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UNIVERSITY OF ALBERTA

THE MEASUREMENT OF ODOUR CONCENTRATION

BY

GUOLIANG QU

A THESIS SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF **DOCTOR OF PHILOSOPHY**

IN

BIORESOURCE AND FOOD ENGINEERING

DEPARTMENT OF AGRICULTURAL, FOOD AND NUTRITIONAL SCIENCE

UNIVERSITY OF ALBERTA

EDMONTON, ALBERTA

Spring, 2000

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled **THE MEASUREMENT OF ODOUR CONCENTRATION** submitted by **GUOLIANG QU** in partial fulfillment of the requirements of the degree of **DOCTOR OF PHILOSOPHY** in **BIORESOURCE AND FOOD ENGINEERING.**

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IN LOVING MEMORY OF
MY PARENTS WHO PASSED AWAY MORE THAN TEN YEARS AGO
TO
QING FENG, MY WIFE, THE LOVE OF MY LIFE AND
ZHAIBAI, MY HANDSOME AND TALENTED SON
FOR THEIR LOVE AND ENCOURAGEMENT

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ABSTRACT

An eight-panelist-station, single-sniffing port, triangular forced-choice ascending concentration series olfactometer (UA olfactometer) was designed and constructed. Compared with the most recently designed conventional olfactometers, the UA olfactometer has advantages of less odour contamination potential, economy of sampling, time saving, low manufacturing cost, and less psychological bias. The system can analyze ten-samples per hour. Carbon dioxide was used as a tracer gas to calibrate and test the UA olfactometer. The results show that a neutral air and an odour mixes well in the UA olfactometer, the distribution of the flow rates among eight panelist ports is uniform, and the UA olfactometer operates as it was designed to and satisfies the requirements of the ASTM and the draft CEN standards.

Assuming that the traceability among human panelists/panels exists, a model was developed to quantify the relationship of olfactory responses among panelists/panels to environmental odours and to a reference odour (n-butanol). A data set containing 252 cases was established from forty-four persons hired to evaluate odour samples on the UA olfactometer. The correlation of the model to observed data is significant at the level of α=0.0001, and should be applicable to the general population having normal olfactory sense for all environmental odours. By using the model, the panel's response is normalized. Thus the measurement variance is decreased, and the longstanding problem of measurement of odour concentration being arbitrary is solved.

To get rid of the clumsy olfactometry system, a method of less labour-intensive, non-human-organ-dependent, mobile odour concentration measurement has been developed. This involved creation of predictive function by combining the Adaptive Logic Network

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(ALN), a type of artificial neural networks (ANNs), with a commercially available electronic nose. The function can convert electronic nose measurements into odour concentrations. A data set was developed by evaluating odour samples with both the UA olfactometer and an electronic nose (AromaScan), and was preprocessed to reduce dimensionality by using principal component analysis (PCA), which is the crucial procedure for success. The measurements with the UA olfactometer served as observed values and the dimension-reduced responses of the AromaScan together with the humidity of odour sample and reference air served as input variables. A well-trained ALN can convert measurements of the electronic nose to odour concentrations with less than 20% mean absolute percentage error (MAPE).

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LIST OF ABBREVIATIONS

A_d .... Accuracy of dilution instruments

ALN .... Adaptive logic network

ANN .... Artificial neural network

ASTM .... American Society of Testing and Materials

ATP .... Adenosine triplephosphate

c-AMP .... Cyclic adenosine monophosphate

EROM .... European reference odour mass

I_d .... Instability of dilution

MAPE .... Mean absolute percentage error

PCA .... Principal component analysis

ppbv .... Parts per billion by volume

ppmv .... Parts per million by volume

RMSE .... Root mean square error

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LIST OF UNITS USED IN THE TEXT

UNIT	DEFINITION
°C	Degree Celsius
g	Gram
h	Hour
kg	Kilogram
µg	Microgram
kPa	Kilopascal
L	Liter
ml	Milliliter
m	Meter
min	Minute
mol	Molar
µmol	Micromolar
mV	Millivolt
OU_E	European odour unit (odourant)
OU_E/m^3	European odour unit (air)
psi	Pounds per square inch

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Chapter 1 Introduction

Intensive livestock production, notably swine operations, can result in odour problems and therefore become an environmental constraint to expanding the pig industry. It is unlikely that high intensity odour emitted from intensive livestock units will continue to be tolerated by society in the future. The emission of odours from, and the dispersion of the odours downwind from odour sources, such as manure land application or pig facilities like a confined animal building and an earthen manure storage, together will determine the odour concentration caused by these sources. Many research projects in this area have been completed from different points of view. However, implementation of odour abatement programs, odour policy and regulations requires an objective, reliable, and accurate odour measurement with acceptable repeatability within a laboratory and reproducibility among laboratories. The objectives of this research are to improve the instrument used for the measurement of odour concentration, to develop a model for normalizing a panel's olfactory responses, and to find a method that can replace the clumsy olfactometry system and measure odour concentration directly on site.

Currently, an olfactometer is the fundamental instrument used to measure odour concentration. Although research on the development of dynamic olfactometers has been conducted for nearly thirty years, currently only two kinds of olfactometers are commercially available, and neither of them have enough panelist-stations (at least eight). It was imperative to design and build a multi-panelist olfactometer with less contaminant potential, reduced psychological bias, and lower manufacturing cost than the existing systems. This will be discussed in Chapter 2.

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Assuming that traceability among panelists/panels exists, a normalization model of a panel's olfactory response was developed and is discussed in Chapter 3. By using the model, resolution for the long-standing problem that the measurement of odour concentration has been relatively arbitrary could be improved.

A method of less labour-intensive, non-human-organ-dependent, time and sample saving, mobile odour concentration measurement was developed and is discussed in Chapter 4. The method depends on creating a predictive function by combining an artificial neural network with the response from an electronic nose. The function can convert the response of sensors in the electronic nose into odour concentrations.

In this chapter, discussion will be focused on the measurement of odour, especially on the measurement of odour concentration. Literature on the origin and the perceptions of odours will be reviewed.

1.1 Origin of livestock odours

Odour has long been associated with animal production, particularly swine production. In general, feed and body odours are not regarded as offensive, but those generated from manure and its decomposition during collection, handling, storage, and spreading are considered offensive. A series of complex biochemical reactions occur during the periods from the feed-waste conversion inside animals to the decomposition of swine wastes in confinement buildings and storage units. Thus two stages of degradation occur in the conversion of feed to the swine wastes: a) the passage through the animal yielding urine and feces, as shown in Figure 1-1, and b) the aerobic/anaerobic degradation of the mixture of feces and urine during storage. After excretion, pig manure typically drops through slotted floors of the swine building into drain gutters and in some systems

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remains there 7 to 14 days before being discharged to an earthen manure storage. Manure with moisture content of 85-90% is semi-solid and will flow by gravity, at 90% or greater, it can be pumped. The moisture content of swine manure slurries is usually more than 95% (Midwest Plan Service, 1983). The accumulated volume and high moisture content can guarantee removal of all manure stored in drain gutters. The physical properties and elemental composition of swine manure slurry and sludge stored in an earthen manure storage are shown in Table 1-1. The sludge or solids accounts for 5% of the total volume of manure present in the earthen manure storage. The organic content (C+N+H) of the slurry phase is approximately 50% of the dry weight of slurry, and the inorganic constituents listed in Table 1-1 account for about 15% (Zahn, et al., 1997).

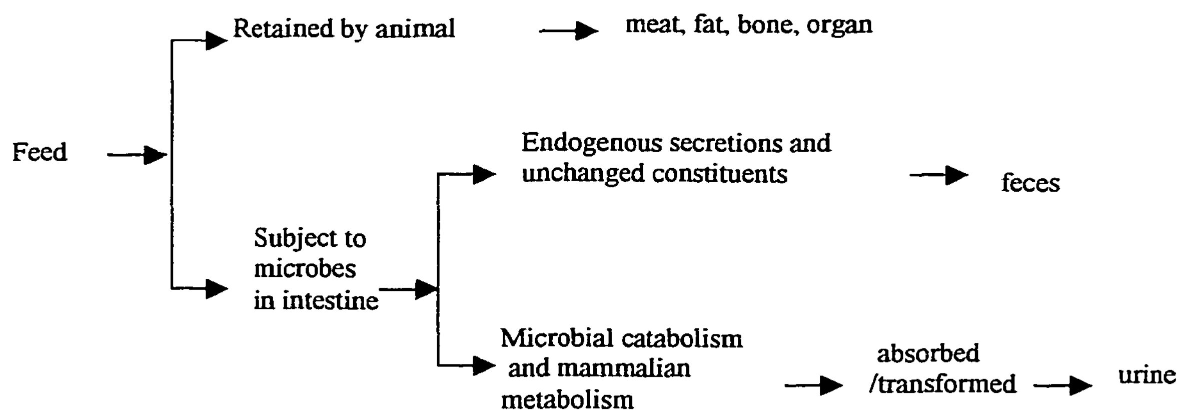

Figure 1-1. The passage of feed through an animal yielding urine and feces

Over 160 odourous compounds have been identified in swine operations (O'Neill et al., 1992). Some principal odourous compounds are ammonia, amines, sulfur-containing compounds, volatile fatty acids, indoles, skatole, phenols, alcohols, and carbonyls (Curtis, 1993). These compounds can be original components of animal urine and feces

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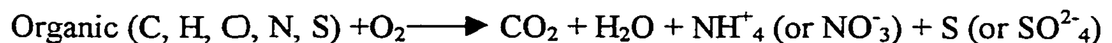
or products of microbial digestion in manure treatment/storage. The microbial digestion in manure treatment/storage is generally anaerobic, and perhaps to lesser extent aerobic on the top surfaces of manure in an earthen storage, or when air is forced into liquid by air-injectors or by submerged air tubes.

Table 1-1. Physical properties and elemental composition of slurry and sludge from swine earthen manure storage (adapted from Zahn et al.1997)

Parameter	Slurry	Sludge	Slurry/Sludge
pH	7.2 ± 0.1	7.2 ± 0.1	1
Solid Content, mg/mL	21.9 ± 3.3	31.2 ± 1.7	0.70
%C of dry mass	37.2 ± 0.4	42.3 ± .2	0.88
%H of dry mass	5.2 ± 0.2	6.2 ± 0.1	0.84
%N of dry mass	3.0 ± 0.1	2.3 ± 0.2	1.30
Ca, mg/mL	280 ± 28	626 ± 51	0.45
Cu, mg/mL	14 ± 4	50 ± 8	0.28
Fe, mg/mL	13 ± 6	51 ± 11	0.25
K, mg/mL	1931 ± 25	1675 ± 14	1.15
Mg, mg/mL	99 ± 8	223 ± 16	0.44
Na, mg/mL	245 ± 31	229 ± 19	1.07
P, mg/mL	612 ± 20	980 ± 36	0.62
S, mg/mL	104 ± 5	158 ± 12	0.66
Zn, mg/mL	7 ± 1	41 ± 9	0.17

Aerobic digestion is the process by which organic matter is digested by bacteria in the presence of free oxygen. A complete biological oxidation of organic compounds in aerobic decomposition can be expressed as:



$$\text{Organic (C, H, O, N, S)} + O_2 \longrightarrow CO_2 + H_2O + NH^+_4 \text{ (or } NO^-_3\text{)} + S \text{ (or } SO^{2-}_4\text{)}$$

Nitrate and sulfate are used as electron acceptors by some aerobic and facultative bacteria and are reduced to nitrogen gas (N_2) and elemental sulfur (S). The nitrogen compounds (proteins, peptides, amino acids and amines) are first converted into ammonium (NH_4^+) by heterotrophic bacteria, then into nitrite (NO_2^-), and finally into nitrate (NO_3^-). As

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shown in Table 1-2, the redox potential of the NO_3^-/NO_2^- couple is 433mV and far higher than that of most odour-causing volatile fatty acids. Because of the low redox potential, odourous compounds are unable to be generated in the presence of free oxygen (O_2/H_2O +818 mV) in aerobic digestion, but may exist originally in manure. Thus, under aerobic conditions, production of odourous compounds in the anaerobic environments is prevented.

Table 1-2 Thermodynamic Sequence of
Important Redox Couples of Biological System
(adapted from Thauer et al., 1977)

Redox	Eo' (mV)
O_2/H_2O	818
NO^-_3/NO^-_2	433
Fumurate$^-$/succinate	33
HSO^-_3/HS^-	-116
$Fe3^+/Fe2^+$	-182
$SO2^-_4/HS^-$	-215
CO_2/CH_4	-244
S^0/HS^-	270
CO_2/CH_3COO-	-300
H^+_2/H_2	-420
$HCOO^-/HCHO$	-450
$CO_2/HCOO^-$	-460
SO^{2-}_4/SO^{2-}_3	-516
CO_2/CO	-524

Anaerobic degradation is the process by which organic matter is fermented by bacteria in the absence of free oxygen. When the substrates contain high concentrations of an organic polymer, the overall anaerobic process can be divided into three phases: the hydrolytic phase; the acidogenic phase; and the methanogenic phase.

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Organic polymers in swine feed are mostly hydrolyzed and absorbed by the intestine of the animal. Thus the acidogenic phase and the methanogenic phase could predominate in manure storage.

Depending on the nature of the organic materials, specific hydrolytic organisms become enriched in manure storage and provide the nutrients for the chain of organisms involved in the overall process by breaking large molecules of polymers into smaller ones. A variety of microorganisms, especially bacteria, possess the ability to hydrolyze the organic polymers involved in this process, and of these, cellulolytic organisms are a most important group. The most important cellulolytic bacteria in anaerobic reactors is Clostridium. Clostridium is also the most important proteolytic organism involved in protein degradation. Clostridium is anaerobic, Gram-variable, spore-forming, rod-shaped bacteria. It has been detected in both mesophilic (20°C to 45°C) and thermophilic (45°C to 65°C) environments (Chynoweth et al., 1987). Clostridium, as well as other cellulolytic bacteria, adhere to plant cell-wall polymers, cellulose, and hemicelluloses and hydrolyze the substrate to oligosaccharides and monsaacharides. A number of oxidation-reduction, decarboxylation, condensation reactions occur and give rise to a variety of products from pyruvate.

Fermentation of organic substrates results in the production of a spectrum of products, including alcohol, fatty acids, and aromatic compounds. A number of bacteria have been identified and characterized that reform the fermentation. These include the hydrogen-evolving, proton-reducing bacteria that utilize substrates such as volatile and higher fatty acids, aromatic compounds, alcohols and lactic acid which cannot serve as substrates for fermentative bacteria. A second group of bacteria, namely, the homoacetogens, produce

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acetic acid or higher fatty acids by the reduction of CO_2. Some of these may also grow autotrophically, using CH_4 and CO_2, while others grow by the fermentation of sugars such as fructose, converting sugars entirely into acetic acid. The hydrogenogenic bacteria include the group of bacteria which decarboxylate oxalate and formate from the acetogenic bacteria. These, however, grow only in the presence of hydrogenotrophic (CH_4 utilizing) bacteria and cannot be grown without a system for the removal of CH_4. In homoacetogens, CO_2 fixation occurs via acetyl coenzyme A (CoA), and by this means, these organisms derive both their energy and carbon requirement from the reduction of CO_2 and CH_4. Carbon monoxide dehydrogenase is the key enzyme of this pathway and the end product is acetate.

Methanogenesis is the terminal step in the anaerobic digestion process, and methane escapes from the system, allowing the digestion process to proceed to completion. A specialized group of bacteria known as methanogens is responsible for this terminal step. These bacteria are strict anaerobes and derive their energy requirements during the production of methane. Obviously, methanogens are chemotrophic bacteria. They are unique in the biological world and grouped in the UrKingdom-Archaebacteria. Methanogens represent most morphological forms of eubacteria, such as the cocci, rods, and the spiral forms. They are mostly free-living, while some are symbiotic with some protozoa. Based on substrate utilization, these are classified into two groups with five families. Group 1 contains 24 species that utilize CH_4/CO_2 and/or format, methanol, or methylamines, while group 2 includes only one family with five defined species and two species not assigned to any family. Organisms in this group utilize methanol and/or

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acetate with or without the ability to utilize CH_4 or CO_2. Typical energy-yielding conversion reactions involving these compounds are shown Figure 1-2.

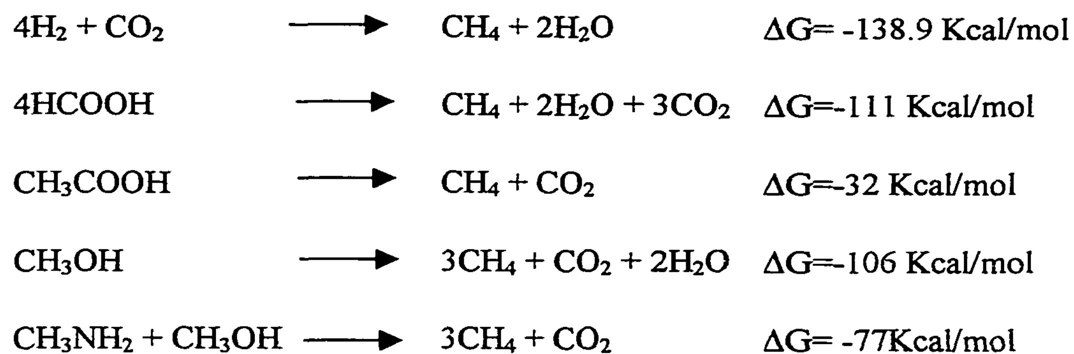

Figure 1-2. Methane formation from 5 substrates utilized by methanogens (modified from Vesiling, 1974).

Nutritional requirements of methanogens range from simple minerals and CO_2/CH_4 as carbon and energy sources to complex specific growth factors such as CoM, yeast extract, trypticase, and digester of rumen fluid. As carbon and energy sources, methanogens can utilize CH_4/CO_2, formate, methanol, methylamines, and acetate. Ammonium salts can serve as a source of nitrogen. Sulfide is used as a sulfur source by all methanogens. pH around neutral is preferred by most of the methanogens, although some can grow at a pH as low as 5.3 and produce methane at pH 3.0 (Bruce et al., 1984). Acid-forming bacteria are quite hardy and resistant to various inhibitors and change in their environment and are not considered to be the rate or process-limiting factor in digestion. Methanogens, on the other hand, are slow growing and are strictly anaerobic and extremely sensitive to changes in their environment such as pH changes, presence of heavy metals, detergents, change in alkalinity, ammonia, sulfides and temperature. In manure earthen storage pits, most molecules of celluloles and polymers have been broken

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down into smaller molecules, and a limiting factor in the degradation of swine manure may be methanogens.

In general the final products of microbial degradation of carbonaceous material in an anaerobic natural ecosystem are CH_4 and CO_2. In stored swine manure, little methane is formed. The rate of methanogenesis under storage conditions is not high enough to prevent the accumulation of products of acid forming fermentation. In other words the acidogenic phase and the methanogenic phase in the microbial degradation of the complex substrates in swine manure may not be in balance. The imbalance between the process of acid formation (the acidogenic process) and methane production (methanogenic process) is the main key to understanding the accumulation of volatile compounds (=malodourous products) in the degradation of swine manure. What actually causes the low rate of methanogenesis in stored swine manure is not clear, but a number of factors are unfavorable to methane fermentation.

The temperature at which anaerobic digestion occurs can significantly affect the conversion, kinetics, stability, and the methane production rate. As shown in Figure 1-3, higher temperatures promote higher reaction rates during anaerobic digestion, thus permitting lower hydraulic retention times and higher loading rates without reduction in conversion efficiency. In the range of 42-50°C, reaction rates decrease below those observed in the mesophilic range and then increase as the thermophilic temperature range is reached; this is because certain methanogenic bacteria are extremely sensitive to temperatures above the mesophilic range. Temperatures in the thermophilic range generally increase the efficiency and rate of organic solids destruction, improve dewatering characteristics of effluent solids, and increase the rates of destruction of

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pathogenic organisms. However, the temperature of anaerobic digestion can markedly affect the stability of the process, even fluctuations in the temperature within relatively narrow ranges under thermophilic conditions can have a significant impact on digester stability. It is reported (Tchobanoglous, et al., 1991.) that thermophilic digesters used in municipal sewage digestion could only tolerate temperature changes of ±0.8°C, and thermophilic organisms were more sensitive to rapid changes in temperature than mesophilic organisms. Thus the optimum temperature for anaerobic degradation is around 35°C. Whereas the storage temperature of swine slurry, depending on the season, ranges from 0 to 20°C. Although methane fermentation occurs below 10°C, it is well known that psychsophilic methanogens produce methane at a lower rate and grow much slower at lower temperatures as correlated to those at mesophilic temperature.

