

# **Analytical Techniques For Odour Assessment**

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# **Analytical techniques for odour assessment**

(Teknik analisis untuk penilaian bau)

## **Abstract**

Assessing odour impacts of primary industries and establishing regulatory standards have been a foremost challenge to authorities due to lack of knowledge regarding odour concentration and rate of emission. Selection of an accurate, scientifically defined and proven technique with high repeatability is essential to fulfil the needs of solving odour nuisance complains and regulatory requirements. While equipment is available for the detection and quantification of selected indicator odorous gases, they do not provide complete data on odour since it is made up of more than a hundred gases. Odour measurement units that are legally acceptable must take into consideration the detection by the human nose since it is the humans that detect an odour that can be a discomfort or even health-threatening. An ideal detection method would be to combine both sensory and instrumental measurements that are usable for establishing odour units and thus regulatory requirements. This paper reviews the basic concepts of odour assessment and the range of technologies available to evaluate odour emissions from agricultural activities.

Keywords: agricultural odour, olfactometry, olfactory

## **Introduction**

The contribution of agricultural production to economic growth in Malaysia is undeniably huge. Agricultural activities are vulnerable to environmental issues, including odour emanation from livestock and food processing industries drawing attentions of different levels of community and stakeholders (Dorling 1977; Carney and Dodd 1989b; Rappert and Muller 2005a). This means farm managers must ensure minimum level of environmental impact to the surrounding areas. Expansion in the number of livestock and food production facilities, increase in human population, urban encroachment and increase in public's concern regarding air quality are contributors of unsolved odour complaints (Willinger 1974; Mackie et al. 1998; Mahin 2001; Centner 2003).

Scarcity of the knowledge, expertise, equipment and documentation impeded local researchers to visualise and understand the concept of odour studies in Malaysia. Increasing public's awareness on environmental pollutants production may be a good starting point for compulsory odour assessment and regulation implementation locally. However, the aim to attain a 'no odour'

situation is unachievable because of nature's phenomena but it is possible to look into ways to reduce the undesirable odours after thoroughly studying the nature of odour contributor. Many refused to accept the fact that measurement of odour is the fundamental step to prevent and control odour emission accurately and economically (Willinger 1974; Powers 2003; Powers et al. 2005; Rappert and Muller 2005a).

Constant odour complaint is a problem for both breeders and local authorities especially when it comes to closure or relocation of any agricultural facilities and operations as this will create loss of employments and incomes and affect national food security. It ought to be noted that agricultural contributions to the economy in many agricultural active countries are huge. It is essential for authorities to synergistically attempt ways to invent new technology or to make use the available technologies to compute and mitigate agricultural odour emissions.

### **Odour generation**

Obnoxious odour generated from animal facilities abundantly contribute to air pollution (Willinger 1974; Skinner et al. 1997; Hobbs et al. 1999; Mahin 2001; Powers et al. 2005; Rappert and Muller 2005a) due to expansion of livestock facilities to meet the demand for meat, egg and milk intake for human diet (Mackie et al. 1998; Mahin 2001; Centner 2003). Generally, these emissions contain dust, a range of micro-organisms and odorants due to a combination of manure storage, bedding materials, ventilation fans, animal and animal feed within the facilities (Carney and Dodd 1989b; Wathes et al. 1997; Mackie et al. 1998; Ullman et al. 2004; Rappert and Muller 2005b). However, the largest proportion of odour is generated from animal manure decomposition during collection, handling, storage and spreading as fertilisers (Edeogu et al. 2001). The strength of odour differs from one type of animal to another and their stages of life cycle (Rappert and Muller 2005b).

Fundamentally, aerobic and anaerobic biodegradation processes of organic materials generate odorous gases at rates depending on the production and accumulation of waste within the facility itself (Willinger 1974; Dorling 1977; McGahan et al. 2002). Micro-organisms break down the abundant nutrients in the waste generating stench mostly due to oxygen depletion. Changes in temperature and humidity, poor animal health, dietary upset, inappropriate drinking lines and inadequate insulation or ventilation create localised wet bedding materials and accelerate odour generations (Willinger 1974). Removal and breaking of thick caked pile manure and bedding materials in farms may enhance immediate rapid volatilisation and production of dusts.

