheds during the batch.



**Figure 15.   Aerial view of farms B (left) and C (right) used for Field Trial 2.**

**Approximate odour sampling points are shown as red dots. A total of 73 samples were collected from these farms.**

# Methodology

## Daily sampling procedure

Sampling sites for ambient odour samples were chosen at various distances downwind, depending on where odour was detectable on the day. Odour samples were concentrated around the sheds. Collecting samples too far downwind proved pointless because their odour concentrations were too weak to obtain a standard olfactometry result (either the majority of the panel could not detect it, or we ran out of sample). On most sampling days, at least one in-shed sample was collected, usually from the same shed, unless it was empty. In-shed samples were either collected via a probe placed into the shed or from directly in front of an active exhaust fan. Figure 16 shows representative odour sampling locations. The lung principle was used to collect samples into bags inside rigid 120 L drums as described in Sohn *et al*. (2008).



**Figure 16.  Odour sampling at fans (top) and downwind (bottom) at farms B (left) and C (right).**

Samples were analysed immediately by AOS and then by dynamic olfactometry within six hours of collection. AOS analysis was conducted from the sample bag to provide a stable downwind sample not affected by fluctuations due to wind and movement of odour plumes. Prior to each AOS sample analysis, the AOS was baselined using a clean, dry air reference gas (instrument grade air) for 20 minutes, followed by a 20-minute analysis of the sample.

**Figure 17. Calibrating mass flow controllers used for AOS sensor calibration.**

## AOS sensor calibration

To quantify and account for sensor drift (both baseline and sensitivity changes) the AOS was periodically calibrated throughout the project using standard gases. This also necessitated calibration of the MFCs used for AOS sensor calibration (Figure 17).

Two calibration gases were used: isobutylene (for the PID sensors) and ammonia (for the ammonia sensor). The MOS sensors require oxygen to function, therefore all calibration gases must contain an air balance. The MOS sensors were not corrected for changes in sensitivity if they occurred but were corrected for baseline drift using a baseline reference in clean (instrument grade) air prior to each sample analysis.

Ammonia calibration gas was originally derived using bottled gas standards (20 ppm with an air balance). This was eventually changed to a certified G-Cal ammonia permeation device (VICI Metronics Inc.) and temperature-controlled oven due to difficulties and lead times in sourcing ammonia standards with air balance. These were configured to provide an air-based calibration gas stream containing approximately 20 ppm ammonia.

All calibration and sample measurements where conducted using a 20-minute analysis time (to allow sensor responses to stabilise).

## Correction of relative humidity effects

Correcting for variation in sample RH was attempted in two ways: (a) by developing an RH calibration model to correct individual sensor output; and (b) by analysing a clean air reference gas at a humidity matching that of the sample. This in effect would allow comparison of sensor response in an odorous sample and a clean (odour-free) sample with identical temperatures and humidities.

The RH referencing system was used to generate an RH correction model for the MOS sensors. The gas stream from the referencing system was mixed at a controlled dilution with an artificial odour generated using permeation devices (which were also used to calibrate the SIFT-MS deployed during the third field trial). This was intended to provide an RH calibration dataset in a representative sample and therefore more likely to be successfully applied to actual measured samples (    Figure 18).
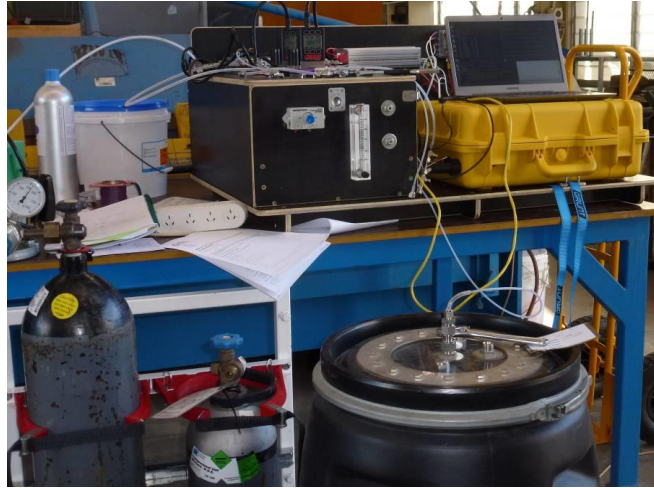


**Figure 18.  Performing RH calibration correction on AOS MK2.**

The AOS MK2 sensor data treatment described is illustrated in    Figure 19.
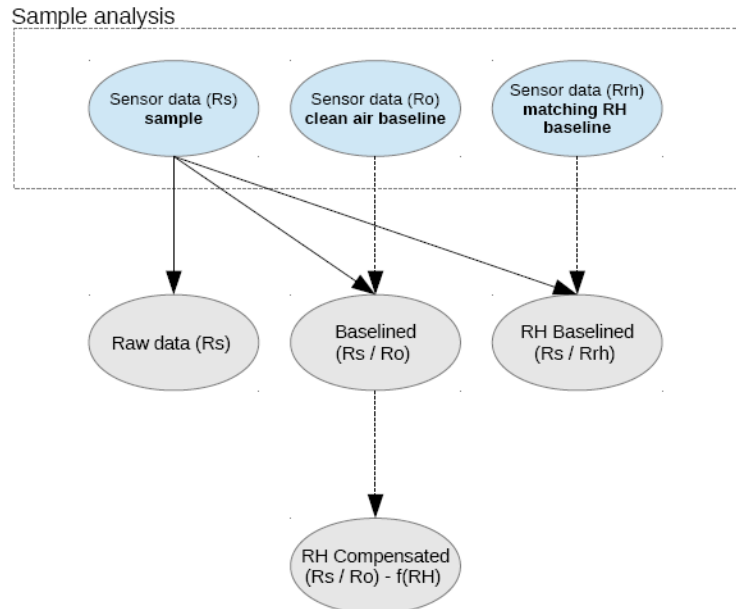


**Figure 19.  Flowchart of AOS MK2 sensor array data treatment.**

## Vehicle fit-out with AOS

It was necessary to mount the AOS MK2, associated power supply and sample delivery system on a custom-built platform (see Figure 18) designed to fit in a station wagon as shown in Figure 20. This was for ease of transport, as well as saving time in the field with re-assembly and minimising the chances of damage to the system and also ensured that no parts were forgotten. Whilst transporting the system it was left running on battery power (charged overnight) to ensure the system was warmed up and stable for field use, and for calibration upon return. When operating the AOS in the field (Figure 21) it was sometimes necessary to close the vehicle up as much as possible and run the car air-conditioning, to maintain optimal operating temperature during analysis.



**Figure 20.  AOS MK2 fitted in a vehicle.**



**Figure 21.  Analysing a bag sample on-site with AOS MK2.**

# Results

A total of 73 samples were collected and analysed. Sample details and corresponding odour concentration measurements are summarised in Appendix D. The concentrations are representative for the industry.

The AOS data analysis is reported in Chapter 7.

## Relative humidity effects on sensor response

The MOS responses were observed to be dependent on sample RH. The response of the TGS2600 sensor is shown in Figure 22. Compensation models of the form in Equation 3:

$$f(RH) = A \times (RH)^B$$

**Equation 3**

were fitted to the RH response curves for each MOS sensor. This was then used to correct a clean air baselined sensor measurement using the Equation 4:

$$MOS\ sensor\ signal = (R_s / R_o) - A \times (RH^B)$$

**Equation 4**

where $R_s$ is the sample response and $R_o$ is the baseline response.
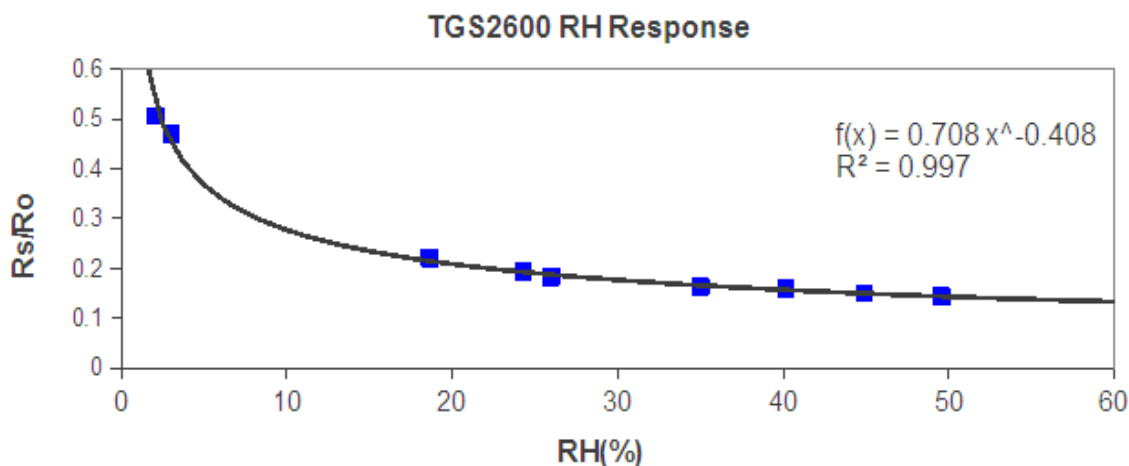
**Figure 22. Observed TGS2600 response change in variable sample RH.**

The response of the 10.6 eV PID sensor was observed to be relatively independent of humidity (Figure 23). This is supported by the manufacturer claims that the sensor is generally insensitive to changes in sample humidity and therefore no humidity compensation of this sensor is required.
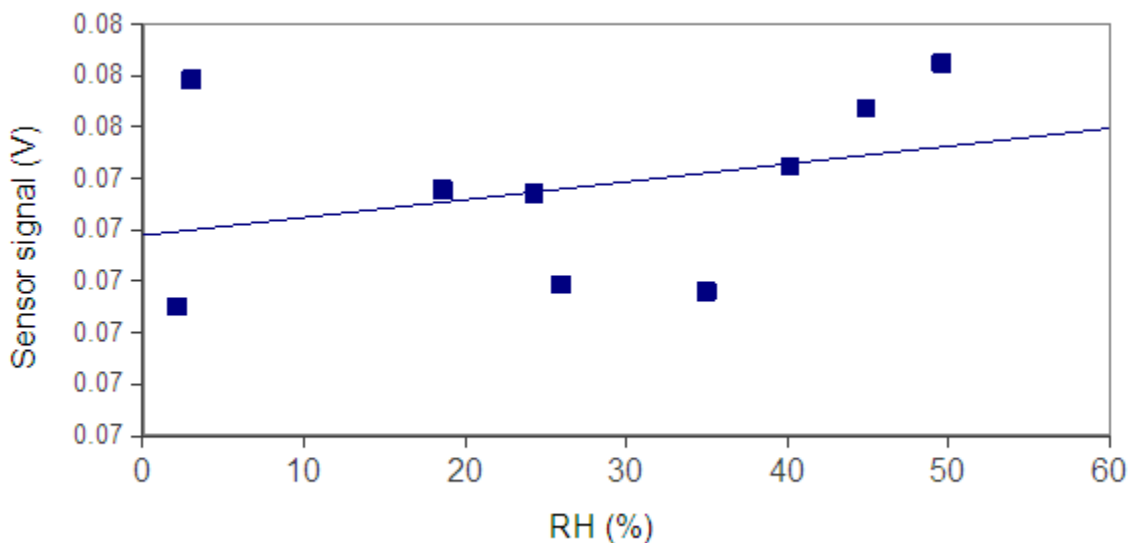


**Figure 23. Observed PID (10.6 eV) response change in variable sample RH.**

It was not possible to observe the effects of humidity on the sensitivity of this sensor due to the difficulties that arose in generating an $NH_3$ calibration standard at varying RH. Since the calibration standard used for the MOS and PID sensor analysis does not contain $NH_3$, it was possible to observe a shift in the $NH_3$ sensor baseline response due to a change in sample humidity. The magnitude of this change translates to an error in measurement of approximately 2.5 ppm $NH_3$ over the full range of tested humidities. It is difficult to

compensate for this empirically without full RH calibration data, however, the effects of RH on the $NH_3$ sensor baseline will be partially accounted for using the RH baselining technique (Figure 24).
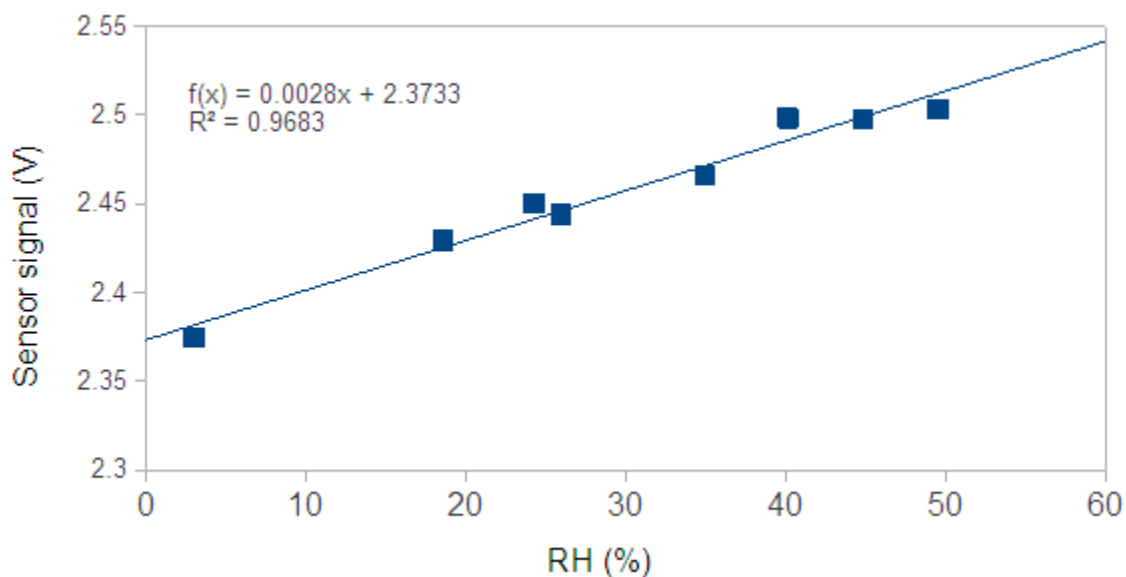


**Figure 24.   Observed electrochemical NH3 sensor baseline change in variable sample RH**

## Key findings

- MOS sensor responses were dependent on sample RH.

- PID 10.6 eV sensor response was relatively independent of humidity.

- We were unable to determine RH effect on the sensitivity of the ammonia sensor.

- RH effects on ammonia sensor baseline are partially accounted for using the RH baselining technique.

- AOS MK2 travelled well and the software and hardware were robust.

# 6. Field trial three (AOS MK2)

## Background

The purpose of this trial was to conduct a comprehensive evaluation of the AOS odour quantification abilities at an independent farm. This trial was used to validate the odour prediction model developed from the field trial two data (see Chapter 5). This trial was also used for gathering samples for GC-MS analysis to add to the database of NMVOCs identified on meat chicken farms and to help identify potential key odorants to target with appropriate sensors in the future. This trial was also timed to coincide with the one month trial of a selected ion flow tube mass spectrometer (SIFT-MS) which was deployed at the same farm.

## Study site

For this trial we returned to farm A, which was used for the AOS MK1 field trial (see Chapter 3).

## Methodology

### Odour sampling

The odour sampling method for AOS and dynamic olfactometry purposes is described in Chapter 5.

Sampling took place during September and October 2013.

### SIFT-MS sampling

A Voice200 SIFT-MS (Syft Technologies, Christchurch, NZ), mounted in a van, was used on-site at farm A. Air analysed by the SIFT-MS included ambient, in-shed, near-shed, downwind and background odour during grow-out and litter cleanout. Additionally, olfactometry samples, litter headspace samples were measured as well.

The SIFT-MS was run in two modes: (1) full scan, to measure all detectable chemicals; and (2) selected ion mode (SIM) scan, to measure pre-selected chemicals. Full scans were used to establish which chemicals should be targeted using the SIM methods. For this trial two SIM methods were set up, namely, one just for sulphides, and a more general one for known poultry odorants (ammonia, sulphides and NMVOCs including those used for odour prediction by Murphy *et al*. (2014)).

## Results

### AOS results

A total of 22 representative samples were collected in and around the sheds. The details are summarised in Appendix D.

These results are reported in 7. Data analysis and model development.

### GC-MS results and discussion

A summary of the GC-MS laboratory analyses is provided in Appendix B. The details of the GC-MC samples collected are given in Appendix C.

Some of the expected odorants identified by others in Australian meat chicken farms (Jiang & Sands, 2000; Pillai *et al*., 2010; Murphy *et al*., 2014) were present but overall there were fewer detectable odorants and their concentrations were much lower. Table 2 lists the chemicals detected and the maximum concentration recorded.

Unlike direct headspace sampling, sorbent tube samples are prone to degradation, contamination and producing artefacts, as well as lower recovery rates (Pillai *et al*., 2010). This could help explain why some of the GC-MS results were anomalous, even between duplicates. Also, the GC-MS sampling time of 30 minutes could be considered as not long enough for the concentration of odorants in the air at the time of sampling. Previously, a similar sampling method was found to be suitable for concentrated in-shed NMVOC measurements collected from the ventilation fans, as described in Dunlop *et al*. (2011). However, it is not suitable for the sulphides, which are key odorants and likely contributors to odour nuisance and, therefore, the GC-MS sampling methodology on poultry farms needs to be revised for any future odour research.

Compounds with the highest concentrations are not necessarily the most dominant in odours and it is important to consider the detection thresholds of the odorants. In the samples collected, 2,3-butanedione and acetoin were the two most abundant odorants. The concentration of 2,3-butanedione was approximately seven times higher than its detection threshold (Table 2).

**Table 2.** **Summary of TD-GC-MS results obtained from field sampling campaign.**

| *Compound* | *Chemical formula* | *Maximum concentration (ppb)* | *Odour detection threshold (ppm)* |
|---|---|---|---|
| Acetoin | C4H8O2 | 64 | Unknown |
| Acetic acid | CH3COOH | 64 | 13.0 |
| 2,3-Butanedione | C4H6O2 | 58 | 8.6 ppb |
| Trichlorofluoromethane | CCl3F | 13 | 45.0 |
| Acetone | C3H6O | 13 | 20.0 |
| Toluene | C7H8 | 5 | 1.6 |
| 2-methyl-butane | C5H12 | 3 | Unknown |
| Xylene | C8H10 | 2 | 1.1 |
| Tetramethylbenzene | C10H14 | 2 | 0.1 |
| 2-butanone | C4H8O | 2 | 5.4 |
| Camphene | C10H16 | 2 | Unknown |
| Cymene | C10H14 | 1 | Unknown |
| γ-Terpinene | C10H16 | 1 | Unknown |
| Benzene | C6H6 | <1 | 4.7 |
| Ethylbenzene | C8H10 | <1 | 2.3 |
| Trimethylbenzene | C9H12 | <1 | 2.4 |
| Nonanal | C9H18O | <1 | 0.53 ppb |

