3. Researches and Developments on Odor Measurement

Development of the Next Generation Dynamic Olfactometer – Dynascent

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Keywords

Odour, dynamic olfactometer, calibration

Abstract

The introduction of an international standard on odour measurement presents a new challenge for olfactometry laboratories to meet tough new instrumental performance and panellist performance criteria. The paper reviews a number of olfactometers used in the last ten years both in Australia and overseas. It has found that the back pressure could contribute a significant error in the delivery of the required dilution ratio and the contamination of the olfactometer was the source of large variation in olfactometry results. The paper discusses the instrumental performance of the newly developed olfactometer. DynaScent is a fourth generation olfactometer which uses no flow measurement device (flowmeter or mass flow controller). The dilution ratios of the DynaScent were calibrated using CO gas and auto calibration feature. The effects of the CO sampling point locations (within the system and above the sniffing cup) could have significant impacts on the dilution ratio. Practically, the manual calibration is both time consuming and labour intensive. Furthermore, it was found that the accuracy and instability results were sensitive to the selection of the CO gas range. It is concluded that the calibration of a dynamic olfactometer should be carried out more frequently than once a year. The DynaScent olfactometer was able to achieve averaged instability of 1.7% and accuracy of 9% over the dilution range of 2 - 65000.

1. INTRODUCTION

Uncertainty in odour measurement is a major concern to environmental regulators, researchers, and stakeholders[1]. The large variations in odour concentrations have limited the use of dynamic olfactometry results in the regulations in the United States and European countries. In some studies, the olfactometry method was considered to be "comparable" within the studies and was not compatible to other studies due to different olfactometry standards being used. The reliability of odour measurements has often been debated in environmental courts. Odour is the most contentious issue in environmental regulations.

Berglund *et al*[2] suggested that "*A substantial proportion of the large variation attributed to the observers in odor studies, originates from olfactometric malperformance*". A more recent study at the University of Minnesota indicated that "*Sample and supplemental airflow rates were significantly different at the beginning and end of a typical session*" with their mass flow based olfactometer[3]. Jiang also confirmed that the ability of dynamic in delivering the required dilution radio over many sessions of

odour testing is one of the two critical factors and suggested that the instrumental calibration should be carried out in more frequent than once per year[4].

The importance of instrument calibration to determine the dilution ratio and panel management to monitor individual panellist performance over standard odorant (such as n-butanol) is also disputed among the professionals. Most commercial olfactometers did not produce the instrumental accuracy and instability from the tracer gas calibration results over the entire dilution range as required in the standards. van Harreveld et al. reviewed 20 years of olfactometry development in Europe and concluded that panel selections and span adjustment were the only two "crucial" steps[5]. Span adjustment meant that "laboratories could adjust their method to achieve the same value for the odour unit: 1 ou/m^3 = 40 ppbv n-butanol." Jiang noted that meeting the instrumental performance and panellist performance criteria was a major challenge for olfactometry laboratories in the implementation of the Australian standard[1].

The calibration of any analytical instrument is the first step toward producing reliable testing results. Odour measurement is no exception and instrumental calibration is the most important step in controlling uncertainty during the measurement to ensure the delivery of the required dilution ratio. There are two assessment criteria. The accuracy of the dilution apparatus ensures that the dilution ratio between five series is delivered within 20% of the set dilution ratio. The instability of the dilution apparatus ensures the same dilution ratio is given to all the panellists within 5% of the expected values in the same dilution step[6, 7].

Unfortunately, some olfactometers are not so easy to calibrate. For example, those olfactometers that use sniffing masks have some difficulties in confirming the dilution ratio at the time when the panellist is sniffing. Consequently, an uncalibrated olfactometer undoubtedly affects the panel selection and results in the panellists' making inappropriate assessment of the odour. As a result, the odour concentrations measured using the uncalibrated olfactometer might suffer from poor accuracy and instability.