Volatiles from animal facilities can be grouped mainly as volatile fatty acids, aromatic compounds, nitrogen and sulphur containing compounds which can be perceived easily even at very low concentration (Persaud et al. 1996) and may have the capability to exert great impacts on the environment, global climate change, human and animal health and their products (Willinger 1974; Mackie et al. 1998; Schiffman 1998; Seedorf et al. 1998; Radon et al. 2001; Tech 2001; De Boer 2003; Krupa 2003; Nimmermark 2004). An individual need not stay near emission areas to be affected by the odorants in the plume as some volatiles are highly stable throughout the emission and dispersion processes which carry the odour from the point source to the sensitive receptors in the nose (Shen and Sewell 1984).

Odour from primary production is not a threat to human health but it constitutes a significant nuisance. Thus, many have called for the livestock odour to be subjected to regulations. In the food processing sector, physical processes as well as biological or chemical reactions in food processing, coupled with waste and decaying materials within the facilities result in odour emanation. Odorous compounds produced by microbial degradations are similar to the ones from livestock except for short-chain alcohols, ketones, aldehydes, aromatic and acids (Rappert and Muller 2005a).

In Malaysia, the Environmental Quality Acts place much emphasis on water quality but do not include odour emission. Other than livestock facilities, there have also been numerous nuisance complaints regarding foul smell emanating from palm oil sludge lagoons as well as rubber processing factories.

## **Perception and properties of odour**

### ***Odour perception***

Odorants are volatile compounds that are responsible for creating odour which stimulates human olfactory system (Gostelow et al. 2001; Stuetz and Frenchen 2001). Odorants must have the ability to volatilise at ambient temperature to ease the absorption of substance in the mucus layer on the sensitive surface of epithelium in the human nose. In addition, these odorants should not be substances existing on the olfactory epithelium in order to avoid errors in identification of stimuli. Smell receptors in human are built in the olfactory epithelium in the roof of the nasal cavity. It has approximately five to six millions olfactory receptor neurons. The olfactory epithelium consists of three types of cells, viz., a) the olfactory cells, b) supporting cells, c) the basal cells. Each cell sends information in the form of electrical signal to the olfactory bulb in the forebrain where it is processed and spread to other parts of the brain that detects and identifies an odour (Doty 1995; Stuetz and Frenchen 2001).

The human nose is an efficient odour detector compared to any scientific instrument. Currently, no analytical instrument can measure or evaluate an odour in the manner a human nose does. The human nose detects and differentiates thousands of volatiles as low as part per billion in concentration in the ambient air (Guyton et al. 1987; Ganong and Coleman 1997). Most importantly, the human nose has the capabilities to attract or reject an odour according to the perceived stimuli (Nimmermark 2004). For instance, individual approaches pleasant odours related to food and taste and aromatherapy but avoids and becomes aware of identified hazards in the environment such as spoiled food, smoke and infections. In fact, a study has been conducted on patients diagnosed with probable and questionable Alzheimer's disease testing on the efficiency of their olfaction system as an early indicator of that particular disease (Morgan et al. 1995). Sense of smell trains and reminds a person of awareness and sensitivity of its surroundings.

### ***Odour intensity***

Intensity of odour is the strength of odour perceived above its threshold level and it is very much related to its odour concentration (Stuetz and Frenchen 2001; Nimmermark 2004; Nicell 2009). Response of an olfactory receptor depends on the intensity of an odour. It is the individual perception on the odour's concentration. A common way of measuring odour intensity is to compare the intensity of an odour to the intensities of different but known concentrations of a reference odorant such as the commonly used n-butanol. However, it can also be described using Fechner's law and/or Steven's power function (Misselbrook et al. 1993). Terms such as not perceptible, weak or strong are used to scale the odour perceptions (*Table 1*).

As mentioned earlier, no devices can detect odours like the human nose. To date, dynamic olfactometer is the most suitable instrument to measure an odour concentration by presenting odorous air samples to an odour panel in a range of dilutions and letting the panellists detect the presence of an odour. According to European air quality standard draft, odour concentration is best expressed in odour units per cubic meter ( $\text{OU}/\text{m}^3$ ) or European odour unit (OUE) expresses as  $\text{OU}_E/\text{m}^3$  (CEN 2003). It defines the volume of diluent needed to dilute a unit volume of odour until the detection threshold of the odour is achieved. Alternatively, odour unit per cubic meter also defines the concentration of odour in one cubic meter of air as the panel detects the threshold of the odour. The European air quality standard (CEN 2003) also defines European Reference Odour Mass (EROM) as equivalent to 123  $\mu\text{g}$  n-butanol evaporated into 1  $\text{m}^3$  of neutral air.