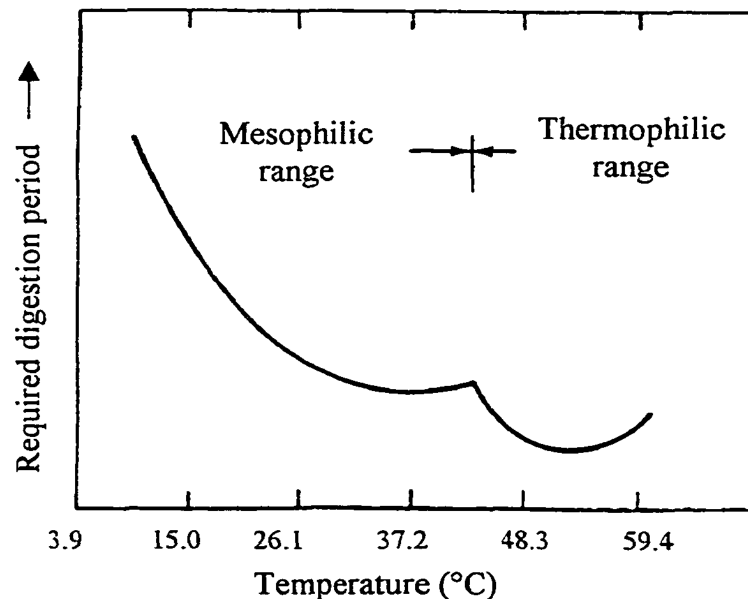

Figure 1-3. Influence of temperature on anaerobic digestion time (modified from Benefield et al., 1980)

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Since the optimum temperature for anaerobic degradation is around 35°C, if the manure earthen storage could be heated to 35°C, odours should be less of a problem. However, it is a costly approach.

The pH of the swine manure slurry stored in lagoons is an important parameter. The methanogens are pH-sensitive and have an optimum pH range of 6.6 to 7.6. Beyond these pH limits, as shown in Figure 1-4, degradation can proceed, but more slowly. At a pH below 6.2, the efficiency drops off rapidly and the acidic conditions produced can become inhibiting to the methanogens. The primary substrate of the methane-forming bacteria is acetic acid. Any sudden environmental change usually results in a pH drop that results in process inhibition. When an earthen storage is in balance, the acetylenic bacteria use acid intermediates as rapidly as they are formed. If the methanogens are not present in suitable numbers or are being inhibited by unfavorable conditions, they will not use the acids as rapidly as they are produced, which results in an increase in the volatile acid concentration. Thus, the increase in acid concentration indicates that the acidogenic are not in balance with the acidogens (acid formers).

This can be caused by overloading with degradable organic material that causes failure of digester installations in municipal sewage digestions (Tchobanoglous, et al., 1991.). Similarly, digestion of swine manure was found to be inhibited at loading rates of 4-8 $kg \cdot m^{-3} day^{-1}$ total solids.

Overloading results in diminished methane production and accumulation of volatile fatty acids in the earthen storage. An overload situation is likely to be present in stored swine manure.

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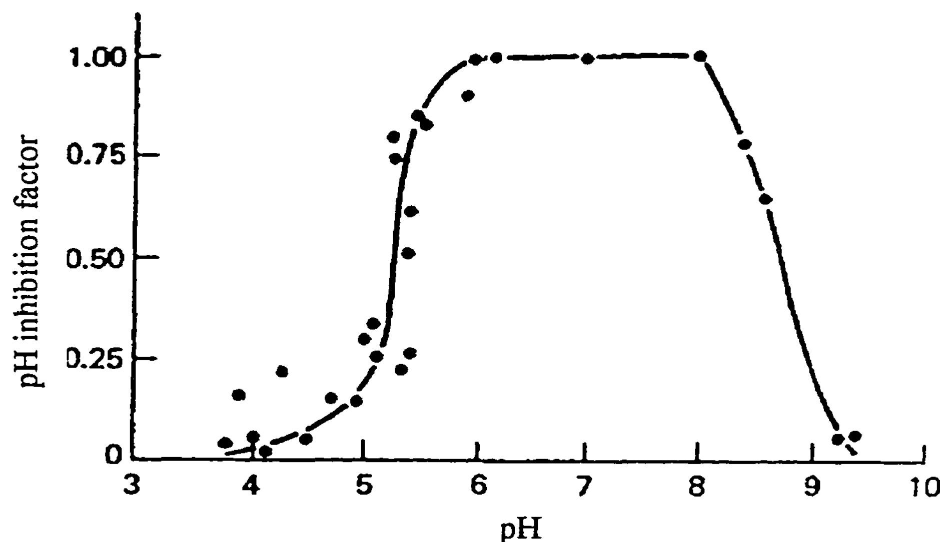

Figure 1-4. Effect of pH on the rate of methane fermentation (adapted from Benefield et al., 1980),

The other unfavorable influence on methanogenesis is any the disturbance of the stored manure which will introduce free oxygen with the incoming slurry. Methanogens are extremely sensitive to changes in their environment and are strictly anaerobic, oxygen, even at trace amounts, is extremely toxic.

Some heavy metals, notably copper, are very toxic to most microorganisms. In aerobic digestion, copper-containing compounds, mainly copper sulfate ($CuSO_4$), are highly soluble, and thus extremely toxic. In anaerobic digestion, the actual concentration of the toxic cation is diminished a hundred-fold or more by complexing reactions and by precipitation as poorly soluble sulphides. Inhibition can also be caused by other metal cations.

The main groups of volatile compounds that occur in anaerobic degradation of swine slurry are the following:

(a). Volatile fatty acids, aldehydes, alcohol and esters

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Of the compounds consisting of only C, H, and O, the volatile fatty acids are the most dominant in swine manure. Total amounts in manure slurry range from 4 to 25g/l. Acetic acid and propionic acid represent about 60 and 25%, respectively, of the total amount of volatile fatty acids, respectively. While butyric, isobutyric, branched valeric and n-valeric acids range from 3 to 10% each.

(b). S-containing volatile compounds

Most of the sulfur-containing compounds detected in the headspace of swine manure are present in trace amounts only. Hydrogen sulfide and methyl mercaptan are most frequently reported as constituents of swine manure and are quantitatively the most important S-containing volatile constituents.

(c). Volatile amines

Anaerobic incubation of protein-containing products with bacteria often leads to the production of volatile amines. The principal volatile amines that are produced during anaerobic degradation include methyl-, ethyl-, propyl-, butyl-, amyl-, iso-butyl-, iso-amyl-, hexyl-, dipropyl-, and dibutyl-amine.

1.2 Odour perceptions

A public awareness environmental issue is becoming more acute, demanding and expecting a clean environment, particularly outdoor air quality. More than half of environmental conflicts are associated with malodours, especially from animal production sites (O'Neill and Phillips, 1992.).

An odour is an organoleptic attribute perceptible by the human olfactory organ upon sniffing air containing certain odourants. Therefore, an odour is an effect or a character of odourant that interferes with people's enjoyment of life and property, while an

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odourant is a substance that stimulates the human olfactory system so that an odour is perceived.

Odourant compounds can cause sensory irritation and stimulate sensory nerves to cause neurochemical changes that can potentially influence health. The main complaints of health symptoms from odours range from irritation of the eye, nose, and throat, to nausea, headache, and vomiting, and further to disturbance, annoyance, and depression (Schiffman, 1998; NRC, 1979). A long-term exposure can even cause mental problems like "Kindling" and "Response Facilitation" (Frey, 1995). High levels of odourous compounds in confinement swine buildings can reduce pig growth performance and increase susceptibility to diseases (Tamminga, 1992). Human and animal studies suggest that expression of health symptoms involves a complex interplay between biological and behavioral/psychosocial influences. Thus, these complaints probably derive from a combination of physiological and psychogenic sources.

The human sense of smell is centered on a cluster of specialized nerve cells (olfactory cells) just above the bridge of the nose, as shown in Figure 1-5, out of the main air stream. Three turbinate bones support the inner nose. Approximately 100 million olfactory cells are located in the olfactory area. Olfactory bulbs are the endings of olfactory nerves that connect olfactory cells with the brain. Normally, only 5% of inspired air passes through the olfactory area but this may be increased to as much as 20% by sniffing, greatly enhancing the sense of smell. The human sense of smell normally increases with age until the early teens and then remains at its most sensitive for a further 30 years (Callan, 1993). Humans can perceive several hundred odours, but, through training, this can increase up to 10,000.

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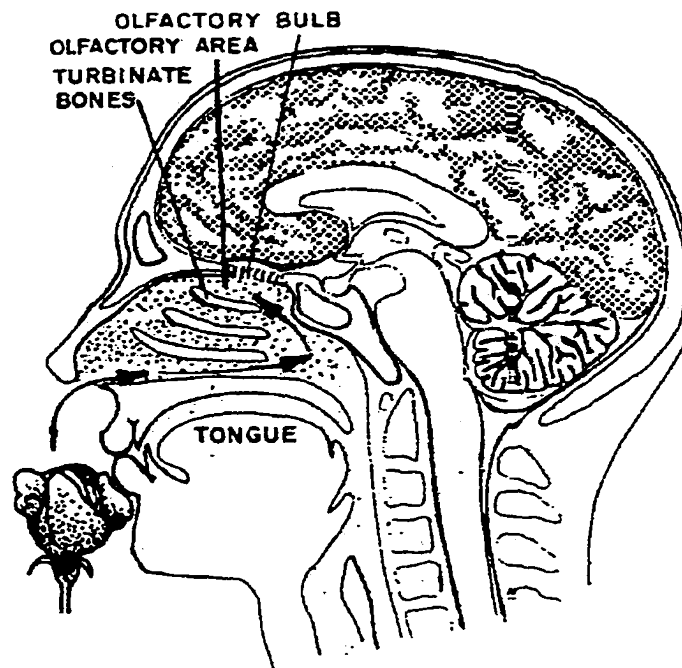

Figure 1-5 Anatomy of Human Olfactory System (adapted from Brobeck, 1974)

Newly discovered receptor proteins, also called seven-time proteins, are located on the surface (membrane) of each olfactory cell (Frey, 1995). The receptor proteins can be grouped into at least seven subfamilies, each of which appears to consist of five to twenty members. There is solid evidence for more than 100 kinds of receptors and it is believed that there are at least 1,000. The receptor proteins and perception operate somewhat similar to the immune system in its detection and identification of bacteria: the receptor proteins are equivalent to antibody proteins, olfactory cells to white blood cells, and an odourous compound to a bacterium invading the blood stream, respectively. Odour perception begins when small volatile molecules bind to receptor proteins that transmit signals to the inside of the cell by interacting with G proteins. The odour molecule is called the first messenger of information to the brain. After it binds to the receptor

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protein, it triggers second, or intracellular, messengers which are the critical link in the signal cascade between odour molecule binding and subsequent brain activity. When an odour molecule binds to a protein receptor on the surface of the cell membrane, the sensory cell induces a G protein in the cell to activate adenylate cyclase to produce c-AMP. The chemical ATP in the cell provides energy for the reaction. The c-AMP opens ion channels in the olfactory cell membrane that let sodium and calcium ions into the sensory cell. These ions induce a voltage, an electrical potential across the cell membrane. This potential fires off an action potential, an electrical pulse. This electrical pulse travels up the olfactory nerve to the brain where the information about the odour is processed. The essentials of this cascade are shown in Figure 1-6.

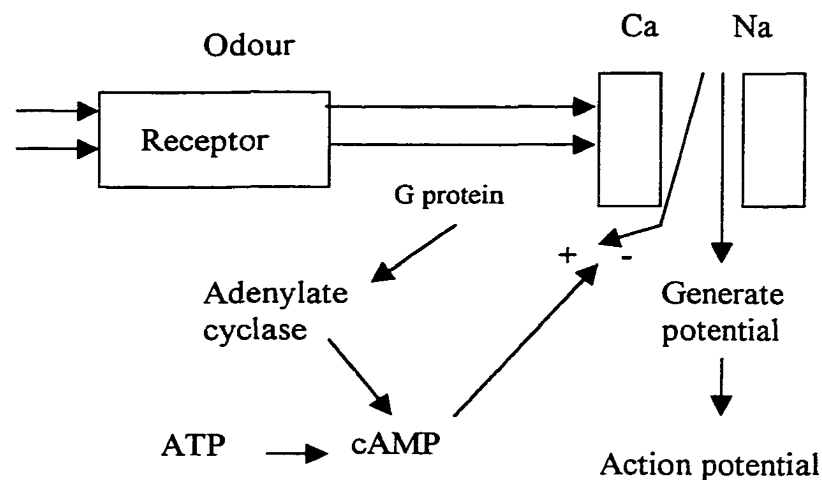

Figure 1-6. Second-messenger signaling in the human olfactory system (adapted from Frey, 1995)

Generally, humans have a very acute sense for odour concentration, and can detect an odour compound at parts per billion levels which is far more sensitive than any existing instrumentation. Table 1-3 illustrates the odour thresholds for chemicals found in livestock, and Table 1-4 shows the odour threshold values (OTV) measured with two methods for odour compounds generated from swine operations: detection threshold and

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lateralization threshold. The detection threshold is the lowest odour concentration that can just be perceived, while the lateralization threshold is the lowest odour concentration that can just be localized which of nostril perceives the odour. The detection thresholds are at least a factor of 500 lower than the lateralization thresholds, and these odours are detected long before their concentration becomes locatable.

Table 1-3. Odour threshold for selected chemicals often found in livestock odours (modified from Kreis 1978)

Type	Chemical	Odour Threshold (ppmv)
Aldehydes	Acetaldehyde	0.21
	Propionaldehyde	0.0095
Volatile Fatty Acids	Acetic acid	1.0
	Propionic acid	20.0
	Butyric acid	0.001
Nitrogen Containing	Methylamine	0.021
	Dimethylamine	0.047
	Trimethylamine	0.00021
	Skatole	0.019
	Ammonia	46.8
Sulfur Containing	Methanethiol	0.0021
	Ethanethiol	0.001
	Propanethiol	0.00074
	t-Buty thiol	0.00009
	Dimeth sulfide	0.001
	Hydrogen sulfide	0.0072

Table 1-4 Thresholds for acetone and butanol in acetone-exposed workers and unexposed subjects (adapted from Wysocki et al. 1997)

Threshold Type	Objective	Acetone(ppmv)	Butanol (ppmv)
Odour Detection	Workers	855	3.17
	Unexposed	41	0.16
Lateralization	Workers	36,669	2,538
	Unexposed	15,758	2,300

Humans are very insensitive in detecting and discriminating the components of odour mixtures. As shown in Figure 1-7, regardless of experience or training, test method, type

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of odour, and complexity of an odour, human beings can only identify up to three or four odourants in a mixture (Laing, 1994). Furthermore, it is also unclear what the relationship

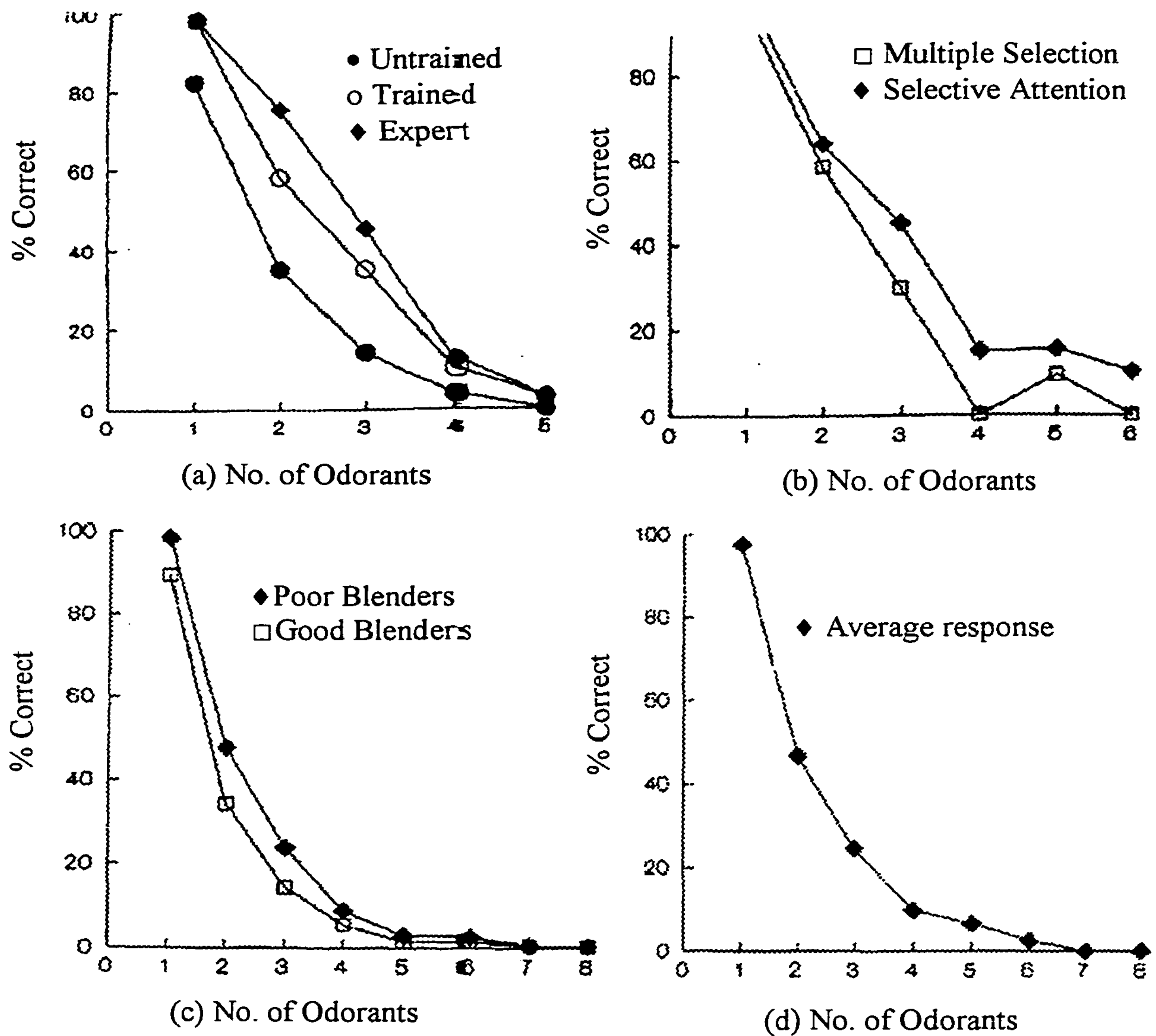

Figure 1-7 Identification of components in Mixed Odour by Human Olfactory System (modified from Laing, 1994).

of the odour concentration of a mixture with the odour concentrations of each component odour could be, such as additive, subtractive, synergistic, or counteractive (suppressing each other). Although several reports (Rosen et al., 1962; Laska et al., 1990) indicated that the odour concentrations became enhanced, most cases supported the hypothesis that

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odour concentrations become suppressed when odours are mixed. The reasons for the properties of mixed odours are unknown. Laing (1994) proposed two mechanisms, Spatial and Temporal Filtering, to account for this loss and to explain this phenomenon of blending. Simply speaking, the spatial filtering function is the result of odourants competing for olfactory cells, while temporal filtering is the result of the different times needed for different odourants to stimulate receptor proteins.

As discussed above, a mixture of odourants may smell different from the unmixed compounds, and in general the offensiveness will increase as the odour concentration increases. Large individual differences exist in reaction to the exposure to odourant concentrations. Various factors like age, gender, hedonic tone (a category judgement of the relative pleasantness or unpleasantness of an odour sample), health, attitudes to the swine industry, and changes in climatic variables (temperature, humidity, and wind speed) (Cavalini, 1992; Mackie, el at. 1998). However, the most significant variation comes from the difference among individuals.