During the 1980s, considerable effort was made in developing olfactometric odour measurement techniques in the Netherlands and elsewhere in Europe[8, 9]. Initially, the application of these olfactometric results was limited mainly to comparing odour emissions from various manure treatment systems in intensive animal production. In 1985, the Victoria EPA in Australia first introduced legislation based on olfactometer results and air dispersion model (Ausplume). In North America, despite earlier interest in olfactometric measurement techniques during the 1970s, it was not until the mid 1990s that North American universities set up olfactometry laboratories to investigate odour from animal production.

The development of olfactometric measurement techniques continued in Europe and resulted in the introduction of the first draft European Standard for odour measurement

by dynamic olfactometer. In Australia, a national workshop on odour measurement standardization was held in 1997 and consensus was reached to adopt the draft European standard. In 2001, Australia published the first official standard ahead of European countries. European countries have officially agreed to adopt the CEN standard in early 2003.

In summary, most olfactometers currently used around the world can be categorised in three groups on the basis how the dilution is achieved:

- static method (syringe method in USA, triangle bags in Japan);
- rotameter/fixed orifice based olfactometers (VIC. EPA B2 in Australia, TO7 in Germany, IITRI in USA) and
- Mass Flow Controller (MFC) based olfactometers (Ac'scent olfactometer in USA, Olfaktomat as used in the Netherlands).

Static method utilises the syringe to make the necessary dilution. The error in reproducing the necessary dilution ratio is so large. The flow rates at the sniffing ports are so low. The manual operation can no long meet the requirements specified in Australia and European standards.

Rotameter based olfactometers are currently used in many laboratories in Australia and elsewhere. The rotameters are extremely sensitive to downstream pressure variations that could result in errors in rotameter readings of up to 25%. Such pressure variations may be occurred during the mixing of clean air and odorous air to create the required dilution radio or subsequently during the sample presentation of the diluted sample. The latter may be accentuated by the use of an enclosed sniffing mask, adversely affecting overall performance of the olfactometer. The manual mode of operation for rotameter based olfactometers makes it impossible to meet stringent instrumental performance criteria, particularly at the high dilution ratio end of the range. Furthermore, high labour costs when using manual data input for monitoring panellist performance and in the data processing used for retrospective screening may also make compliance with the proposed standard excessively expensive.

In contrast, automated, MFC based olfactometers have demonstrated an inherent ability to comply with both instrumental and panellist performance criteria. But the MFC based olfactometers are also sensitive to the downstream pressure of the flow measurement devices. The backpressure occurring during mixing can be compensated for by instrumental calibration. However, pressure variations occurring during the sample presentation stage cannot be predicted and therefore cannot be compensated for by calibration. Backpressures can vary from panellist to panellist. In practice, the reduced flow arising from the specific personal characteristics of a panellist will be sensed by the mass flow meter resulting in the valve being further opened. However, the presentation time for each panellist is long (10 – 30 seconds) in comparison with the response time of the mass flow controller to change the valves (several seconds). These unstable conditions will be repeated many times during the

session. As a result, the actual dilutions of odour samples at the sniffing ports can be highly variable.

Furthermore, the MFCs are susceptible to contamination buildup that can alter the calibration and result in the reduced performance. The tiny space between the temperature elements inside the mass flow meter can be easily contaminated or blocked. The MFC is really designed for single component gas and better suited to a clean and non-sticky gas. In particular, the odour samples can sometime be very sticky and dusty. Therefore, MFC based olfactometers could easily suffer from the poor performance of the MFCs during the operation. This has proved to be a major limitation in the use of MFC based olfactometers. Flushing the MFCs may take hours and is not effective at all. Over a period of usage, dust and residuals eventually become irreversibly adhered to the surfaces of the temperature elements and the MFC must be replaced. The performance of MFC based olfactometer in delivering the required dilution radio can not guaranteed.

2. METHODOLOGY

The DynaScent olfactometer is a fourth generation dynamic olfactometer which is fully computer controlled and uses no flow measurement devices (The DynaScent Olfactometer, EnvironOdour Australia Pty Ltd, 2003). The sample is mixed with odourfree air within a custom designed venturi gas jet. The dilution ratios are adjusted by a series of needle valves controlled by the digital precision motion controller with ±0.0001 revolution accuracy. The repeatability of the dilution is purely based on the mechanical repeatability which is capable to reproduce highly repeatable dilution ratio. The precision needle valves can be dismantled and cleaned to minimise the effects of the contamination. The variation in the dilution ratios during the sample mixture and during the panellist sniffing is minimised by the use of the critical nozzle and the improved sniffing cups.