Table 1. Odour intensity scaling

Odour concentration	Intensity level
Not perceptible	0
Very weak	1
Weak	2
Distinct	3
Strong	4
Very strong	5
Extremely strong	6

Table 2. Odour characters and threshold of compounds (Stuetz and Frenchen 2001)

Compounds	Odour description
Methyl mercaptan	Decayed cabbage
Dimethyl disulphide	Putrefaction
Acetic acid	Vinegar
Acetone	Fruit, sweet
Indole	Faecal, repulsive
Ammonia	Sharp, pungent
Valeric acid	Sweat

All odours are only detectable at a concentration of  $1 \text{ OU}_E/\text{m}^3$ . The Malaysian Standard MS 1963:2007 corresponds to the European Standard EN13725: 2003.

Odour intensity is a way to compare the strength of an odour perceived with or to another. At a higher concentration, some odours may be perceived as very weak while others may be perceived as distinct. The Weber-Fechner law (Eq 1) is used to develop the relationship between intensity and concentration as

$$I = k_w \log (C/C_o) + K$$

(Eq 1)

where  $I$  is the intensity of perceived odour,  $k_w$  is the dimensionless of Weber-Fechner constant,  $C$  is the concentration of odorant,  $C_o$  is the concentration of odorant at the detection threshold and  $K$  is a constant which relates to the use of mean intensity levels.

Generally, all volatiles have their own threshold limits. Threshold is the minimum concentration required by the sensory property to detect an odour. It is often determined by 50% of the odour level determined by a panel consisting of a specified number of people (5 – 8 persons) using olfactometer (Voorburg and Kroodsma 1992). Two levels of threshold existing in the olfactometry science are the detection threshold and the identification threshold. The threshold for detection is the minimum concentration needed by an assessor to identify between a sample and blank without any need to identify the odour. The threshold for identification or recognition is the minimum concentration needed by an assessor to identify accurately and correctly character of a volatile compound. This is often difficult as odour exists in the form of mixture and some compounds have the tendency to mask the other compounds in the mixture (Nimmermark 2004). However, odour threshold aspect varies among the human population due to nature of the chemical itself, sensitivity, age, gender, social habits, occupation and state of health of panellist (Bliss et al. 1996; Nimmermark 2004). Mostly women are much more sensitive and have lower odour threshold detection limits as compared to men and the ability to detect an odour declines with the increase in age especially after 60 years old.

### ***Odour characters***

An odour characters explain how an odour smells like (*Table 2*). Usually odour descriptors based on source of odour will be provided to panel to help them to describe the odour perceived. As odour descriptors vary from one to another and no one descriptor can satisfy or match another completely. The American Society of Testing and Material (ASTM) has the most collection of descriptor for over 800 compounds (Stuetz and Frenchen 2001). Characteristics of odour can be revealed using proper methods such as the gas chromatography-mass spectrometry (GC-MS).

Hedonic aspect of an odour is related to its pleasantness or unpleasantness which is directly related to odour intensity and concentration. Unpleasantness increases proportionally with the increase in odour concentration. While evaluating an odour in the laboratory for its hedonic tone using an olfactometer, panels are exposed to a controlled stimulus in terms of intensity and duration. The hedonic scale ranges can be set according to experimentation purpose, for instance from 0 to 10, -4 to +4 or -10 to +10. Negative sign indicates the most unpleasant and the positive sign indicates the most pleasant odour. Nevertheless, most pleasant odour may still become unpleasant with the increase in intensity and concentration causing annoyance to sensitive receptors. The degree of pleasantness or unpleasantness is very subjective as it is very much influenced by panel's experiences, psychological and emotional factors associated with a particular odour.