## SIFT-MS results and discussion

SIFT-MS measurements helped confirm the conclusion that the VOCs in meat chicken odour are at very low concentrations (parts per billion, ppb). Figure 25 shows the concentrations measured for 18 key odorants during a litter harvesting event at farm A. All odorants were at less than 70 ppb, acetic acid being the highest (32-69 ppb) and most odorant concentrations were less than 15 ppb.
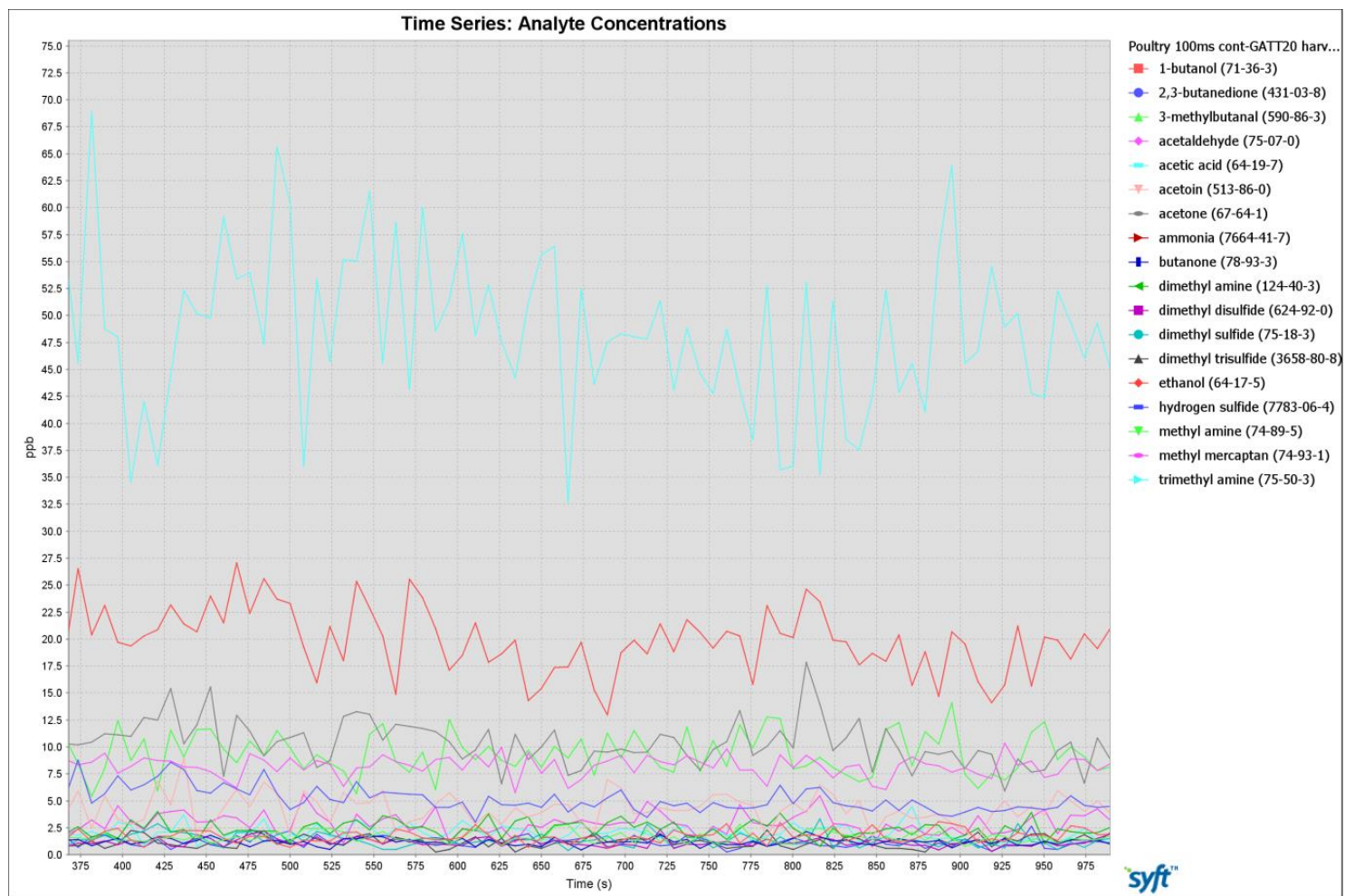
**Figure 25. Example of SIFT-MS output during monitoring of litter harvesting.**
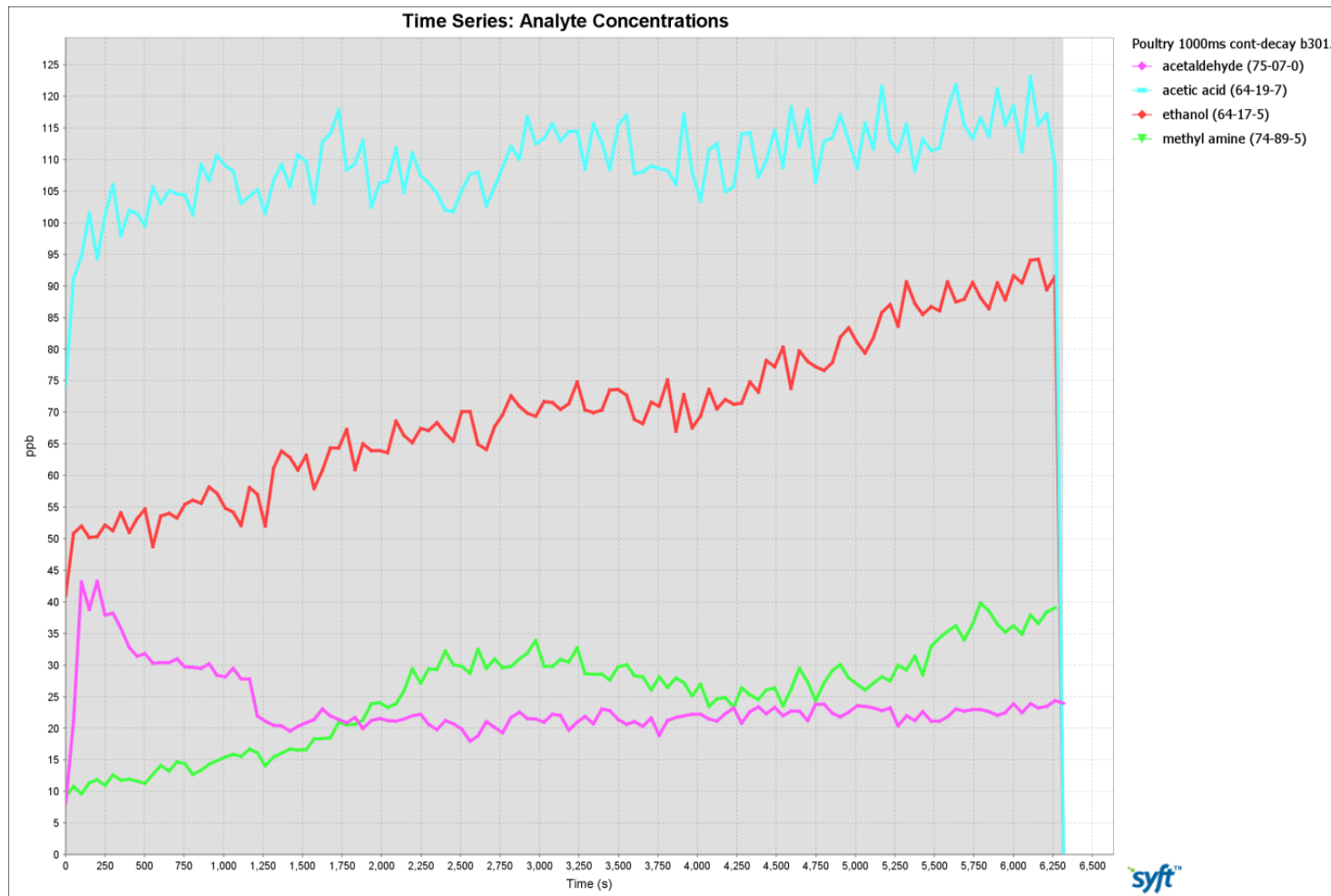
**Figure 26. Odorant changes over 1.5 hrs in a bagged sample typical of those collected for olfactometry.**

Figure 26 illustrates changes in concentration of four key odorants over a 90-minute period in a bagged odour sample, typical of those collected for dynamic olfactometry. The sample was an in-shed sample collected late in the batch. Note the increasing ammonia, acetic acid and methylamine and decreasing acetaldehyde. These changes are of concern because olfactometry is usually delayed several hours and such changes possibly influence the perceived odour strength depending on the detection thresholds for these volatiles.

This has implications for the accuracy of the AOS odour prediction models which are based on near-instant AOS analyses and olfactometry analyses performed approximately four to six hours later.

## Key findings

NMVOCs are at sub-ppm concentrations; these would be considered extremely difficult to measure using available sensors (such as the MOS and PID sensors that were used in the AOS). This is especially the case when the sample matrix is further complicated by fluctuating RH and $NH_3$ that fluctuate independently of odour.

SIFT-MS showed there are odorant composition changes in olfactometry bags over time. Such changes need to be known and understood as they may have significant ramifications for the accuracy and validity of odour prediction models which are based on calibrations with olfactometry data.

# 7. Data analysis and model development

## Background

### Sensor-based models

In an attempt to adapt electronic noses to environmental monitoring of complex, dynamic odours (such as those from treatment plants, landfill sites and particularly intensive livestock facilities), discriminating and quantifying the target nuisance odours has proved challenging.

A variety of traditional and newly-developed data analysis techniques have been used to model the relationships between sensor array data and nuisance odours. Such data is typically noisy and vague, lending itself to non-linear methods including fuzzy logic and artificial neural networks. For example, di Francesco *et al.* (2001) used a fuzzy logic-based pattern recognition system on waste water samples to obtain an 87% recognition rate on their test set.

Sohn *et al.* (2009) collected 39 odour samples (of which 25 were used) and deployed a commercial enose to determine the major contributing source to odour nuisance in an affected rural community. They used PCA for data compression and visual representation. An artificial neural network (similar to the more-traditional parametric discriminant analysis) proved capable of discriminating the odour sources. However, these 'black box' approaches are prone to over-fitting (resulting in poor predictive performance), and provide no real understanding of the odour composition and dynamics.

During a major study to determine dust and odour emissions from Australian meat chicken facilities (Dunlop *et al.*, 2011), Sohn *et al.* (2008) analysed 174 odour samples using a custom-built AOS containing a sensor array of 12 MOS sensors. The raw voltages were converted to sensor resistance values before being pre-processed and normalised. Principal components analysis (PCA) was used for data exploration, and partial least-squares regression (PLS) for analysis. Reasonably good PLS predictions were found using three latent variables.

### Odorant-based odour models

To investigate the relationship between the NMVOCs and perceived odour, Murphy *et al.* (2014) analysed the results of 49 GC-MS samples collected from five Queensland meat chicken sheds during autumn (April-May 2008). They then used multivariate calibration models to predict in-shed odour concentration during the peak period of the batch (aged 32-36d) using the concentrations of eight prevalent NMVOCs. Box-Cox power transformations were first used to approximately normalise the data. The recent PLS regression method of 'orthogonal projection to latent structures' (O-PLS) was adopted, with backwards-elimination selection and cross-validation. Good predictions were achieved with two latent variables, as they warn that more variables may lead to over-fitting.

In a Danish study of piggery odour composition in grower-finisher facilities, Hansen *et al.* (2012) used the odorant concentrations measured by proton transfer reaction mass spectrometry (PTR-MS) to demonstrate the feasibility of predicting odour concentration this way. Odour was log-transformed to stabilise the degree of variation. Again PCA was used for data exploration and presentation. Standard PLS regressions only produced a reasonable degree of fit for this system, with $R^2$ of 53%.

# Methods

## Data processing

The sensor array data was processed and screened before final data analysis and development of an odour prediction algorithm could be done. Sensor response values were calculated by averaging the last five seconds of the time-series data (a 20-minute measurement). Sensor calibration and data treatment was applied as follows.

## MOS sensors

Measured MOS sensor signals were firstly converted to sensor resistance ($R_s$).

MOS sensor measurements were calculated using Equation 5:

$$MOS = R_s \: / \: R_o$$

**Equation 5**

where $R_o$ is measured at the same temperature and RH as the sample, using the relative humidity baselining technique.

## PID sensor (10.6 eV)

Sensor signals were converted to concentration (ppm isobutylene equivalent), which was calculated using the calibration data, and baseline-referenced in clean dry instrumental-grade air prior to sample analysis. No humidity compensation was applied to this sensor.

## NH$_3$ sensor

NH$_3$ sensor signals were converted to ammonia concentration (ppm). This was calculated using the ammonia calibration data and the sensor is baseline-referenced in clean dry air prior to sample analysis. Humidity compensation was also not applied to this sensor.

Measurements from the 9.6 eV PID were excluded from the analysis as the sensor did not appear to be working. While this sensor is insensitive to ammonia, the sensitivity of the sensor to VOCs is also approximately 1/10th that of the 10.6 eV PID. Based on VOC concentrations measured by the SIFT-MS and GC-MS, it is highly unlikely that any meaningful response from the 9.6 eV PID would have been measured. Sensor changes due to relative humidity and other effects would have dominated and given the appearance of a faulty response.

Some outliers were identified and corrected; others (including some notably high values) were retained as 'believable' (given the researcher's notes), representing extreme conditions. In the end, there were 64 samples with all the necessary data. The skewed variables, namely OU, NH$_3$ and the PID readings, were log-transformed prior to the analyses (hence the statistical leverage of the biological extremes was lessened). Responses from the TGS MOS sensors were approximately 'normal', so were not transformed. Readings for tgs2600 and tgs2620 correlated 'almost perfectly' (r = 0.994). Multiple general linear models using both were trialled, with the tgs2600 then being used for the other statistical analyses due to a slightly superior result for validation.

For multivariate comparisons, the principal components analysis was conducted on the correlation matrix. So whilst the data set does need to be approximately normal (log-transformations were used to achieve this), there was no need to standardise these variables to unit variance, as has also been done by some previous researchers.

# Results

Of the three available temperature variables, MOS temperature reading was considered the 'most applicable' (to the sensor readings) for the analyses. It correlated reasonably well ($R^2 = 0.88$ and $0.81$) with the sample and chamber temperatures respectively, as shown in Figure 27.
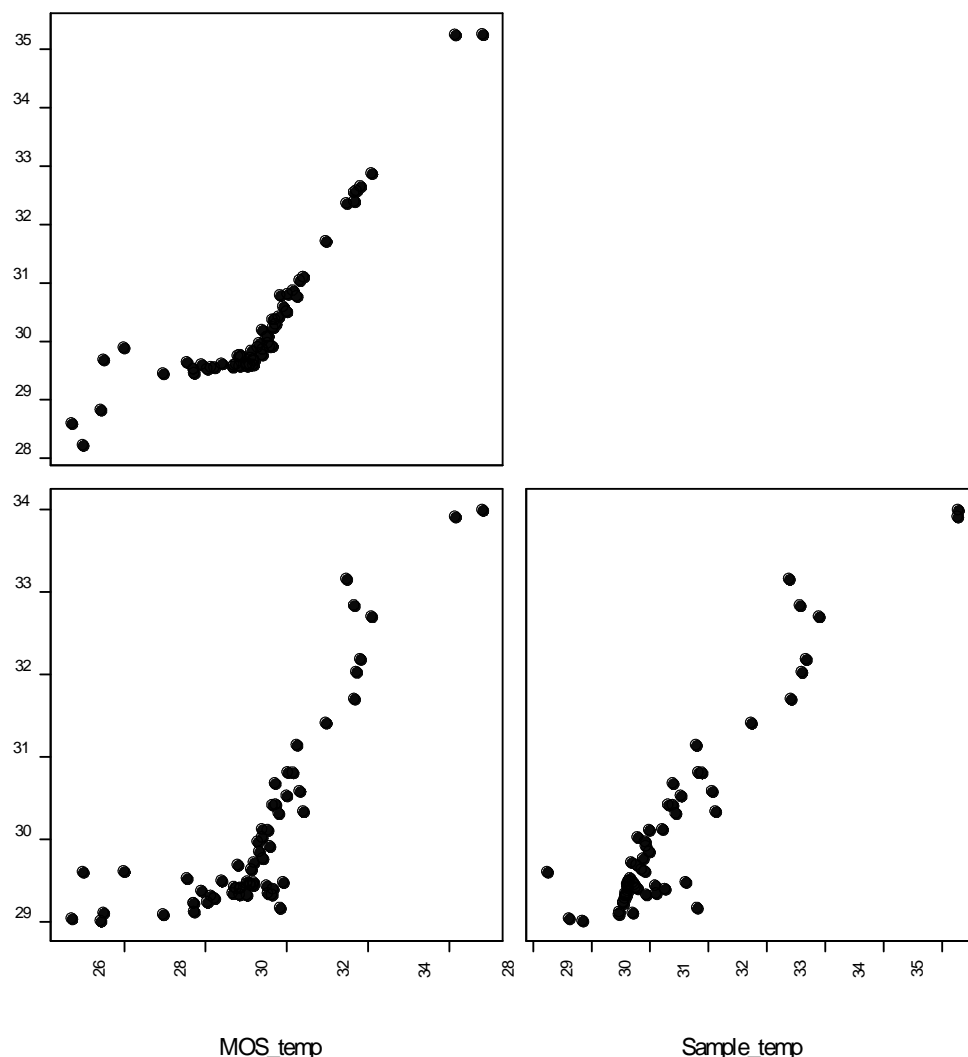


**Figure 27. Temperature monitoring measurements within AOS MK2.**

Odour units (OU) was adopted as the key dependent variable, and this was log-transformed to statistically correct for positive skewness and heterogeneous variance. This log was on base-10, so each unit on this scale represents a ten-fold increase in OU. $NH_3$ and pid10.6 were similarly $\log_{10}$-transformed, with the additional benefit of approximately linearising their relationships with log (OU). A double-log was required to achieve this for pid10.6, after adding a constant of 1.71 to avoid negative numbers prior to the log transformation (this constant was determined, by nonlinear regression, to provide the best overall fit).

As shown in Table 3 and Figure 28, there was a considerable degree of correlation amongst the different sensors. Most of the sensors also correlated reasonably well with log (OU), with r-values of up to 0.75. Log (OU) was negatively correlated with the tgs variables, and positively with pid10.6 and log ($NH_3$). There was no significant degree of curvature ($P > 0.05$) between log (OU) and any of the

sensor variables; the apparent 'downward trend to the left' in the pid10.6 figure is countered by the considerable scatter above. The linear nature of these relationships certainly assists with some of the more complex statistical methods.

**Table 3.    Correlation matrix (r-values) - T for TGS (MOS sensors); P for PID.**

| | OU | logOU | logNH₃ | l-l-P106 | T2600 | T2602 | T2610 | T2611 | MOS_t |
|---|---|---|---|---|---|---|---|---|---|
| **logOU** | 0.874 | 1 | | | | | | | |
| **logNH₃** | 0.722 | 0.685 | 1 | | | | | | |
| **loglogP106** | 0.743 | 0.690 | 0.863 | 1 | | | | | |
| **T2600** | -0.469 | -0.515 | -0.805 | -0.708 | 1 | | | | |
| **T2602** | -0.644 | -0.745 | -0.789 | -0.773 | 0.858 | 1 | | | |
| **T2610** | -0.704 | -0.727 | -0.795 | -0.884 | 0.763 | 0.839 | 1 | | |
| **T2611** | -0.290 | -0.345 | -0.241 | -0.270 | 0.292 | 0.366 | 0.607 | 1 | |
| **MOS_temp** | -0.048 | 0.091 | -0.190 | -0.135 | 0.112 | -0.090 | 0.063 | -0.214 | 1 |
| **rh** | 0.479 | 0.471 | 0.744 | 0.579 | -0.724 | -0.574 | -0.594 | -0.131 | -0.326 |
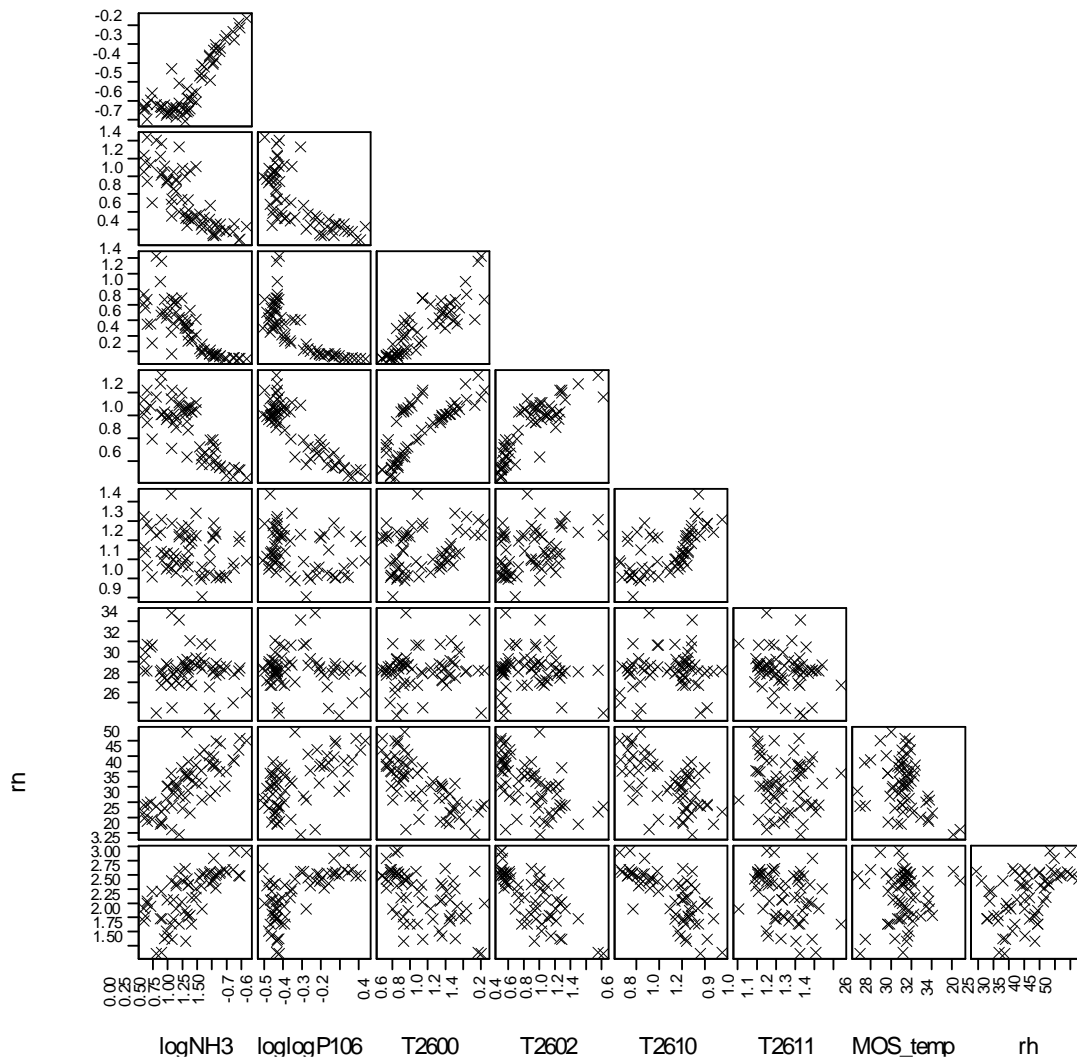
**Figure 28.  Correlation matrix scatterplots cross-sensor correlations and relationship with odour concentration.**

For log (OU), the best linear predictor ($R^2 = 55.5\%$) was tgs2602, as shown in Figure 29. Here, the sites are shown separately (along with their respective lines). These were not significantly different ($P > 0.05$), and the data points show a continuum with good degree of overlap. Similarly, the locations (in-shed, beside-shed and downwind) also displayed overlapping patterns.