Carbon monoxide was chosen as a tracer because CO is a non-reactive gas and because of the reliability of CO gas monitors. Due to the limited detection range in the gas monitor, a series of CO gases were used so that the final gas concentrations at the sniffing cup were within the detection range of the gas analyzer. The CO gas was loaded as a normal sample and placed within the sampling drum. A Monitor Labs 9830 CO analyzer (range from 0 to 200 ppm), calibrated by an accredited laboratory, was used to determine the CO concentration at the sniffing cup.

The following table lists the range of CO gases used in the calibration.

Table 1 CO concentrations at source and cup

Dilution step	Dilution range	CO conc. at source, ppm	Expected CO conc. at cup, ppm
1 – 4	2 – 16	162	81 – 10
5 – 8	32 – 256	4,840	150 – 20
9 – 12	512 – 8,192	100,000	195 – 24
13 – 19	16,384 – 524,288	1,000,000	122 – 2

The signal output of the CO analyzer was connected into the DynaScent olfactometer for direct display of CO readings and data logging. The Dynascent automatically starts a dilution step, takes 10 CO readings over 100 seconds (one reading every 10 seconds), then moves to the next dilution step and takes 10 CO readings, until all the steps are completed. This process is then repeated five times. The CO results are used to calculate the accuracy and instability of the olfactometer as per the Australian and New Zealand standard[6].

Figure 1 Calibration setup

Figure 1 illustrates the calibration setup. A second CO monitor was used to monitor the CO concentration in the room to keep it below 15 ppm at all times for safety reasons. A flow meter with a range of 4 – 40 Liters Per Minute (LPM) was used to calibrate the flow rate at the sniffing ports. Before the calibration of the dilution ratio, the flow rates were checked to be 20 LPM at both sniffing ports before and after the completion of the dilution adjustment.

3. RESULTS AND DISCUSSION

Instrumental calibration of an olfactometer is time consuming and labour intensive. Calibration of the olfactometer involves hundreds of measurements (950 single measurements comprising 50 measurements for each dilution step for a 19-step

olfactometer). The process could take up to five days if adjustment of the instrument setting is required for a MFC based olfactometer. Consequently, the inclusion of an automated calibration feature in the olfactometer is necessary.

The location of the sampling tubing could have an effect on the CO readings. As shown in Figure 2, CO concentrations at the sniffing cups (Figure 2, right) were 40% smaller than those collected in the tubing (Figure 2, left) which was below the sniffing cup. Initially, it was suspected that extra gas went into the gas analyzer to cause the large bias. A bulb flow meter was used to measure the sampling rate of the CO analyzer. It was found that there was no difference between these two arrangements. The only possibility was that the open space in the cup might dilute the CO concentrations. In this study, the sampling at the sniffing cup was used.
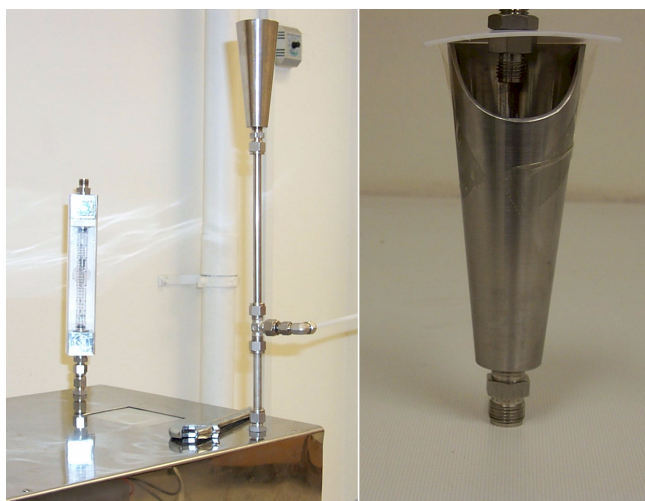

Figure 2   Sampling arrangement

Figure 3 shows the consecutive CO concentrations for dilution steps 5 – 8 over 45 minutes. The results are distributed over a narrow range which depends on the expected concentrations. The results show a higher CO variation (130 – 162 ppm) at the higher expected CO concentration (150 ppm) and a lower CO variation (19 – 22 ppm) at the lower expected CO concentration (20 ppm). These variations were likely caused by the CO gas analyser.