### **Impact of odour on human health**

Due to the presence of hazardous volatile compounds and micro-organisms, livestock odours are now deemed as serious toxicants affecting human and animal health rather than just as a nuisance. According to the World Health Organisation (WHO), health is defined as a state of complete physical, mental and social well being. Health of an individual may not be predicted merely with the absence or non-appearance of diseases. Consequently, regulators and environmental groups have shown great concern on volatiles from livestock area as they are classified as hazardous pollutants (Turan et al. 2007) and have the capability to remain chemically stable. Recent research has suggested adverse effects of odour on the health of neighbours from large animal production facilities (Schiffman 1998; Wing and Wolf 2000; Nimmermark 2004).

Odour is often regarded as an environmental stressor because of its psychological and depressive impact to sensitive receptors (Nicell 2009). Such symptoms include loss of appetite, nausea, fatigue, vomiting, headache and insomnia. In long time perception stress, illnesses may lead to heart and blood vessel diseases depending on the decline in the immune defence. Odorants may also have physical impact on individuals due to presence of gases as well as micro-organisms, causing sensory irritation, tears, asthma-like reaction and allergic symptoms.

Long term exposure to a particular odorant may lead to a decrease of sensitivity to that odorant due to adaptation. Workers exposed to odours from livestock facilities regularly for long term may have a changed or different odour perception due to high level concentrations of chemicals which might have decreased their sensitivity and lead to a non-understanding of odour complaints from neighbours with irregular exposure. Adaptation is faster and greater to unpleasant than to pleasant odours. At some point, these odours cause serious uneasiness in human activities and social enjoyment. These may be exhibited by avoidance of outdoor recreational programme, mood swing, impairment of food preparation, reluctance to receive guests, compulsive house cleansing, increased laundry frequency and decline in business (Jones et al. 1992; Nimmermark 2004).

### **Odour assessment techniques**

Odour analysis can be conducted using both sensory and/or instrumental measurement methods. Selection of the right method for odour measurement depends very much on the objective of the particular analysis.

#### ***Olfactometer***

Sensory study that has been conducted for many years employs the static or dynamic dilution olfactometer. The dynamic dilution olfactometer serves better compared to static one due to its efficacy in transferring sample to the smelling port with minimum impact of sample being adsorbed onto the instrument surface (Stuetz and Frenchen 2001). This instrument went through

many phases of development in the 1980s in Netherlands before protocols were established on the application of the instrument (Jones et al. 1992). Usually diluted odour samples are presented to a group of trained assessors to determine the dilution factor at the 50% of detection threshold in an odour free environment. These assessors are of various ages from both genders to produce data representing average community.

Currently, there are two different modes of data recording namely, a) yes or no and b) forced choice. In the yes-no method, the assessors are asked to judge whether an odour is detected or not. In the forced-choice method, assessors are forced to make a choice out of two or more air flows, one of which is the diluted sample. Forced choice mode is more reliable than the yes-no mode as panel members detect and describe the intensity of the perceived odour compulsorily (Jones et al. 1992).

The most tedious part of olfactometer study is the sample collection. Equipment used for sample collection must be able to provide samples that are representative of the odour emanating in the field with minimum interference. Two types of sampling can be considered: a) dynamic sampling, and b) sampling for delayed olfactometry. With dynamic sampling, the sample is ducted directly to the olfactometer, without storage in a sample container. In sampling for delayed olfactometry, a sample is collected and transferred into a sample container for analysis by delayed olfactometry. The Malaysian Standard MS 1663: 2007 provides detailed procedures for sampling odour.

Two different approaches are used for determining odour emission rate i.e. indirect and direct methods (Gostelow et al. 2001; Hudson and Ayoko 2008; Frechen et al. 2004). Indirect method provides meteorological measurement which is mostly suitable for odour dispersion and modelling studies. Direct method measures the emissions rates of odour directly from the sources using suitably designed devices such as the static flux hood chamber, dynamic flux hood chamber, conventional wind tunnel or low speed wind tunnel. With these methods of measurement, it is very essential to select the correct sampling device to attain reproducible data at ambient.