40
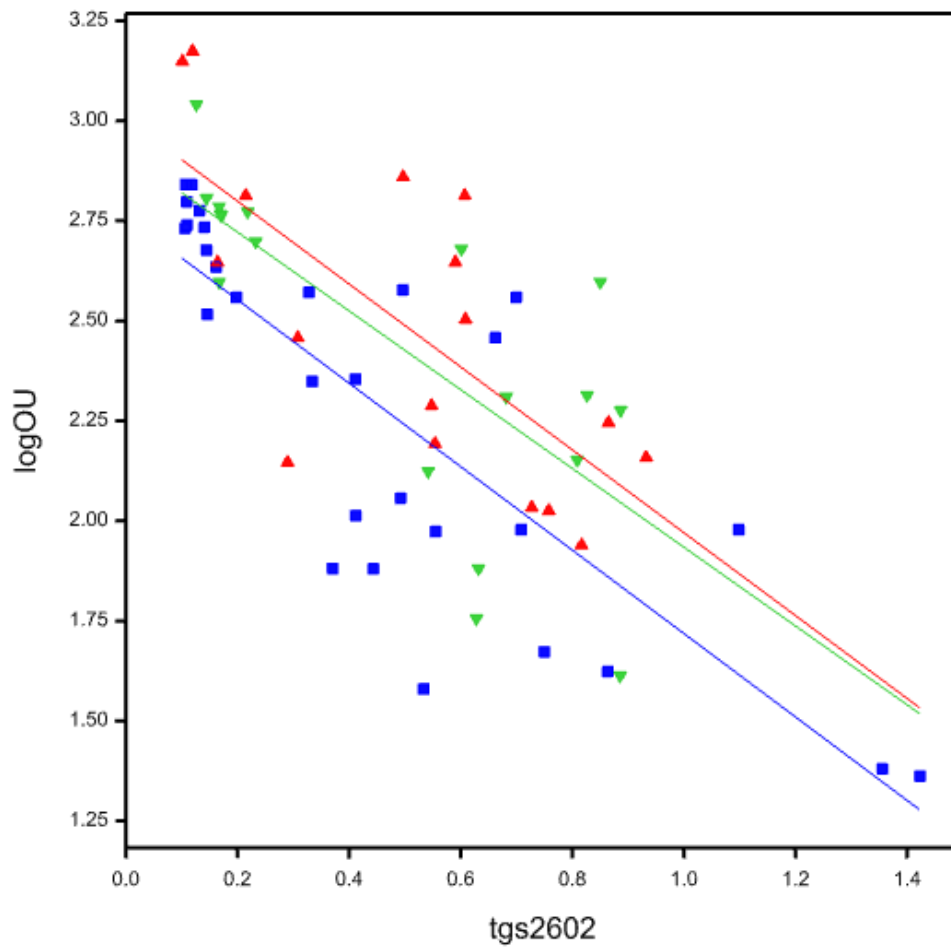
**Figure 29. Linear relationship between odour units and the tgs2602 sensor (green, red and blue = farms A, B and C, respectively).**

The principal components overall summarisation of the multivariate data is shown in Figure 30, with each point labelled by site and location. Again, there appears to be a good degree of overlap for the eight combinations of these (there were no data points for 'beside' at the farm A). Figure 31 shows the corresponding discriminant analysis results. Note that whilst this method forces the maximum possible separation between the groups, there is still a good degree of overlap between these groups.
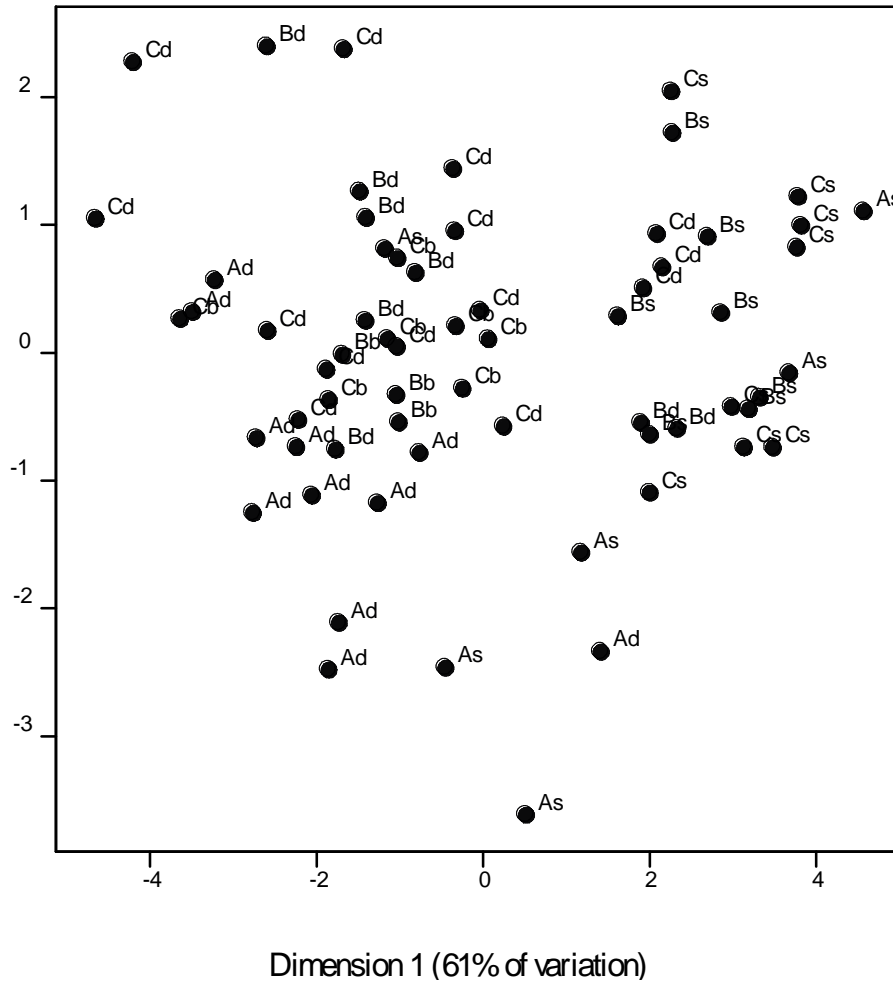
**Figure 30.** **Plot for two-dimensional principal components in a PCA analysis on the odour and sensor data, by sampling locations across three broiler farms (A, B and C).**

**s = in-shed, b = beside shed, d = downwind**
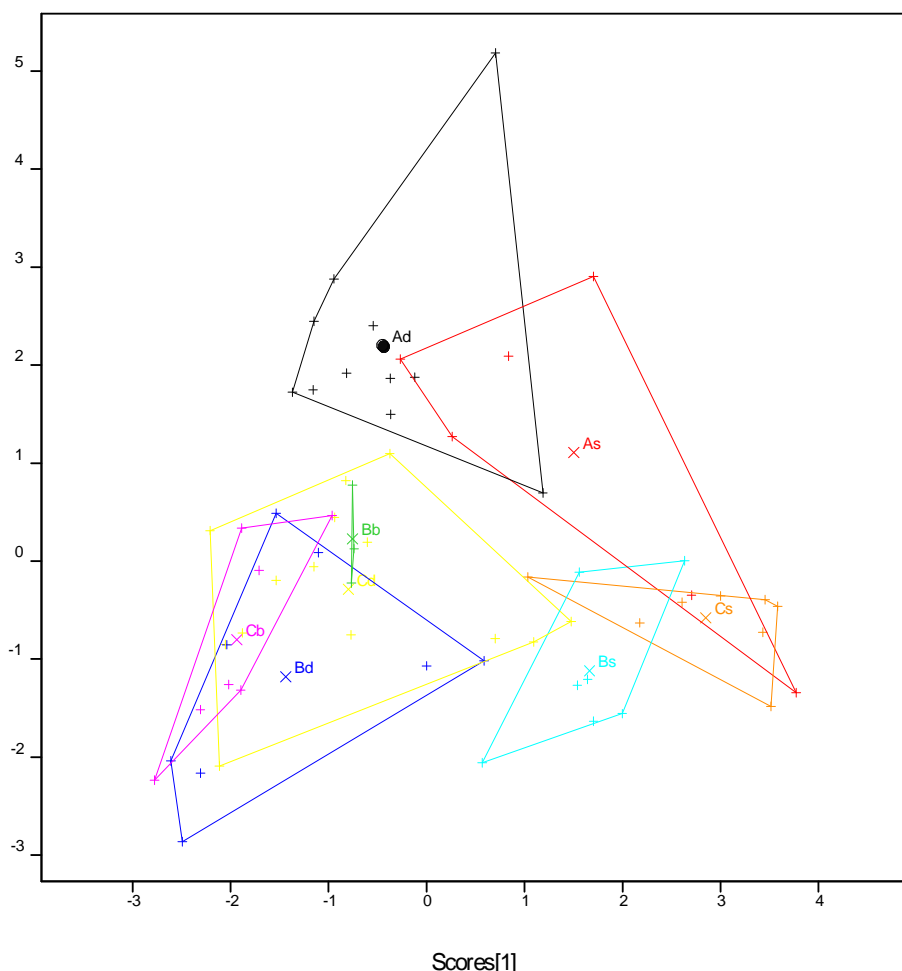
**Figure 31.  Linear discriminant analysis. X = group centroids with labels (same combinations as in Figure 30).**

**+ = data points with the encompassing polygons showing the ranges.**

Importantly, on both graphs there appears no overall clustering, hence, the data set has good coverage of the desired ranges and combinations of all of the variables, and there are no 'discrete, different groupings'. If one (or more) group of data points was spatially separated from the rest it would mean that 'different combinations' of the variables were occurring, for which different prediction models might be required. However, these results indicate that a single model should be robust across the sites and locations.

## Predicting odour concentration

As outlined earlier, there is quite a range of statistical methods that have been used in this and other disciplines. As outlined below, we have investigated and tested most of these available methods, with the exception of the recently developed orthogonal projection to latent structures (O-PLS), as the necessary software is not readily available. Trygg and Wold (2002) show that O-PLS is an extension and modification of 'traditional' partial least squares (PLS). Whilst O-PLS may have some interpretational advantages, predictions from an O-PLS model are identical to those obtained using PLS.

There are noted statistical problems dealing with multivariate data sets with high degrees of collinearity amongst the 'independent' variables. Whilst latent methods such as PLS are usually adopted here, general linear models can still be used. Notably, general linear models (GLM) will

function reliably (regarding predictive performance) 'as long as the collinearity between variables remains constant' (Dormann *et al.*, 2013). Given the overlapping distributions between farms and locations evident in the multivariate representations, this property would appear to hold for our data set.

In their key review, Dormann *et al.* (2013) recommend comparing and contrasting the available modelling methods. Interestingly, their extensive simulation study demonstrated that 'methods specifically designed for collinearity, such as latent variable methods did not outperform the traditional GLM'. Their results highlighted the value of using GLMs in combination with penalised methods, particularly ridge regressions. Similarly, Vajargah *et al.* (2012) 'recommend PLS/OPLS methods as complimentary rather than alternative to the available classical regression models such as linear regression'.

## General linear models (GLM)

The best-fitting multiple linear model was identified using all-subsets regression with RSEARCH in GenStat (VSN 2013), with an adjusted $R^2$ of 68.8%. This model is listed in Table 4. The only other contender four-parameter model replaced tgs2610 with relative humidity (which is not desirable), and had a lower adjusted $R^2$ of 67.6%. There were no significant improvements for five-term and higher-order models ($P > 0.05$). No interactive terms were investigated, as these models are often prone to over-fitting, and each would need to be based on a sound understanding and explanation of the mechanics involved. With these sensors, we simply do not have this understanding of how the sensor readings might interact (with regard to their responses to differing odour concentrations).

**Table 4.  Best general linear model for $\log_{10}(OU)$.**

| Parameter | Coefficient | Standard error |
|---|---|---|
| **Constant** | 2.407 | 0.273 |
| **tgs2600** | 0.946 | 0.201 |
| **tgs2602** | -1.090 | 0.223 |
| **tgs2610** | -0.490 | 0.256 |
| **logNH₃** | 0.390 | 0.132 |

It is interesting that whilst pid10.6 was one of the stronger linear predictors (r = 0.69), it did not appear in any of the best multiple models. This indicates that whatever 'OU signal' that pid10.6 was measuring is being adequately (and preferably) explained by the other sensors selected in this model.

Considering the in-shed data separately (despite the considerable overlap in the multivariate representations) it was suggested that the in-shed data may behave 'differently', in that $NH_3$ might be the only driving variable for OU. In GLM analyses taking only the in-shed data, $NH_3$ was in fact the strongest single predictor ($R^2 = 43.0\%$), however multiple models (with and without $NH_3$) then paralleled the 'all-data' analyses. For example, the best three-parameter model ($R^2 = 63.1\%$) had tgs2600, tgs2611 and tgs2620. In conclusion, there was no evidence that the 'OU dynamics' were any different between in or out of the sheds, so the overall regression models remain valid for all locations.

## Ridge (penalised) regression

Based on principal components vectors (or latent variables), ridge regressions adjust the fitted coefficients towards their best expectations, had the data set been uncorrelated. It effectively does this by sacrificing some of the degree of fit to achieve more stability in the estimated parameters, and therefore in any future predictions from the model. Backwards elimination of the non-significant variables arrived at the same four variables as used in the GLM. For this data set, the adjustment (ridge k-value) of 0.20 appeared most appropriate, approximately minimising ISRM (the index of stability of

relative magnitudes) whilst maintaining NLMS (the numerical largeness of more significant regression coefficients). This model gave $R^2 = 64.8\%$.

## Partial least squares (PLS)

As is common in PLS, all variables were first standardised to unit variance (GenStat, VSN 2013). For the maximal model (of all eight latent variables, using eight PLS dimensions), the first dimension explained 55% of the variation in log (OU), with the second and third dimensions each explaining a further 6%. The total $R^2$ was 72.4%, however this maximal level is probably over-fitted. When the model was reduced to only the four variables found by the GLM and ridge regressions, and hence four latent variables, $R^2$ remained quite similar at 70.8%.

## Classification and regression trees (CART)

This method sequentially splits the dependent data using binary break-points amongst the independent variables, with each split maximising the amount of variation explained. There is no formal statistical test of significance here, and these methods can be prone to over-fitting. Taking five nodes (thus using about the same number of coefficients as the previous regression models), the adopted regression tree gave an $R^2$ value of 60.6%, being notably lower than for the other regression methods. As the relationship between the sensors and OU is expected to be continuous (rather than falling into discrete groups either sides of cut-off values), as well as having lower $R^2$, this method was not taken forward to the validation study.

## Boosted regression

The relatively new boosted regression techniques (Hastie *et al*., 2009), or 'random forests', come from the data-mining and machine-learning sciences. They develop self-tuning ensembles of regression tree models, and these have shown improved predictive behaviour in a number of areas. We used the gbm procedure (Ridgeway, 2013) of the R statistical language (R Core Team 2013), forming the models on the 'training dataset' (farms B and C) and then testing these predictions against the validation dataset (farm A).

A range of operational parameters for the boosted regression were trialled, with quite similar results. The best models utilised 40,000 trees, a training fraction of 0.9, and five for the interaction depth (the number of nodes or variables for each tree). As there are many models (trees), an $R^2$ value for the overall degree of fit (to the training dataset) cannot be obtained. However, $R^2$ for the predicted against the observed values for the independent (validation) dataset forms the crucial value for 'how well' this technique performs.

## GLM ensembles

This recent statistical development (Song *et al*., 2013) effectively extends the successful 'multiple models' concept from boosted regressions into the GLM framework. In climate forecasting, the average of multiple models has repeatedly been shown to outperform any of the individual models. These ensemble predictors 'are known to lead to highly accurate predictions' (Song *et al*., 2013).

We are yet to investigate and incorporate the more complex operational parameters of these techniques available in the R language (R Core Team 2013), however a baseline implementation of GLM ensembles was investigated using GenStat (VSN 2013). We used all-subsets regressions (again, on the training dataset only) to find the best eight models, taking three, four, or five predictors at a time, as well as an extra set which included some of the two-way interactions between these.

As expected, the more complex models (five predictors and the interactions ensemble) showed evidence of over-fitting, giving a higher degree of fit for the base-data but lower for the validation set. Despite simpler models often performing best in boosted regressions, the ensemble of GLMs that only used three predictors was also inferior (on both the original fit and the validation).

The ensemble of models with four predictors was individually the best, however the optimal overall validation came from taking the average predictions from the ensemble of the four best 3-predictor models with all eight 4-predictor models. Obviously this warrants further investigation, but for now this approach appears the most promising for any future use.

## Validation

All statistical models were fitted using only the Field Trial 2 data from farms B and C, and their predictions compared with the actual OU measures from farm A. Table 5 lists the $R^2$ values for the data combinations ('all farms', 'B and C only' and 'A'), along with key measures for validation: the mean average error (on this $\log_{10}(OU)$ scale); and the bias-test (Mayer and Butler, 1993) of whether the fitted slope differs from the expected value of one.

**Table 5.    Regression model results and validation statistics.**

| Model | No. X's | $R^2$ (all farms) | $R^2$ (farms B & C data only) | $R^2$ (prediction. vs. farm A data) | Mean average error | Bias test (P-level) |
|---|---|---|---|---|---|---|
| GLM | 4 | 68.8 | 67.4 | 58.3 | 0.242 | 0.59 |
| Ridge | 4 | 64.8 | 67.4 | 45.1 | 0.323 | 0.44 |
| PLS | 8 | 72.4 | 72.8 | 58.2 | 0.275 | 0.09 |
| PLS | 4 | 70.8 | 70.2 | 58.3 | 0.242 | 0.59 |
| BR[a] | - | - | - | 58.7 | 0.247 | 0.58 |
| Ens.[b] | - | - | - | 71.3 | 0.210 | 0.86 |

a Boosted regression; b Ensemble of 12 GLMs.

The first four rows of Table 5 show the 'traditional' (single model) regression methods. Amongst these, PLS with eight variables had the highest degree of fit to the training and all data, but showed over-fitting as this advantage was not maintained for the independent validation dataset. Ridge regression was notably disappointing, as theory indicates it should produce good validation fits. Perhaps the small data set (17 observations) was insufficient to fully test this. Also, it was previously demonstrated that the farm A data had a considerable degree of data overlap with the other two sites, with presumably similar correlations amongst the variables, so there may not have been the 'separation' to allow ridge regression to sufficiently adjust the coefficients.

For the multiple model methods, boosted regressions were clearly inferior, which was to be expected; these being based on CART techniques, which we had earlier abandoned because of its unsuitability for these types of data. The GLM ensemble, whilst only 'basic' at this stage, proved to be surprisingly good at forming predicted values for the validation dataset, reducing the unexplained variation from 41.7% (for the GLM) to 28.7%, and reducing the mean absolute error by 13%. As expected, the variability and biases present in any individual GLM (from the individual combinations of the variables for each data-point) have been somewhat evened out in this 'multiple models' approach. This is clearly the preferred regression method here.

Figure 32 shows the degree of fit for the GLM ensemble. The 95% confidence intervals, for the 'true value' of $\log_{10}(OU)$, indicate this model is quite reasonable at predicting 'bands' rather than actual likely values. Using the direct back-transformation from these $\log_{10}$ values (that is, $10^x$), a low predicted value of 100 ou has a 95% confidence interval of 67 to 158 – being asymmetrical (as expected), with a span of 91 ou, but still clearly in the 'quite-low' range. At mid-levels (approximately 400 ou), this interval is 300 to 516 ou, a still reasonable range of 216 ou. At higher levels (1000 ou), the 95% confidence interval is 596 to 1518 ou (922 ou wide), so whilst we would be quite confident that the true value is still 'high-range', there appears little accuracy here.

Predicted log(OU)
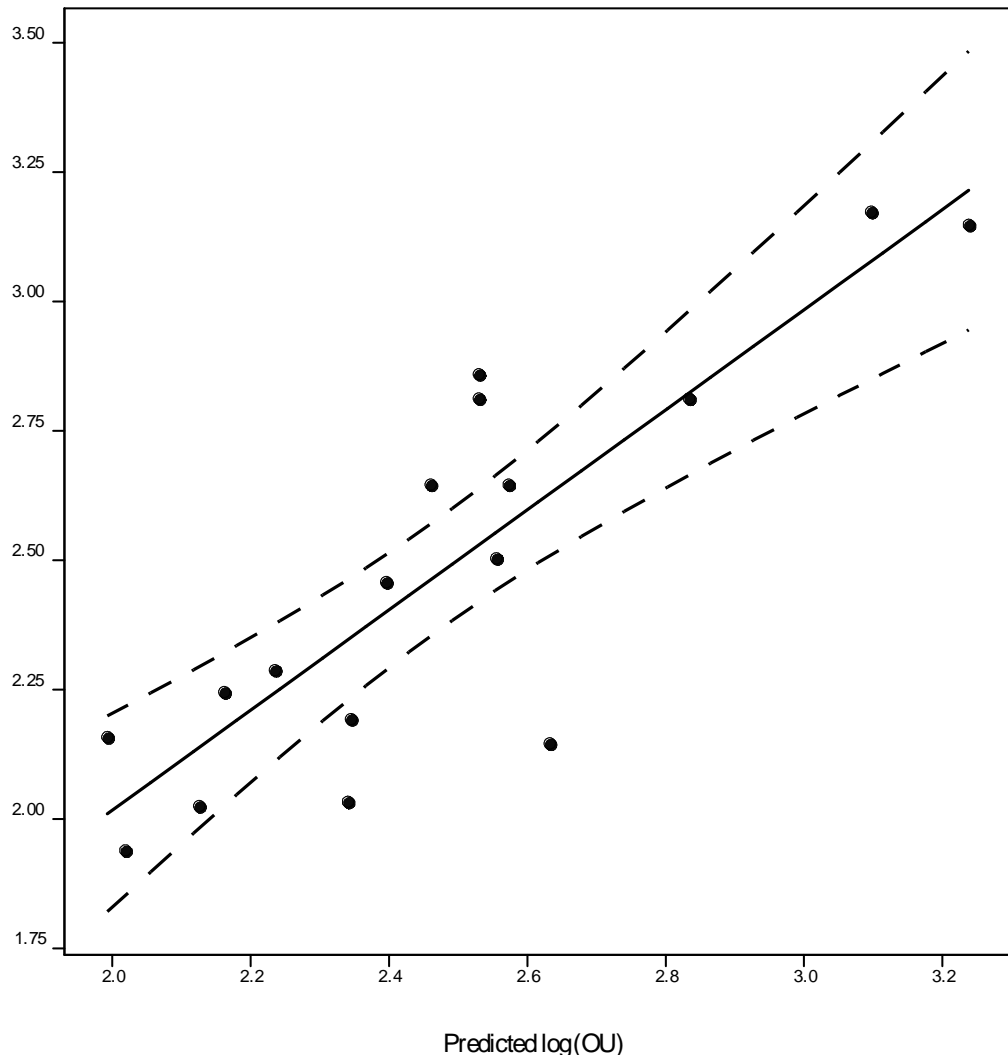
**Figure 32.  Validation data (farm A) vs. predicted values from the GLM ensemble using data from farms B and C.**

# 8. Development of a prototype field olfactometer (FO)

## Background

The aim was to develop a single port (single panellist) dynamic olfactometer that is field-portable and a more robust and refined approach to the problem than the field olfactometers available on the market. It is acknowledged that analyses would not be considered "to Standard", and that its application would be restricted to a limited range of odour unit measurements.