The sensitivity of human olfactory sensory system changes along with the length of period of exposure to odourant concentration, and is characterized by either adaptation or sensitization. Adaptation is the reduction in responsiveness during or following repetitive exposure. Sensitization, conversely, is the increased responsiveness during or following exposure (Wachs et al., 1989). Adaptation to odours can occur on either a short-term or a long-term basis. The short-term adaptation is also called fatigue. During short-term adaptation there is a transient reduction in response to odours during or immediately after exposure. This is generally due to the fact that the activation of receptors to odourants induces a short refractory period during which further stimulation can not occur. This

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short-term adaptation (fatigue) has a significant influence on the behavior of odour panelists; a break must be taken after 15 to 20 min of sniffing to maintain the maximum sensitivity of panel members. During long-term adaptation, there is a more persistent reduction in response that can be measured hours, or even days, following exposure. Long-term adaptation to animal odours occurs in persons who work daily in highly odourous environments. As shown in Table 1-4, odour detection thresholds of workers exposed to acetone and butanol can be 20 to 200 times higher than those unexposed. This chronic exposure to an odour over a long time can modify a person's perceptual world. It explains why persons who work with livestock cannot fully understand the complaints from neighbors who only receive odours intermittently.

Sensitization can be understood as two phenomena, kindling and response facilitation (Frey, 1995). Kindling is a special type of time-dependent sensitization in which repeated, intermittent, sub-threshold stimuli induce an amplification of nerve responses to a convulsive endpoint. Once kindling has occurred, the same low-intensity stimulus that originally evoked little electrophysiological response, now triggers a full seizure. Response facilitation is a type of sensitization in which the threshold for response to a stimulus is lowered when unpleasant stimuli are repeated. Whether the stimuli are perceived as 'strong' depends upon the person. There are wide individual differences in that mild stimuli for most people can be strong for some. Once response facilitation has occurred, such as by a variant of the kindling route, a person can be super-sensitized to a stimulus odour. Thus, there can be a bizarre emotional response to what most of us would perceive as a trivial level of contaminant (Frey, 1995).

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Psychological factors can also play a role in odour perception. Historical experiences and relationship to some odours can change sensitivity and attitude to specific odours. Investigations have shown that implication could also influence odour perception(Frey, 1995).

1.3 Odour measurement

Parameters used to describe odours are odour intensity, odour character, odour hedonic tone, odour persistence, and odour concentration. Odour intensity is the relative strength of the odour above the detection threshold (suprathreshold). The method for determining intensity of an odour is given in ASTM E544 (1988). The odour referencing is accomplished by a comparison of the odour intensity of the odourous air sample to the odour intensity of a series of concentrations of a reference odourant, which is n-butanol. The odour intensity of an odourous air sample is expressed as the best-matched concentration of the n-butanol in parts per million by volume (ppmv). The odour intensity increases as a function of the odour concentration. A larger value of n-butanol means a stronger odour, but not in a simple linear proportion. The dependence may be described as a theoretically derived logarithmic function or as a power function. Researchers (Cain, 1969; Dravnieks et al., 1972; Moskowitz et al., 1974) gave estimates of the perceived odour intensity ratios for n-butanol odours of different concentrations as:

$$I = \left(\frac{X}{Y} \right)^{0.66} \qquad \text{(1-1)}$$

where: I = the perceived odour intensity ratio; and

X, Y = odour concentrations equivalent to the concentrations of n-butanol.

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Suppose there are two odourous samples A and B with intensities equivalent to n-butanol concentrations of 10,000ppmv and 1,000ppmv, respectively. The intensity of sample A in terms of equivalent n-butanol concentration is ten times more than that of sample B, but the perceived intensity ratio is 4.57 (i.e., $10^{0.66}$), which means that sample A smells 4.57 times stronger that sample B. The odour intensity is also influenced by both the odour character and the odour hedonic tone, in addition to the odour concentration.

The odour character is reported by using 'odour descriptors', i.e. what the substance smells like, i.e., fishy, earthy, etc. Odour character is also known as the odour quality.

The odour hedonic tone is a category judgement of the relative pleasantness or unpleasantness of an odour sample. The odour hedonic tone is independent of its character. An arbitrary but common scale for ranking odours by hedonic tone is the use of a 10-point scale: +5 for the most pleasant odour, –5 for the most unpleasant odour, and 0 for a neutral odour. Assigning of a hedonic tone value to an odour sample by an odour panelist is 'subjective' to that panelist. Panelists use their personal experience and memories of odours as a referencing scale. The panelists, during training, become aware of their individual odour experience and memory referencing. The arithmetic average value of the odour panel is the reported 'Hedonic Tone' for the odour sample.

Odour persistence is a term used in conjunction with odour intensity. The perceived intensity of an odour will change in relation to its concentration. However, the rate of change in intensity versus concentration is not the same for all odours. This rate of change is termed odour persistency. The persistency of an odour can be represented as a 'dose-response' function. The dose-response function is determined from intensity measurements of an odour at full strength and at several dilution levels above the

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detection threshold level. As shown in Figure 1-8, the relationship of the logarithm of odour intensity and the dilution ratio are the dose-response function. The slope illustrates the persistency. In Figure 1-8, Odour 'A' with a flat slope, compared to the steep slope of Odour 'B', would have a greater persistence or 'hang time' in the ambient air.

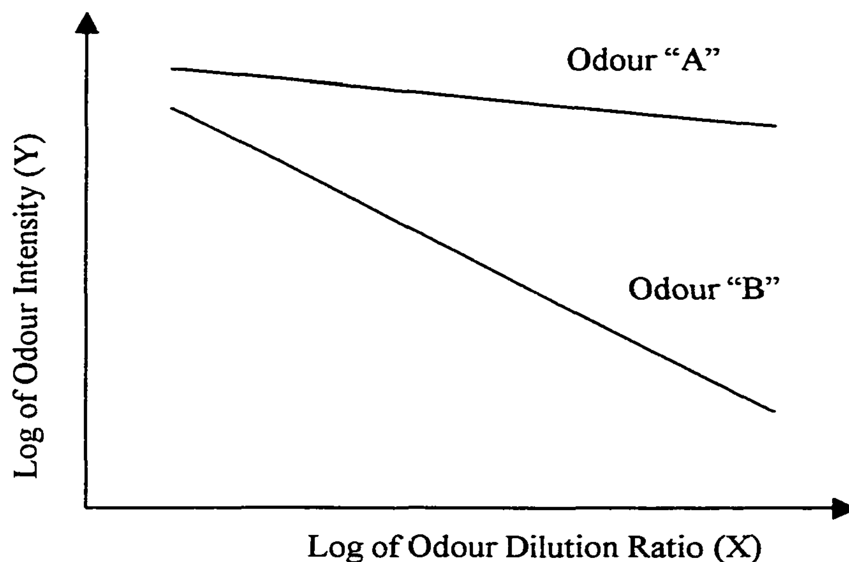

Figure 1-8. Odour Persistence Expressed as Dose-Response

The most important and objective parameter is odour concentration. Parameters of odour intensity, odour persistence, hedonic tone, and odour character are generally applied to the food, beverage, perfume, and cosmetics industry. The odour concentration parameter is applied to all areas related to odour, flavor, and smelling.

The odour concentration, as defined by the ASTM standard (ASTM E-758, 1991) and the draft CEN standard (draft prEN, CEN/TC264/WG2/N222/e, 1998), is measured by determining the mass concentration of pure odourous substances or the odour dilution factor of mixtures of odourants required to reach the detection threshold. At the concentration of the detection threshold, the odour sample, either a pure odourous substance or an environmental odour (a mixture of odourants), has a detection probability

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of 50% under the conditions of the test. The odour concentration is then expressed in terms of the multiple of the detection threshold. The method used in measuring odour concentration is called olfactometry, essential elements of which are a human panel and an olfactometer. Olfactometry uses the human nose as the sensor of odours.

However, the use of human odour panels to evaluate odour samples is labour intensive, time consuming, prone to errors and is difficult to use on-site. An electronic nose could be a candidate apparatus for measuring odour concentration. Electronic noses which are computer-based instruments use a sensor array to mimic the human olfactory system. Many types of materials and technologies have been developed for various sensor arrays. These include metal oxides, lipid layers, phthalocyanines, and conducting polymers (Bartlett et al. 1997). The most popular sensors used in the development of electronic noses are Metal Oxide Semiconductor (Chemoresistive Sensors), Quartz Crystal Microbalance sensors, and Conducting Polymers. The main commercially-available electronic nose based on Chemoresistive Sensors is the Intelligent Electronic Nose that consists of 6, 12, 18 or 32 metal oxide sensors (Alpha M.O.S.America, Inc., Bell Mead, N.J.). A popular alternative to a chemoresistor appears to be the quartz resonator sensor. The unique electrical properties of conducting polymeric materials have been exploited by many research and groups. At least two types of electronic nose are based on conducting polymer arrays: the e-Nose TM 4000 Aroma Analysis System (Neotronics Scientific Inc., Flowery Branch, Ga.) and the AromaScan (AromaScan, Inc., Hollis, N.H.). This sensing technology is based on the adsorption and subsequent desorption of volatile chemical compounds onto an array of proprietary conducting polymers. Adsorption is dynamic and reversible. Each polymer in the sensor array exhibits specific

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changes in electrical resistance upon exposure to different odours and aromas. One constituent of chemicals exposed to the array may interact with certain individual sensors, but not with others. This selective interaction produces a pattern of resistance changes that exhibit a 'fingerprint' of an odour. When an odour is comprised of multiple chemicals, the 'fingerprint' is the sum of their combined interactions with all sensors in the array. An Odour-Map can be made for sample-to-sample comparisons by reducing the dimensionality (the number of sensors in a array) of sample's "fingerprint" to one point on a two or three dimensional space which will be used in the classification, discrimination and recognition of chemical patterns occurring in various kinds of sample(Persaud et al., 1991; Hatfield et al., 1994; Hobbs et al., 1995; Schiffman et al., 1996; Kalman et al., 1997; Byun et al., 1997; Oshita et al., 1999). Recently, researchers (Persaud et al.,1996; Misselbrook et al.,1997) used electronic noses for measuring odour concentration, and preliminary results were obtained. In this project, research was conducted that combining an artificial neural network with an electronic nose to measure odour concentration which will be discussed in Chapter 4.

Difference exists between the ASTM standard and the draft CEN standard in the measurement of odour concentration. In the ASTM system, odour concentration is expressed either in terms of the odourant concentration (g/m^3 or $\mu g/m^3$), or in terms of the dilution factor at the detection threshold and is represented by symbol Z_{OL} (ASTM E679-79, 1979). The panel selected is considered to be representative of the general population (ASTM E-758, 1991). Thus, the odour concentration should be the best estimate of the general population response. In physiological research, panels of 50 or more panelists were used to study olfactory characteristics of the general population

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(Punter, 1983). Obviously, this would be too expensive as a routine approach. Thus, the ASTM standard (ASTM E679-79/91, 1991) recommends that a panel should contain at least eight individuals. An important question is raised as to the level of confidence required about the odour concentration measured when the number of panelists is reduced sharply from 50 to 8. The issue is exacerbated since the variability in olfactory sensitivity between individuals and within an individual in time is considerable (Mills et al., 1963; Wilby, 1969). Actually, the measurement of odour concentration has long been relatively arbitrary.

In the draft CEN system, by defining the European odour unit (OU_E), odour concentration of environmental samples were made traceable to an accepted reference value of a reference material. A European odour unit (OU_E) is that amount of odourant(s) that, when evaporated into 1 m^3 of neutral gas at standard conditions, elicits a physiological response from a panel (detection threshold) equivalent to that elicited by 1 European reference odour mass (EROM) evaporated in 1 m^3 of neutral gas at standard conditions. The European reference odour mass (EROM) is the accepted reference value for the European odour unit, equal to a defined mass of a certified reference material. One EROM is equivalent to 123 μg n-butanol. Evaporated in 1 m^3 of neutral gas, this produces a concentration of 0.040 μmol/mol (ppmv). The odour concentration (OU_E/m^3) is defined as the number of European odour units (OU_E) in 1 m^3 of neutral gas at standard conditions. A strict criterion for selection of panelists was set in the draft CEN standard: The individual detection threshold for n-butanol, using the forced-choice mode, must fall within the range of 20 to 80 ppbv.

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The draft CEN standard is actually a set of strict performance criteria for the dilution instrument, the selection and performance of panelists, the definition of odour concentration, and the olfactometric measurement procedure as a whole. This resulted in a significant decrease of measurement variance for odour concentration. Thus, terminology used in the thesis is adapted from the draft CEN standard.

Table 1-5 Possible data from three panels

Sample	Panel A	Panel B	Panel C
n-Butanol (ppbv)	25	40	60
E-odour (OU_E/m^3)	1,000	2,000	3,000

However, the arbitrary state of odour concentration measurement persists even when using the draft CEN standard. For example, three panels in different laboratories could evaluate a n-butanol and an environmental odour and obtain the results illustrated in Table 1-5. All three detection thresholds for n-butanol fall in the acceptable range. Thus, according to the traceability criterion, all three data values of environmental odour (1,000, 2,000, and 3,000 OU_E/m^3) would fall in the acceptable range with 95% confidence. Thus, all the three data values are "correct", and they must be accepted as being similar.

1.4 Summary

The rate of degradation of swine manure slurry in drain gutters under slotted floors of swine buildings is negligible. The typical digestion process in swine manure earthen storage is anaerobic degradation. Generally, the anaerobic process can be divided into three phases: the hydrolytic phase, the acidogenic phase, and the methanogenic phase. In the degradation of swine manure slurry in earthen storage, the acidogenic phase dominates, thus the acidogenic phase and the methanogenic phase are not in balance.

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The rate of methanogenesis is not high enough to prevent the accumulation for products resulting from the acid forming fermentation. The imbalance between the process of acid formation (the acidogenic process) and methane production (methanogenic process) is the main reason for the accumulation of volatile compounds (i.e., malodourous products) in swine manure. The reasons for the low rate of the methanogenic process are likely low temperature, overloading, low pH, and the presence of heavy metals.

Exposure to unpleasant odour can cause potential health symptoms. The human olfactory system is very sensitive to odour concentration, but very poor in discriminating odour components (at most four) in a mixture. The human olfactory system operates on a model of "second-messenger signaling". A short-term exposure to odours causes fatigue of the olfactory sense which will significantly affect the behavior of an odour panel. A long-term exposure to odours might cause adaptation or sensitization, either of which will permanently modify a person's olfactory world.

Parameters used to describe odours are odour concentration, odour intensity, odour character, odour hedonic tone, and odour persistence. The most important and objective parameter is the odour concentration. Odour concentration is defined as the mass concentration of pure odourous substances or the dilution factor of mixtures of odourants at the detection threshold, which means that, at this concentration, a sample has a probability of 50% of being detected. The method used in measuring odour concentration is olfactometry dependent on a human panel and an olfactometer, and in which the human nose is the sensor of odours. The panel selection method is different between the ASTM system and the draft CEN standard and the interpretation of the odour

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concentration measured is different. Terminology used in the thesis is from the draft CEN standard if not specifically stated.

1.5 Objectives for thesis

The objectives of the work represented in this thesis are:

1) to design and build a multi-panelist olfactometer satisfying requirements of the ASTM and the draft CEN standards with less residual odour, less psychological bias, and lower manufacturing cost;

2) to develop a normalization model of a panel's olfactory response to decrease the variances in odour concentration measurements; and

3) to develop a less labour-intensive, non-human-organ-dependent, and mobile way of measuring odour concentration with reasonable accuracy.

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Chapter 2 Design and Construction of UA Olfactometer

As discussed in Chapter 1, the most important and objective parameter for describing odour is the odour concentration. Odour concentration is measured with an olfactometry method consisting of a human panel and an olfactometer, which uses the human nose as the sensor of odours. An olfactometer is an apparatus in which a sample of odourous gas is diluted with neutral gas by a defined ratio and presented to the panelists.

A variety of olfactometry techniques have been developed to measure odour concentration (Sweeten, 1988). They include the syringe dilution method, the scentometer, the butanol olfactometer, and various dynamic olfactometers (Jones et al., 1992, 1994). Many of these devices do not produce reliable results (Prokop, 1978) and this has limited the effectiveness of past odour research and regulation. Research on current dynamic dilution olfactometers started in the United States in the 1970's (Dravnieks and Prokop, 1975; Dravinieks et al., 1978; Dravinieks et al., 1986) which led to the American Society of Testing and Materials (ASTM) Standard E679 (ASTM E679-79) at the end of 70's. In most research and regulatory institutions in Europe and Australia, dynamic-dilution olfactometry (Dravnieks et al., 1978; Bulley and Phillips, 1980; Franz, 1980; Den Hartigh, 1985; Voorburg, 1986; Hangartner et al., 1991; van Harreveld, 1991; Janes et al., 1992) is now accepted as the standard.

Dynamic olfactometry is a technique in which a stream of odourous air is continuously diluted with a stream of neutral air to a known dilution factor before being presented to a panel of people through a sniffing port. Each panelist performs the odour evaluation task by sniffing the diluted odour from the olfactometer. The response of panelists could be either a "Yes/No" style or a "Forced" one. In the "Yes/No" style, each panelist has only

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one sniffing port and must indicate if an odour is perceived or not (Yes/No) in the air stream. This is an absolute judgment. While in the "forced" style, a panelist is presented with more samples at each of the dilution levels, and may have more than one sniffing ports, usually three. Only one of these samples is odourous, and the other two are neutral air. The location of the odours in consecutive presentations is randomly distributed among the three ports. Panelists are forced to make a choice of which ports contain odour even if no difference is observed. Thus the "forced" style involves a relative judgment. The human olfactory sense is more capable of making a relative judgment than an absolute one (Laing, 1994), and also the "forced" style can eliminate the conservative response bias. Hence, the odour concentration in terms of dilution factor at threshold measured with the "Yes/No" method is lower than that with the "forced" method (Thiele, 1984). The ASTM standard (ASTM E679) only recommends the "forced" style while the proposed European standard (draft prEN, CEN/TC264/WG2, 1998) accepts both.

The odour concentration of samples presented to panelists could be random or presented from low to high concentration. Both of these methods are accepted by the draft CEN standard. Since the human olfactory system may adapt to high concentration, the latter method (odour concentration progressing from low to high) is recommended by both the ASTM standard (ASTM E679) and the draft CEN standard. The statistical approach combining the three-sniffing-option and increasing levels of sample presentation is called "triangular forced-choice ascending concentration series", and is the standard method recommended by ASTM E679.

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Though research on the development of a dynamic olfactometer has been conducted for nearly thirty years, currently only two kinds of olfactometers are commercially available, the St. Croix Sensory ISO olfactometer (one-panelist station) (AC'SCENT, Inc. Website) in the United States, and the TO7 olfactometer (four-panelist stations) (Ecoma GmbH Website) in Germany. Both olfactometers do not have enough panelist-stations (at least eight are required), and samples must be run two or more times to satisfy the requirements of the ASTM standard (ASTM E679) and the draft CEN standard (draft prEN, CEN/TC264/WG2/N222/e, 1998). Thus it is imperative to design and build a suitable olfactometer for odour control research or for regulatory application. The objective of this work was to design and build a multi-panelist olfactometer satisfying requirements of both the ASTM standard and the draft CEN standard with less residual odour, less psychological bias, and lower manufacturing cost.

2.1 Characteristic of the UA Olfactometer

Triangular forced-choice ascending concentration series olfactometers range from single panel-station to multi-panel-station units, each panel-station having either a single sniffing port or three sniffing ports.

2.1.1 Conventional olfactometers

The most recently reported conventional olfactometers (Jones et al., 1994; Huang et al., 1996; Li, et al., 1997; Choiniere and Barrington, 1998) are multi-station, three-sniffing port, triangular forced-choice ascending concentration series devices, and can be simplified as shown in Figure 2-1. A pre-determined flow rate of a neutral air is delivered through Lines 1 and 2, and the odourous air, according to the defined dilution ratio of neutral air and odour sample at each dilution level, is delivered through Line 3.