Odour samples are usually collected in odour free bags made of Tedlar, polytetrafluoroethylene (PTFE) or Nalophan from the sites using lung principle before being analysed. However, at present analysis of sample collected using Tedlar material provides outstanding reproducibility with minimum interference and degradation of samples with longest processing time. To attain ambient representative results, collected air samples should be presented to assessor within 24 hours of collection. Equal attention must be given regarding tubing materials connecting sampling devices to collection container and from collection container to all inlets and outlets of olfactometer in order to avoid absorption, deformation and loss of sample along the tubes during sampling and analysis process. Teflon and stainless tubing materials act better in accordance to fulfil these purposes compared to ordinary plastic or poly vinyl chloride (PVC) materials in which odour samples precipitate (CEN 2003; Epa 2007).

Simple precautions taken can make results obtained accurate and reliable. It is always better to allow sufficient odour free air to flow into the olfactometer prior to and upon completion of analysis to eliminate dust and carry overs from previous and current sampling sessions. In addition, testing or calibrating the sense of panel members prior to analysis with *n*-butanol gas confirms the reliability of results obtained by using respective members and assists to identify biasness. Odour samples are diluted in descending order in the mixing chamber in the olfactometer with odourless air before being distributed to the smelling ports for panels to assess. This process is conducted to avoid intensified odour to stay along in the lines of the olfactometer tubing in order to produce better results by introducing less intensified to more intensified air sample to the panels (CEN 2003).

Dynamic olfactometer has been in use in the waste, livestock and wastewater treatment areas for many years (Carney and Dodd 1989a) to identify the relationship between the odour



concentrations, dust particles and intensity (Misselbrook et al. 1993; Hobbs et al. 1995). This instrument is useful and low in price compared to very technical instruments. Generically, one can handle it easily and does not require any additional equipment for it to function. Except for some intermittent cases, the olfactometer operator must be familiar with the idea of dilution estimation especially when dealing with highly concentrated samples beyond the levels of dilution.

In most cases, odour concentration obtained from dynamic dilution olfactometer can be used to estimate odour emission rates, dispersion modelling and impact assessment (Jones et al. 1992). A research team found huge variations of odour concentration and odour emissions' rates from poultry units over various growth period of animals using olfactometer (Hayes et al. 2006). However, this sensory odour measurement is rather subjective and has its limitations. Most importantly, it has to be calibrated regularly to enhance accurate measurement and this step is not easily done with some olfactometers. The accuracy of the odour intensity and odour concentration results obtained may be doubtful as they depend on the panels' assessment. For this reason, panels have to be trained and screened before becoming an assessor using standard odorous gas, commonly *n*-butanol. In addition to this, detailed quantitative and qualitative study of concentration and components of an odour sample cannot be performed using the dynamic dilution olfactometer. Studies have reported on inability of correlating odour intensity with volatile compounds in the emissions samples using olfactometer (Schaefer 1977; Misselbrook et al. 1993).

### ***Electronic nose***

The first concept of an electronic nose or artificial nose was proposed and developed at the University of Warwick, UK in 1982 that can be used in the wastewater, livestock, and landfill odour emission analysis to detect and measure odour (Persaud and Dodd 1982; Schaller et al. 1998). The device has built in electronic chemical sensors and proper recognition system to identify simple and complex odours and to discriminate sources of odour emanating. The result obtained can be significantly different based on the source of odour. The intensity of the sensor response is proportional to the concentration of the volatile compounds (Hobbs et al. 1995). The correlation of the sensors response against odour strengths shows that a reasonable range of odour concentrations between 100 and 800,000 OU/m<sup>3</sup> can be obtained.

However, the sensor baselines and sensitivity of the electronic nose may be notably affected by the environmental aspects such as the humidity and temperature. The selection of a suitable gas for different environment odours and the requirement for timely calibration weaken the use of the electronic nose to produce valid and reliable findings using this system (Nimmermark 2001).

### ***Gas chromatography-mass spectrometry***

Gas chromatography-mass spectrometry (GC-MS), developed in the 1940s, is an instrumental method that combines the features of gas chromatograph and mass spectrometer to identify different substances within a test sample for both quantitative and qualitative purposes. Reliability of GC-MS as a tool of choice for tracking organic pollutants in the environment increased tremendously over the years (Wang et al. 2008).

The advantage of GC-MS lies in its superiority of the sampling and separation method suitable for complex mixtures which is due to the rapid development of capillary column that provides better peak resolution (Sandra et al. 1980). Increase in retention and decrease in phase ratio produce better peak resolutions too. Separations of compounds of a mixture introduced into the GC system occur as the sample travels the length of the column.