### Justification

Despite its limitations for odour assessment, AOS technology is relatively inexpensive, easier to operate, and has greater portability and adaptability for field applications, compared to analytical instruments. It is being successfully used for many other applications (Wilson, 2013). With improvements in sensor sensitivity and specificity, using AOS for routine odour measurement is becoming increasingly viable for more industries.

Regardless of the application, being able to recalibrate these instruments conveniently is important. Currently, that is not the case; we rely on dynamic olfactometry for standard odour measurements to calibrate an AOS.

A serious drawback with using gas sensor arrays is that the sensors will need to be replaced from time to time and there is no guarantee the replacement will behave identically, or even be available. Changing any sensor will require recalibration/retraining of the AOS. Currently, that implies more dynamic olfactometry, more corresponding AOS samples, and more modelling. From past experience, the development of a robust predictive odour concentration model requires in excess of 50 olfactometry samples. In practice, it is simply not viable to do so.

If a simple field olfactometer could be shown to measure odour from a bagged sample at the same concentration as a standard olfactometer, then a low-cost alternative would be available for AOS purposes. This could be used to develop predictive models quickly and revise them more often.

A FO would be particularly useful when determining the ideal configuration of the sensor array, as any previous olfactometry data becomes redundant once a sensor is replaced with another one. Rather than waste funds on standard laboratory-based dynamic olfactometry, the FO potentially provides a far more efficient and cost-effective way to build a database.

## Hardware

The prototype field olfactometer that was developed is an automated system with the following features:

- mass flow controller for repeatable odour dilution and staggered presentation

- concentration measurement range of 32 to 2048 ou.

The initial prototype was functional but was revised after laboratory testing showed panellists could not achieve the same sensitivity as they could with standard olfactometry (using an *n*-butanol reference). This was attributed to minor background odour in the clean air supply and air-odour mixing problems.

## Operating principle

### Forced choice dynamic olfactometry

The principle of operation is analogous to a two-port, forced choice dynamic olfactometry (see Australian/New Zealand Standard for Dynamic Olfactometry (AS4323.3) (Standards Australia/Standards New Zealand 2001)). The operation is controlled by purpose-built software run on a laptop. The user interface is shown in Figure 33.

To prevent desensitisation of panellists operating in the field, the system was designed to provide a continuous flow of clean air to the mask (using a modified *continuous positive airway pressure* (CPAP) pump with carbon filtered air) at a rate of 20 L/min. Olfactometry is performed by either injecting or not injecting odour into this clean air stream (with mixing occurring in a chamber prior to the mask) to simulate two olfactometry sniffing ports (virtual ports).

Odour is diluted at a nominated starting dilution and randomly presented via one of the two virtual ports, for each dilution used. The panellist has to indicate (via the user interface) in which 'port' the stimulus (odour) is presented and how sure they are: inkling or certain. Otherwise, they must indicate that they cannot detect it.

The system could be operated from 12V for field operation (from a battery or vehicle).
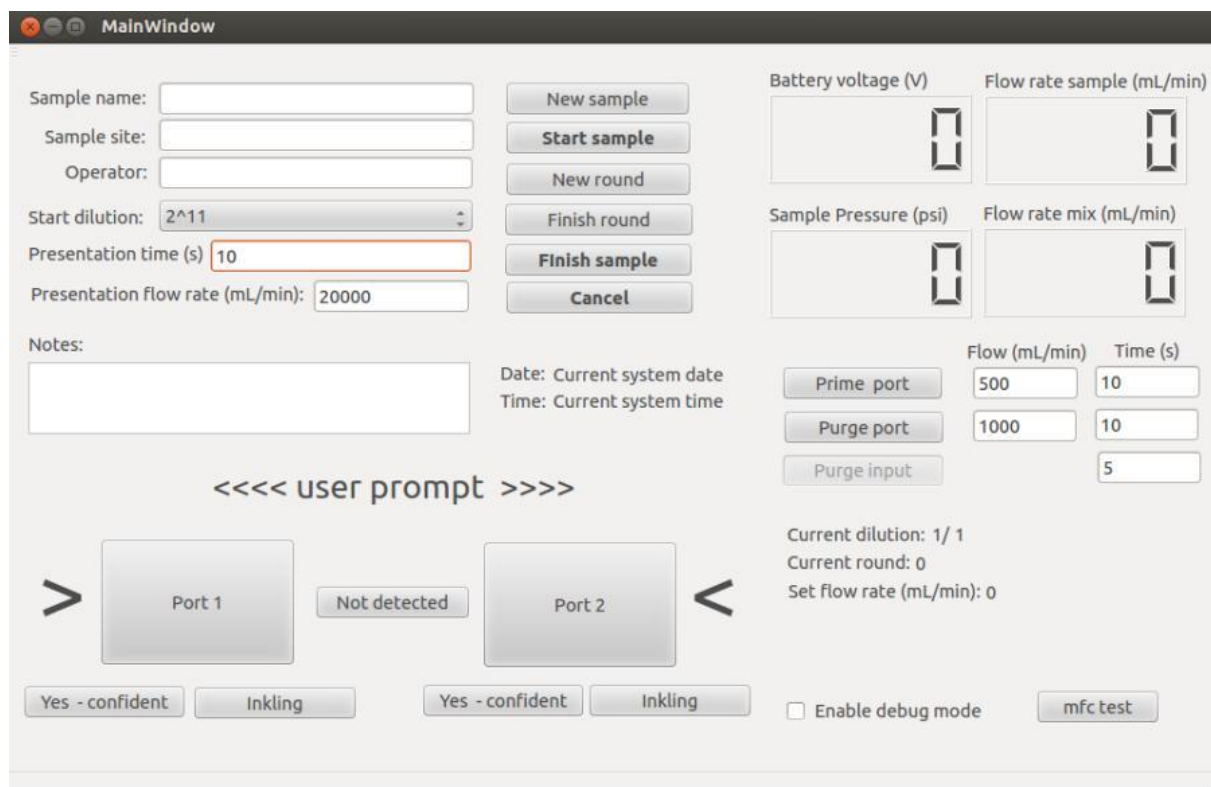


**Figure 33.    User interface for field olfactometer.**

### Staggered dilution series

Traditional olfactometry has limited resolving capabilities and relies on averaging of multiple panellist results. Once the dilution step is set for the first round, the range between dilutions is predefined and

static as shown in Figure 34. A novel technique based on a staggered dilution series was tested, resulting in a narrower range as shown in Figure 35.

True odour = 300 OU

Presented dilution series ➜ **2048 – 1024 – 512 – 256 – 128 – 64 – 32**

Panellist could not detect odour

Panellist did detect

*Odour lies between 256 and 512 OU*
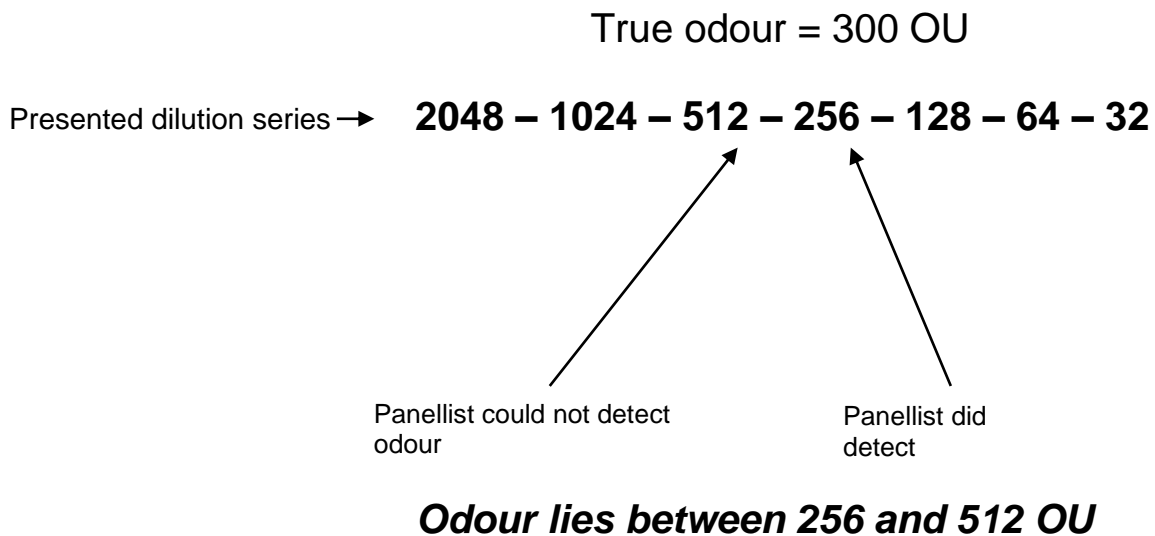
**Figure 34.  Dynamic olfactometry dilution series concentration intervals are predetermined by the step factor (two in this case) and are static.**

**2048 – 1024 – 512 – 256 – 128 – 64 – 32**

1536 – 768 – 384 – 192 – 96 – 48

1280 – 640 – 320 – 160 – 80 – 40

*Odour lies between 256 and 320 OU*
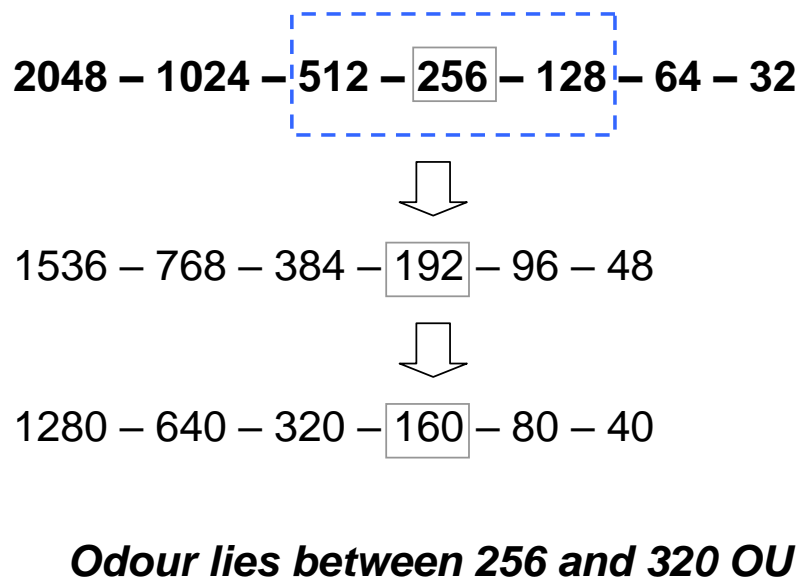
**Figure 35.  Field olfactometer uses a staggered dilution series to narrow the concentration range of the sample.**

**Single boxed values are the dilutions odour is first detected in the dilution series.**

In an effort to eliminate background odour, the CPAP pump was later bypassed using carbon filtered air provided by a small 12V DC pump and finally using externally supplied compressed air (used by the laboratory olfactometer). These provided little or no improvement and it is likely that background odour was a result of the mask, air lines, or both. The current FO shown in Figure 36 relies on an air-

odour mixing unit in a modified ResMed SoftEdge™ Mirage FX mask to ensure an even presentation of diluted odour to the panellist.



**Figure 36.   Laboratory evaluation of field olfactometer using n-butanol reference gas.**

## Methodology

### Calibration

Calibration accuracy of dilution was verified using Sulphur Hexafluoride ($SF_6$) tracer gas and an Innova 3434i photo-acoustic analyser (LumaSense Technologies).

### Laboratory evaluation

The laboratory evaluation of the current field olfactometer was carried out in two steps. Firstly, using an *n*-butanol reference gas, two panellists were screened for their detection thresholds, which was then compared to results obtained from DAFF's standard dynamic olfactometer. Secondly, simulated odour samples (using varying concentrations of *n*-butanol) were generated to test the resolving capabilities of the field olfactometer with and without a staggered dilution series.

## Results

### *n*-butanol detection threshold

Using the FO and *n*-butanol, the two panellists consistently required a stronger concentration (typically three to four dilutions stronger) to reach detection threshold than they did using DAFF's laboratory forced-choice olfactometer. Despite efforts to address perceived background odour, both panellists felt there was still a background odour impacting their *n*-butanol detection. Future efforts will need to focus on finding and removing the source of this odour.

The FO resolving capabilities using a single panellist were successfully demonstrated using *n*-butanol. The results shown in Table 6 are representative of the FO results for one of the panellists using the staggered dilution series method. In this example the *n*-butanol detection threshold range was narrowed from 470 - 940 ppb to 660-790 ppb. However, *n*-butanol was detected by this panellist much earlier (up to four dilutions earlier) when using normal dynamic olfactometry. The reasons for this difference are still to be resolved.

**Table 6.    Staggered dilution n-butanol detection results for a panellist.**

**Underlined values = first dilution detected with certainty. Bolded values = revised threshold limits.**

| Round | Dilution series | Revised detection threshold range (ou) | Revised detection threshold (ou) | n-butanol detection range (ppb) | n-butanol detection threshold (ppb) |
|---|---|---|---|---|---|
| 1 | 2048, 1024, 512, 256, **128**, **64** | 64-128 | 91 | 470-940 | 660 |
| 2 | 1448, 724, 362, 181, **91**, 45 | 64-91 | 76 | 470-790 | 790 |
| 3 | 2432, 1216, 608, 304, 152, **76** | 76-91 | 83 | 660-790 | 720 |
| 4 | 1722, 861, 430, 215, 108, 54 | 76-91 | 83 | 660-790 | 720 |

# Proposed field evaluation

A proposed demonstration of in-field operation of the prototype FO and a limited evaluation (small sample set) could not be carried out because of the abovementioned unresolved matters. The aim of this proposed field evaluation was to verify a panellist's ability to analyse odour on-site (at the odour source) where they would be subject to olfactory desensitisation, temperature and humidity variation.

# Proposed methodology

Had the field evaluation proceeded, then the following methodology would have been used. It is included in this report as the recommended method for field evaluation in the event that future development work delivers a FO worth demonstrating in the field.

Method:

- in-field collection of bulk DO samples and FO samples (six samples)

- in-field analysis of FO samples (six samples) using two in-field panellists

- bulk odour samples are collected into 120L olfactometry sample drums. Sample bag is pre-sampled and purged prior to collection to minimise absorption of VOCs to bag surface

- a subsample (1.2 L) for FO analysis is sampled from the bulk sample using the same technique (pre-sample and purge)

- the subsample is analysed by the first field panellist using the FO

- the FO subsample is then purged and refilled from the bulk sample and then analysed by the second field panellist

- this procedure is completed for all six samples, and with the bulk samples, which are then returned for laboratory analysis using the DAFF dynamic olfactometer within six hours of sample collection

- the two field panellists shower and change clothing before joining the laboratory olfactometry panel.


# Key findings

The important findings are:

- the modified CPAP machine can help address the problem of panellist odour desensitisation (as can happen in the field), by providing a continuous clean air flow in between odour presentations

- mass flow controller allows for repeatable odour dilution and staggered presentation

- concentration range of 32 to 2048 ou can be achieved

- a clean air supply is important to avoid background odour

- unresolved background odour and sensitivity issues limited further testing

- the developed FO was robust and easy to use and is potentially a useful cost-effective proxy for traditional olfactometry, provided background odour and sensitivity issues can be resolved.

# 9.  Development of a prototype low-cost field portable VOC pre-concentrator

## Background

VOCs in meat chicken emissions are at sub-parts-per-million concentrations. In practice, the sensitivity of the MOS sensors tends to be no better than ppm levels. Therefore, the total VOCs response from a grab sample may not even register, or is lost in the 'noise'. Either way, the MOS sensors provide little useful information about the VOCs.

Perhaps the only situation odorants would reach ppm levels is if they were 'pre-concentrated' in an unventilated, closed shed containing used litter; for example, after the birds are taken out. This is an atypical in-shed situation which the current AOS was unable to handle because of high levels of ammonia saturating the sensor and masking the total VOC sensor response.

Beyond the shed, ammonia is generally not a problem. Pre-concentrating the odorants to register a meaningful AOS response that could, for example, allow estimation of the ambient total VOCs, would be a better outcome. However, sensor sensitivity would still need to improve for a pre-concentrator to make any significant difference to the current AOS application; for example, pre-concentrating odorants from ppt to ppb levels is of no help if the sensor can only register ppm levels.

Another consideration is that once new odorants start being introduced to the sensors as a result of pre-concentration, the odour prediction algorithm may be compromised. The same applies for the non-odorous VOCs that are also being concentrated. It may be that another odour prediction algorithm is necessary for use with pre-concentrated samples.

With pre-concentration, the question still remains as to what percentage of the total VOC response was caused by odorants and what the odorants are. That question may be resolved by collecting duplicate pre-concentrated samples and having them analysed by SIFT-MS or PTR-MS, for example. Pre-concentration may become increasingly relevant to AOS applications due to: improving sensor technology; a shift to narrow-range sensors to target specific odorants, or classes of odorants; and demand for application-specific AOS instruments (Wilson, 2013). The ability to pre-concentrate key marker chemicals, particularly at receptor locations where odours are weaker, intermittent and mixed with non-target odours, is also useful for odour discrimination.

The VOC pre-concentrator's greatest potential would be to use it in conjunction with an AOS and SIFT-MS or similar technology in helping to identify nuisance odorants. The pre-concentrator could be modified to operate as a remotely triggered sampling device; for example, at a receptor, when an AOS at source indicates odour could impact. The collected samples could then be analysed for VOC content.

## Operating principle

The principle of operation is similar to that for collecting GC-MS samples onto Tenax tubes, only automated. Ambient air was drawn through purpose-built Tenax tubes at a user-defined flow rate and duration.
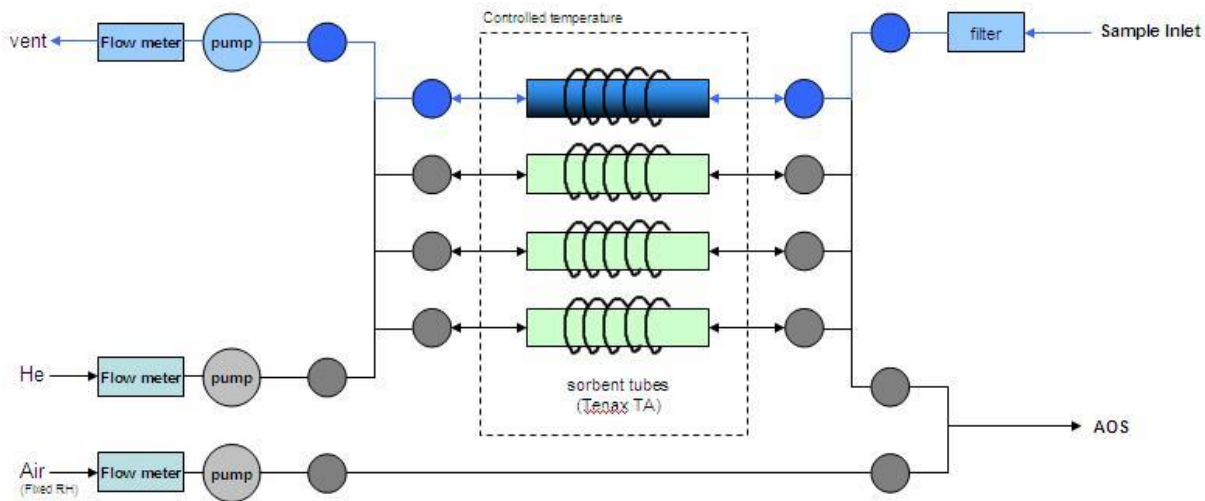
**Figure 37.  Schematic of the pre-concentrator. Sample can be collected on to one of four available sorption tubes and stored for later analysis by the AOS.**
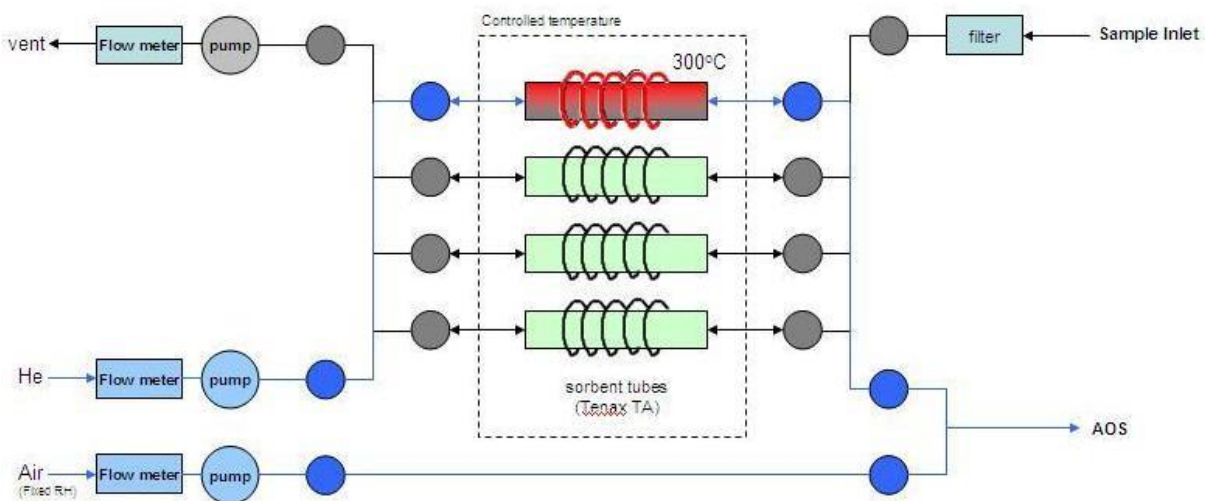