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Lines 1 to 3 are connected randomly, by a sample-assignment device or a combination of distribution valves, to manifold chambers A, B, and C, at each dilution level. Samples, either neutral air or odourous air, reach sniffing ports a, b, and c at each of the panel stations. Panelists must make a decision which port among a, b, and c is odourous after sniffing samples from each sniffing port at each of the dilution levels either by a guess or by a positive detection.

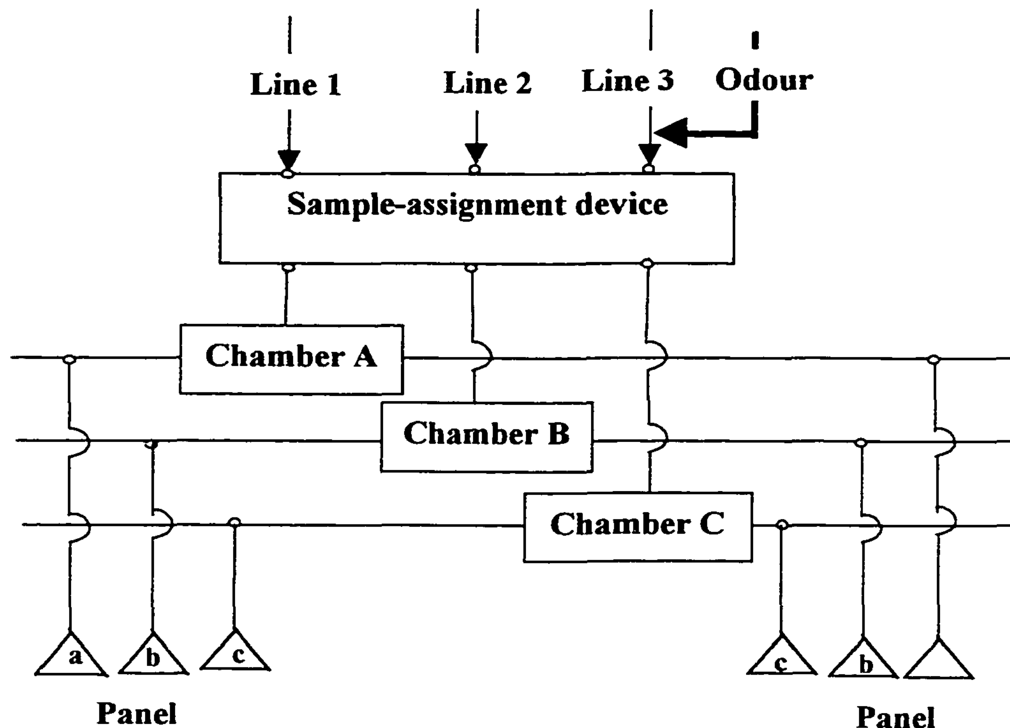

Figure 2-1. Simplified schematic of conventional-design olfactometer

2.1.2 UA olfactometer

The Olfactometer of the University of Alberta (UA Olfactometer) was designed and constructed to satisfy the objectives outlined above. A simplified schematic diagram of the UA olfactometer is shown in Figure 2-2.

The UA olfactometer is a multi-station (eight), single-sniffing port, triangular forced choice ascending concentration series olfactometer. The pre-determined flow rate neutral

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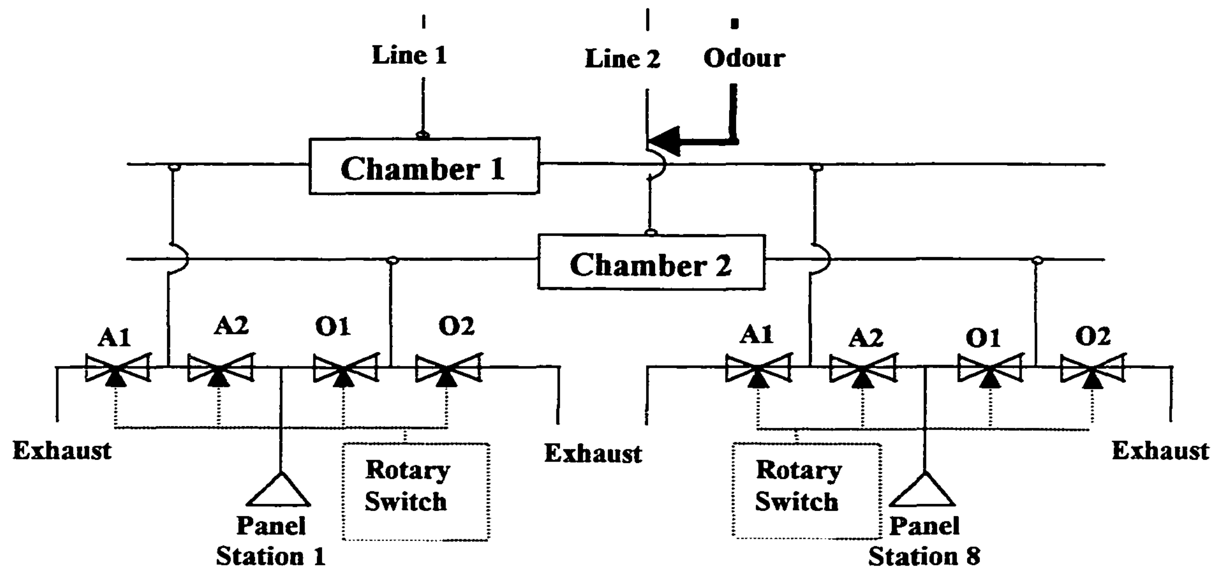

Figure 2-2. Simplified schematic of the UA olfactometer

air is delivered by Line 1, and the odourous air, according to the defined dilution ratio of neutral air to odour sample at each dilution level, goes to Line 2. Lines 1 and 2 are connected to manifold chambers 1 and 2, respectively. Samples, either neutral air or odourous, are presented to each of eight panel stations. Each panelist station consists of a single sniffing port, two rotary switches (for forced-choice decision and hedonic tone), and three buttons. Three samples are represented by three positions on the decision rotary switch. A computer decides randomly which switch position at each panelist station will deliver odourous sample at each dilution level. For example, at a given dilution level, the computer might decide that position 2 will deliver odourous sample. If the panelist puts the rotary switch in position 1 or 3, valves A1 and O1 are closed and A2 and O2 are open, and neutral air from Chamber 1 is presented to the sniffing port. Otherwise, valves A1 and O1 are open, valves A2 and O2 are closed, and odourous air from Chamber 2 is presented to the sniffing port. At each of dilution levels, panelists must decide which, among the three positions, is odourous after sniffing samples from

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the sniffing port. The decision can be reached either by a guess or by a positive detection.

The advantages of the UA olfactometer over the conventionally designed olfactometer have been demonstrated to be as follows:

1). Reduced odour contaminant potential in the system.

The fundamental requirements of an olfactometer are to dilute odourous samples accurately and to have no residual odours. In conventional designs, all tubing, chambers, and valves after and including the sample-assignment device can be assigned to either neutral air or odour samples, thus all these components are at risk of being contaminated by odourous samples. In the UA system, independent of the number of panelist stations, there are only two air chambers: one for neutral air, and the another for odourous air. If the three-way rotary switch is rotated by the panelist to a position disagreeing with the control-program's 'decision', the sample presented to that panelist port is neutral air from the chamber 1, otherwise odourous air is presented from chamber 2. Except a two-feet ¼" tubing to a sniffing port, components for the neutral air are never in contact with odours.

2). Less sample and time required, and no need to purge between dilution levels.

For both systems, when the dilution level ascends to the next one, the odourous sniffing port or the odourous position for the decision-rotary switch generally changes. In the conventional design, a neutral air chamber could have been odourous in the previous dilution level. Thus, a purging of lines and chambers has to be done between dilution levels to remove remaining sample, which means consuming sample and time. In the UA olfactometer, the neutral air chamber always contains neutral air and the odourous

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chamber always contains odourous air, and the odour concentration of the odourous sample in chamber 2 becomes two or three times stronger in the next dilution level. Thus there is no need to purge chambers between dilution levels.

3). Decreased manufacturing cost.

In the conventional designs, all components after and including the sample-assignment device are subject to contact with odourous samples. Thus, they must be made of expensive non-odour-absorbed materials, such as stainless steel or Tedlar ™. In the UA system, only the one chamber is in contact with the odourous samples.

4). Less psychological bias.

In the conventional design with multi-sniffing ports, if a panelist thinks that a port is odourous but the same port was odourous in the previous dilution level, the panelist may hesitate in making this decision. Some panelists sniff in order of sniffing ports, e.g., first sniffing port A and then B and C. This could produce bias. In the UA olfactometer, a panelist always sniffs the same sniffing port. Thus less psychological bias is expected.

2.2 Overall layout and design

The design specifications for the UA olfactometer were as follows:

Duration of sampling: four to five minuses per sample;

Capability: ten samples per hour;

Method: triangular forced-choice;

Order of dilution steps: ascending concentration series;

Scale step of dilutions: 2;

Dilution range: 2^3 to 2^{15} ;

Maximum number of panelist: 8;

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Sniffing ports per panelist: 1;

Amount of sample: three, 10L bags in a pressurized delivering 'lung';

Flow rate from each port: 10L/min;

Method of indicating hedonic tone: 11-position rotary switch;

Method of choosing odourous or neutral samples: turning a three-position rotary switch to different zones (totally three zones).

A detailed schematic is presented in Figure 2-3. Photographs of the UA olfactometer, the pressurized sample lung, and the flow control board are shown in Figure 2-4, Figure 2-5, and Figure 2-6, respectively.

The UA olfactometer consists of a circular table partitioned radially into eight stations to ensure privacy for each panelist. Each panelist station is 470 mm wide and 460 mm in depth and the partitions are 610 mm in height. When seated at a station, panelists cannot see the response buttons or faces of adjacent panelists. The center of the table is occupied by a cylindrical housing (600 mm in diameter, 610 mm in height) for the distribution valves and mixing components. Needle valves are installed on the down-stream lines of both chambers to increase air pressures in the chambers and to adjust the pressure drops from chambers to sniffing ports among eight-panelist stations equally. Thus, the flow rates on the eight sniffing ports are equal. In order to ensure that odours are not absorbed, all the components that contact odours are made of stainless steel or Tedlar ™. The sniffing port consists of a Teflon ™ funnel (70 mm in diameter). A computer with appropriate software is used to control the type of air (odourous or neutral) presented at each station depending on panelist's choice, and the dilution of the odourous air. The computer also scans the output responses from each panelist station by checking

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if a decision button either a 'Guess' or 'Detect' response button is pressed, and stores the response data for each dilution level.

Repeated for each panel station (i.e. 1 to 8)

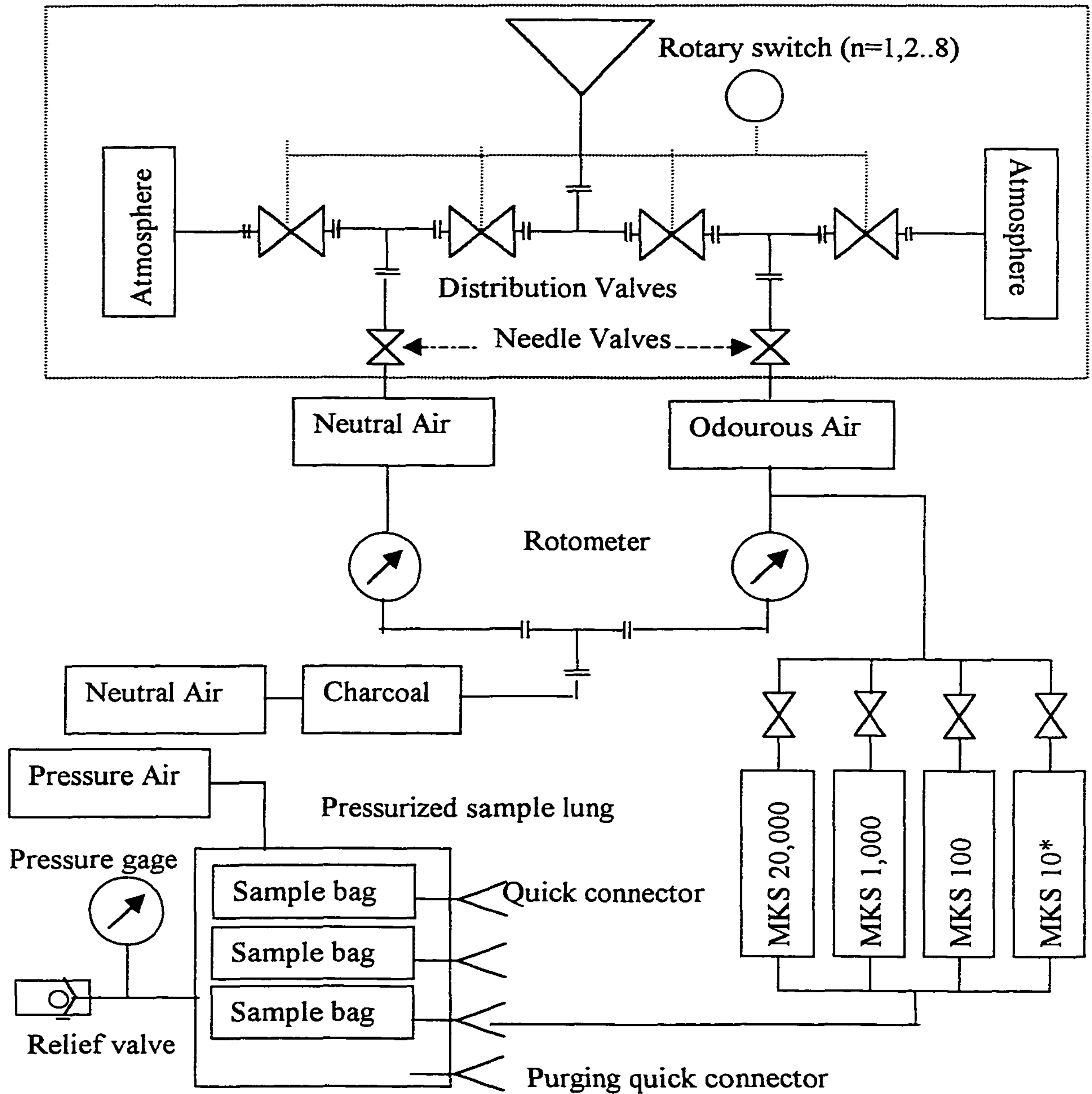

Figure 2-3 Schematic diagram of UA Olfactometer

* mass flow controller, ml/hr

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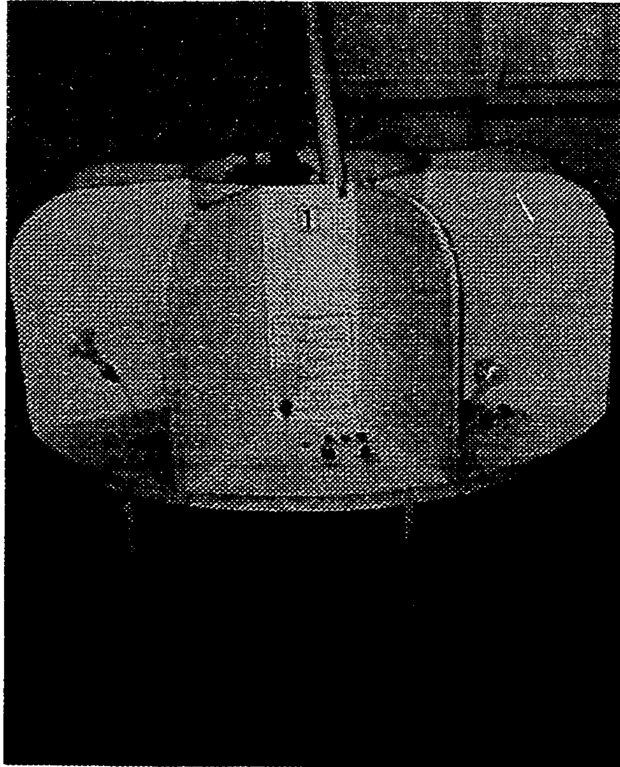

Figure 2-4 UA Olfactometer

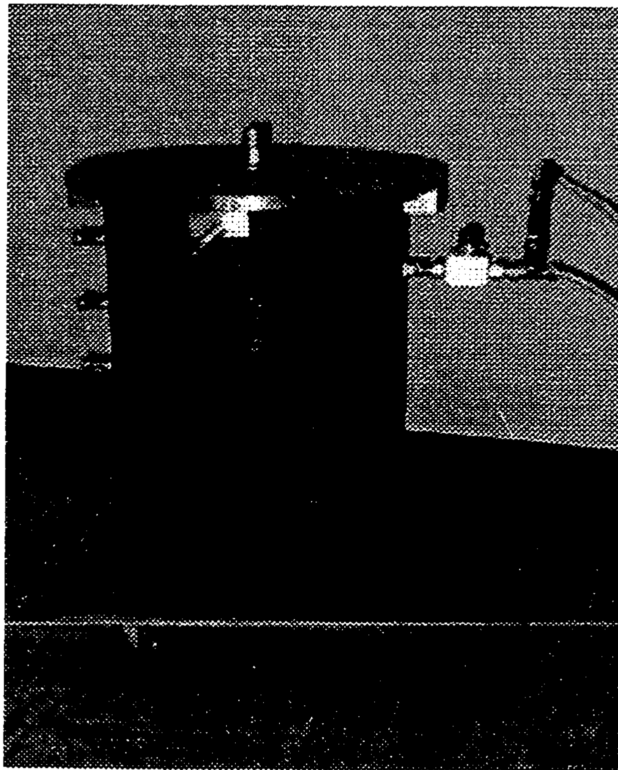

Figure 2-5 The pressurized sample lung

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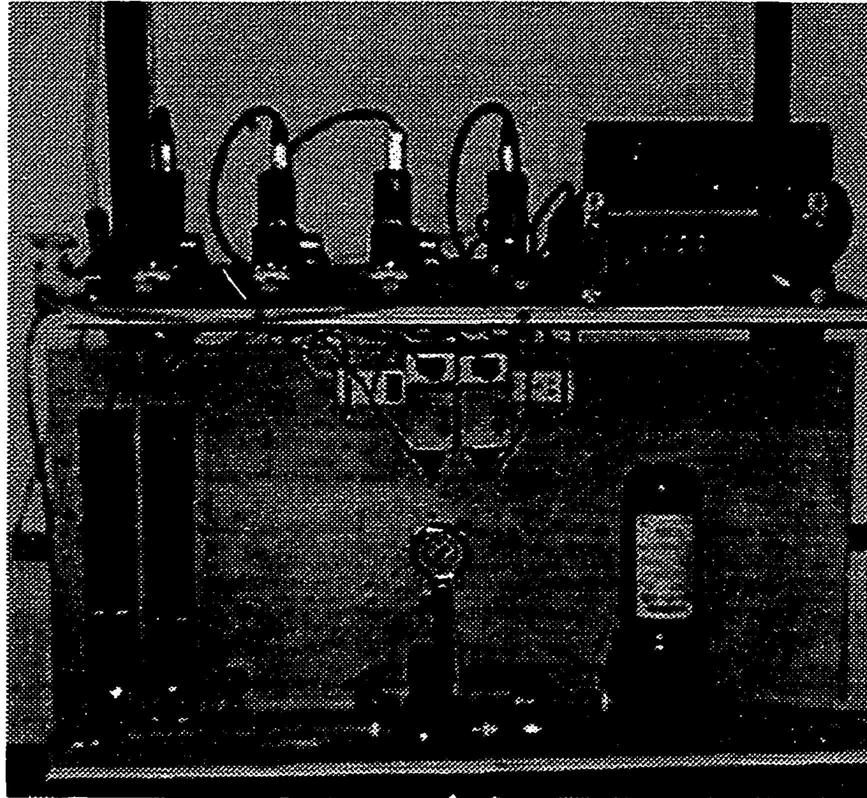

Figure 2-6 Flow rate controller board of UA olfactometer

Computer-actuated mass flow controllers (MKS, Andover, MA) are used to deliver the correct odour flow rate at each dilution level. The controllers deliver the odour sample to the odourous chamber (Chamber 2 in Figure 2-2) at the given rates. Four mass flow controllers are used, and the measurement ranges are 0 – 10 ml/min, 0 – 100 ml/min, 0 - 1,000 ml/min, and 0 - 20,000 ml/min, respectively. If the required flow rate of the odour sample exceeds the capacity of a mass flow controller, the computer software activates the larger flow controller and shuts off the others. These flow controllers can adjust the flow rates for 14 dilution levels, at dilution factors of 2^3 to 2^{15}. The maximum error for MKS mass flow controllers is 2% of the full measurement range. In addition to this error leakage will significantly impact the accuracy of the odourous airflow rate, especially when the system works at high dilution levels. Thus, a valve is installed on the down

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stream side of each mass flow controller to prevent leakage by shutting down it when the mass flow controller tuns off.