Such columns will have excellent ability to detect and separate substances with minimum impact of oxidation and adsorption of sample during the process. Usually a column supplier would provide suggestions and advices on column selection based on the research to be performed.

Generally there are two types of columns available for GC use, namely: a) packed column and b) capillary column. Packed column is glass or stainless steel coil with dimension of 1 – 5 m total length and 5 mm inner diameter. It is filled with the stationary phase, or a packing coated with the stationary phase. Capillary column is thin fused-silica capillary with dimension of 10 – 100 m in length and 250  $\mu$ m inner diameter with stationary phase coated on the inner surface. Most odour studies are conducted by using capillary column as it provides better separation efficiency than that of packed column.

Molecules take different retention time to elute out of the gas chromatograph before the mass spectrometer captures, ionises, accelerates, deflects and detects the ionised molecules separately. The molecules are broken into ionised fragments and detected using their mass to charge ratio ( $m/z$ ). It is almost impossible to make an accurate identification of a particular molecule by gas chromatography or mass spectrometry alone as both these instruments are interdependent. At present the coupling of mass spectrometer with gas chromatograph works significantly better than any other detectors available in the market such as the electron capture detector (ECD), flame photometric detector (FPD) and the traditionally used flame ionisation detector (FID). However, the selection of the most suitable detector coupling with the GC depends very much on the aim and substances in research (Hobbs et al. 1995; Stuetz and Frenchen 2001). Even though mass spectrometer is expensive, it has a high compatibility with capillary column used in GC. Identification of compounds may be difficult and time consuming especially when two different molecules have a similar pattern of ionised fragments that behave in the same way in both a gas chromatograph and a mass spectrometer or low quality of matching probability with library. However, very few detectors can be used to detect odour compounds. For instances, FID is robust and sensitive mainly to hydrocarbons compared to other types of detectors. Nevertheless, its sensitivity to sulphide compounds is less than other detector. Sulphur chemiluminescence detector (SCD) can be applied to gas chromatograph with additional equipments to identify sulphur containing substances such as dimethyl sulphide, dimethyl disulphide and dimethyl trisulphide as it works more sensitively and selectively for sulphur detection. It may still detect compounds other than sulphur in the sample introduced at a low sensitivity level.

Despite advancement in the GC techniques, there is still a lack of reliable odour measurement method resulting in a gap of information that correlates between sensory and instrumental data leaving either data less authoritative for estimating odour impact or establishing regulations.

### ***Gas chromatography-mass spectrometry-olfactometry (GC-MS/O)***

The human nose identifies odorants in a mixed mode describing perceived odour in different perceptions. Thus, gas chromatography-mass spectrometry-olfactometry is a great tool to measure volatiles using both sensory and instrumental measurements (Friedrich and Acree 1998; Delahunty et al. 2006). This method was first proposed and used by Fuller and his co-workers in the 1960s to select odour active compounds from a complex mixture. Due to problems of reproducibility and hot effluent from the GC, this method was not put into practice (Friedrich and Acree 1998; Van Ruth 2001; Delahunty et al. 2006) till it made a comeback in 1971. Headspace sampling techniques suit conveniently with the GC-MS-O analysis for odorant detection. Chromatograms from GC response differently to the chromatogram collected from olfactometry studies. This system enables identification of odour active compound since it has insignificant peak area from gas chromatogram which is an advantage for abatement studies.

This type of study has been used vastly in the food, perfumery and wine industries for many years to identify compounds responsible for taste and aroma production. (Heiler and Schieberle 1997; Curioni and Bosset 2002; Garruti et al. 2006; Komthong et al. 2006; D'acampora Zellner et al. 2008). Fewer studies have been carried out in the agricultural sector.

It is suggested that GC-O has mainly four data processing categories as mentioned below (Van Ruth 2001; Delahunty et al. 2006):

**Dilution analysis** Charm and aroma extraction dilution analysis (AEDA) are two different dilution analysis commonly used to detect potent odour compounds in food as quality control markers (Song et al. 2008). In charm analysis the detection of a volatile begins and ends with data of threshold and intensity as perceived by a panel. This technique is usually time consuming and requires many sniffers. AEDA determines the last dilution in which odour compounds are detected. Results from AEDA are represented in logarithm of the factor of dilution against retention index. Both Charm and AEDA are similar in that samples are subjected to dilutions and both are based on the odour detection threshold principle.