**Figure 38.  Sample regeneration using the VOC pre-concentrator. Thermal desorption principle is used to volatilise VOCs at 300°C into airflow at known flow rate.**

# Hardware

The pre-concentrator design and operation is illustrated in Figure 37 (for the sample collection process), and Figure 38 (for the sample regeneration process). The actual device is pictured in Figure 39.
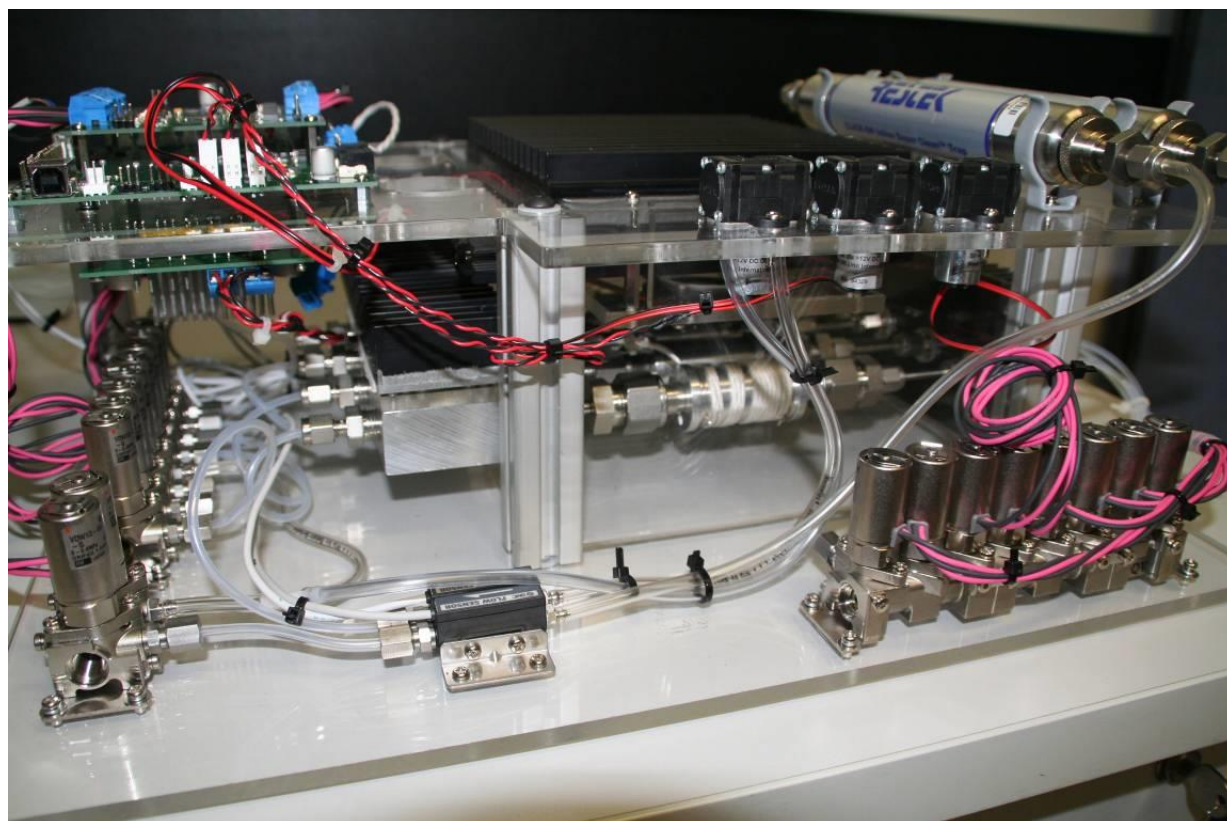


**Figure 39.   VOC pre-concentrator.**

# Discussion and conclusions

Using a pre-concentrator has advantages, but also introduces another level of complexity to odour measurement because the odours need to be thermally desorbed before they can be analysed by olfactometry and AOS.

As stated in the introduction, the best use of the pre-concentrator could be in conjunction with an AOS (with improved sensors), and SIFT-MS, PTR-MS or similar technology, to help identify nuisance odorants. The pre-concentrator could be modified to operate as a remotely triggered sampling device; for example, at a receptor when an AOS at source indicates odour could impact. In this case, an AOS may only have to predict the concentration of an odorant or group of odorants (an easier task than predicting odour) to be an effective monitoring tool that spawns more informative odour assessment. The collected samples could then be analysed for VOC content.

# Discussion and conclusions

Success and utility of AOS applications for odour nuisance monitoring depends largely on the chemical composition and concentration of odorants. AOS have been demonstrated relatively successfully for waste water treatment facilities (Capelli *et al*., 2008) and pulp and paper industries (Deshmukh *et al*., 2014), where the analysis conditions are far more favourable than for intensive livestock odours. Bulk changes in chemical composition are far easier to measure.

Odour from broiler farms are characterised by odorants at very low (ppb) concentrations. These are challenging to detect and quantify by even high-end laboratory equipment. The situation is further compounded by the presence of interfering compounds, such as ammonia, at concentrations an order of magnitude greater. The low cost sensors available and used in this study are generally sensitive to this compound, making it difficult to objectively measure fluctuation in odorants. Furthermore, in-shed ammonia fluctuates independently of odour.

Though a reasonable prediction model was developed for the three meat chicken farms studied, its accuracy for other farms cannot be assumed with any confidence. Even a small change to a farm's operation (such as reusing the litter) may invalidate the model for the farm concerned. A diet change introduced to farms B and C could potentially render the model invalid for both those farms.

From experience gained in this project, development of a reliable AOS for measurement of poultry odour — and intensive livestock odours in general — is unlikely to succeed in the foreseeable future for the following reasons:

- VOC quantities emitted from broiler sheds are very low (in the order of 500 ppb in total). Instrumented measurement of VOCs at these concentrations is challenging and there are no low-cost sensors available with sufficient sensitivity for the job. High-end instrumentation such as GC-MS, SIFT-MS or PTR-MS is required.

- The sensors used by the AOS are not sufficiently selective. The sensors also respond to non-odorant VOCs, humidity, temperature and ammonia (highly variable at 100 times the concentration levels of VOCs). These are not correlative with odour (and this is well understood). Measuring fluctuating odorant concentrations is masked by these other components.

- The odour prediction model may fail if a small change in odour composition occurs. Small changes in odorants can significantly alter the odour strength and characteristics with no change detected by the sensors (the human nose is many orders of magnitude more sensitive for some odorants). This could make use of an AOS on other poultry sites independent of the 'training' sites problematic or impossible.

The following additional considerations concerning long-term use of AOS technology are also flagged:

- An odour prediction model is highly dependent on the sensors used during the 'training' phase. A change in sensor characteristics or commercial availability of sensors would likely render the model useless and the entire modelling process (including collating another database of corresponding olfactometry and AOS measurements) would need to be repeated.

- The sensors are prone to drift and degradation which would cause the odour prediction model to fail.

- The odour prediction model may fail if a small change in odour composition occurs.

- For research applications, if the odour measurement capabilities of the AOS are unsatisfactory, the sensor data itself is of no use because it provides no insight into the odour's chemical composition (that is, identifying and quantifying VOCs).

To conclude, measurement of broiler shed odour has proven very challenging. While the initial results of this study were promising, the true nature of the problems became apparent as the project progressed. The SIFT-MS findings further highlight the difficulties and reinforce these conclusions.

In limited circumstances, AOS technology may prove useful in odour measurement (for example, for research applications with local calibration), however, it is unlikely in the near future that an enose based on low-cost sensors could provide an on-site odour measurement tool for monitoring meat chicken shed odours.

# Implications

The implications for the industry and community are that there is no convenient, affordable, objective odour measurement solution for the foreseeable future. Traditional assessments using the human nose will continue to be the norm, and any commercial sensor-based tool currently offered to 'measure' poultry odour needs to be treated with a large degree of scepticism until it can be proven it works. The level of proof required would include extensive validation against olfactometry data for a number of farms, and repeated under different circumstances (for example, season, bird age, diet) for each farm.

The implications for policy makers are that the funding of AOS research has not returned a viable solution. However, the project has taken AOS research to a new level and provided clear direction for future researchers. Future success of AOS is dependent on the development of more sensitive and selective sensors.

The implications for potential users are that AOS has too many limitations for measuring chicken shed odour, making it an unviable commercial proposition currently. In the course of this AOS research, much better analytical instruments capable of measuring individual odorants in real time have emerged like SIFT-MS and PTR-MS. These instruments are cost-prohibitive for most stakeholders but once their utility is proven, they could be leased or provided on a fee-for-service arrangement. These analytical instruments will allow well-targeted, unambiguous odour analyses in future that will aid development of better odour reduction strategies, and more robust and defensible odour prediction models.

# Recommendations

Future odour assessment and abatement research should focus on identifying and acquiring a much better understanding of the key odorants within poultry odour that cause odour nuisance. The cause of those odorants needs to be identified in developing better odour reduction strategies.

The ability to identify and measure these key odorants in real-time in the field has improved with the advent of new analytical instruments such as SIFT-MS (Španěl and Smith, 2011; Van Huffel *et al*., 2012) and PTR-MS (Hansen *et al*., 2012). These analytical instruments are expensive and still largely unproven for intensive livestock odour measurement and impact studies, particularly, at the odour receptor. Trialling such instruments in parallel with GC-MS and odour measurements (for example, using DO, FO, AOS) is critical to improve understanding of instrument accuracy and relationships between odour chemistry and perceived odour concentration. We need to verify what the instrument is claiming to measure is correct, and if not, determine why it is not correct.

To assess abatement strategies which suppress specific odorants, an affordable, objective AOS solution may be still desirable but sensors targeting the suppressed odorants would have to be developed before revisiting AOS. Analytical instruments like SIFT-MS and PTR-MS taken and used on-site may prove a better choice.

More reliance should be put on field olfactometry to develop cost-effective odour prediction models. The working models can be later confirmed and strengthened with standard dynamic olfactometry measurements.

Seeking expert opinion from researchers with a strong environmental chemistry background is essential before committing funds and resources to future odour assessment research projects.

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# Appendix A   Market research report

The information in this report is an edited version of an interim project report of the same name submitted to RIRDC in August 2009.

## Towards Artificial Olfaction System Solutions for On-site Odour Assessment

by Michael Atzeni and Jae Ho Sohn

### Executive Summary

An artificial olfaction system (AOS) is under development through collaboration between the project team led by the Department of Employment, Economic Development and Innovation (DEEDI), Queensland Government and Rural Industries Research and Development Corporation (RIRDC).

As the final outcome of the project is a commercialisable prototype AOS capable of objectively measuring and discriminating poultry odours, it is important to conduct market research to evaluate prospective clients' needs and expectations in determining the market potential and optimum benefits and features of a new product.

An online market research survey was distributed to around 200 prospective AOS users worldwide. A total of 92 responded and, of those, 86 completed the survey in full.

The survey revealed a general lack of confidence in the reliability of current field odour measurements, and dissatisfaction with the cost-effectiveness of both lab and field odour assessments. Even though suitable AOS devices for environmental monitoring are not yet available commercially, recognition of this technology is high and there are strong demands for reliable, portable odour sensing devices.

With regards to the important features for design and development of the AOS, accuracy and reproducibility/precision are considered the two most important factors, followed by sensitivity, portability and cost. A portable field device could expect to retail for $10,000-$15,000 and should weigh around 10kg or less.  Portability is secondary to reliability of the measurements provided the instrument can also be left *in situ.* Incorporation of Global Positioning System (GPS) and meteorological data collection functionality is desirable but not imperative. Incorporation of additional sensors for measuring certain other chemicals in tandem with odour is highly desirable, if cost-effective.

In summary, there is a ready retail market for the AOS and also a rental market.  This level of interest identified with this market research augurs well for fast-tracking the commercialisation of the proposed AOS.

### Introduction

Market research is necessary to evaluate prospective clients' needs and expectations, and therefore the market potential and optimum benefits and features of a new or revised product. For the Rural Industries Research and Development Corporation (RIRDC)-funded Artificial Olfaction System (AOS) project, a commercialisable, prototype portable AOS tailored to the poultry industry is the main expected outcome.

AOS is a tool for objectively measuring odour and discriminating between odours from different sources. Although the technology is based on electronic nose (eNOSE) technology, **the proposed AOS has the following distinguishing features**:

- **Poultry odour quantification**. Combining with odour concentration prediction models that employ artificial intelligence (AI) algorithms, the AOS is able to measure odour concentrations on a real-time basis with a significant level of accuracy. There is no commercially available eNOSE for quantifying odours of any type.

- **Poultry odour discrimination**. The AOS is capable of discriminating poultry odour from the odours emitted from other sources, using the customised pattern recognition engine. The pattern recognition engine has been developed using the poultry odour database established over three years of odour sample collection from various commercial poultry farms.

- **Continuous odour assessment**. While eNOSEs instruments are used mainly for qualitative work, requiring only discrete measurements, the proposed AOS is specifically designed for continuous poultry odour assessment.

- **In-field capability**. Conventional eNOSEs are lab-based instruments. In contrast, the new AOS will be able to operate under in-field conditions.

The AOS project will span three years (April 2009 to March 2012). This preliminary market research will provide significant input into design considerations and which consumer preferences can be accommodated to deliver the best possible solution within the time and budget allocated.

For in-field monitoring applications, the AOS market is considered to be mostly consultants, regulators, odour assessment service providers and researchers. Within the poultry industry, and other intensive livestock industries, a market also exists with producers who would use it as an odour management tool.

## Methodology

The survey was developed using the Survey Monkey online survey development software (http://www.surveymonkey.com/). Before being released, the survey was beta-tested in-house. The survey is shown in Appendix A-A.

An explanatory email with a link to the final version of the survey was distributed to around 230 prospective AOS users worldwide on 19 September 2008. Those contacted were predominantly Australian and international environmental consultants, odour assessment service providers and odour researchers, Australian and New Zealand government regulatory authorities, including all councils in Victoria and Queensland councils with poultry industries in their shires.

The contact list was compiled from internet searches, personal contacts and industry magazines. For the purpose of the survey, "AOS" terminology was deliberately avoided in favour of "Electronic Nose" terminology, as it is the better known terminology representing this technology and would minimise confusion. In this report, the term 'eNOSE' is, however, used for representing the existing instruments, to distinguish it from the proposed new AOS technology.

It was optional for the respondent to supply their personal details, that is, they could remain anonymous.

The survey questions were grouped to evaluate the following:

- Current odour assessment practices;

- Needs for alternative solutions for odour measurement;

- Ideal design for an AOS device;

- Market demands for an AOS; and

- AOS applications.

## Results and discussion

### Response level and categorisation

A total of 92 responded and, of those, 86 completed the survey in full. Anonymity was offered, yet 50%, including some key international odour researchers, chose to provide their name and contact details. A number of respondents expressed their personal interest in the project, some requesting further information. Several New Zealand respondents also asked to be kept up to date with progress on the project.

Of the 83 respondents who nominated their location, 77% were from Australia and 10% from New Zealand. The remaining 13% were from Germany, Belgium, Japan, Ireland, UK, USA, Canada, Netherlands and Spain.

Figure 1 and Figure 2 show regulatory staff (41%) and government organisations (49%) dominated the response sources, respectively. Private businesses (consultants/odour assessment providers) accounted for 35% of respondents and researchers (13%).
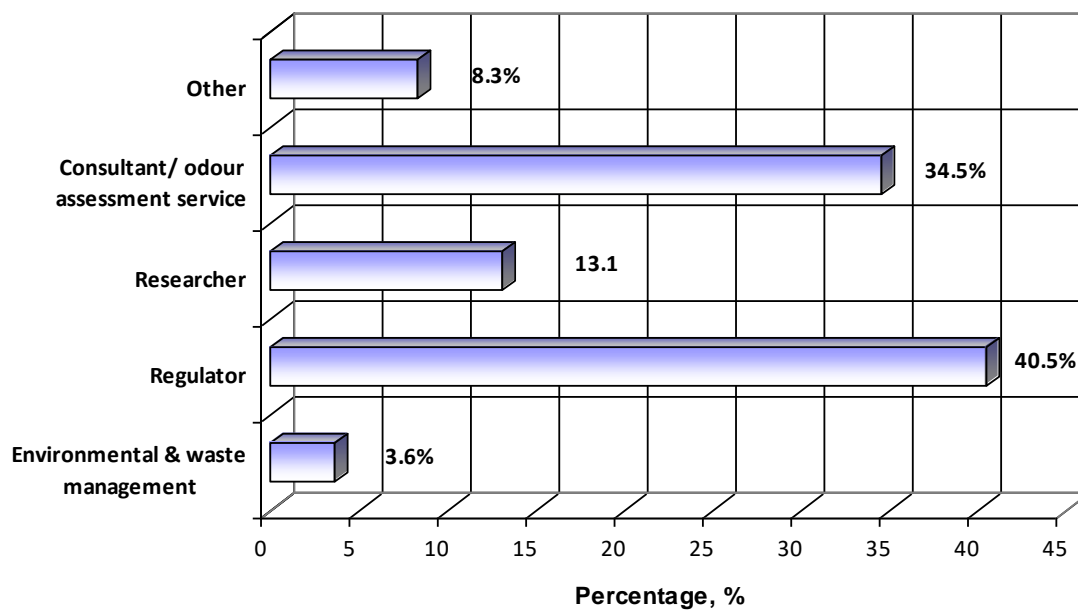


**Figure 1.    Occupation of respondents**
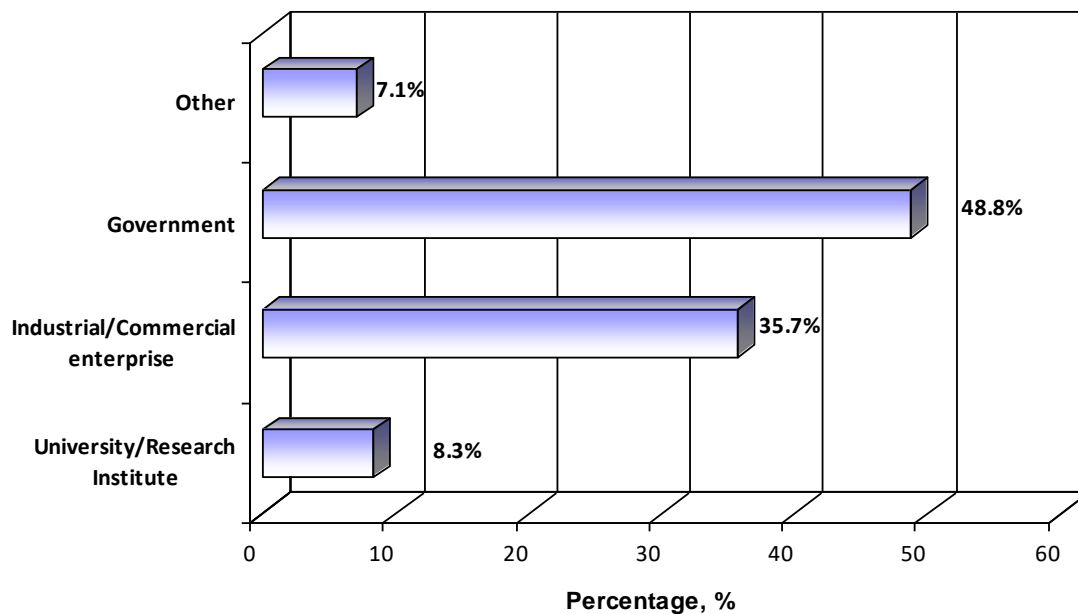
**Figure 2.  Business/organisation of respondents**

Of the 46 respondents who provided optional details about their place of work, 46% were from the private sector, 46% were from government and the remaining 8% were from universities/research institutions.

## Current odour measurement practice

### *Respondents' knowledge level on odour measurement*

The results shown in Figure 3 indicate 80% of respondents had at least a fair knowledge of odour monitoring and measurement techniques, while 51% indicated their knowledge was good or better. Therefore, the sampling pool for our target audience is well-defined, allowing us to address their needs in developing the AOS.

### *Frequency of dealing with odour problems*

The results in Figure 4 indicate 87% of respondents deal with odour problems at least seasonally and 46% at least weekly; with 20% indicating a daily involvement. These results indicate a significant commitment of time and resources already exists, despite the various shortcomings of existing odour measurement techniques. In many situations, odour measurements using the currently available instruments/techniques are not an option because of logistical problems, availability of facilities or prohibitive costs.

Therefore, a more convenient, cost-efficient assessment instrument that was reliable and objective would provide more advantages to solve odour-related issues. This bodes extremely well for the commercial success of a suitable AOS solution.
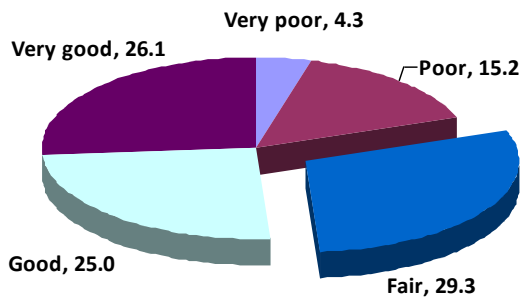
**Figure 3.** Odour measurement and monitoring knowledge level
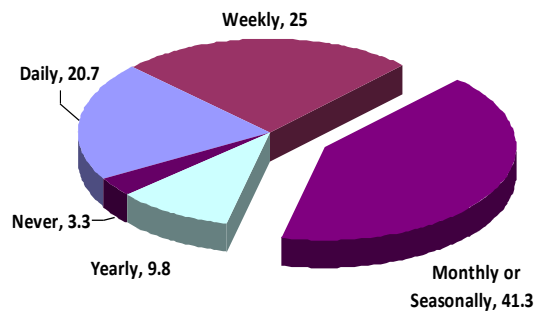


**Figure 4.** Odour measurement and monitoring frequency

*Current odour measurement techniques*

Of the instruments used for odour assessment, the human nose (75%) was the most common, followed by olfactometry (53%) as shown in Figure 5. Multiple answers were allowed for the question because multiple odour measurement techniques are often used together to investigate odour-related issues. The olfactometry figure is interesting because it indicates there are olfactometry services close enough to at least half the respondents.