Odourous air samples are contained in 10L-sample bags, up to three of which, as shown in Figure 2-5, can be placed in the pressurized sample lung. The lung pressure is maintained at 48.3 kPa (7 psi) to ensure sufficient sample flow from the flow controllers to the odourous chamber for all the dilution levels. A five-minute refreshment break for the panel and sample changing by the panel leader is taken after these three samples have been evaluated within, i.e., every 12 to 15 minutes.

Computer software was written in QBASIC to control the operation and scan the input signals from the I/O boards (P10-24 Keithley Metrabyte, Staunton, MA) installed in the computer. The software program reads the required dilution level from the keyboard and then communicates the odour sample flow rate to the mass flow controllers. A green light comes on at each panelist station when the samples (two neutral one odourous) are ready to be presented. The panelist operates the three-way rotary switch to sample among three options, and presses either the guess or detection button to indicate his/her decision on which switch position corresponds to the odourous sample. The green light will remain off as long as a decision button is pressed. Once all of the panelists have responded, the next ascending dilution level is selected and the process is repeated, until all panelists have detected odour correctly at two consecutive dilution levels.

Hedonic tone can also be measured with the UA olfactometer. A 10-point scale of -5 to +5 is used to rate the offensiveness of odours: +5 the most pleasant odour, –5 the most unpleasant odour, and 0 a neutral odour. The 11-point scale is expressed by the eleven positions on the hedonic rotary switch. The non-diluted agricultural odours are normally

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very offensive and not significantly different in hedonic tone, thus the dilution level for hedonic tone measurement is set at two levels higher than the first detection level. A panelist uses his/her personal experience and memories of odours as a referencing scale to make judgement by setting the hedonic rotary switch and pressing the hedonic button. The computer software scans the hedonic rotary switches and the hedonic tone buttons and records the result.

About ten samples, including refreshment and sample changing time, can be evaluated per hour.

2.3 Calibration

In addition to no odour residual, two additional fundamental requirements for an olfactometer are: 1). Odourous and neutral air are well mixed; 2). The flow rate to each of eight panelist-ports is accurate and uniformly distributed. A calibration was conducted to verify that the UA olfactometer satisfies these requirements.

Since its concentration can be readily measured, carbon dioxide (CO_2) with a concentration of 99% (v/v) was used as tracer gas in place of odourous samples to investigate mixing performance, stability, repeatability, accuracy, and uniformity of flow rates among the eight panelist ports. The CO_2 concentration was measured with a non-dispersive, infrared gas analyzer (Model 846, Beckman, Fullerton, CA).

As shown in Table 2-1, the CO_2 concentration at each sniffing port was very uniform and the maximum standard deviation (SD) was only 1.04%. The SD could be primarily a result of the measurement error. Thus, the neutral air and odour sample are well mixed in the UA olfactometer. The average relative error between the expected and measured CO_2 concentration is 2.2% with an extreme value of 5.9%, satisfying the draft CEN standard's

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requirement of less than 10% error. Since the precision (small SD) is much higher than the accuracy (Dilution ratio error), this error could be caused by the flow rate controllers and the operating program. As long as the flow rate of an odourous sample is set accurately, the error should be near zero. The sniffing ports also were tested by measuring CO_2 concentration to ensure that the neutral air from the room was not drawn into the sniffing ports to further dilute the sample to the panelists. Again, the CO_2 concentration near nostril locations was within 2% of the CO_2 concentration upstream from the port opening.

Table 2-1. Carbon dioxide concentrations at each port and dilution level

Dilution level		6	7	8	9	10	11	12	13	14
Port number	1	*0.025	0.051	0.101	0.205	0.377	0.800	1.602	3.326	6.53
	2	0.025	0.052	0.101	0.204	0.378	0.799	1.601	3.235	6.651
	3	0.024	0.052	0.101	0.204	0.378	0.798	1.602	3.326	6.591
	4	0.024	0.052	0.101	0.203	0.377	0.800	1.603	3.326	6.591
	5	0.025	0.052	0.101	0.204	0.378	0.799	1.602	3.295	6.621
	6	0.026	0.052	0.101	0.204	0.378	0.799	1.602	3.326	6.621
	7	0.026	0.052	0.102	0.204	0.378	0.798	1.603	3.265	6.561
	8	0.025	0.052	0.102	0.205	0.378	0.799	1.602	3.265	6.561
Measured	Conc.	0.025	0.052	0.101	0.204	0.378	0.799	1.602	3.296	6.591
	Dil-rat	3968	1920	987.4	490	264.7	125.15	62.42	30.3	15.2
	SD(10^{-2})	0.59	0.32	0.27	0.53	0.54	0.45	1.04	36.14	39.58
Predicted	Conc.	0.025	0.05	0.1	0.2	0.4	0.8	1.587	3.124	6.25
	Dil-rat	4000	2000	1000	500	250	125	63	32	16
Dil-rat error(%)		0.8	4	1.26	2	5.9	0.12	0.92	5.3	5

*: unit %
Each port reading is averaged from three readings taken on three separate days
Conc.: concentration of CO_2 for each port at different levels
Dil-rat: dilution ratio of (Neutral air + Sample)/ Sample
SD: standard deviation

Since the odourous sample presented to all panelists comes from the same sample chamber, and odour and neutral air are well mixed, both the instability parameter I_d and the accuracy parameter A_d (examples of how to calculate I_d and A_d are demonstrated in the draft CEN standard) of the UA olfactometer should be close to zero. The

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Table 2-2 Flow rate (L/min) at each of panelist ports

Msmt[1]	PORT								Average	SD[2]
	1	2	3	4	5	6	7	8		
1	10.21[3]	10.15	10.15	10.18	10.17	10.32	10.15	10.18	10.19	0.05693
2	9.97	9.92	9.91	9.99	9.92	10.07	9.91	9.93	9.95	0.055742
3	10.08	10.05	10.07	10.08	10.06	10.25	10.02	10.06	10.08	0.069885
4	10.24	10.25	10.23	10.29	10.20	10.10	10.15	10.18	10.21	0.06071
5	10.06	10.07	10.03	10.03	10.06	9.95	10.01	10.05	10.03	0.038822
6	10.01	10.02	10.00	10.01	9.97	10.21	9.92	9.93	10.01	0.089672
7	10.22	9.80	10.00	10.21	10.09	9.87	10.07	10.39	10.08	0.193349
8	10.16	10.20	10.15	10.13	10.14	10.11	10.09	10.06	10.13	0.043425
9	10.24	9.82	10.01	10.23	10.09	9.87	10.07	10.40	10.09	0.195041
10	10.13	9.70	10.20	10.00	9.59	9.60	10.30	9.66	9.90	0.291927
11	10.03	9.57	10.05	9.96	9.52	9.53	10.30	9.59	9.82	0.301541
12	10.17	9.74	10.23	10.02	9.63	9.69	10.48	9.60	9.95	0.327109
13	10.15	9.72	10.21	10.00	9.63	9.69	10.45	9.57	9.93	0.321459
14	10.16	9.73	10.22	10.01	9.62	9.66	10.43	9.58	9.93	0.321778
15	10.18	9.73	10.21	10.01	9.63	9.66	10.46	9.57	9.93	0.329651
16	10.20	9.73	10.21	10.02	9.62	9.66	10.46	9.58	9.94	0.332007
17	10.17	9.73	10.21	10.00	9.63	9.66	10.46	9.57	9.93	0.328261
Average	10.14	9.88	10.12	10.07	9.86	9.88	10.22	9.88	10.00	0.197489
SD	0.0819	0.2049	0.1046	0.1000	0.2508	0.2560	0.2080	0.3050	0.1889	

[1] Msmt: Measurement;
[2] SD: Standard Deviation
[3] Unit: L/min

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performance that should be investigated is the uniformity of flow rates among the eight panelist ports.

As shown in Table 2-2, the average flow rate over 17 measurements at eight ports was 10.0L/min that highly agree with the design value 10L/min. The maximum and minimum flow rates were 10.48L/min (port seven at measurement 12) and 9.52L/min (port five at measurement 11), respectively. Thus, the maximum flow rate error is 4.8%. The average standard deviation within a panelist port was 0.189 with a maximum 0.305 (port eight), and the average standard deviation among eight panelist ports was 0.197 with a maximum of 0.332 (measurement 16). Thus, the distribution of flow rates among panelist ports is uniform. Since needle valves for fine adjustment of flow rates were installed in the system after the above measurements, even better results can be expected. The calibration and test results show that the UA olfactometer operates as it was designed to, and satisfies the requirements of the ASTM standard and the draft CEN standard.

2.4 Conclusions

The following conclusions can be drawn:

1). An eight-panelist-station, single-sniffing port, triangular forced-choice ascending concentration series olfactometer (UA olfactometer) was designed and constructed. Compared with the most recently designed conventional olfactometers, the UA olfactometer has advantages of less odour contaminant potential, sample and time saving, low manufacturing cost, and less psychological bias.

2). The UA olfactometer can analyze ten samples per hour, and it can also be used to measure the hedonic tone of odour.

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3). The neutral and odourous air are mixed well in the UA olfactometer. The distribution of the flow rates among eight panelist ports is uniform.

4). The calibration and test results show that the UA olfactometer works as it was designed to and satisfies requirements of the ASTM standard and the draft CEN standard.

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Chapter 3 Normalization of an Odour-Panel's Olfactory Response

A method for reliable and accurate measurement of odour concentration with acceptable repeatability, reproducibility, and traceability that provides valid odour characterization data that remain stable with time is imperative for the implementation of odour policy, abatement programs, and regulations. In the past, variance of odour concentrations for pure substances published by different researchers remained notoriously large, spanning several orders of magnitude (Ruth 1986). Even during the mid '80s, Harden et al. (1984) reported that, in tests on the same odour sample, the largest ratio between the highest and the lowest odour concentration measurements was 2.5 for the same panel, same day, same olfactometer, and 200 for the same panel, same day, and different olfactometers.

Researchers in developed countries struggled for decades to reduce the analytical variances by standardizing odour measurement (van Harreveld, et al., 1999). Variances were found to be mainly due to: the dilution equipment used; the variability caused by using different panels, and the variability of individual results within a panel (Thiele et al., 1981; Thiele, 1982). A breakthrough in odour measurement was made in the Netherlands in 1993 (Heeres et al., 1993; Heeres et al., 1996) by strict selection of panelists using n-butanol as a reference and by defining the odour unit as:

$$1\ OU_E / m^3 = 40 \text{ parts per billion n-butanol by volume (ppbv)}$$

This led to a significant improvement in repeatability to a factor of 1.5 to 3, with a factor of 5 in an isolated case, and of reproducibility to a factor of 3.3. Thus, n-butanol is now accepted universally as a standard reference odour and is required by the draft CEN standard (draft prEN, CEN/TC264/WG2/N222/e, 1998) as a reference material to serve

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as the basis for quality control and assessment structure, including regular performance evaluation of panelists and of the measurement procedure.

The method that uses n-butanol as a reference is based on the traceability, or transferability, of measurements of environmental odours. Traceability is the property by which a measurement result can be related through an unbroken chain of comparisons to appropriate reference materials, generally national or international reference standards. The need for traceability of odour panelist's performance to different substances was recognized early in the development of odour measurement (Mills, et al., 1963; Wilby, 1969; Dravnieks and Jarke, 1980), and was supported by research conducted in the 1990's (Laska and Hudson, 1991; Harreveld and Heeres, 1995).

To ensure that olfactometry, consisting of an olfactometer and an odour panel, can be generally applied to the assessment of samples of environmental odours, it must be confirmed that the selection of panelists, on the basis of their performance on the reference odour, is also predictive for their performance on environmental odours. The property of traceability indicates that if the sensitivity of a panelist/panel to the reference odour (n-butanol) is high/low, that their relative performance should be the same with environmental odours.

The draft CEN standard requires that measurements of the reference n-butanol by a panel be evaluated with two parameters: accuracy (A) and repeatability (r). The accuracy, A, must be less than 0.217 (calculated from the logarithms of detection threshold expressed in ppbv). If the geometric mean value of thresholds is 40ppbv, the accuracy criterion implies that 95% of results must be in the range of 25 to 65 ppbv. The repeatability, r, must be less than 0.477 (calculated from the logarithms of detection threshold expressed

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in ppbv) (draft prEN, CEN/TC264/WG2/N222/e, 1998). This implies that two single-panel threshold results may not be more than a factor of three apart in 95% of the cases. Thus, if the accuracy and repeatability of measurements on reference n-butanol by a panel fall in the required range, the accuracy and repeatability of measurements on environmental odours by the panel should fall, with 95% confidence, in that range.

Traceability discussed above is actually the property within a panelist/panel to respond predictably to different odours. It can be used for selecting and evaluating panelists, or correlating the odour concentration of a reference with known-concentration odours, but can never be used for correlating the odour concentration of a reference with unknown-concentration odours such as environmental samples (normalizing the unknown odours). The principal requirement for correlating the odour concentration of a reference with unknown-concentration odours is traceability among panelist/panels. The traceability among panelist/panels means that if the sensitivity of a panelist/panel to the reference is higher/lower than that of other panelist/panels, their sensitivity to environmental odours should be higher/lower than that of other panelist/panels.

A question is raised as to whether or not traceability exists among panels. Also if the traceability among panelist/panels is confirmed, a second question is raised: Can the olfactory responses to environmental odours among panelist/panels be modeled by their measured responses to the reference n-butanol? If it can, measurements of environmental odours by different panelist/panels can be normalized and, thus, the variability of odour concentration can be decreased.

The objectives of the work discussed in this chapter were to investigate the assumption that the traceability among panelist/panels exists and, if the assumption was confirmed, to

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develop a model describing the quantitative relationship of olfactory responses among panelists/panels to environmental odours and to the reference n-butanol. A secondary objective was to study panelist behavior and to investigate the impact of gender and age on olfactory response to odours.

3.1 Development of a Normalization Model

Suppose both an environmental odour and n-butanol with a concentration of 40 parts per million by volume (ppmv) are measured for odour concentration on an olfactometer by m panels, and each of panel consists of n panelists. The individual detection thresholds for an environmental sample and a 40 ppmv n-butanol measured by *i*th panelist (*i*=1, 2, ...,n) in the *p*th panel (*p*=1, 2, ..., m) are noted as $x_{p,i}^{env}$ and $x_{p,i}^{nbut}$, respectively. Since the concentration of odour sample is defined as the group detection threshold, which is the geometric mean of the individual detection thresholds of all panelists in the group (ASTM E-679-91, draft prEN, CEN/TC264/WG2/N222/e, 1998), the following two equations are obtained:

$$x_p^{env} = \left(\prod_{i=1}^{n} x_{p,i}^{env} \right)^{\frac{1}{n}} \qquad \text{(3-1)}$$

$$x_p^{nbut} = \left(\prod_{i=1}^{n} x_{p,i}^{nbut} \right)^{\frac{1}{n}} \qquad \text{(3-2)}$$

where: x_p^{env} = the odour concentration (the group detection threshold) of an environmental sample measured by the *p*th panel consisting of n panelists (p=1,2,...m); and

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x_p^{nbut} = the odour concentration (the group detection threshold) of a 40 ppmv n-butanol measured by the *p*th panel consisting of n panelists (p=1,2,...m).

In order to obtain continual variables, $x_{p,i}^{env}$ and $x_{p,i}^{nbut}$ are transformed to logarithm format. The logarithms of both $x_{p,i}^{env}$ and $x_{p,i}^{nbut}$ are independent random variables assumed to be normally distributed:

$$\ln(x_{p,i}^{env}) \sim (\mu_{env}, \sigma_{env}^2) \qquad (3\text{-}3)$$

$$\ln(x_{p,i}^{nbut}) \sim (\mu_{nbut}, \sigma_{nbut}^2) \qquad (3\text{-}4)$$

where: μ_{env} and μ_{nbut} = the true values (the general population expectations) of odour concentrations for the environmental odour and the 40ppmv n-butanol, respectively; and

σ_{env}^2 and σ_{nbut}^2 = the variances of odour concentrations for the environmental odour and the 40ppmv n-butanol, respectively.

From equations (3-1), (3-2), (3-3) and (3-4), the logarithms of both x_p^{env} and x_p^{nbut} should be normally distributed or independent random variables:

$$\ln(x_p^{env}) \sim (\mu_{env}, \frac{\sigma_{env}^2}{n}) \qquad (3\text{-}5)$$

$$\ln(x_p^{nbut}) \sim (\mu_{nbut}, \frac{\sigma_{nbut}^2}{n}) \qquad (3\text{-}6)$$

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The normalization model of panelist olfactory response is based on the assumption that the traceability among panelist/panels exists, which means that if the sensitivity of the *i*th panelist in the *p*th panel to the reference 40ppmv n-butanol is higher/lower than other panelists in the panel, their performance with environmental odours should have a predictable deviation relative to the other panelists. If the group detection threshold is considered to be the neutral, the above assumption can be readily expressed as:

$$y_{env,i} = f\left(y_{nbut,i}\right) \qquad (3\text{-}7)$$

where: $y_{env,i} = \dfrac{x_{p,i}^{env}}{x_{p}^{env}}$, named the environmental odour ratio, and

$y_{nbut,i} = \dfrac{x_{p,i}^{nbut}}{x_{p}^{nbut}}$, named the n-butanol ratio.

The form of the function f is unknown. It could be linear, quadratic, power, exponential, logarithmic or other. Researchers (Cain, 1969; Dravnieks et al., 1972; Moskowitz, 1973; and Moskowitz et al., 1974) gave estimates of the perceived odour intensity ratios (suprathreshold) for n-butanol odours of different concentrations as a function of exponential form. Thus, a reasonable, also a simple, assumption can be made that the function of f is of the form:

$$\ln\left(y_{env,i}\right) = A + B * \ln\left(y_{nbut,i}\right) \qquad (3\text{-}8)$$

where: A and B are constant.

From equation (3-8), equation (3-9) can be derived:

$$\tilde{x}_{p}^{env} = x_{p,i}^{env} * \exp\left\{ \left(A + B * \ln(y_{nbut,i})\right)\right. \qquad (3\text{-}9)$$

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where: $\tilde{x}_p^{env}$ = the estimate of x_p^{env} based on the *i*th panelist measurement of $x_{p,i}^{env}$ and $x_{p,i}^{nbut}$.

Equation (3-9) can be used to estimate the *p*th panel's measurement of the group detection threshold of an environmental odour based only on the measurement of the *i*th panelist. However, it should be stressed that an estimate based only on the measurement of one panelist is not reliable and is not of interest. What is interesting is how to estimate the true value of the group detection threshold of an environmental odour based on the measurement of one panel. The group detection threshold of an environmental odour measured by m panels is the geometric mean of the measurement of each of panels (ASTM E679-91), thus the following two equations are obtained:

$$x^{env} = \left(\prod_{p=1}^{m} x_p^{env} \right)^{\frac{1}{m}} \qquad \text{(3-10)}$$

$$x^{nbut} = \left(\prod_{p=1}^{m} x_p^{nbut} \right)^{\frac{1}{m}} \qquad \text{(3-11)}$$

where: x^{env} = the group detection threshold (the odour concentration) of an environmental odour measured by m panels; and

x^{nbut} = the group detection threshold (the odour concentration) of the 40 ppmv n-butanol measured by m panels.

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When the panel number m tends to be infinity, both x^{env} and x^{nbut} tend to the true values (the general population expectations) of odour concentrations of an environmental odour μ_{env} and of the 40ppmv n-butanol μ_{nbut} , respectively.