**Detection frequency analysis** This technique centres on detection of threshold with a group of assessors to detect odour active compounds from undiluted sample at the sniffing port. Assessors evaluate the sample at the sniffing port while simultaneously measuring the intensity of compounds. Described peaks or compounds by the assessors are analysed as frequency of the presence with description recorded for each volatile and not based on the intensities of the compounds.

**Posterior intensity analysis** Posterior intensity analysis is not frequently used as it requires the assessors to perform quite complex tasks. It records odour intensity as volatiles are eluted from GC system. Scale used by the assessor differs and this complicates the assessment process.

**Time intensity analysis** This analysis was developed to estimate odour intensity. An extracted sample is injected into a GC column for the compounds to separate and simultaneously these compounds are fed to two detectors which are the mass spectrometer and the olfactory detection port. Volatiles eluted from GC column are assessed by a trained panel at the sniffing port with no dilution being required. Assessor evaluates the intensity using modes given (i.e light, mild, high and very high) and describes verbally the smell. Results are computed as intensity versus retention time and are matched with GC chromatogram of the sample examined.

## **Development of olfactometry laboratory**

### ***Laboratory layout***

Since dynamic olfactometry uses the human nose as a sensor, a 'room within a room' type of facility is provided in order to minimise the factors that affect the sense of smell. These include the room ambient room quality, drinking water and equipment interference. The project staff and panel members first enter an air conditioned large room where samples are prepared and stored. Within this room is the olfactometry laboratory which consists of a rest room and an evaluation room. Air from this laboratory passes through a carbon filter, which removes background odours from the rooms.

The rest room is provided for panel members during the time between sniffing. Bottled drinking water is provided for panel members in the rest room. The olfactometer is placed in the evaluation room. As the panel members enter the evaluation room one at a time, they place their nose into the sniffing port of the olfactometer and indicate whether they detect any odour.

### ***Method of analysis***

The dynamic olfactometer used is the CrossScent™ which is automated and computerised. It allows the precise dilution of gases to be presented to panel assessors for the determination of odour thresholds. It caters for six odour samples per hour, with a panel of six trained odour

Table 3. General olfactometer specification

Testing modes	Binary forced choice
Ascending concentration series	Two fold dilution increments
Dilution range	4 to 200000
Presentation flow rate	20 litres/min or thereabout
Presentation device	Teflon nose port
Presentation face velocity	Approximately 0.5 m/s
Air mixing time	10 – 30 s
Air supply	Strictly deodorised air
Wetted parts	316 stainless and Teflon
Working pressure	7 psi for olfactometer, 4 psi for pressure vessel
Average throughout	4 – 6 samples/hr with a panel of 6 assessors

Table 4. Expression of panel responses in forced choice method

Response	Choice results	Certainty
False	Incorrect	Guess
False	Correct	Guess
False	Incorrect	Inkling
False	Correct	Inkling
False	Incorrect	Certain
True	Correct	Certain

assessors. The method is able to comply with EU Standard EN13725:2003 for determination of odour concentration. The general specifications of this instrument are shown in *Table 3*.

The binary forced choice method is used in the odour evaluation. After the odour samples are collected from a source, a panel of trained assessors is presented with a series of the samples which are diluted over a range of 4 – 200000. Each assessor is presented with two samples at a time, one containing the odour while the other containing the odour free air serves as a placebo. The assessors are asked to indicate the presence of odour in each, and the computer then captures and processes the data to generate the different threshold levels.

To reduce variability, the assessor is asked whether his/her choice is guess, inkling or certain. The response is classified as true or false from the combination of choice results (*Table 4*). The given time to a panel member to evaluate the presented sample is 15 s. The odour concentration is defined as 1 odour unit per cubic metre of odour free air (OU/m<sup>3</sup>) at the dilution factor of 50% detection threshold. Materials used for sampling equipment are those that do not emit odour, such as polytetrafluoroethylene (PTFE), tetrafluoroethylene hexafluoropropylene copolymer (FEP) or polyethylene terephthalate (PET). Materials such as silicon and natural rubber that emit odour are not used. Similarly, materials used as sampling bags are odourless materials such as FEP, PET or polyvinylfluoride (PVF). Sampling bags are not reused to eliminate residual odours. Neutral gas is used to dilute odour samples. The reference odorant material used is *n*-butanol.