Only 25% used GC-MS and 23% used field panellists, a reflection of their more technical and labour-intensive natures, respectively. Not surprisingly, eNOSE had only been used by 6.5% of respondents mainly due to the lack of commercially available eNOSE for in-field odour assessment.

The 24 % "Other" result is misleading in that of the 19 responses, only five mentioned different odour measurement techniques to those already listed.  Of these, three used complaints registers, one used a Draeger hand pump system, one used weather bureau data and poultry farm production logs, and one used absorbing tubes for collecting VOC samples for GC-MS-O tests. Three respondents did not use the techniques but used the results of others.
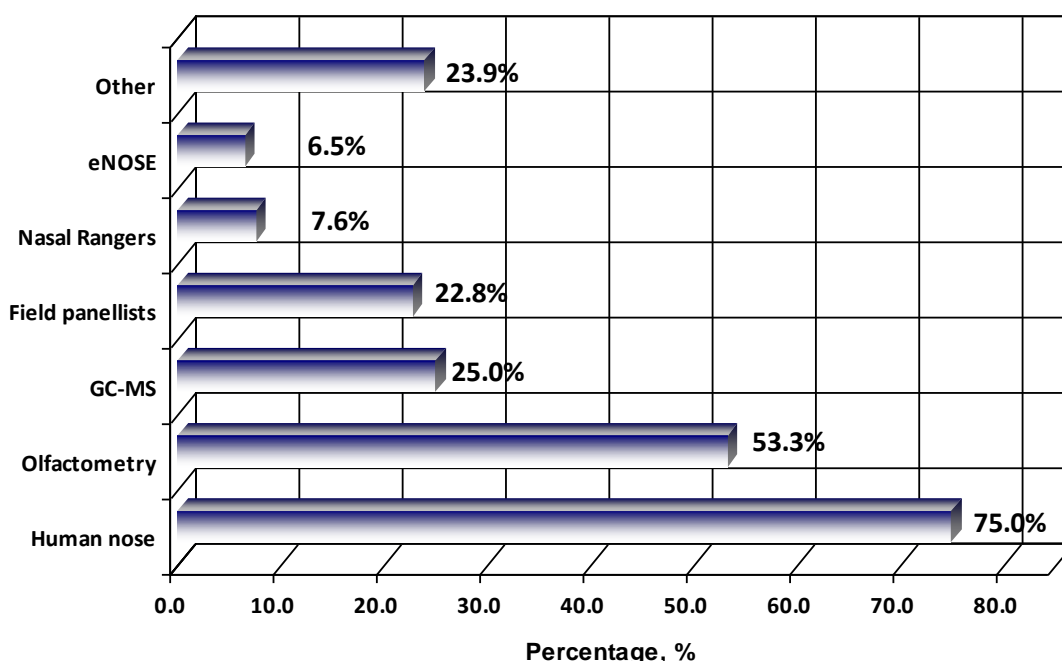
**Figure 5.   Odour measurement and monitoring techniques used**


*Respondents' satisfaction levels on current odour measurement techniques*

For laboratory data, 61 respondents gave an opinion (Table 1). Half of them indicated the data was good to very good. Only five of them rated the data as excellent.

For field results, the majority who offered an opinion considered them fair (35%) to good (21%) as shown in Table 1. The satisfaction level of field results is less than that of laboratory results. Only 16% considered them any better. There is clearly room for marked improvement in providing options for improving field data quality, based on current satisfaction levels, which the AOS solution should be able to fulfil.

**Table 1.     Reliability of odour data assessments**

|  | Poor | Fair | Good | Very good | Excellent | Not Applicable | Total |
|---|---|---|---|---|---|---|---|
| Laboratory results | 4.3 % (4) | 15.2 % (14) | **20.7 %** **(19)** | **20.7 %** **(19)** | 5.4 % (5) | 33.7 % (31) | 100 % (92) |
| Field results | 7.6 % (7) | **34.8 %** **(32)** | **20.7 %** **(19)** | 13.0 % (12) | 3.3 % (3) | 20.7% (19) | 100 % (92) |

Those that commented on the costs of services were mainly of the opinion they are fair for both field-based and lab-based assessments (Table2). Clearly, there is much scope to improve wide held opinion by developing affordable field-based instrumentation that can operate continuously and thereby reduce the number of lab tests required.

**Table 2.    Cost-effectiveness of odour assessments**

|  | Poor | Fair | Good | Very good | Excellent | Not Applicable | Total |
|---|---|---|---|---|---|---|---|
| Laboratory | 15.2 % (14) | **26.1 % (24)** | **19.6 % (18)** | 6.5 % (6) | 1.1 % (1) | 31.5 % (29) | 100 % (92) |
| Field | 13.0 % (12) | **25.0 % (23)** | **21.7 % (20)** | 17.4 % (16) | 4.3 % (4) | 18.5 % (17) | 100 % (92) |

The usefulness of the odour measurements was generally considered good to very good for lab results, but only fair to good for field results (Table 3). This reinforces the need for improvement in the field; a niche which AOS technology can fill.

**Table 3.    Overall usefulness of odour assessments for performing work duties**

|  | Poor | Fair | Good | Very good | Excellent | Not Applicable | Total |
|---|---|---|---|---|---|---|---|
| Laboratory results | 3.3 % (3) | 14.1 % (13) | **26.1 % (24)** | **17.4 % (16)** | 8.7 % (8) | 30.4 % (28) | 100 % (92) |
| Field results | 5.4 % (5) | **21.7 % (20)** | **35.9 % (33)** | 13.0 % (12) | 7.6 % (7) | 16.3 % (15) | 100 % (92) |

## Needs for alternative solutions for odour measurement

The majority (65%) had at least some knowledge of existing eNOSE technology (Figure 6) and 11% were familiar or very familiar with it (Figure 6).  It demonstrates that there is a high recognition of existing eNOSE technology, despite the general lack of access to it and the complete lack of commercially available eNOSEs for environmental monitoring purposes.

When asked if they had ever used an eNOSE before, most (88%), for the reasons outlined above, had not. Of the 12% that had, their additional comments revealed some had been involved with the earlier lab-based eNOSE technologies such as Aromascan, Alpha-MOS and z-Nose, including a respondent who evaluated eNOSE and similar technology in connection with poultry and rendering plant odours.
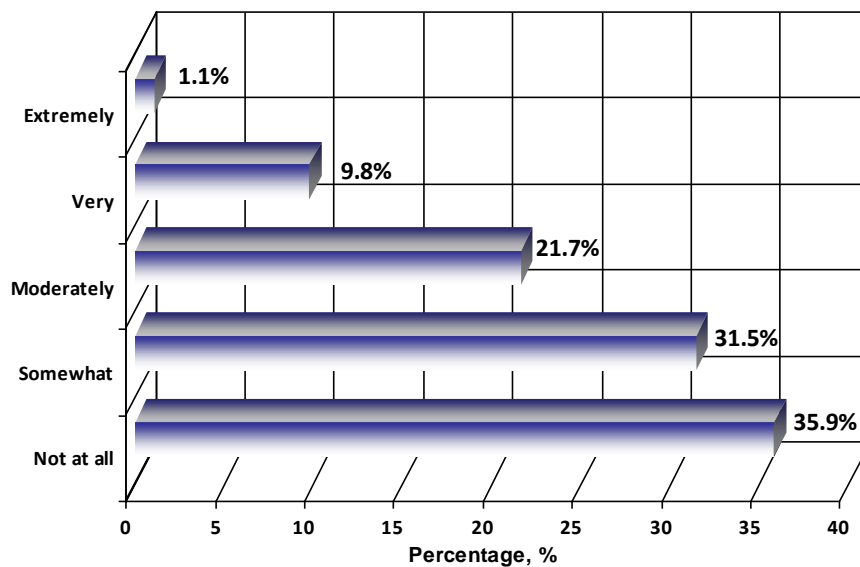
**Figure 6.    Respondents' familiarity with existing eNOSE technology**

## Ideal design for an AOS solution

To achieve the best possible outcome, it was imperative to determine what the most important features of an AOS solution are considered to be, and therefore what trade-offs need to be made to deliver an optimal product within time and budget.

To gather this critical information, the respondents were given definitions for the following five factors, which they then had to rank in order of importance: sensitivity, accuracy, reproducibility/precision, portability and price.

The definitions given were as follows:

- **Sensitivity** – the minimum magnitude of the input signal required to produce a meaningful output signal, after taking any "noise" into account;

- **Accuracy** – degree of closeness of the measured odour to the actual (true) value as determined by standard procedure using a human panel, that is, olfactometry;

- **Reproducibility/precision** – ability to achieve a consistently accurate measurement of the same odour in independent tests;

- **Portability** – general characteristic of being readily transportable from one location to another; and

- **Price** – cost of retail item.

The collated results of the 86 respondents to this question (Table 4) indicate that accuracy is ranked highest, followed by reproducibility/precision, sensitivity, portability and price.

**Table 4.    Rankings for five key factors in development of a portable AOS**

|  | 1 (most) | 2 | 3 | 4 | 5 (least) | Total |
|---|---|---|---|---|---|---|
| Sensitivity | 10.5 %<br>(9) | 18.6 %<br>(16) | **40.7 %**<br>**(35)** | 14.0 %<br>(12) | 16.3 %<br>(14) | 100 %<br>(86) |
| Accuracy | **41.9 %**<br>**(36)** | 27.9 %<br>(24) | 17.4 %<br>(15) | 7.0 %<br>(6) | 5.8 %<br>(5) | 100 %<br>(86) |
| Reproducibility/precision | **33.7 %**<br>**(29)** | **33.7 %**<br>**(29)** | 17.4 %<br>(15) | 11.6 %<br>(10) | 3.5 %<br>(3) | 100 %<br>(86) |
| Portability | 7.0 %<br>(6) | 8.1 %<br>(7) | 12.8 %<br>(11) | **40.7 %**<br>**(35)** | 31.4 %<br>(27) | 100 %<br>(86) |
| Price | 7.0 %<br>(6) | 11.6 %<br>(10) | 11.6 %<br>(10) | 26.7 %<br>(23) | **43.0 %**<br>**(37)** | 100 %<br>(86) |

For sensitivity (Figure 7), 30% nominated the most sensitive option, that is, 2.5 ou/m$^3$, which is the standard requirement for many of the environmental air quality regulations. Almost the same percentage (28%) did not know or had no opinion about the sensitivity. Of the 21% "other" responses, that is, 18 respondents, 11 of them specified a sensitivity of $\leq 2$ ou. Of these, five specified 1 ou. The seven others made more general responses but a need for single digit sensitivity was the overall message conveyed.

One pertinent comment made was the AOS should be able to discriminate a similar change in odour unit concentration to the human nose. Importantly, the AOS is being calibrated against the human nose by way of olfactometry.

For accuracy (Figure 8), the majority (33%) were happy with $\pm 10\%$, although 23% indicated $\pm 5\%$ level was the acceptable level for their purposes.
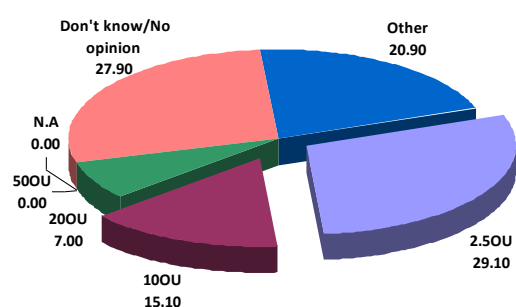


**Figure 7.    Preferred sensitivity levels for AOS**
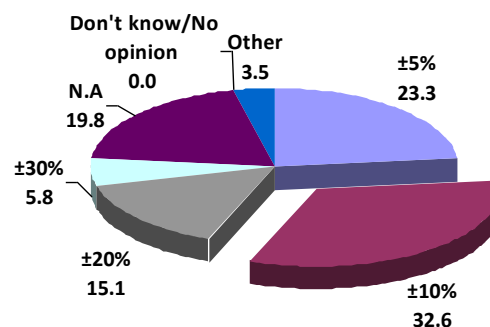


**Figure 8.    Preferred accuracy levels for AOS**

A precision level of $\pm 5$ to 10% would appear acceptable (60%) (Figure 9). As one respondent commented, precision is likely to be far more problematic for complex odours than single compounds and its importance needs to be considered on a case-specific basis.

For portability, 70% indicated a weight of 10 kg or less was acceptable and 39% preferred a weight of 5 kg or less (Figure 10). Weight considerations brought about some interesting, sometimes conflicting, comments from the "Other" 6%:

- For work, health and safety reasons, the weight should be 2-16 kg, depending on how long it is needed to be carried.

- If accuracy was significantly improved by additional equipment, up to 20 kg would be acceptable.

- Weight becomes a non-issue if the portable unit is to be left *in situ.*
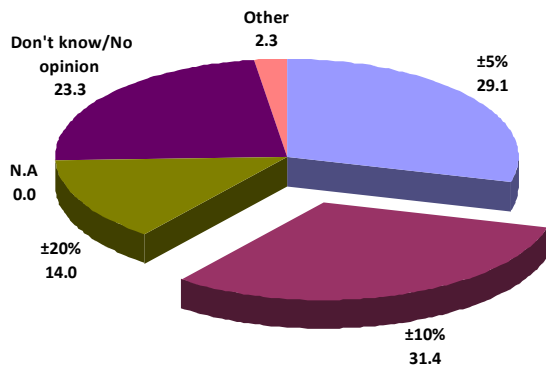
**Figure 9.** **Preferred precision levels for AOS**
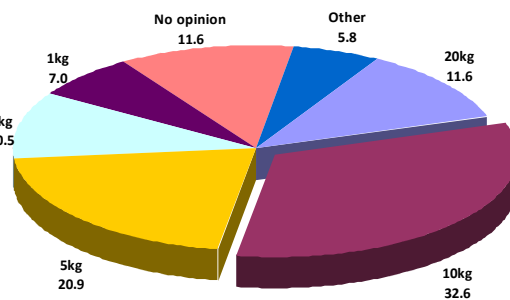
**Figure 10.** **Preferred weight for portability of an AOS**

As shown in Figure 11, opinion on cost was well spread, but 22% indicated $10,000 was acceptable and 80% were prepared to pay $5,000 or more, to a maximum of $15,000.
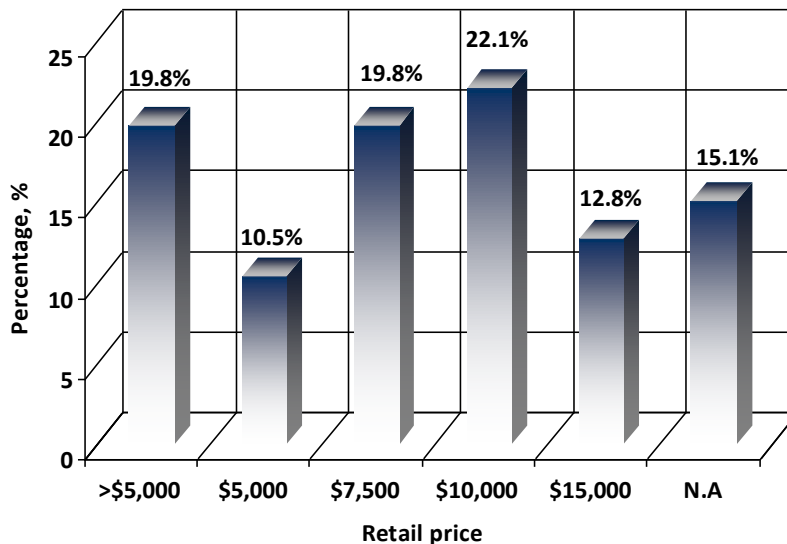
**Figure 11.  Preferred retail cost for portable AOS**

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The apparent message in this section is that the market is interested in an odour measurement instrument/technique provided it is reliable, and they are willing to pay the price, and compromise on portability for more reliable measurements. From our perspective, it means we should focus on the AOS's reliability, that is, its reproducibility/precision and accuracy.

With regard to additional functionalities, the majority agreed a Global Positioning System (GPS) (85%) and meteorological data collection device (92%) (Figure 12) would be useful inclusions. For convenience alone, this comes as no surprise.

Most of the 13 who commented further about GPS indicated they already had GPS instrumentation so it was not a necessity for the AOS application. It may indicate that those who do not have a GPS already probably answered in favour of including a GPS with the AOS unit. Including a GPS unit would enable electronic recording of position at the time of odour measurement, removing the chance of human transcription errors in associating the odour level with the location.

With respect to incorporating a meteorological data collection capability into AOS, several commentators queried the logistics of including this in a portable device and how reliable/relevant the data would be. The general consensus was crucial data like wind speed and direction needs to be collected in the standard way using a 2m or 10m weather station. Therefore, including a data link port or wireless communication module to the AOS for data synchronisation with a standard weather station would be considerable option.

Incorporating GPS and meteorological data collection capabilities into the AOS is however, beyond the scope of the project. Nevertheless, serious consideration should be given to incorporating connectivity which allows off-the-shelf units to be plugged into the AOS.
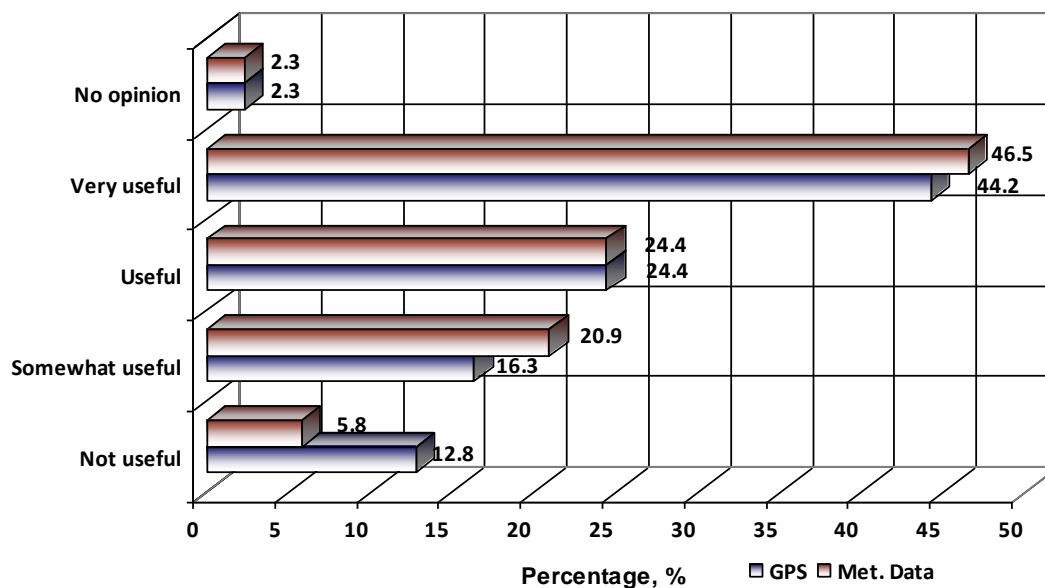


**Figure 12. Usefulness of incorporating a GPS and meteorological data collection function into a portable AOS**

When performed properly, odour mapping is a logistically difficult, time-consuming and costly exercise. It is rarely undertaken for these reasons, yet odour mapping is necessary especially for:

- verifying dispersion modelling predictions; and

- validating the various dispersion models being routinely used in Australia and overseas.

Using multiple AOS devices in the field over extended periods may greatly enhance the accuracy of odour mapping and also reduce the cost involved in assessing odour using humans.

Using multiple AOS devices at a site appealed to 87% of respondents with 37% indicating they would use more than one at a site and 50% indicating maybe they would (Figure 13). Cost of multiple units would be a reason for the high "maybe" response, whereas the others see it as a necessity, regardless of cost. Had all the underlying issues been spelt out in the survey, as above, it is our contention many more respondents would have been convinced of the benefits and answered affirmatively instead.
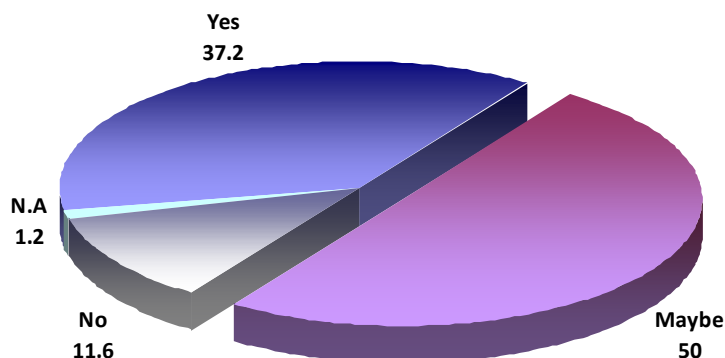


**Figure 13. Likelihood of using multiple AOS at a given site**

Most (71%) believe it is important to also measure VOCs in conjunction with odour and to a lesser extent $H_2S$ (67%) and Ammonia (54%) as presented in Table 5. There were various other specific chemicals nominated that were considered to be useful in conjunction with odour, depending on the odour source. Sulfur dioxide, reduced sulfides, mercaptans, organic acids, aldehydes, ketones and other carbonyl groups were nominated. There would be considerable benefits incorporating VOC specific sensors into the AOS, although this is not a part of the project requirements as it stands.

There is a clear message here that it would be beneficial to include suitable dedicated sensors for at least VOCs, $H_2S$ and ammonia in the AOS, if available and affordable, and collect data in parallel with the odour data.