Comparing equations (3-1) and (3-2) with (3-10) and (3-11), (3-3) and (3-4) with (3-5) and (3-6), and assuming that traceability exists among panelist/panels, equation (3-12) can be obtained:

$$x^{env} = x_p^{env} * \exp\left\{-\left(A + B * \ln \frac{x_p^{nbut}}{x^{nbut}}\right)\right\} \qquad \text{.........................(3-12)}$$

Now, if the panel number m tends to infinity, equation (3-12) can be written:

$$\widetilde{\mu}_{env} = x_p^{env} * \exp\left\{-\left(A + B * \ln \frac{x_p^{nbut}}{\mu_{nbut}}\right)\right\} \qquad \text{.........................(3-13)}$$

where: $\widetilde{\mu}_{env}$ = the estimate of the true value (the general population expectation) of the detection threshold of an environmental odour based on the *p*th panel measurements : x_p^{env} and x_p^{nbut} .

3.2 Data Collection and Processing

An experiment was conducted to collect data for testing the hypothesis, determining constants A and B in the model equation (3-8) by regressing and for training an electronic nose to measure odour concentrations (Discussed in Chapter 4). Eight sets of samples were collected from ventilation exhaust fan outlets of swine buildings in the Edmonton vicinity and from a slurry storage center on the Edmonton Research Station of the University of Alberta, as illustrated in Figures 3-1, and 3-2. Two sample sites were

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chosen on each farm, as shown in Table 3-1. n-Butanol with a concentration of 40ppmv was used as the reference as recommended by the draft CEN standard. Forty-four persons were hired to evaluate these nine samples (eight environmental sources and one 40ppmv n-butanol) on the UA olfactometer as described in Chapter 2 and shown in Figure 3-3. In fact, forty-eight persons were hired initially, however two persons decided to withdrew during the experiment and two were dismissed due to insensitivity to odours.

Figure 3-1 The sample collecting vehicle and equipment

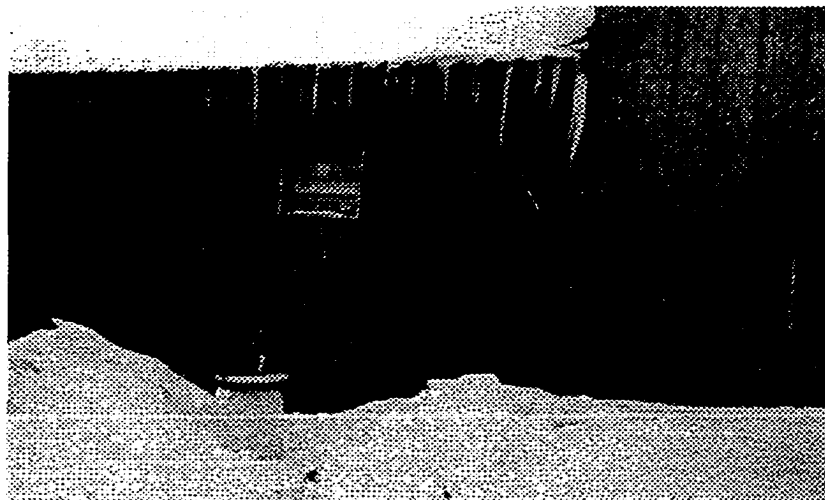

Figure 3-2 Collecting sample from the exhaust of swine building

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Table 3-1 Sources of odour samples

Farm	Sample Site I	Sample Site II
Farm 1	Slurry Processing Center	Sow/Weaning
Farm 2	Weaning	Finishing
Farm 3	Finishing	Growing
Farm 4	Finishing	Growing

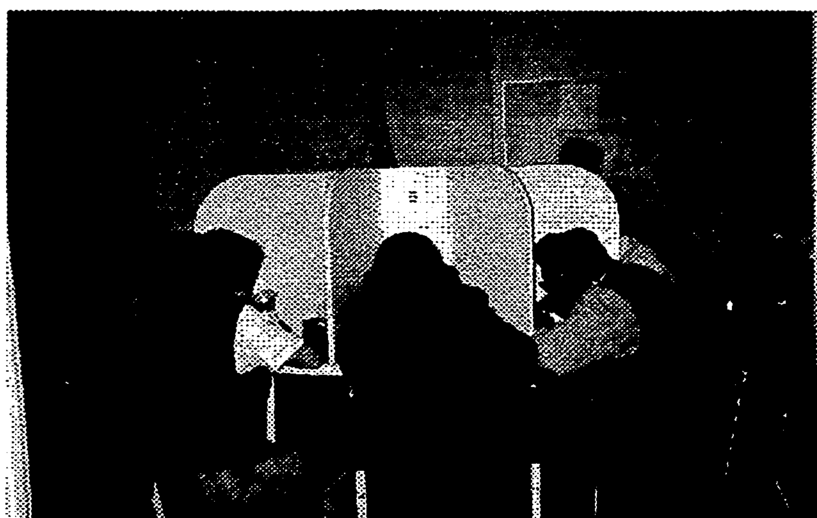

Figure 3-3 Panelists sniffing samples on the UA olfactometer

The requirements for being a panelist were good health, being a non-smoker, and having a 'normal nose' judged by the panelists themselves. The draft CEN standard gave strict requirements for selecting qualified panelists in terms of sensitivity and stability as discussed later. However, the number of such qualified panelists is small, and if only qualified panelists were included, both the size of the data set and the range of variable (sensitivity) would decrease, which would result in decreasing reliability of regression. Thus, panelists hired in the experiment were not strictly screened and did not all meet the ASTM and the draft CEN requirements.

The experiments lasted for three weeks from February 23 to March 12, 1999 and were conducted on Tuesday and Friday of each week. The forty-four persons were randomly

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assigned to six panels on each of the experimental days for six evaluation sessions. Each of the odour samples was evaluated on the UA olfactometer by these six panels.

To determine parameters A and B in equation (3-8), data collected in the experiment had to be processed into the form of the n-butanol ratio (y_{nbut}) and the environmental odour ratio (y_{env}). Consequently, nine data values were collected for each panelist each day, i.e., eight environmental odours and one 40ppmv n-butanol sample. Since each measurement for these eight environmental odours and the same 40ppmv n-butanol were not independent, the geometric mean of these eight odour measurements was used as the equivalent odour measurement. The geometric means for each individual and each panel were calculated with equations (13-4) and (3-15), respectively:

$$x_{p,i}^{env} = \left(\prod_{s=1}^{8} x_{s,p,i}^{env} \right)^{\frac{1}{8}} \quad \text{.......(3-14)}$$

$$x_{p}^{env} = \left(\prod_{s=1}^{8} x_{s,p}^{env} \right)^{\frac{1}{8}} \quad \text{.......(3-15)}$$

where: $x_{s,p,i}^{env}$ = the individual detection threshold for the *s*th environmental odour (s=1,2...8) measured by the *i*th panelist (*i*= 1,2,....n) in the *p*th panel (p=1,2,...m);

$x_{s,p}^{env}$ = the group detection thresholds for the *s*th environmental odour (s=1,2...8) measured by the *p*th panel (p=1,2,...m).

Thus, dilution ratios of eight environmental odours were condensed into one equivalent odour dilution ratio, and the data set was processed into one equivalent odour dilution

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ratio (compacted environmental odours) and one n-butanol dilution ratio. In total, the data set consists of 252 cases.

3.3 Regression results

A linear regression was conducted to determine parameters A and B, and the results are shown in Figure 3-4. The regression is significant at the level of α=0.0001 and the residuals are homogenous, as illustrated in Figure 3-5.

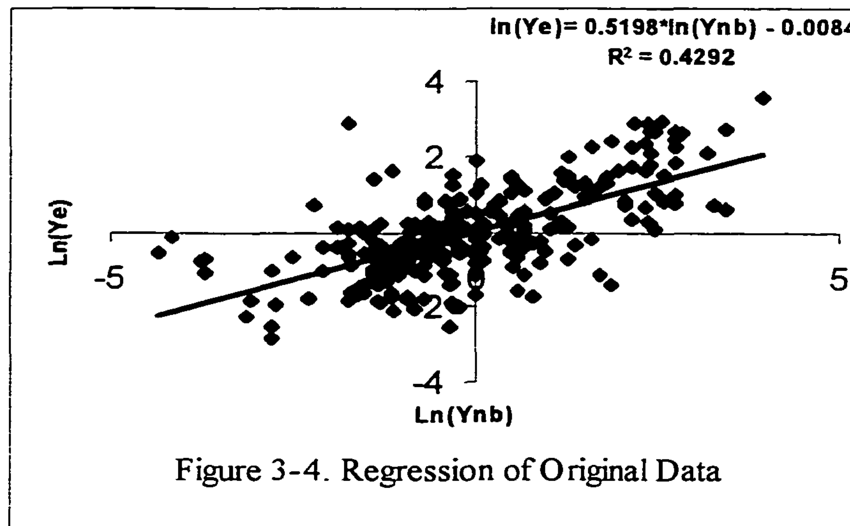

Figure 3-4. Regression of Original Data

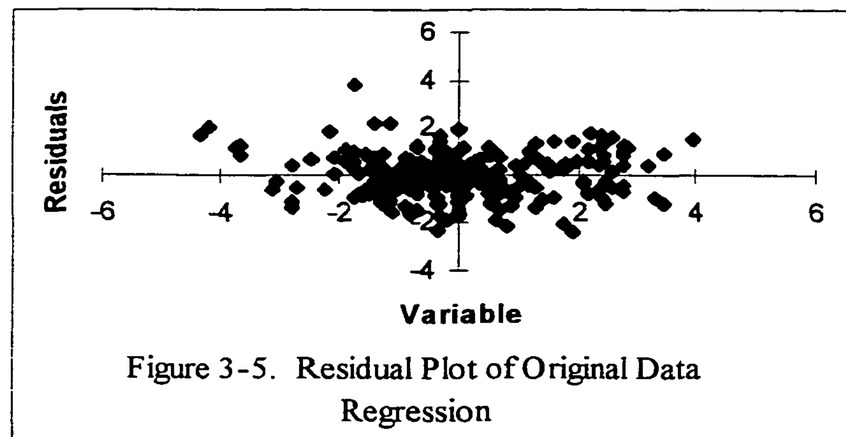

Figure 3-5. Residual Plot of Original Data Regression

However, outliers can be observed in both Figure 3-4 and Figure 3-5. Residuals of regression were checked with a standard deviation (SD) of 2σ as follows:

$$R = y_{env} - \hat{y}_{env} \quad \text{......(3-16)}$$

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where: $\hat{y}_{env}$ = the predicted y_{env}

If a residual is larger than or equal to a SD of 2σ (σ=1.0635), that point was considered to be an outlier with 95% confidence and was removed from the data set. In total, ten points were removed. Since the intercept constant in equation (3-12) should be zero to ensure that the normalization parameter was equal to 1 when $x^{nbut} = x_p^{nbut}$, and since it was close to zero in the regression of the original data shown in Figure 3-4, the option was taken to force the intercept constant to zero (A=0). The regression results for the data set after removing outliers are shown in Figures 3-6, 3-7, and 3-8. The regression is significant at the level of α=0.0001, and the residuals are homogenous, and the distribution of residuals is normal.

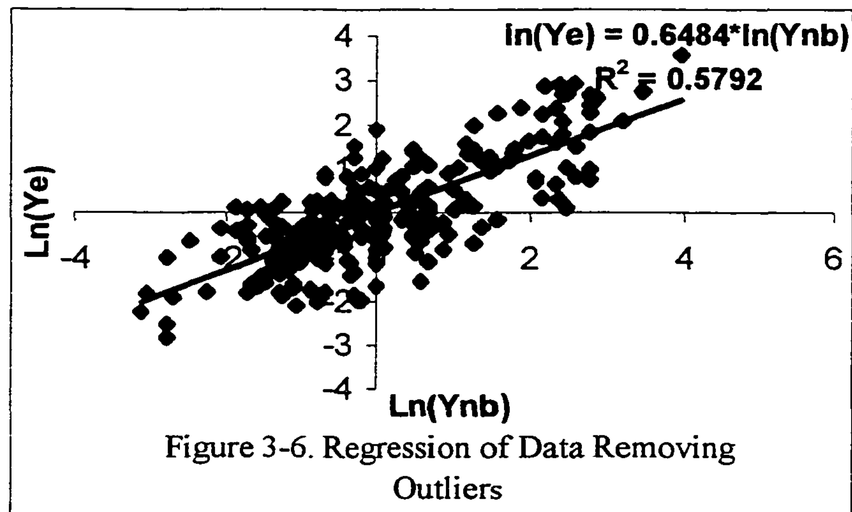

Figure 3-6. Regression of Data Removing Outliers

From Figure 3-6, equation (3-13) can be written in the form of:

$$\tilde{\mu}_{env} = x_p^{env} * \left(\frac{x_p^{nbut}}{\mu_{nbut}} \right)^{(-0.6484)} \quad \text{.. (3-17)}$$

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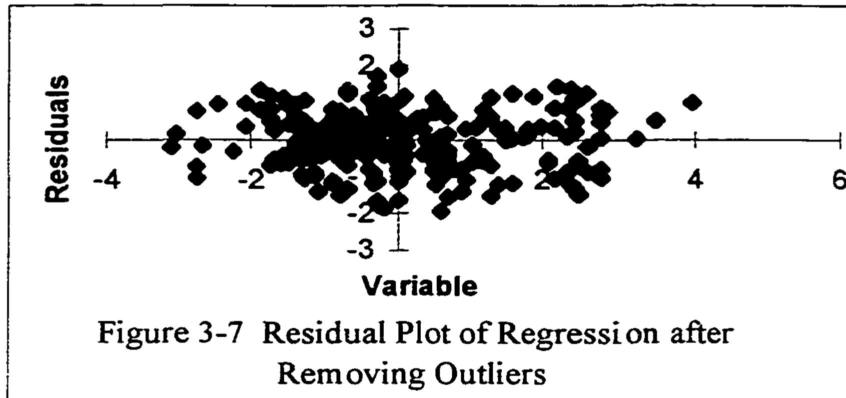

Figure 3-7 Residual Plot of Regression after Removing Outliers

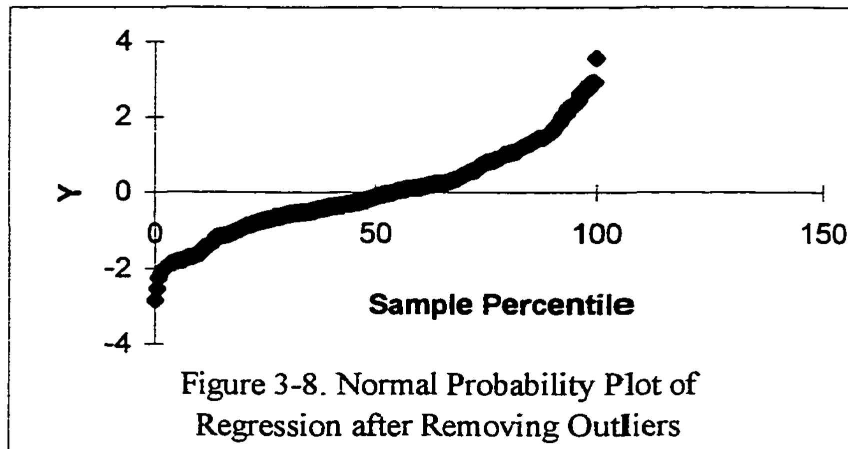

Figure 3-8. Normal Probability Plot of Regression after Removing Outliers

Equation (3-17) can be used to estimate the true value of the odour concentration of an environmental sample based on the measurements of one panel to the environmental sample and to the 40ppmv n-butanol, or, in other words, to normalize the measurement of an environmental sample to a standard value with a reference of 40ppmv n-butanol.

3.4 Verification and Discussion

Since the regression of equation (3-17) is significant at the level of α=0.0001, the conclusion can be drawn that the response of panelists/panels to environmental odours is a function of their response to a standard material (n-butanol) and, therefore, the assumption of traceability among panelists/panels is confirmed.

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Equation (3-17) can be written in the form: $\tilde{\mu}_{env} = K * x_p^{env}$, where K is the normalization parameter, and

$$K = (R_{nbut})^{-0.6484} \qquad (3\text{-}18)$$

where: $R_{nbut} = \dfrac{x_p^{nbut}}{\mu_{nbut}}$, the n-butanol ratio.

The equation (3-18) is plotted in Figure 3-9. The normalization parameter K for qualified panelists whose n-butanol detection thresholds were within 20 to 80 ppbv is listed in Table 3-2. The range of n-butanol ratio for qualified panelists is by a factor of four from 0.5 to 2, but the normalization parameter K only changes 2.457 times from 0.634 to 1.567. This result agrees with the conclusion (Harreveld and Heeres, 1995) that the response to environmental odours has a smaller variability than that to the reference n-butanol.

Equation (3-17) describes the traceability of olfactory sensitivity among panelists/panels; thus it should be independent of samples. As discussed in Chapter 1, researchers (Cain, 1969; Dravnieks and Laffort, 1972; Moskowitz, 1973; Moskowitz et al., 1974) gave estimates of the perceived odour intensity ratios for n-butanol odours of different concentrations as:

$$I = \left(\frac{X}{Y}\right)^{0.66} \qquad (3\text{-}19)$$

where: I = the perceived odour intensity ratio;

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X, Y = odour concentrations equivalent to the concentrations of n-butanol.

Comparing the exponent 0.66 with -0.6468 in equation (3-17), and ignoring the sign, an inference can be made that equation (3-17) may represent all environmental odours. However, further experiments are required to confirm this inference.

Table 3-2 Normalization Parameter K vs. N-Butanol Ratio R_{nbut} for Qualified Panelist

ppbv	80	70	60	50	40	30	20
R_{nbut}	2.00	1.75	1.50	1.25	1.00	0.75	0.50
K	0.634	0.696	0.769	0.865	1.00	1.205	1.567

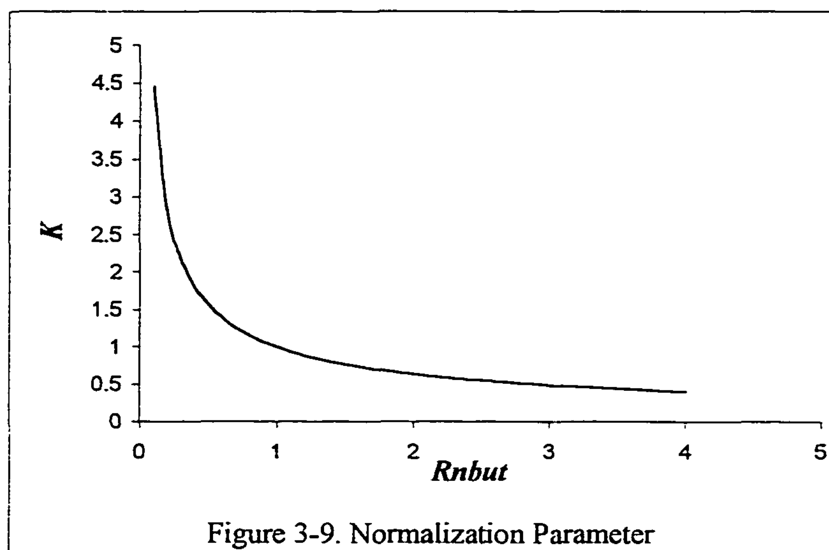

Figure 3-9. Normalization Parameter

The draft CEN standard requires two parameters, sensitivity and stability, to select panelists. To meet this requirement, an experiment must be conducted to measure the response to ten n-butanol samples during three sessions, each session being separated by at least 24h. The detection threshold for qualified panelists should be within the range 20 to 80 ppbv (sensitivity), and the logarithm of the variation should be less than or equal to 2.3 (stability). These criteria imply the selection of a well-defined subset of the general population, not necessarily representative of the general population. Also, the 40ppbv n-

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butanol standard is just a consensus value, not necessarily the true value for the general population. In this experiment, the odour concentration of the 40ppmv n-butanol measured by the 44 panelists was 761 OU_E/m^3 (60ppbv), with a logarithmic standard deviation $\sigma = 0.681$. Thus, this super-panel consisting of 44 panelists satisfies the criterion (20-80ppbv) required by the draft CEN standard, although not all individuals do separately.