The accuracy (expressed as a bar chart) and instability (expressed as a single line chart) of the dynamic olfactometer are shown in Figure 4. The results show an excellent instability of 1.7% for the dilution step of 1 - 16 and an increased instability for the dilution step of 17 - 19. This was caused by the small CO concentrations, less than 10 ppm. The averaged accuracy of the olfactometer is 9% over the range of 2 - 65000. The accuracy of the instrument seems to be more sensitive to the absolute CO concentration levels at the sniffing cup.
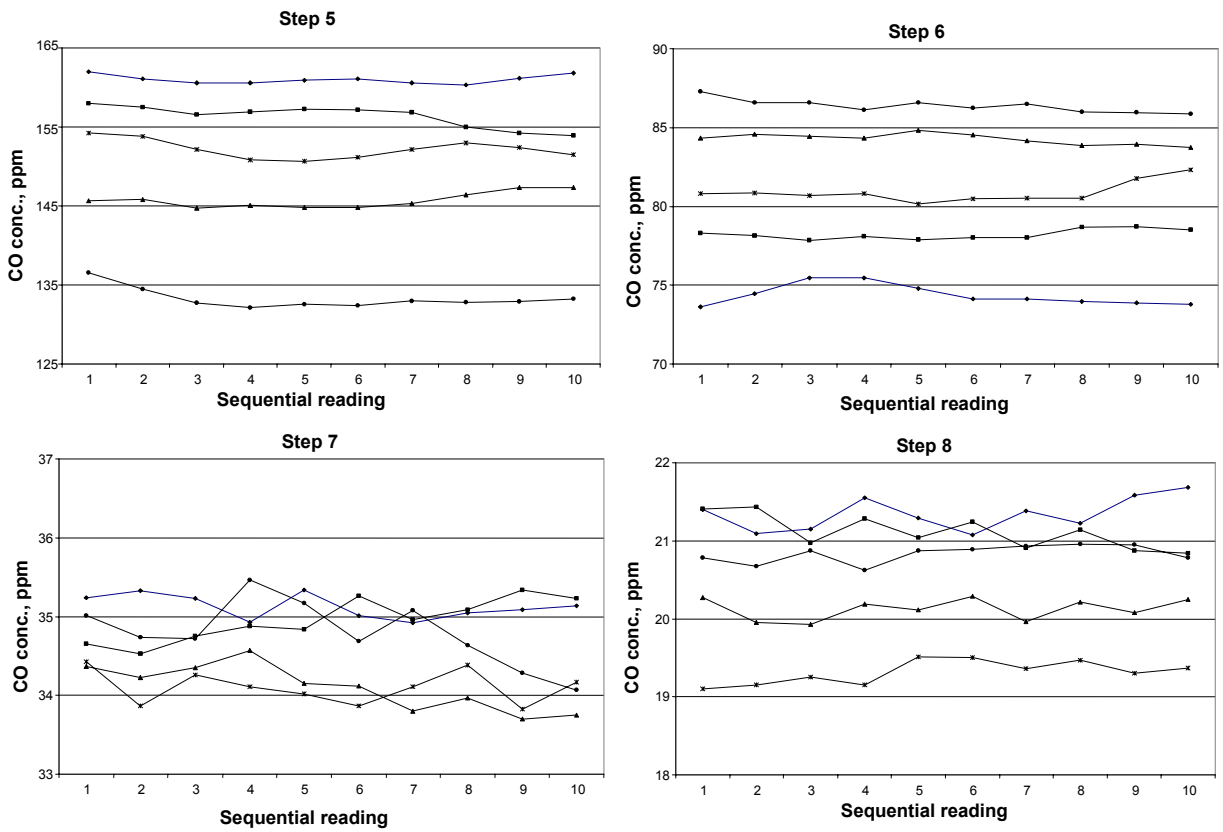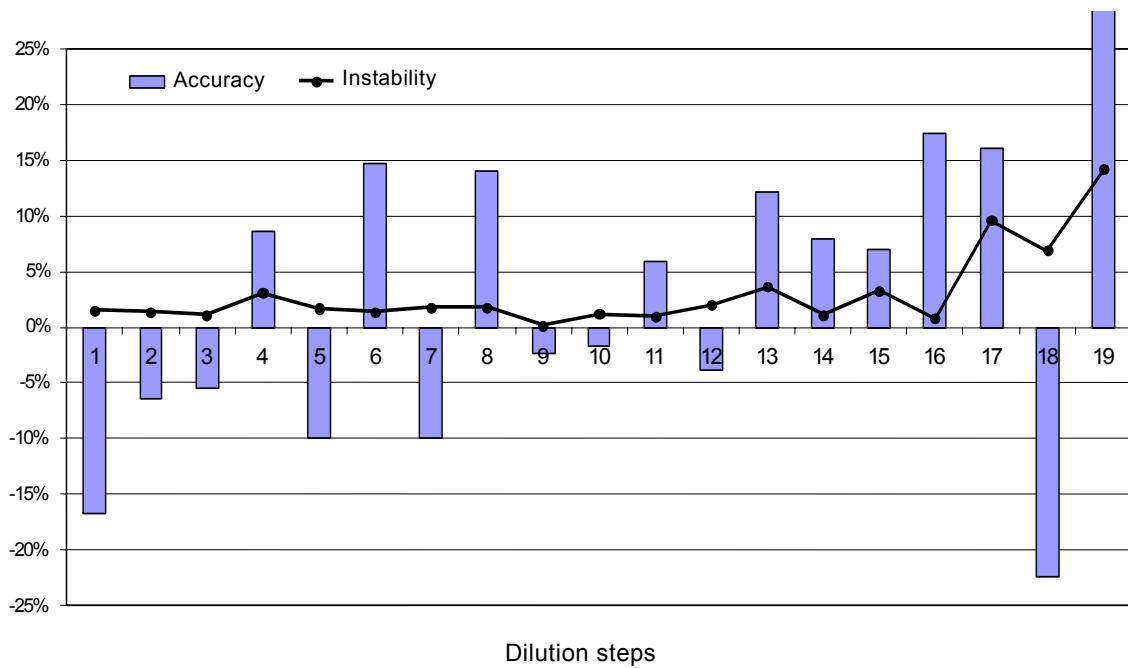
Figure 3 CO concentrations at dilution steps 5 – 8