### ***Panel selection***

A panel consists of a group of panel members or panellists who are utilised to determine odour threshold of odour samples. They can also be considered as persons who are trained to judge samples of odorous gas, using dynamic olfactometer. The general population shows a typical bell

shaped curve in terms of olfactory sensitivity, with 96% within the normal sense of smell. The remaining portion is allotted for hypersensitive and anosmic conditions equivalently. The panellists selected should represent the general population. However, in order to ensure repeatability, the sensitivity variability of panel members selected is much narrower than the variability within the population. To achieve this, panel members with a specific sensitivity to the reference gas, i.e *n*-butanol is selected. The following groups of people are excluded as panel members: smokers, drug addicts, pregnant women, people with allergies, sinusitis patients, people suffering frequent colds and chewers of betel nuts or other odorous substances. Panel members also agree to the following code of behaviour:

- a) Be motivated to carry out his/her job conscientiously
- b) Be available for a complete measuring session (a series of measurement on a day, interrupted by short breaks only)
- c) Be willing to participate for a sufficient period to build up a history of measurement
- d) Be willing to refrain from food and drinks except water 30 minutes before and during measurement
- e) Be willing to refrain from using perfumes, deodorants, body lotions or other cosmetics on the day of measurement
- f) Be willing to be present in the odour room 15 minutes before the measurement start in order to get adapted to the odour room conditions

## **Conclusion**

Currently, there is an urgent need to conduct research on agricultural odour impact assessment and regulation in Malaysia since potential sources of odour remain uncharacterised resulting in a lack of scientifically proven data on odour emissions. This is an important step towards proper waste management that takes odour reduction into consideration. Towards this end emphasis must be given to integrate research that focuses on analytical techniques and sensory technology to generate reliable odour evaluation data. Reliable data are needed to assist local authorities to establish odour standards that are acceptable socially and legally. Reliable data are also required in establishing minimal separation distances between an agricultural centre and the next public housing area, thus avoiding future nuisance complaints and giving land security to primary producers.

In line to fulfil the scarcity in odour research in Malaysia, a dynamic olfactometry laboratory for the objective measurement of odours is being established at Malaysian Agricultural Research and Development Institute (MARDI) in Serdang. The method is principally based on the human nose as the odour detector and not based on the detection of indicator gases. Therefore, a panel of trained odour assessors is required for such measurements. Dynamic olfactometry is needed to help establish buffer zones between primary production and residential areas, set regulatory standards suitable for local situations, settle legal disputes, study odour dispersion and serve as cross reference for international standardisation. An ISO standard for the determination of odour concentration is being adopted in Malaysia. It specifies procedures for the determination of the odour concentration of a gaseous sample using dynamic olfactometry with human assessors.

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### **Abstrak**

Menilai kesan bau industri primer dan mewujudkan standard kawal selia telah menjadi cabaran utama kepada pihak berkuasa kerana kekurangan pengetahuan mengenai kepekatan bau dan kadar pelepasan. Pemilihan teknik yang tepat, ditakrifkan saintifik dan terbukti dengan keterulangan yang tinggi adalah penting bagi memenuhi keperluan menyelesaikan masalah bau. Walaupun terdapat peralatan yang berkebolehan untuk mengesan dan menentukan kuantiti bau berdasarkan gas rujukan atau gas terpilih, kaedah tersebut tidak dapat memberikan data yang lengkap mengenai bau kerana ia terdiri daripada lebih daripada seratus gas. Unit pengukuran bau yang boleh diterima mengikut undang-undang perlu mengambil kira pengesanan oleh hidung manusia kerana manusia yang dapat mengesan bau yang menyebabkan ketidakselesaan atau mengancam kesihatan. Satu kaedah pengesanan yang ideal menggabungkan kedua-dua ukuran deria dan instrumentasi yang boleh digunakan untuk mewujudkan unit bau dan dengan itu keperluan kawal selia. Laporan ini mengkaji konsep asas penilaian bau dan pelbagai teknologi yang sedia ada untuk menilai pelepasan bau daripada aktiviti pertanian.