Since Equation (3-17) was developed on the data of olfactory responses from 44 persons (sub-sample of the general population having normal olfactory sense), the model should be applicable to the population who have normal olfactory sense. If all 44 panelists satisfied the requirements of the draft CEN standard, since they belonged to a subset of the population who have normal olfactory sense, the model would be the same. However, further experimentation is needed to collect data based on qualified panelists.

As defined in the draft CEN standard, 1 OU_E/m^3 is equivalent to 40ppbv n-butanol, and μ_{nbut} is defined as 1000 OU_E/m^3 for reference n-butanol with a concentration of 40 ppmv. As discussed above, both σ^2_{nbut} and μ_{nbut} are characteristics of a well-defined subset of the general population, and not necessarily the characteristics of the general population.

The original data set developed in the experiment conducted during March to April 1999 was processed to verify equation (3-17). Though the same original data set was processed to regress equation (3-17), the methods and principles of data processing were different. In processing data for regression, data were processed by calculating geometric means of odour concentrations of eight environmental samples and the odour

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concentration of 40ppmv n-butanol measured by each of the panelists. Thus the experimental unit was the individual panelist, and forty-four odour dilution ratio data points could be collected per experimental day since forty-four panelists were involved in the experiment. In processing these data for verification, the geometric means were calculated of odour concentrations of eight environmental samples and the odour concentration of 40ppmv n-butanol measured by each of the panels. Thus the experimental unit was the odour panels, and only six data values could be collected per experimental day because there were only six sessions (panels) per experimental day.

By combining all the measurements of the eight samples over the experimental period into one, a comparison was made among different procedures: the original data measured by each of panel, the super-panel data, and the original data normalized by equation (3-17). The results are shown in Table 3-3.

Table 3-3 Comparison among Original, Super-panel, and Normalized Data

Objects		Original	Super-panel	Normalized
Average	log	6.6346	6.66	6.909
	Anti-log	760.6	778.0	991.9
Log(Stdev)		0.567	0.155	0.500

As discussed above, the odour concentration of the 40ppmv n-butanol measured by the 44 panelists (i.e. the super-panel) was 778 OU_E/m^3, 22.2% lower than 1000 OU_E/m^3, though not statistically significantly different. The "true" value of the odour concentration (measured by an infinitely large panel consisting of qualified panelists) for the environmental samples was unknown, but must be higher than the value measured by the super-panel. As shown in Table 3-3, the normalized odour concentration for the

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environmental odours was 992 OU_E/m^3, 27% higher than that of the super-panel, which suggests that this value is closer to the "true" value. The logarithm standard deviation, compared to the original one-panel data, is decreased. Thus, the measurement results are improved by normalization.

The normalization of the human olfactory response could significantly decrease the variance of measured odour concentration, and it is suggested that a series of experiments covering main sources of agricultural odours should be conducted in several laboratories to verify this model. If this model, after modification according to all laboratories involved, is confirmed, it should be standardized as a method of the measurement of odour concentration.

3.5 Survey of Panelist Behavior

In the experiment conducted during March to April 1999, 17 out of 44 persons, 39% of total panelists, satisfied the requirements of "normal panelist" defined in the draft CEN standard. An experiment was also conducted to recruit more panelists. In this case, 8 out of 27 (29%) satisfied the requirements. On the basis of these limited samples, roughly only 35% of the general population are qualified to be panelists. Thus, every qualified panelist is a very precious resource in odour concentration measurement.

In terms of sensitivity and stability of olfactory response, people can be categorized, as shown in Table 3-4, into two classes and six groups.

Table 3-4 Sensitivity and Stability of Human Olfactory Response

	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6
Sensitivity	H	H	N	N	I	I
Stability	S	U	S	U	S	U

H, N, and I = High sensitivity, normal sensitivity, and insensitive, respectively.
S and U = Stable and unstable, respectively.

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Groups 1, 3 and 5 belong to the stable class, and Group 3 consists of qualified panelists. In this class, the olfactory response to odours is stable, and every member works sincerely with high commitment. Thus, Group 1 and Group 5 could be used as panelists by normalizing their measurements with equation (3-17) if the number of people in Group 3 is not enough. Groups 2, 4, and 6 belong to the unstable class. The reasons for instability could be physiological or psychological. The physiological reasons include fatigue- oriented olfactory character, unhealthy conditions, and impaired olfactory organs similar to a person with impaired hearing. Such people normally do not hear but occasionally do. The psychological reasons could include competition or absent-mindedness. Persons within the unstable class should never be included in odour panels, because the measurements by this class are not reliable, and no method exists for processing these measurements to obtain reliable results.

In addition to "sensitivity" and "stability", "condition" also should be added to the requirements for being a qualified panelist. A qualified panelist, who passes the recruiting test, does not necessarily qualify every time. Cases were observed during the experiment that the detection threshold measured by qualified panelists was far beyond the normal range just because these panelists were tired or distressed even in a "healthy" state. If this occurs, these panelists should be excluded from the panel. As recommended by the draft CEN standard, a qualified panel should be composed of no less than four panelists after removing panelists whose condition is not acceptable.

A statistical analysis was conducted to investigate the influences of gender and age on the olfactory response of the panelists. Data were processed in the form of $\ln(y_{env})$,

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logarithm of the environmental odour dilution ratio, similar to that carried out in regression. The summary of the statistical analysis is shown in Table 3-5.

Table 3-5 Influence of Gender and Age on Human Olfactory Response

	Gender		Age				
	Male	Femal	18-25	26-30	31-35	36-40	Over 40
No. of Panelists	23	21	14	12	9	5	4
Mean	-0.035	0.062	-0.337	0.332	-0.125	-0.236	0.735
Std Deviation	1.541	1.491	1.156	1.383	1.537	1.421	1.690
Statistics Test	Not significant		Significantly different ($\alpha = 0.005$)				

Although it is usually assumed that females are more sensitive to smell and odour than males, females are involved with more odours such as perfume and cooking in routine life than males. Statistical analysis of these data shows that there is no significant difference between males and females in responding to the experimental odours.

Based on ages, the forty-four panelists were divided into five groups from under 25 to over 40. Statistical analysis shows that the means of logarithms of individual panelist measurements were statistically significantly different among groups. The lowest mean value occurred in the group under 25 while the highest value occurred in the group over 40. This result is contrary to expectations and two reasons may be involved:

1). Small sample size.

Forty-four people is a large sample size for regression and training electronic nose, but it is small for investigating the influence of age on human olfactory sensitivity to odours, because, after dividing by five, each group has only 8.5 persons on average.

2). Uneven distribution of persons on each group.

The number of persons in each group were very different, and the biggest group, the group under 25, consisted of 14 persons, while the smallest group, the group over 40,

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consisted of only 4 persons. This uneven sample size will cause non-homogeneous residuals and unreliable results. The fluctuation among groups suggests there is no clear tendency based on age. However, the data doesn't allow a confirm assessment of effect of age on the olfactory sensitivity to odours.

3.6 Conclusions

The following conclusions can be drawn from this study:

1). The traceability among panelists/panels exists which means that the response to environmental odours of panelists/panels can be related to their response to a standard odour (n-butanol);

2). Based on the property of traceability among panelists/panels, n-butanol can be used as a reference odour to normalize the human olfactory response to odours. The regression of this model is significant at the level of α=0.0001. The normalization model is as follows:

$$\tilde{\mu}_e = x_p^e * \left(\frac{x_p^{nb}}{\mu_{nb}} \right)^{(-0.6484)}$$

3). The true value of the odour concentration can be estimated on the basis of measurements of one panel on an environmental odour and n-butanol. By using the model, the measurement results are improved;

4). The detection threshold of the 44 panelists for the 40ppmv n-butanol was 761OU_E/m^3, satisfying requirements of the draft CEN standard as a whole, even though not all-individual panelists satisfied selection criteria;

5). Since the model was developed from data within the general population, it should be applicable to the population who have normal olfactory sense, not necessarily only to the

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well-defined subset of the population by the draft CEN standard. The model would be the same even if all panelists satisfied the criteria;

6). Variability of response to environmental samples is smaller than response to reference n-butanol.

7). The normalization model equation (17) should be valid for all environmental odours. However, further experiments are needed to confirm this inference.

8). A series of experiments covering the main sources of agricultural odours should be conducted in several laboratories to verify this model. If this model, after modification, is confirmed, it should be standardized as part of the method of measurement of odour concentration.

9). Gender does not have significant influence on human olfactory sensitivity to odours. . However, the data do not confirm the assessment of effect of ages from under 25 years old to over 40 on the olfactory sensitivity to odours.

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Chapter 4 Combining an Electronic nose with an Artificial Neural Network to Measure Odour Concentration

At present, olfactometry, consisting of an olfactometer and a human odour panel, is the most precise method for quantifying odours, since the human nose can detect compounds at low concentrations that cannot be detected by any other method. However, the use of human odour panels to evaluate odour samples is labour intensive, time consuming, prone to errors and is difficult to use on-site.

The accuracy of odour concentration measurement depends on both the olfactometer and the odour panel. Huge variations of human olfactory sensitivity exist, and considerable variations exist among qualified persons with time (Harden et al., 1984). Even if the variation was decreased significantly by panelist selection, panel olfactory response normalization, and by using an olfactometer with high reproducibility among laboratories and repeatability within a laboratory, the measurement of odour concentration is still labour intensive, time consuming, difficult to use on-site, and requires large numbers of panelists to achieve confidence. Thus, there is a clear need for a less labour-intensive, non-human-organ-dependent, and mobile way of measuring odour concentration that is at least as precise and accurate as the olfactometry method.

An electronic nose becomes a candidate for measuring odour concentration, since it uses an array of sensors to mimic the human olfactory system in the classification, discrimination and recognition of chemical patterns occurring in various kinds of samples (Persaud et al., 1991; Hatfield et al., 1994; Hobbs et al., 1995; Schiffman et al., 1996; Kalman et al., 1997; Byun et al., 1997; Oshita et al., 1999). This sensing technology is based on the adsorption and subsequent desorption of volatile chemical compounds onto

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an array of proprietary conducting polymers. Each polymer in the sensor array exhibits specific changes in electrical resistance upon exposure to different odours and aromas. One constituent of chemicals exposed to the array may interact with certain individual sensors, but not with others. This selective interaction produces a pattern of resistance changes exhibiting a 'fingerprint' of an odour. When an odour is comprised of multiple chemicals, the 'fingerprint' is the sum of their combined interactions with all sensors in the array. In addition to odour composition, odour concentrations can generate different responses in an electronic nose. Figure 4-1 shows the 'raw' response of the two samples, grapefruit (A) and orange essence oil (B), on a commercially available electronic nose (AromaScan). The different responses are an indication of the difference in odour concentration, thus sample 'A' shows higher odour concentration than sample 'B'. The AromaScan electronic nose has an array of 32 conducting polymer sensors, and Figure 4-2 represents the overlay of the normalized 32 sensor responses, the 'fingerprints', for the samples 'A' and 'B'. The height of the bars on the histogram represents the average degree of difference between the two samples at each individual sensor.

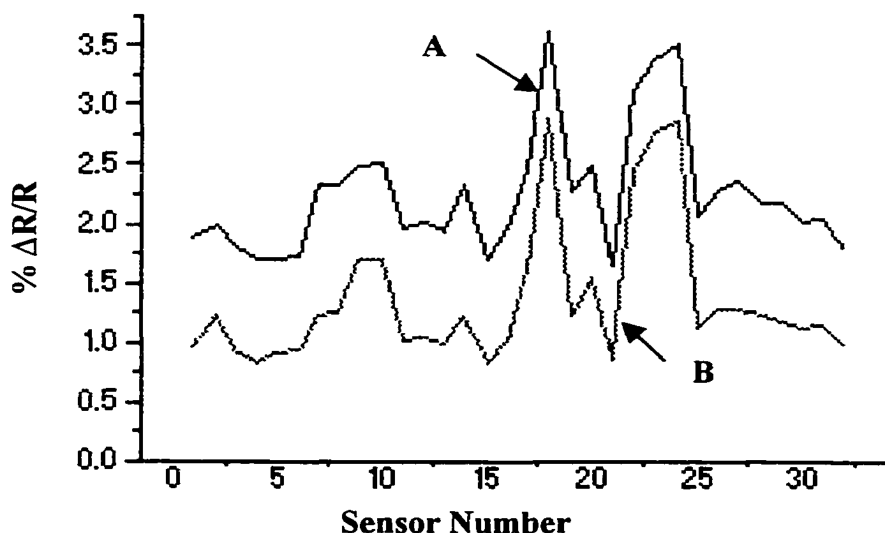

Figure 4-1. Raw responses of grapefruit oil (A) and orange essence oil (B) on an electronic nose (AromaScan website 1999)

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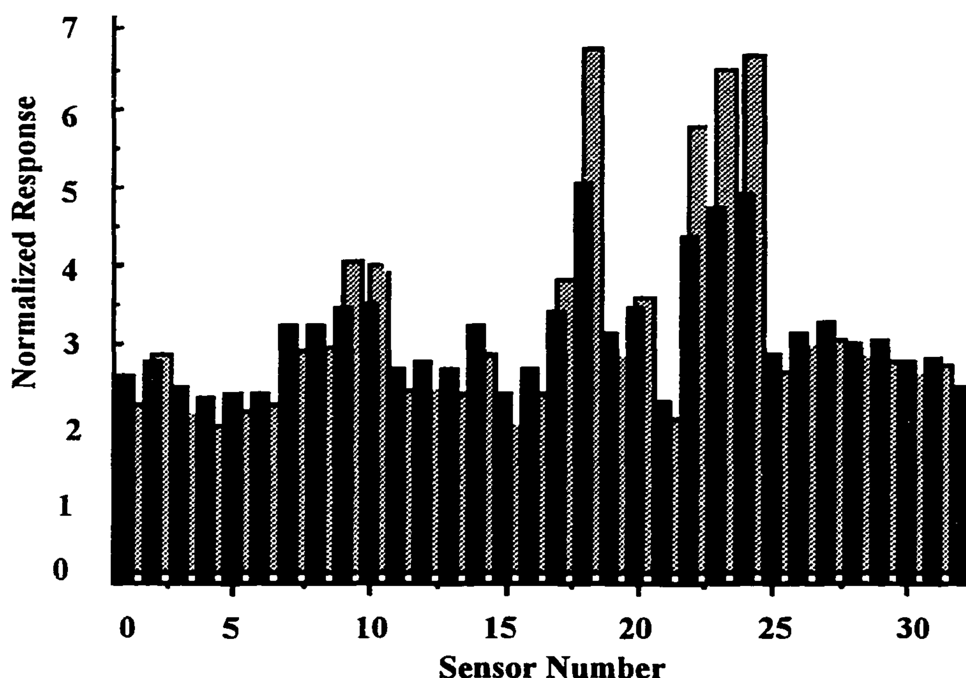

Figure 4-2. The 'fingerprints' for samples 'A' and 'B' in Figure 4-1.
(AromaScan website 1999)

This suggests some potential for utilizing an electronic nose to measure odour concentration. As discussed above, the standard method for the measurement of odour concentration is that an odour sample is evaluated on an olfactometer by an odour panel. If the relationship between odour concentration measured with an olfactometer and the response of a sensor array in an electronic nose to the same odour sample is established, the goal of using an electronic nose to measure odour concentration could be accomplished. Some researchers have attempted this. Persaud et al. (1996) found that an Odourmapper (the previous name for AromaScan) responded well to volatile compounds in the headspace above swine slurries and gave reproducible results over a three-month period. The ability of the Odourmapper to discriminate between two different pig slurries has been demonstrated and a correlation found between sensor response and odour concentration as measured by olfactometry at very high concentrations ($1.5*10^6$

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OU_E/m^3). Misselbrook et al. (1997) demonstrated the ability of an AromaScan to discriminate between cattle slurries at odour concentrations of 10-1200 OU_E/m^3- a level that is similar to those from a variety of agricultural sources. They also found a significant linear relationship between odour concentration and average sensor response with about 60% variance. The above research demonstrated that using an electronic nose to measure odour concentration was a promising approach and that further investigation was warranted.

The objective of the current work was to extend the preliminary work of previous researchers and to use an artificial neural network (ANN) in conjunction with an electronic nose to obtain reliable measurements of odour concentrations. The use of ANN was considered because it is the most powerful tool for nonlinear questions, which is what the sensor's responses of an electronic nose are.

An artificial neural network used in conjunction with an AromaScan can map the 32 parameters of data (generated for each sample by the 32 sensors) to a point in a two- or three-dimensional space (2D or 3D). However, the "fingerprints" and 2D/3D odour-maps represent the normalized response values of the sensor array in an electronic nose, which are independent of a sample's odour concentration. Thus, an external artificial neural network is required to combine with the electronic nose to measure odour concentration.

The responses of different sensors in the sensor array of an electronic nose to the same compound are different. This selective interaction produces a nonlinear pattern of resistance change. The sensor array in an AromaScan is first subjected to carbon filtered room air (reference air), that has the same temperature and humidity as the odour to be

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sampled. The humidity of both the odour and reference air will influence the response of the sensor array, which is polarized. However, the requirement of equal humidity in the odour and reference air is not always satisfied, because the sensor array could be compromised by excessively low or high humidity. Therefore, solving the original problem requires a regression of 34 variables, which are the humidity of odour and reference air and the responses of the 32 sensors.

4.1 Data Collection

As discussed in the Chapter 3 and illustrated in Figure 3-1, Figure 3-2, and Table 3-1, air samples were collected, prior to measurements being carried out from two locations at each of four farms in the Edmonton, Alberta, area. The samples were taken from the ventilation exhaust streams of three swine houses and one slurry processing building. Thus, eight sets of samples were collected.

Forty-four people were hired to evaluate these eight samples on the UA olfactometer described in Chapter 2. The requirements for being a panelist were good health, being a non-smoker, and having a 'normal nose' judged by the panelists themselves. n-Butanol with a concentration of 40 ppmv was used as a reference to evaluate the olfactometry system as recommended by ASTM (ASTM E679-91) and the draft CEN standard (draft prEN, CEN/TC264/WG2/N222/e, 1998). Measurements were carried out over a period of three weeks from February 23 to March 12, 1999, and were conducted on the Tuesday and Friday of each week. The forty-four persons were randomly assigned to six panels on each of the experimental days so that six evaluation sessions could be carried out on each day. Each of the odour samples was evaluated with the UA olfactometer, shown in Figure 3-3, and then with an electronic nose (AromaScan), shown in Figure 4-3.

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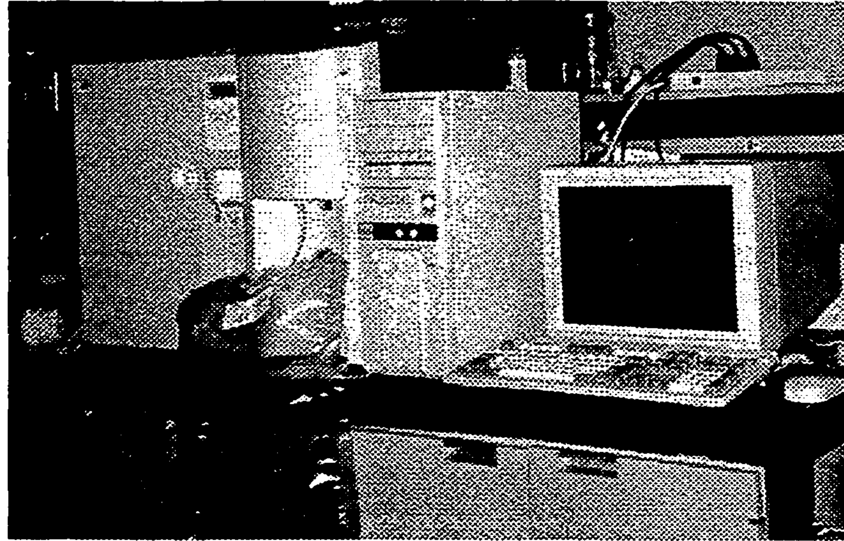

Figure 4-3 A sample being evaluated on the AromaScan

The variations of odour concentration measured on the UA olfactometer, both between the different panels in the six sessions on the same day, and by the same individual panelists for the same sample but on different days, were very large. Table 4-1 shows the variation in odour concentrations which resulted from measuring the same sample with different panels on the same day, and the variation of individuals which resulted from measuring the same sample (40 ppmv n-butanol) by the same person but on different days. The worst ratio between the highest and lowest odour measurements for the panel variation was 29, and the mean of ratios was 6. The worst ratio between the highest and lowest concentrations for the individual variation in this experiment was 512, and the mean of ratios was 76. This result agrees with the report of Harden et al. (1984).