**Table 5.    Chemical measurements considered important in connection with odour measurements**

|  | CO$_2$ | NO$_x$ | NH$_3$ | H$_2$S | VOCs | N.A/ no opinion | Other | Total |
|---|---|---|---|---|---|---|---|---|
| Percentage, % | 15.1 | 26.7 | **53.5** | **67.4** | **70.9** | 15.1 | 20.9 | - |
| Response count | 13 | 23 | 46 | 58 | 61 | 13 | 18 | 92 |

**Market demands**

When asked if they'd consider purchasing an in-field AOS, nearly half (40%) indicated they would and 41% indicated maybe (Figure 14). When asked if they would consider renting an in-field AOS, 53% indicated they would and 46% indicated maybe (Figure 15).

This level of interest augurs well for fast-tracking commercialisation of the AOS system once proof of concept can be shown.
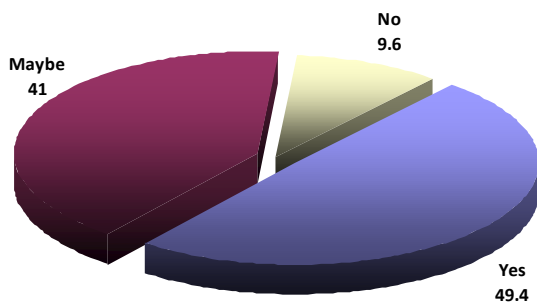


**Figure 14. Consideration to purchasing a commercially available in-field odour monitoring device**
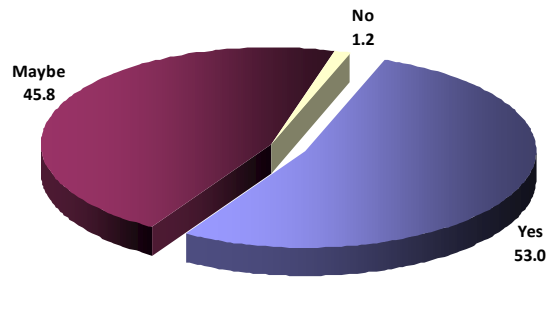


**Figure 15. Consideration given to renting a commercially available in-field odour monitoring device**

## AOS applications

Respondents indicated intensive livestock production was where they were mostly likely to use AOS for (80%). Also ranking highly were odour-emitting industries (64.3%) and waste management practices (65.5%). The other specific applications mentioned were:

- Geothermal emissions and horticultural emissions;

- Solvent type odours;

- Emissions of odour and VOCs from combustion processes *e.g.*, traffic, home heating and industrial for air quality and health impacts;

- Mining industry odours; and

- Commercial food shops (that is, restaurant, charcoal chicken shops, etc.).

## Conclusions

The conclusions from this preliminary market research in relation to current odour measurement practice are:

- There is a general lack of confidence in the reliability and thus usefulness of current field odour measurements; and

- There is dissatisfaction with the cost-effectiveness of both lab and field odour assessments.

In relation to alternative solutions, the conclusions are:

- There is a high recognition of AOS technology, however, its uptake is limited by a general lack of access to the technology and there being no commercially available AOS for environmental monitoring in the field;

- There is significant interest in the development of reliable, portable AOS devices for on-site measurement for various odour emissions; and

- **There are industry needs for reliable odour measurement devices tailored for specific odours.**

In relation to the ideal design for an AOS solution, the conclusions are:

- Accuracy and reproducibility/precision are the two most important factors, followed by sensitivity, portability and least of all, cost;

- A retail price of $10,000-$15,000 for a portable AOS appears acceptable;

- A portable AOS should weigh around 10kg or less;

- Portability is of secondary importance provided the AOS is reliable and can be left operating *in situ;*

- Incorporation of GPS and meteorological data collection functionality is desirable but not necessary, but market forces could change with the need to eliminate human transcription errors and affordable GPS units in future; and

- Incorporation of additional sensors for measuring certain other chemicals in tandem with odour is highly desirable.

From this market research survey of those involved in odour assessment services, odour regulation and odour research and funding of that research, it can be concluded:

- There is a market for retail of AOS devices, and also a rental market, particularly given the need for multiple units on site in many cases; and

- This level of interest augurs extremely well for fast-tracking the commercialisation of the proposed AOS system.

## Acknowledgements

## References

SurveyMonkey (2009) http://www.surveymonkey.com. Last accessed 21 August 2009.

# Appendix A-A. On-line survey form entitled "Towards Electronic nose (eNOSE) Solutions for On-site Odour Assessment"

## Towards Electronic Nose (eNOSE) Solutions for On-site Odour

### Introduction

Welcome to our survey. The Department of Primary Industries and Fisheries in Queensland, Australia, is a leading organisation in air quality and odour research.

You have been contacted because we believe you can help us in creating a portable odour measurement and monitoring device that can serve you well in the future.

The questionnaire should take no more than 15 minutes to complete. This is your opportunity to relate your own odour monitoring needs and preferences so that we can potentially address them sooner rather than later. Your feedback will help us deliver the best tool possible within our means, and within your budget.

Your information will be treated with complete confidentiality. It will only be used for improving our knowledge, and to provide anonymous collated market research findings to our funding bodies.

### Current Odour Measurement in Practice

**1. How do you rate your current level of knowledge about odour measurement and monitoring techniques?**

○ Very poor    ○ Poor    ○ Fair    ○ Good    ○ Very good

**2. What frequency best reflects how often you deal with odour-related problems?**

○ daily    ○ weekly    ○ monthly/seasonally    ○ yearly    ○ never

**3. In dealing with odour problems, what techniques and instrumentation do you use to measure and/or monitor odour?**

☐ Human nose                     ☐ Field panellists

☐ Olfactometry                   ☐ Nasal Rangers

☐ GC-MS                          ☐ eNOSE

☐ Other (please specify)

**4. Thinking about previous odour assessments, if any, done in the lab (e.g. olfactometry) or in the field (e.g. field panellists, Nasal Rangers), how do you rate the overall reliability of the odour data obtained?**

|  | Poor | Fair | Good | Very good | Excellent | Not applicable |
|---|---|---|---|---|---|---|
| Lab results | ○ | ○ | ○ | ○ | ○ | ○ |
| Field results | ○ | ○ | ○ | ○ | ○ | ○ |

**5. Thinking about previous odour assessments, if any, how do you rate the overall cost-effectiveness of odour measurements in the following categories?**

|  | Poor | Fair | Good | Very Good | Excellent | Not applicable |
|---|---|---|---|---|---|---|
| Laboratory | ○ | ○ | ○ | ○ | ○ | ○ |
| Field | ○ | ○ | ○ | ○ | ○ | ○ |

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# Towards Electronic Nose (eNOSE) Solutions for On-site Odour

**6. Thinking about previous odour assessments, if any, how do you rate the overall usefulness of odour measurements in performing your work?**

| | Poor | Fair | Good | Very Good | Excellent | Not applicable |
|---|---|---|---|---|---|---|
| Lab results | ◯ | ◯ | ◯ | ◯ | ◯ | ◯ |
| Field results | ◯ | ◯ | ◯ | ◯ | ◯ | ◯ |

## Looking for alternative options?

We now wish to determine your interest in field-based odour measurement and monitoring using eNOSE devices, also known as artificial olfaction systems.

An eNOSE utilises an array of non-specific chemical sensors and pattern recognition techniques to identify a target odour's "fingerprint".

In the field, an eNOSE should be able to discriminate the target odour from other ambient odours. Furthermore, it should be able to measure odour concentration.

### 7. How familiar are you with eNOSE technology?

◯ Not at all

◯ Somewhat

◯ Moderately

◯ Very

◯ Extremely

### 8. Have you used an eNOSE system before?

◯ Yes

◯ No

If yes, please elaborate?

[text box]

## Design your ideal eNOSE system.

Your needs are our needs. From a practical perspective, please think about your ideal in-field odour monitoring capability.

In relation to eNOSE technology, the following **definitions** apply:

**Sensitivity** - the minimum magnitude of the input signal required to produce a meaningful output signal, after taking any "noise" into account.

**Reproducibility/precision** - ability to achieve an accurate measurement of the same odour in independent tests.

**Accuracy** - degree of closeness of the measured odour to the actual (true) value as determined by standard procedure using a human panel i.e. olfactometry.

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**Portability** - general characteristic of being readily transportable from one location to another.

**Price** - cost of retail item.

**9. Consider the definitions above, then rank these five factors in order of importance.**

**N.B. This question requires a unique ranking for each factor. You will not be able to rank two or more factors equally, so will need to make a value judgement.**

|  | 1 (most) | 2 | 3 | 4 | 5 (least) |
|---|---|---|---|---|---|
| Sensitivity | O | O | O | O | O |
| Reproducibility/precision | O | O | O | O | O |
| Accuracy | O | O | O | O | O |
| Portability | O | O | O | O | O |
| Price | O | O | O | O | O |

**10. An odour unit (OU) is the standard unit of measurement for odour concentration. The higher the number of odour units the stronger the odour. What level of sensitivity would you find acceptable for your purposes?**

- O 2.5 OU
- O 10 OU
- O 20 OU
- O 50 OU
- O Not applicable
- O Do not know/No opinion
- O Other (please specify)

**11. What level of accuracy would you find acceptable for your purposes?**

- O +/- 5%
- O +/- 10%
- O +/- 20%
- O +/- 30%
- O Do not know/No opinion
- O Not applicable
- O Other (please specify)

**12. What level of precision would you find acceptable for your purposes?**

- O +/- 5%
- O +/- 10%
- O +/- 20%
- O Do not know/No opinion
- O Not applicable
- O Other (please specify)

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**13. What do consider a manageable weight for field use?**

◯ 20 kg      ◯ 10 kg      ◯ 5 kg      ◯ 2 kg      ◯ 1 kg      ◯ No opinion

◯ Other (please specify)

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│                     │
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**14. Taking into account the performance level you require, what price would be prepared to pay (Australian dollars) for an eNOSE that meets your needs?**

◯ Under $5,000           ◯ $7,500           ◯ $15,000

◯ $5,000                 ◯ $10,000          ◯ Not applicable

**15. You generally need to record your sampling locations. How useful would it be to you if a GPS (Global Positioning System) capability was incorporated into the eNOSE?**

◯ Not useful      ◯ Somewhat useful      ◯ Useful      ◯ Very useful      ◯ No opinion

Comments

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┌─────────────────────┐ ▲
│                     │
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**16. Meteorological conditions can influence odour emissions. How useful would it be to you if meteorological data collection was incorporated into the eNOSE?**

◯ Not useful      ◯ Somewhat useful      ◯ Useful      ◯ Very useful      ◯ No opinion