Figure 4 Accuracy and instability of the dynamic olfactometer

4. CONCLUSIONS

Instrumental calibration is a fundamental issue for dynamic olfactometers in addressing uncertainty. Unfortunately, the calibration of such instruments is both time consuming and labour intensive. It is understood that most laboratories around the world do not perform the calibrations of dilution ratios. Some olfactometers do not have a feature to allow the end-users to adjust the olfactometer.

The performance of the flow meter and MFC based olfactometers was found to degrade over time. This could be caused by the accumulation of dirt on the contact surfaces and the change of operating conditions. The calibration of a dynamic olfactometer should be carried out more frequently than once a year as suggested in the standard. A procedure should be in place to check the accuracy of the olfactometer frequently so that the performance of the olfactometer can be monitored. A full instrumental calibration should be carried out if necessary.

The study discussed several important aspects of instrumental calibration. The selection of sampling points during the calibration might produce different calibration results. Sampling within the sniffing cup is recommended since the arrangement is similar to the nose's position during the sniffing. The selection of CO gas concentrations can also affect the measured accuracy and instability. The fact that the calibration results are subject to the selection of the CO concentrations suggested that the use of CO to calibrate the olfactometer may not be the best technique. A more sensitive analytical instrument with sensitivity to a low ppb level should be used.

The instrumental performance of the DynaScent olfactometer has been demonstrated by CO gas calibration. Overall, the DynaScent olfactometer could achieve an averaged instability of 1.7% and an averaged accuracy of 9% over the dilution range of 2 – 65000 (step 1 – step 16).

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