Table 4-1. Variations of odour concentrations as determined by panels and individuals

	Ratio (Max. /Min.)	Mean of Ratio
Panels	28.7	6.1
Individuals	512	75.7

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The large variation indicates that the odour concentrations measured by different panels could not serve as the desired output for training an ANN. Thus, the geometric mean of the odour concentrations measured by all 44 panelists as one big "super-panel", rather than panels comprised of 7 to 8 panelists, was used as the desired output for training an ANN. This method indicates that, though we never know the true value (the expected response of the general population) of the concentration of an odour sample, the measurements resulting from a very large panel consisting of 44 persons are statistically the best approach to the expected response of the general population.

As discussed in Chapter 3, the selection of panelists as a well-defined subset of the population indicates that the assumption that the panel should be representative of the general population was explicitly abandoned. Thus, the measurements made by the qualified panelists are not necessarily those expected of the general population. It would be nice if all panelists satisfied the requirements of the draft CEN standard. However, the proportion of qualified persons is only about 30%, and seeking a large number of qualified panelists is practically difficult. No research results have been reported on the general population response to n-butanol. A detection threshold of 40ppbv for n-butanol is achievable in most laboratories in Europe (Harreveld and Heeres, 1997) and should be close, if not equal, to the population expectation. The approach used here did not disagree with the hypothesis that the detection threshold of n-butanol is 40ppbv. Thus, using a large panel consisting of 44 panelists provides a practical approach to achieving similar measurements to the qualified panelists who form a well-defined subset of the general population.

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The outputs of the electronic nose for each of the samples are the responses of a thirty-two-sensor array. Together with sample and reference air humidity, these thirty-four parameters were used, as shown in Figure 4-4, as the input-variables to an artificial neural network. By setting different reference air humidity on the AromaScan and by changing the sample humidity (injecting water to sample bags), a 35-dimensional data set (an odour concentration together with 34 input variables) containing 480 cases was developed.

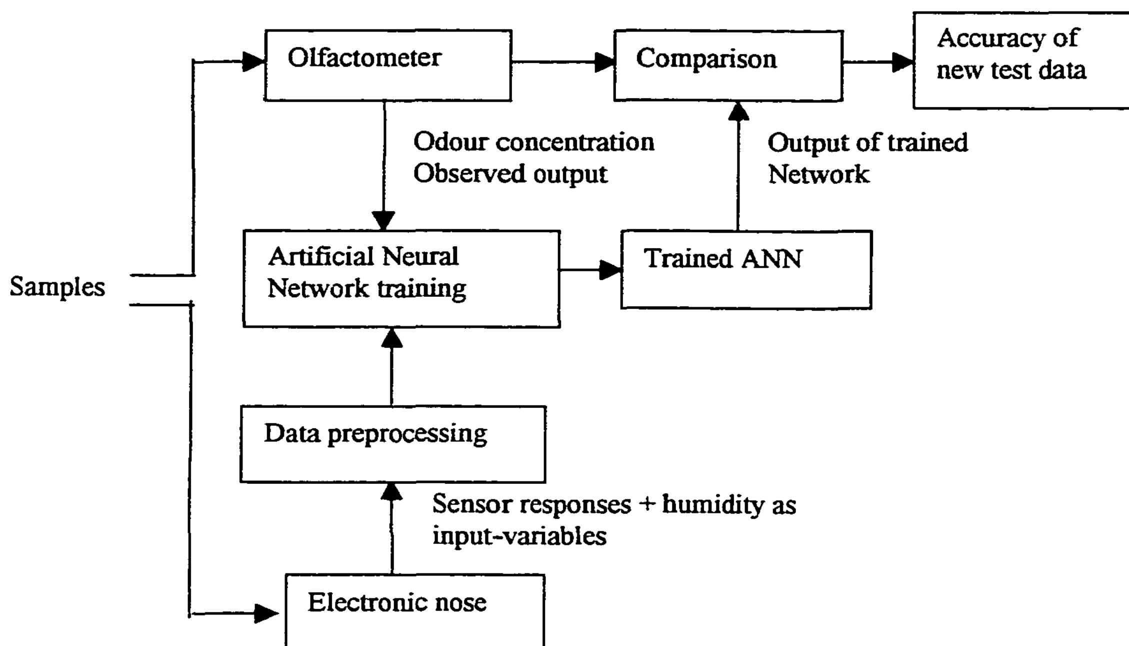

Figure 4-4. Combining an ANN with an electronic nose to measure odour concentration

4.2 Data Processing

In order to find the best expression to relate the input and output variables, the 480-case data set was converted into four forms: original, logarithm of original (both input and output variables), input-variable ratio (the changes of electrical resistance of sensors compared to their base electrical resistance), and logarithmic ratio.

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Not all of the 34 input variables were significantly influenced by the odour concentration, and these insensitive variables didn't contribute to training the ANN. Principal component analysis was used to overcome the problem of insensitive variables. As shown in Table 4-2, the first three principal components could explain 99% of the variance. Thus, the original data set was simplified to a four-dimensional one (odour concentration and the three principal components) without losing any information. Parts of eigenvectors (X_1 to X_7) for the first three principal components are shown in Table 4-3. The remaining parts (X_8 to X_{34}) have the same tendency as X_3 to X_7.

Table 4-2 Eigenvalues of the First Three Principal Components

PC No.	Eigenvalue	Difference	Proportion (%)	Accumulative (%)
1	0.567791	0.507634	87.2	87.2
2	0.060157	0.044163	9.2	96.5
3	0.015994	0.010761	2.5	98.9

Table 4-3. Parts of Eigenvector (X_1-X_7) of the First Three Principal Components

PC No.	X_1	X_2	X_3	X_4	X_5	X_6	X_7	...
1	.01469	.05062	.18682	.19385	.17398	.17407	.18069	...
2	.99721	-.00084	.00107	-.00636	-.01207	-.01112	-.00076	...
3	.00574	.97973	.03939	-.02574	.04020	.01223	.00138	...

The first principal component, explaining 87% of the variance, is the general response of the sensor array (denoted as x3-x34 in the input variables). This indicates that it is possible to develop an inexpensive electronic nose with fewer sensors for measuring odour concentrations from a specific source, such as agricultural odours. However, odours from different sources, or from the same source but generated at different seasons (when the temperature is different), could activate different sensors and, thus, have

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different "fingerprints". Thus further research is required to explore this. The second principal component, explaining 9% of variance, comes mainly from x1 which is the reference air humidity, and the third principal component, explaining 2.5% of variance, represents the influence of x2, the sample humidity.

The reduction of the variable dimension from 34 to 3 appears to be an essential procedure for the successful training of a neural network. This process can not only remove dependent-variable-noise, but it can also increase accuracy of the training result. Since the larger the ratio of the number of experimental data to the dimension of variables, the higher is likely to be the accuracy. The main reason that previous researchers (Persaud et al., 1996; Misselbrook et al., 1997) could only measure samples with very high odour concentration or with high variance could be either that the 34 inputs were not significantly independent resulting in too much noise, or that a lot of information was lost by using the average response of sensors. Also, successfully training a neural network needs an impracticably large data set if high input space is used.

4.3 Adaptive Logic Networks and training result

Variety combinations of ANN and an electronic nose could be used in this application. In this case, for reasons of convenience and accessibility, the Adaptive Logic Network (ALN) (Armstrong et al., 1998) was combined with an AromaScan to estimate odour concentration. The ALN used in this study is a kind of machine learning software of ANNs with feedforward, multi-layer perception which uses linear functions in the first hidden layer, and Maximum and Minimum operators in other hidden layers, and in the output layer. ALNs have been successfully used in rehabilitation of persons with spinal cord injury (Armstrong et al., 1995), real-time control of mechanical systems (Armstrong

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et al., 1994; 1999; Gorodnichy et al., 1999), athletic sport (Armstrong, 1998), and facial feature detection (Gorodnichy et al., 1997). An appropriate ALN can approximate, to arbitrary precision, any continuous real-valued function:

$$Y = f(X)$$

on a compact set of D-dimensional space. Given a set of noisy data samples (X, Y) from the function, an ALN can be trained to approximate the function, whereby the tree adds maximum and minimum nodes and the linear functions change the values of their coefficients (weights). The samples are of the form:

$$X_n \in R^D, \quad Y_n \in R, \quad n = 1, 2, \ldots N.$$

In neural networks, these points are usually referred to as cases or patterns. The main difference and advantage of the ALN over other approximation techniques is that it utilizes piecewise linear surfaces with fillets for smoothness. As explained by Armstrong et al. (1994), linear functions:

$$Li: \; Xn = W_{i,0} + W_{i,1}X_1 + \ldots . + W_{i,n-1}\, X_{n-1}$$

are combined using the Minimum and Maximum operations to form a piecewise linear function:

$$X_n = f(X_1, \; \ldots X_{n-1})$$

Quadratic fillets that never deviate from the piecewise linear part by more than a predefined amount smooth the result to be continuously differentiable while, at the same time, not requiring additional parameters. Each linear piece L_i is learned by weighted least-squares fitting on the data points in a certain portion of the input space. In training, the ALN does least-squares fitting using many linear pieces at once: as linear pieces shift

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during training, each of them gains or loses points of the training data set according to which piece is active for a given training point.

ALN geometry can be of two types: fixed geometry and growable. A fixed geometry tree is a tree with a given number of layers. The fixed geometry type should only be used on data whose structure is known and fits this model. If the structure of the data is not known, a growable tree should be used. In this study, because of the unknown geometry type, a growable tree was used. Fillets were made so small they could be neglected. A growable tree starts with one linear piece and grows by splitting a piece into two if it exceeds a given error found after adaptation of weights has had time to fit the data as best it can. The depth of a trained ALN is dependent on both the complexities of the training data and the error tolerances. The larger the error tolerances are set, the shallower is the depth of an ALN. If the error tolerance is set large enough, an ALN becomes a linear regression. An approximation is shown in Figure 4-5.

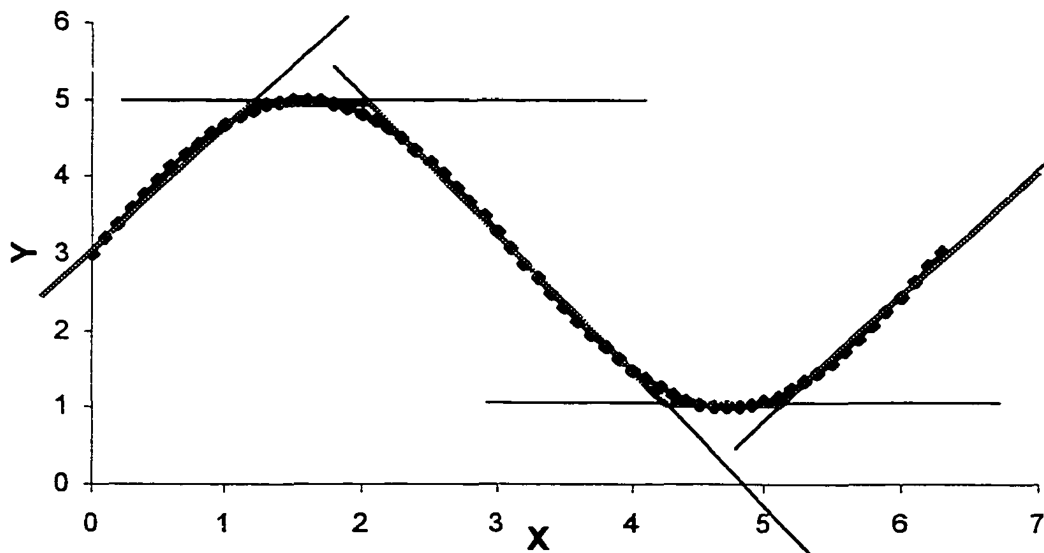

Figure 4-5. Using Linear Pieces to fit a Sinusoid (dark lines)

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After training, a decision tree is created that partitions the input space into blocks, in each of which the function is represented by a simple expression containing a few linear functions connected by Max and Min operations. Each of these expressions is much smaller than the expression of the whole ALN, thus the arithmetic is much simpler and faster.

Similarly to other feed-forward ANNs, a well-trained ALN will have low Root Mean Square Error (RMSE) on a test set not used in training, and thus can be expected to receive good generalization. Finally, a well-trained ALN will have few linear pieces. A large tree is likely to be over-trained. Jitter is used in ALN to overcome over-training. Jitter takes every point in the training set and extends its influence out a certain distance in the input axes, thus creating an augmented training set. Each input variable is changed up to some specified tolerance, and the output value is not changed, thus creating a new-supply of data points whose least squares fit is likely to generalize better than without jitter.

The program used in this study was ALNBench 1.0, a free demonstration software package developed by Dendronic Decisions Limited of Edmonton, Canada.

The 35-dimensional data set containing 480 cases was randomly divided into three subsets: the training subset containing 360 cases, the validation subset containing 60 cases, and finally a test subset containing the remaining 60 cases. The test subset was never used in training and validation, and was only used for final estimation of the generalization error. The parameters Root of Mean Square Error (RMSE) and Mean Absolute Percentage Error (MAPE) were used to quantify the ALN performance. The MAPE was calculated as:

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$$MAPE = \left|\frac{\text{observed output - ALN output}}{\text{observed output}}\right| * 100\%$$

Among the four expression forms, the logarithm original performed best. The results for the training, validation, and test data sets of the logarithm original data are shown in Table 4-4.

Tale 4-4 Resulting Errors

Name	LFs*	Training		Test		Validation
		MAPE	RMSE	MAPE	RMSE	MAPE
Trial 1-1	487	0.1644	0.2163	0.2114	0.2764	0.1622
Trial 1-1	654	0.1402	0.1839	0.1953	0.2023	0.2110
Trial 2-1	485	0.1378	0.2124	0.1853	0.2482	0.1690
Trial 2-2	459	0.1395	0.2095	0.1619	0.2142	0.1853
Trial 3-1	509	0.1580	0.1871	0.1650	0.2164	0.1771
Trial 3-2	574	0.1659	0.1985	0.1749	0.2277	0.1626
Average	528	0.1510	0.2013	0.1823	0.2309	0.1779

LFs: Number of effective linear functions in a well trained ALN

On average from six trials, a well-trained ALN is composed of 528 effective linear pieces, and the MAPE for the validation and test data set are 18.2 % and 17.8%, respectively. Thus, the well-trained ALN can correctly predict odour concentration with less than 20% Mean Absolute Percentage Error.

Due to its portability, high accuracy, and operation independent of a human panel, an electronic nose can make on-site field odour concentration measurements possible.

4.4 Conclusions

1). The variations of odour concentration determinations by panels and individuals are very large. The best practical approach to obtaining the true value of the general population expectation and the one measured by qualified panelists of odour concentration is to use a large panel (44 persons in this study) to evaluate odour samples;

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2). The 34-dimensional input space obtained from the electronic nose data can be mapped to a three-dimensional input space by using principal component analysis almost without losing any information, and this appears to be an essential procedure to the ANN's success. However, the loss of non-linearity and other methods for dimension reduction were not investigated.

3). The first principal component, explaining 87% of variance, is the average response of the 32-sensor array of the AromaScan. This indicates that it may be possible to develop an inexpensive electronic nose with fewer sensors for measuring odour concentrations from a specific source, such as agricultural odours;

4). A well-trained ALN combined with an electronic nose, can measure odour concentration with less than 20% Mean Absolute Percentage Error, and allows for immediate field measurements;

5). The testing was done in samples not used in training the ALN, thus a good generalization for the new samples can be expected.

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Chapter 5 Synthesis

Odour emitted from intensive livestock, notably swine operations, has become an environmental constraint to expanding the pig industry. A reliable and accurate measurement with acceptable repeatability within a laboratory and reproducibility among laboratories to provide valid odour characterization data that remain stable with time is imperative for the implementation of odour policy, abatement programs, and regulations.

The understanding of the odour origins and the health symptoms caused by odours will help to choose parameters for describing odour and methods for odour measurement. The offensiveness of animal malodour is determined by multiple sources, such as a confined animal building, an earthen manure storage, and a manure land application. The main reason for the malodour generation is that the manure slurry storage and degradation is an unbalanced anaerobic digestion, on which only the acid formation dominates and large amount of odourous volatile compounds accumulate.

The human olfactory system operates on the basis of a model of "second-messenger signaling". It is very sensitive to odour concentration, but very poor in discriminating odour components (at most four) in a mixture. Exposure to unpleasant odour may cause health symptoms, and complaints include irritation of the eye, nose, and throat, to nausea, headache, and vomiting, disturbance, annoyance, and depression. The sensitivity of human olfactory system changes after being exposed to odours. A short-term exposure causes fatigue of olfactory response which will significantly affect the behavior of an odour panel. A long-term exposure might cause adaptation or sensitization; either of them will permanently modify a person's olfactory world.

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Parameters used to describe odours are odour intensity, odour character, odour hedonic tone, odour persistence, and odour concentration, among which the most important and subjective parameters for describing odour is the odour concentration. Odour concentration is defined as the mass concentration of pure odourous substances or the dilution factor of mixtures of odourants at the detection threshold, which means that at this concentration a sample has a 50% probability of being detected. Odour concentration is measured with an olfactometry method consisting of a panel and an olfactometer, which uses the human nose as the sensor of odours. Thus, the accuracy, repeatability, and reproducibility of odour concentration measurement are dependent on both an olfactometer and a human panel.

The first research project under taken in this research was the design and construction of an eight-panelist-station, single-sniffing port, triangular forced-choice ascending concentration series olfactometer (UA olfactometer). Compared with the most recently designed conventional olfactometers, the UA olfactometer has advantages of less odour contaminant potential, economy of sampling, time saving, low manufacturing cost, and less psychological bias. The UA olfactometer can analyze ten samples per hour, and it can also be used to measure the hedonic tone of odour. Calibration results show that the neutral air and an odour are mixed well in the UA olfactometer, and the flow rate to each of ports meets the accuracy of the draft CEN standard.

Huge variance exists in the responses of the human olfactory system to odours. Even among qualified persons selected by strict screening criteria, the ratio between the most sensitive and insensitive could be four. Based on an assumption that the traceability among panelist/panels exists, a model correlating the olfactory responses among

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panelist/panels to environmental odours and to a reference n-butanol was developed. The model is significant at the level of α=0.0001, and can be used to normalize the panel's olfactory response to decrease the measurement variance, thus solving the problem that the measurement of odour concentration has long been relatively arbitrary.

The olfactometry method is labour intensive, time consuming, and cannot be used on-site. A new method for measuring odour concentration was developed, which is to create a function to convert responses of an electronic nose, AromaScan, to odour concentrations by combining an artificial neural network, the Adaptive Logic Network (ALN), with the electronic nose. A data set containing 480 cases was developed by evaluating odour samples on the UA olfactometer by forty-four hired persons and on the electronic nose. The data set was used, after preprocessing, to train, test, and validate an ALN. In the data preprocessing, the 34-dimensional input space obtained from sensor responses of the electronic nose, the humidity of sample and the reference can be mapped to a three-dimensional input space by using principal component analysis almost without losing any information. The first principal component, explaining 87% of variance, is the average response of the 32-sensor array of the AromaScan. This indicates that it may be possible to develop an inexpensive electronic nose with fewer sensors for measuring odour concentrations from a specific source, such as agricultural odours. A well-trained ALN combined with an electronic nose, can measure odour concentration with less than 20% Mean Absolute Percentage Error, and allows for immediate field measurements. A reduction in dimensionality, i.e. principal component analysis, appears to be an essential procedure for the success of training the Adaptive Logic Network.

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