Comments

```
┌─────────────────────┐ ▲
│                     │
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**17. Would you consider using more than one eNOSE at a site, e.g. to measure odour simultaneously from different directions to the odour source, or taking into account prevailing wind directions and surrounding topograghy?**

◯ Yes           ◯ Maybe           ◯ No           ◯ Not applicable

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**18. Which of the following specific chemicals do you consider important to measure real-time, in conjunction with odour measurements?**

☐ CO2 (Carbon Dioxide)

☐ NOx (Nitrous oxides e.g. NO, NO2, N2O)

☐ NH3 (Ammonia)

☐ H2S (Hydrogen Sulphide)

☐ VOCs (Volatile Organic Compounds)

☐ Not applicable/no opinion

☐ Other (please specify)

## Odour services and cost

This section is related to your past expenditure, if any, in relation to odour assessments.

**19. In the past, which of the following services have you used?**

☐ Olfactometry

☐ Neighbourhood surveys

☐ Field assessments with panellists

☐ Gas chromatography-mass spectrometry

☐ Other (please specify)

**20. Would you consider purchasing an in-field odour monitoring device if commercially available?**

◯ Yes

◯ Maybe

◯ No

**21. Would you consider renting/hiring an in-field odour monitoring device?**

◯ Yes

◯ Maybe

◯ No

**22. What would be the maximum price (Australian dollars) you would be prepared to pay for a device capable of:**

- **discriminating a target odour in a mixture of odours;**
- **measuring odour concentration (in odour units) real-time;**
- **operating semi-continuously; and**
- **operating on battery power**

○ < $5000

○ $5000

○ $7500

○ $10000

○ $15000

○ >$15000

**23. What do you consider the single most important factor as to whether you would buy an eNOSE?**

**Just recapping:**

**Accuracy - degree of closeness of the measured odour to the actual (true) value as determined by standard procedure using a human panel i.e. olfactometry.**

**Sensitivity - the minimum magnitude of the input signal required to produce a meaningful output signal, after taking any "noise" into account.**

**Reproducibility/precision - ability to achieve an accurate measurement of the same odour in independent tests.**

○ Accuracy

○ Sensitivity

○ Reproducibility/precision

○ Cost

○ Other (please specify and explain why)

**24. What type of emissions would you be likely to use eNOSE technology for?**

☐ Intensive livestock (e.g. piggeries, feedlots, dairies, poultry farms)

☐ Industrial (e.g. tanneries, abattoirs, food manuafacturers, pulp mills)

☐ Waste management (e.g. waste water treatment plants, landfill sites)

☐ Not applicable

☐ Other (please specify and explain why)

**25. If you wish to comment further about your current or potential odour assessment needs, please do so below.**

## Categorising potential users

Finally, for classification purposes please tell us about yourself.

**26. What best describes your occupation?**

○ Primary producer e.g. farmer

○ Environmental management/non-primary production/waste management e.g. wastewater treatment, abattoir

○ Regulator

○ Researcher

○ Consultant/odour assessment service provider

○ Other (please specify)

**27. What best describes your business/organisation?**

○ Farm

○ University/College/Research Institute

○ Industrial/commercial enterprise

○ Government

○ Other (please specify)

[text box]

**28. What's your location?**

City/Town: [____]

State: [____]

Country: [____]

**29. Which of the following best describes your odour assessment requirements?**

○ Infrequently (e.g. no more than once or twice a year)

○ Occasionally (e.g. once a month)

○ Regularly (e.g. every week)

○ Not applicable

○ Other (please specify)

[text box]

**30. The following are optional. What are your contact details?**

Name: [____]

Company: [____]

Email Address: [____]

Phone Number: [____]

**Thanks!**

Thanks for your time and feedback. It is greatly appreciated.

We will do our best to meet your needs.

Please click the **DONE** button when ready to submit.

# Appendix B.   GC-MS report

Dr Gavin Parcsi
UNSW Odour Laboratory
Water Research Centre
School of Civil & Environmental Engineering
The University of New South Wales
Sydney, NSW, AUSTRALIA

**NMVOC Speciation of Poultry House Emissions with Thermal Desorption**

### Introduction

The following report summarises the results from the non-methane volatile organic compound assessment of sorbent tubes as supplied to the Atmospheric Emissions & Odour Laboratory of the Water Research Centre (UNSW, Sydney Australia) and discusses the implications of these results to the globally perceived odour. The experimental methodologies engaged for this work were based upon extensive work performed for unrelated poultry emissions projects. The validation of the laboratory methods had previously yielded significant reliable, reproducible, and robust data; however the most significant result of all previous work emphasised the variability of the emissions. The identification of the key odorant (2,3-butanedione) within the samples collected for this project reflect the dominance of this compound within other broiler house emissions. The presence of sulphides within the TD-GC-MS results is indicative of the potential for the presence of hydrogen sulphide and methyl mercaptan at the source of the emissions; however without the use of reliable sulphur gas sample collection and analysis methodologies the accurate identification and quantification of the sulphur species is not possible.

### Methodology

Conditioned and cleaned sorbent tubes (Tenax TA) were supplied to Queensland DAFF (Australia). Field sampling was the responsibility of DAFF Queensland. The sampled tubes were returned to the AE&O Lab and analysis was undertaken. It should be noted that the methodology of sampling differed from that of previous works and was not under the control of the AE&O laboratory. It was understood that the samples collected were all of approximately 3 L in volume.

Thermal desorption-gas chromatography-mass spectrometry was used for the chemical speciation of the sorbent tubes; the parameters were as follows.

Thermal Desorption was used to liberate the analytes captured on the sorbent tubes, followed by secondary trapping on a sub-ambient narrow bore quartz general purpose graphitised carbon sorbent tube (*cold trap*) prior to rapid heating of the cold trap to inject an analyte *slug* into the gas chromatogram. Tube Desorb 5 min at 275 ℃, Cold Trap at -10 ℃, Trap Desorb -10 ℃ to 290 ℃ @ 40 ℃.min$^{-1}$ hold 290 ℃ for 5 minutes.

Gas Chromatograph: Oven Program 2 min at 50 ℃, increase @ 10 ℃.min$^{-1}$ to 175 ℃, followed by an increase @ 25 ℃.min$^{-1}$ to 225 ℃, 2 min hold (18.5 min total Run Time)

The separation of the compounds was on an intermediate polarity GC Column (J&W Scientific DB-VRX 30 m x 0.250 mm x 1.40 μm).

The identification of analytes within the matrix was performed by mass spectrometry with the mass spectrometer operating in constant scan mode (35 - 335 m/z) with a 1.25 minute solvent delay.

Spectra matching NIST 4.0 and Wiley375 databases and quantification by standard injections.

The results of the individual samples have previously been supplied and will not be reproduced in this document. Amount of analyte on the tube were calculated to the nearest ng (as quantified through standard calibration); a summary table is presented which includes the dominant compounds, the quantities of the compounds presented are expressed in ppb for comparability to results generated by the other technologies. Odour detection thresholds (where available) are also presented to infer potential for given compounds to contribute to the perceived odour.

It is imperative to stress that the inclusion of sulphur compounds within the results would be erroneous as the methodology of sample collection and analysis does not favour the accurate reproduction of the sulphur species that are present at the source of the emission. It has been demonstrated that the thermal degradation of thiols (mercaptans) to disulphides yields unreliable quantification of both parent (thiol) and daughter (disulphides) compounds. Similarly the use of Tenax TA sorbent tubes precludes the accurate analysis of nitrogenous species (trimethylamine, indole, skatole.)

## Results

The results of the samples collected in Queensland reflected those seen in previous studies, with due consideration given to the extensive variability previously observed, the rationale and implication of these findings are discussed in the following text. As observed in previous work (Poultry CRC Project 04-45 Dust and Odour) there is a significant variability in the non-methane volatile organic compound emissions between facilities, temporally, seasonally, and with respect to the bird density. Without consistency of sampling methodology it would not be possible to compare quantifiably the results from different projects. It is stressed that the inference of results of sampling events being comparable would be erroneous.
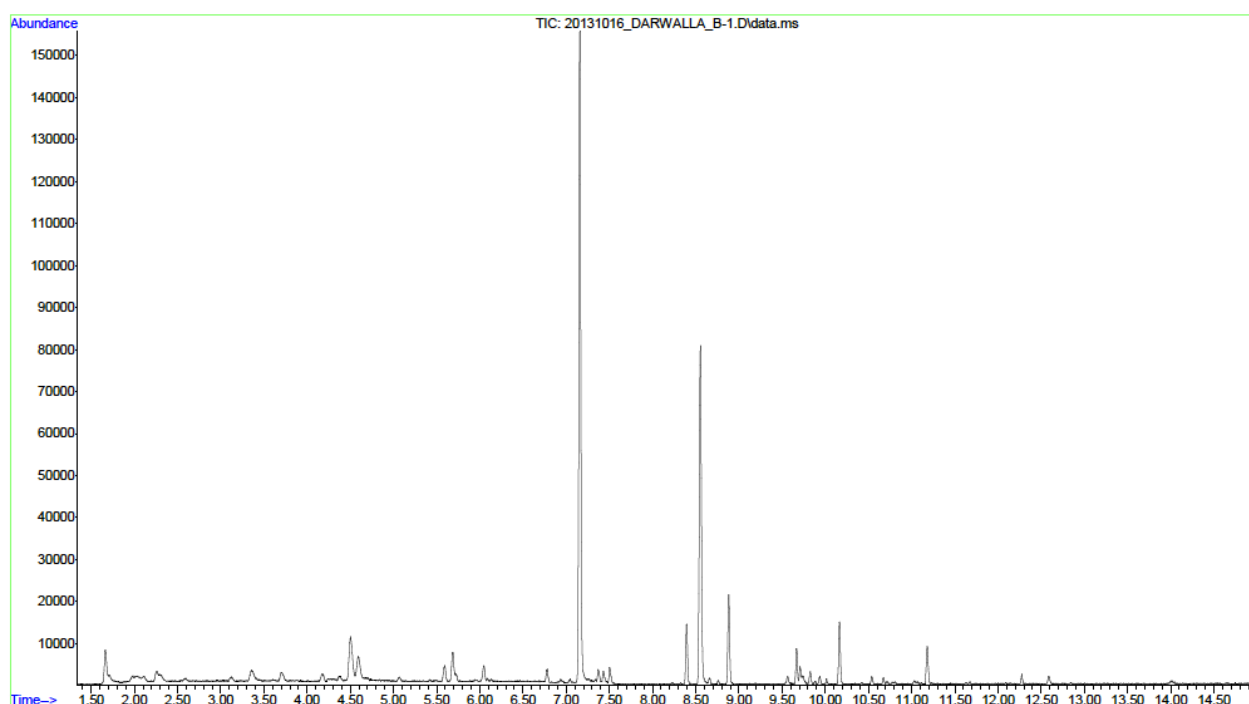


**Figure 1**. **Typical total ion chromatogram obtained from sorbent tube sample collected from a poultry house in Queensland.**

**Table 1. Summary of the results obtained from the field sampling campaigns.**

| Compound | Chemical Formula | Maximum Concentration (ppb) | Odour Detection Threshold (ppm) |
|---|---|---|---|
| 3-hydroxy-2-butanone | C4H8O2 | 64 | Unknown |
| Acetic acid | CH3COOH | 64 | 13 |
| 2,3-Butanedione | C4H6O2 | 58 | 8.6 **ppb** |
| Trichlorofluoromethane | CCl3F | 13 | 45 |
| Acetone | C3H6O | 13 | 20 |
| Toluene | C7H8 | 5 | 1.6 |
| 2-methyl-butane | C5H12 | 3 | Unknown |
| Xylenes | C8H10 | 2 | 1.1 |
| Tetramethylbenzene | C10H14 | 2 | 0.1 |
| 2-butanone | C4H8O | 2 | 5.4 |
| Camphene | C10H16 | 2 | Unknown |
| Cymene | C10H14 | ~1 | Unknown |
| γ-Terpinene | C10H16 | 1 | Unknown |
| Benzene | C6H6 | ~1 | 4.68 |
| Ethylbenzene | C8H10 | ~1 | 2.3 |
| Trimethylbenzene | C9H12 | ~1 | 2.4 |
| Nonanal | C9H18O | ~1 | 0.53 **ppb** |

*Sulphur compounds (dimethyl sulphide. dimethyl disulphide, dimethyl trisulphide, and sulphur dioxide) were identified within the samples; however the methodology precludes accurate quantification.

## Discussion

Many of the chemical compounds present within the samples were in quantities well below their reported odour threshold values: however this does not imply that they do not contribute to the global odour perceived at the source (Ryan *at al*., 2008). It is difficult to infer an accurate interpretation of the total odour perceived owing to the innate combinatorial complexities of odour interaction. It should also be noted that many of the compounds that were identified within the samples do not have

published odour detection thresholds, this is not a reflection of the compounds potential odour impact, however emphasises the difficulty in interrelating chemical quantity and odour value.

*The quantification of the species that were present it can be inferred that 2,3-butanedione and nonanal would be dominant in the perceived odour at the source as they were both present in quantities well above their reported odour detection thresholds.*

The selection of the sorbents for the analyte capture were based on literature and recommendations, however in nominating the sorbent it should be anticipated that not all analyte will have an equal affinity and therefore the retention of the specific analytes may not give a precise and accurate representation of the composition of the source matrix. The majority of the compounds identified within the matrices of the samples collected for this project were C4 ~ C10 hydrocarbons, aldehydes, alcohols, and ketones. It should be emphasised that the sorbent tubes used (Tenax TA) in the field sampling are documented for the reliable and reproducible results with C6-C32 compounds, however extensive work performed over numerous sampling campaigns have produced excellent results with compounds from ethanol ~ C16, as well as many of the chlorofluorocarbons. However it is worth noting that due to the potential analyte competition for active sites within the sorbent tube matrix some of the more volatile compounds (*id est* those with a lower boiling point, lower vapor pressure, or lower molecular mass) may not be observed in the analytical results, or may not be represented in their respective abundancies.

It should also be noted that the use of thermal desorption results in inherent inaccuracies when quantification of sulphur compounds is attempted. As previously stated; there is a strong correlation between the presence of sulphides (mono-, di-, and tri-) in the thermal desorption-gas chromatography-mass spectrometry results and the presence of thiols (mercaptans) at the source of sample collection.

The non-methane volatile organic compounds are ostensibly volatile, hence due consideration should be engaged when interpreting the results from the mass selective detector. The innate volatility of some of the species could result in the decreased yield within the analytes detected at the mass selective detector as they may have reacted within sampling lines, or even on the sorbent bed during sample collection, that is, before the sample reaches the laboratory it may have undergone transformation rendering the results of the analysis inaccurate.

It should be duly noted that as with all experimental methods there are limitations that need to be accepted when interpreting the results to ensure accurate understanding. The heating of the sorbent tubes to facilitate thermal liberation of the analytes from the sorbent bed and then the rapid heating of the cold trap within the thermal desorber places the highly labile chemical species under potential risk of thermal oxidation or breakdown.

The chemical analysis of the poultry house emissions was undertaken utilising thermal desorption, gas chromatography coupled mass spectrometry detection, whilst the chemical selectivity of the sorbents within the thermal desorption tubes has been discussed, the chemical selectivity of the analytical detector needs to be included. A mass selective detector can provide adequate levels of chemical speciation within its operating parameters, chemicals with smaller ion fragments (<35 *m/z*) may not get detected.

The intrinsic volatility of the chemical species that were being collected during the poultry house study should be accepted to influence the analytes being capture and detected from the different detectors. The fate of chemicals in the environment are subject to the environmental conditions, temperature, humidity and sunlight will all impact on the stability of these chemicals; consequently it should be anticipated that exists a potential for differences in species at the source to those that could potentially reach a receptor, and have a different olfactory impact than that of the source compound.

NMVOCs identified from the research undertaken for this study included several alcohol, aldehydes, ketones and carboxylic acids (Table 1) which could readily be related to each other through known oxidation pathways.

The oxidation of a diol (2,3-butandiol) to a hydroxy-ketone (3-hydroxy-2-butanone) to a dione (2,3-butanedione) is similar to the oxidation of a secondary alcohol, however there is a second hydroxy functional group that can potentially oxidise, thus forming an additional intermediate. Although 2,3-butanediol was not detected in any of the samples it is plausible that it may be present at the source and its volatility caused it to oxidise before or during sample collection or analysis thus giving the predominant presence of 3-hydroxy-2-butanone.
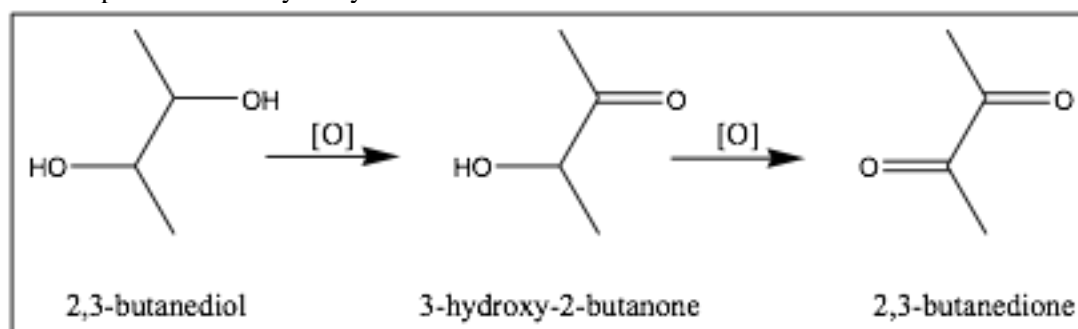


**Figure 2. Reaction pathway from diol to dione, via hydroxy ketone intermediate**.

Ergo, it is reasonable to conclude that the presence of one of these chemical species within a specific sample could potentially indicate the presence of its respectively related chemical compounds at the source.

The global odour will be composed of all the chemical species interacting in different manners, of which there is three principle interactions that the odorants can undergo; additive, antagonistic and synergistic. The additive mechanism will yield a global odour that is composed of the chemicals in their respective ratios, the antagonistic mechanism will yield a global odour that is less than the total of the components, and the synergistic mechanism will yield a global odour that is greater than the total of the individual components. Through these mechanisms it is plausible that when assessed in isolation a low impact chemical species may not be detected from the olfactory detection port, however may constitute a significant impact when combined with other odorants or non-odorous chemical species. (Ryan *et al*., 2008).

Beyond the global definition of non-methane volatile organic compound yet having an undeniable significance of presence within the analytes captured were pungent sulphur odorants of dimethyl trisulphide, dimethyl disulphide and dimethyl sulphide. A posteriori speculation tends to the presence of thiols (methyl mercaptan, hydrogen sulphide - known potent disagreeable odorants) from the source emissions; thermal desorption methodologies do not favour these highly labile sulphur species.

Variability and dynamicity were substantial throughout the sampling campaign and therefore development of a simulated emission would be thwart with erroneous conclusions. However having elucidated the composition of many different samples it is plausible that the emissions from other poultry houses will comprise a similar family of chemical compounds, with emphasis upon the nuisance compounds notably the aldehydes (nonanal) and ketones (2,3-butanedione).

### References

Ryan, D, Prenzler, PD, Saliba, AJ, and Scollary, GR. 2008. The significance of low impact odorants in global odour perception. *Trends in Food Science & Technology* 19(7):383-389.

# Appendix C   GC-MS sample details

**Table C1   GC-MS sample details. Samples are listed as their duplicate pairs.**

| Sample Tube | Sample Date | Source Reference | Farm | Bird age (days) | Source description |
|---|---|---|---|---|---|
| Mi 181813 | 19/09/2013 | A22 | A | 26 | downwind 23m; from bag |
| 204093 | 19/09/2013 | A22 | A | 26 | downwind 23m; from bag |
| 204082 | 19/09/2013 | A22 | A | 26 | downwind 70m; from bag |
| Mi 181826 | 19/09/2013 | A22 | A | 26 | downwind 70m; from bag |
| 204018 | 26/09/2013 | B4 | B | .. | downwind 20m |
| 204037 | 26/09/2013 | B4 | B | .. | downwind 20m |
| 204061 | 26/09/2013 | B4 | B | .. | at fan; from bag |
| Mi 181852 | 26/09/2013 | B4 | B | .. | at fan; from bag |
| 204030 | 26/09/2013 | B4 | B | .. | in-shed |
| 204010 | 26/09/2013 | B4 | B | .. | in-shed |
| Mi 181808 | 2/10/2013 | A4 | A | 5 | in-shed |
| 204091 | 2/10/2013 | A4 | A | 5 | in-shed |
| 204076 | 2/10/2013 | A32 | A | 9 | in-shed |
| 204035 | 2/10/2013 | A32 | A | 9 | in-shed |
| 204038 | 2/10/2013 | AT6 | A | na | in-shed; empty; disinfected |
| 204060 | 2/10/2013 | AT6 | A | na | in-shed; empty; disinfected |
| 204058 | 2/10/2013 | AT6 | A | na | in-shed; near-empty |
| 204033 | 2/10/2013 | AT6 | A | na | in-shed; near-empty |
| 204036 | 3/10/2013 | A25 | A | 14 | at fan |
| 204043 | 3/10/2013 | A25 | A | 14 | at fan |
| 204096 | 3/10/2013 | A14 | A | 27 | in-shed |
| 204086 | 3/10/2013 | A14 | A | 27 | in-shed |
| 204013 | 3/10/2013 | A24 | A | 34 | in-shed |
| 204024 | 3/10/2013 | A24 | A | 34 | in-shed |
| 204053 | 3/10/2013 | A21 | A | 41 | in-shed |
| 204072 | 3/10/2013 | A21 | A | 41 | in-shed |
| M 181816 | 14/10/2013 | A31 | A | 21 | at fan |
| M 181860 | 14/10/2013 | A31 | A | 21 | at fan |
| Mi 181836 | 14/10/2013 | A9 | A | 35 | in-shed |
| Mi 181861 | 14/10/2013 | A9 | A | 35 | in-shed |
| M 181846 | 16/10/2013 | A20 | A | 50 | in-shed |
| M 181871 | 16/10/2013 | A20 | A | 50 | in-shed |
| Mi 181821 | 16/10/2013 | A19 | A | 48 | at fan; from bag |
| Mi 181811 | 16/10/2013 | A19 | A | 48 | at fan; from bag |

# Appendix D  Olfactometry results

**Table D1**  **Summary of olfactometry samples and measured concentration (ou).**

| Date | Time | Farm | Bag | Concentration (ou) | Type | Bird age | Shed # | Distance downwind (m) |
|---|---|---|---|---|---|---|---|---|
| 12/04/13 | 11:16:00 | B | 2982 | 609 | in-shed | 28 | 4 | 0 |
| 12/04/13 | 11:55:00 | B | 2984 | 189 | downwind | 28 | 4 | 13 |
| 12/04/13 | 12:39:00 | B | 2985 | 152 | downwind | 29 | 5 | 18 |
| 12/04/13 | 12:14:00 | B | 2990 | 825 | in-shed | 29 | 5 | 0 |
| 22/04/13 | 10:58:00 | B | 2983 | 519 | in-shed | 38 | 4 | 0 |
| 22/04/13 | 11:26:00 | B | 2986 | 402 | downwind | 38 | 4 | 30 |
| 22/04/13 | 09:55:00 | B | 2987 | 803 | in-shed | 39 | 5 | 0 |
| 22/04/13 | 10:23:00 | B | 2992 | 416 | downwind | 39 | 5 | 18 |
| 23/04/13 | 11:45:00 | B | 2976 | 100 | downwind | 40 | 5 | 39 |
| 23/04/13 | 12:09:00 | B | 2977 | 664 | in-shed | 40 | 5 | 0 |
| 23/04/13 | 10:35:00 | B | 2978 | 594 | in-shed | 39 | 4 | 0 |
| 23/04/13 | 11:10:00 | B | 2988 | 104 | downwind | 39 | 4 | .. |
| 26/04/13 | 11:15:00 | B | 3019 | 568 | downwind | 42 | 4 | 10 |
| 26/04/13 | 12:23:00 | B | 3020 | 416 | downwind | 42 | 4 | 30 |
| 26/04/13 | 10:43:00 | B | 3021 | 609 | in-shed | 42 | 4 | 0 |
| 26/04/13 | 11:58:00 | B | 3022 | 395 | downwind | 42 | 4 | 15 |
| 30/04/13 | 09:51:00 | B | 2989 | 592 | downwind | 47 | 5 | 10 |
| 30/04/13 | 09:20:00 | B | 3001 | 579 | in-shed | 47 | 5 | 0 |
| 30/04/13 | 11:15:00 | B | 3006 | 640 | in-shed | 47 | 5 | 0 |
| 30/04/13 | 10:47:00 | B | 3014 | 499 | downwind | 47 | 5 | 10 |
| 2/05/13 | 08:44:00 | B | 3003 | 799 | in-shed | 49 | 5 | 0 |
| 2/05/13 | 10:13:00 | B | 3005 | 85 | beside | 49 | 5 | |
| 2/05/13 | 10:43:00 | B | 3007 | 565 | in-shed | 49 | 5 | 0 |
| 2/05/13 | 09:02:00 | B | 3016 | 87 | beside | 49 | 5 | 2 |
| 7/05/13 | 11:32:00 | C | 2995 | 362 | in-shed | 20 | 1 | 0.5 |
| 7/05/13 | 09:25:00 | C | 3002 | 497 | in-shed | 20 | 1 | 0 |
| 7/05/13 | 10:52:00 | C | 3010 | 47 | beside | 20 | 1 | |
| 7/05/13 | 10:06:00 | C | 3024 | 94 | downwind | 20 | 1 | 10 |
| 9/05/13 | 10:20:00 | B | 2998 | 583 | in-shed | 55 | 4 | 0.5 |
| 9/05/13 | 09:44:00 | B | 3000 | 204 | downwind | 56 | 5 | 28 |
| 9/05/13 | 09:05:00 | B | 3036 | 395 | in-shed | 56 | 5 | 0 |
| 9/05/13 | 11:05:00 | B | 3037 | 57 | downwind | 55 | 4 | 33 |
| 15/05/13 | 09:54:00 | C | 2999 | 242 | downwind | 28 | 1 | 20 |
| 15/05/13 | 09:15:00 | C | 3008 | 596 | in-shed | 28 | 1 | 0 |
| 15/05/13 | 10:30:00 | C | 3038 | 39 | downwind | 28 | 1 | 35 |
| 15/05/13 | 11:17:00 | C | 3039 | | beside | | | |
| 16/05/13 | 09:22:00 | C | 3032 | 23 | downwind | 27 | 2 | 65 |
| 16/05/13 | 09:54:00 | C | 3035 | 42 | downwind | 29 | 3 | 70 |
| 16/05/13 | 16:20:00 | C | 3041 | 627 | in-shed | 29 | 1 | 0 |

| Date | Time | Farm | Bag | Concentration (ou) | Type | Bird age | Shed # | Distance downwind (m) |
|------|------|------|-----|-----|------|------|------|------|
| 16/05/13 | 10:38:00 | C | 3042 | 24 | downwind | 29 | 3 | 140 |
| 21/05/13 | 10:32:00 | C | 3023 | 95 | downwind | 34 | 2 | 25 |
| 21/05/13 | 11:19:00 | C | 3034 | 95 | beside | 34 | 1 | |
| 21/05/13 | 09:10:00 | C | 3040 | 542 | in-shed | 34 | 1 | 0 |
| 21/05/13 | 09:43:00 | C | 3044 | 287 | downwind | 34 | 1 | |
| 23/05/13 | 10:32:00 | C | 2996 | 373 | downwind | 36 | 2 | 25 |
| 23/05/13 | 08:23:00 | C | 3055 | 693 | in-shed | 36 | 1 | 0.5 |
| 23/05/13 | 09:04:00 | C | 3057 | 362 | beside | 36 | 1 | |
| 23/05/13 | 09:48:00 | C | 3058 | 596 | in-shed | 36 | 1 | 0.5 |
| 23/05/13 | 11:07:00 | C | 3060 | 693 | in-shed | 34 | 4 | 0.5 |
| 28/05/13 | 09:26:00 | B | 3051 | 206 | beside | 4 | 4 | |
| 28/05/13 | 11:10:00 | B | 3053 | 142 | beside | 4 | 4 | |
| 28/05/13 | 08:39:00 | B | 3056 | 478 | in-shed | 4 | 5 | 0 |
| 28/05/13 | 10:31:00 | B | 3059 | 395 | beside | 4 | 4 | |
| 30/05/13 | 10:54:00 | C | 3049 | 38 | beside | 43 | 1 | |
| 30/05/13 | 11:22:00 | C | 3052 | 223 | beside | 43 | 1 | |
| 30/05/13 | 10:02:00 | C | 3061 | 549 | in-shed | 43 | 1 | 0 |
| 30/05/13 | 12:00:00 | C | 3063 | 103 | beside | 43 | 1 | |
| 4/06/13 | 09:24:00 | B | 3012 | 76 | downwind | 11 | 4 | 5 |
| 4/06/13 | 10:25:00 | B | 3046 | 883 | in-shed | 11 | 4 | 0 |
| 4/06/13 | 08:53:00 | B | 3050 | 41 | downwind | 11 | 4 | 30 |
| 4/06/13 | 11:16:00 | B | 3062 | 81 | downwind | 11 | 4 | 50 |
| 6/06/13 | 11:20:00 | C | 2997 | 42 | beside | 49 | 1 | |
| 6/06/13 | 08:35:00 | C | 3033 | 76 | downwind | 49 | 1 | 13 |
| 6/06/13 | 09:09:00 | C | 3047 | 328 | downwind | 49 | 1 | 20 |
| 6/06/13 | 09:56:00 | C | 3048 | 475 | downwind | 48 | 4 | 16 |
| 6/06/13 | 10:44:00 | C | 3054 | 538 | in-shed | 49 | 1 | 0 |
| 13/06/13 | 10:27:00 | C | 3011 | 226 | downwind | 55 | 4 | 20 |
| 13/06/13 | 08:56:00 | C | 3026 | 1002 | in-shed | 54 | 2 | 0 |
| 13/06/13 | 09:35:00 | C | 3027 | 378 | downwind | 55 | 5 | 10 |
| 13/06/13 | 11:40:00 | C | 3064 | 431 | downwind | 55 | 4 | 4 |
| 5/07/13 | 11:08:00 | B | 3087 | 133 | downwind | 42 | 3 | 55 |
| 5/07/13 | 09:47:00 | B | 3089 | 189 | downwind | 42 | 4 | 50 |
| 5/07/13 | 10:30:00 | B | 3090 | 1097 | in-shed | 42 | 4 | 0.5 |
| 6/09/13 | 11:42:00 | A | 3097 | 443 | downwind | 14 | 21 | 9 |
| 6/09/13 | 09:34:00 | A | 3098 | 1407 | in-shed | 13 | 22 | 0 |
| 6/09/13 | 10:28:00 | A | 3100 | 1491 | in-shed | 14 | 21 | 0 |
| 6/09/13 | 10:45:00 | A | 3101 | 724 | downwind | 13 | 22 | 11 |
| 19/09/13 | 09:51:00 | A | 3094 | 156 | .. | | | |
| 19/09/13 | .. | A | 3095 | 443 | in-shed | 41 | 22 | 0 |
| 19/09/13 | 09:30:00 | A | 3102 | 176 | downwind | 26 | 22 | 70 |
| 4/10/13 | 12:44:00 | A | 3016 | 192 | in-shed | 42 | 21 | 0 |
| 4/10/13 | 12:00:00 | A | 3045 | 197 | in-shed | 42 | 21 | 0 |

| Date | Time | Farm | Bag | Concentration (ou) | Type | Bird age | Shed # | Distance downwind (m) |
|------|------|------|-----|--------------------|------|----------|--------|-----------------------|
| 4/10/13 | 13:01:00 | A | 3104 | 181 | downwind | 42 | 21 | |
| 4/10/13 | 09:53:00 | A | 3109 | 147 | downwind | 42 | 21 | 48 |
| 9/10/13 | 12:05:00 | A | 3084 | 194 | downwind | 47 | 21 & 22 | |
| 9/10/13 | 13:10:00 | A | 3096 | 287 | in-shed | 47 | 21 | 0.5 |
| 9/10/13 | 11:29:00 | A | 3105 | 106 | downwind | 47 | 21 & 22 | 10 |
| 9/10/13 | 10:42:00 | A | 3107 | 87 | downwind | 47 | 21 & 22 | 12 |
| 9/10/13 | 09:47:00 | A | 3110 | 144 | downwind | 47 | 21 & 22 | 27 |
| 10/10/13 | 11:35:00 | A | 3072 | 140 | downwind | 47 | 21 & 22 | |
| 10/10/13 | 12:30:00 | A | 3078 | 108 | downwind | 47 | 21 & 22 | |
| 10/10/13 | 10:25:00 | A | 3111 | 319 | in-shed | 47 | 22 | 0.5 |
| 16/10/13 | 09:43:00 | A | 3130 | 362 | in-shed | 50 | 20 | 0 |
| 16/10/13 | 11:00:00 | A | 3132 | 650 | in-shed | 48 | 19 | 0.5 |
| 16/10/13 | 11:40:00 | A | 3133 | 650 | downwind | 48 | 19 & 20 | 10 |

Artificial olfaction system for on-site odour measurement

By Michael Atzeni, Craig Lobsey, David Mayer and Gavin Parcsi

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