CHEMICAL ENGINEERING TRANSACTIONS
VOL. 30, 2012
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at: www.aidic.it/cet
Guest Editor: Renato Del Rosso

ISBN 978-88-95608-21-1; **ISSN** 1974-9791
DOI: 10.3303/CET1230020

Comparison of Odour Concentrations Obtained by Yes/No and 2AFC Mode

André van Boheemen

Witteveen+Bos Raadgevende ingenieurs B.V., Postbus 233, 7400AE, Deventer, The Netherlands
A.vBoheemen@WitteveenBos.nl

The international standard EN 13725 allows two different modes to be used for determination of odour-concentrations. Both modes are assumed to yield equal results. To verify whether this assumption is correct, a comparison have been carried out. In the experimental design, the nAFC mode was fully implemented using a 2AFC procedure. To prevent from adverse effects on the panellists' performance, the yes/no mode was performed without presentation of blanks. No main effect of method was established ($p > 0.10$). Furthermore, there is a tendency that odour concentrations obtained with the yes/no method are slightly higher than those obtained with a 2AFC-method.

1. Introduction

In the 1970's odours emanating from industrial, agricultural and other sources became a serious problem in the Netherlands. Since it was realized in the 1980's that good correlations between the olfactory properties of an odour emission and the analytical concentration of the various compounds were hard to find, sensory techniques, such as dynamic olfactometry, became the method of choice for testing odours for environmental management purposes. With dynamic olfactometry, the dilution factor at the 50 % detection threshold is determined. At that dilution factor the odour concentration is 1 ou_E/m^3 by definition. At present, odour concentrations in the Netherlands are exclusively estimated using procedures conform EN 13725 (2003).

According to EN 13725 methods for establishing odour concentrations require qualified panellists, a dilution apparatus and a procedure on how to present the odorants to the panellists. As far as presentation of odorants is concerned, two different modes are permitted: the yes/no mode and the n-alternative forced-choice mode (nAFC). Although the psychophysical models involved are different, both modes are supposed to yield an equal result. Experiments carried out by Ueno et al. (2009) show that the results of both modes are within the limits of uncertainty inherent to dynamic olfactometry. Since the experiments comprised singular odorants, n-butanol, ethyl acetate and hexanal, it is uncertain whether this finding is transferable to the complex mixture of day-to-day odour emissions. In this paper odour concentrations of singular and multiple mixtures, established by a 2-alternative forced-choice method (2AFC) and a yes/no method, are compared. It was supposed that both methods of establishing odour concentrations would not differ significantly.

Please cite this article as: van Boheemen A., 2012, Comparison of odour concentrations obtained by yes/no and 2afc mode, Chemical Engineering Transactions, 30, 115-120 DOI: 10.3303/CET1230020

2. Materials and methods

The study comprises of a number of experiments in which the odour concentration of various samples, singular and multiple component mixtures, was established. Experiments were conducted in an air conditioned room (21 ± 2) °C by employees of the odour laboratory of Witteveen+Bos. The quality system of the laboratory is accredited by Dutch Accreditation Council (RvA). One of the activities recorded in the scope concerns the analysis of odour concentration according to EN 13725 using a 2AFC method. In connection with the accreditation, all procedures of the quality system are reviewed by qualified external auditors on annual basis.
Dilutions were prepared using a dynamic olfactometer fitted with two sniffing ports (Figure 1). Air flow at the sniffing ports was 20 L/min. Speed of the air flow leaving each sniffing ports was 2 m/s. The dilution system of the olfactometer comprised 15 settings which were spaced using step size 2.0. The lowest setting was 6 dilutions. Dilutions settings were regularly calibrated using a FID and butane as tracer. Before and after each session, air flows making up dilutions were checked using a calibrated flow meter. In case a targeted flow was out of bound, the olfactometer was scheduled for further inspection and subsequently repaired if necessary. Control and calibration procedures used were part of the accredited quality system. All dilutions were traceable to (inter)national standards.

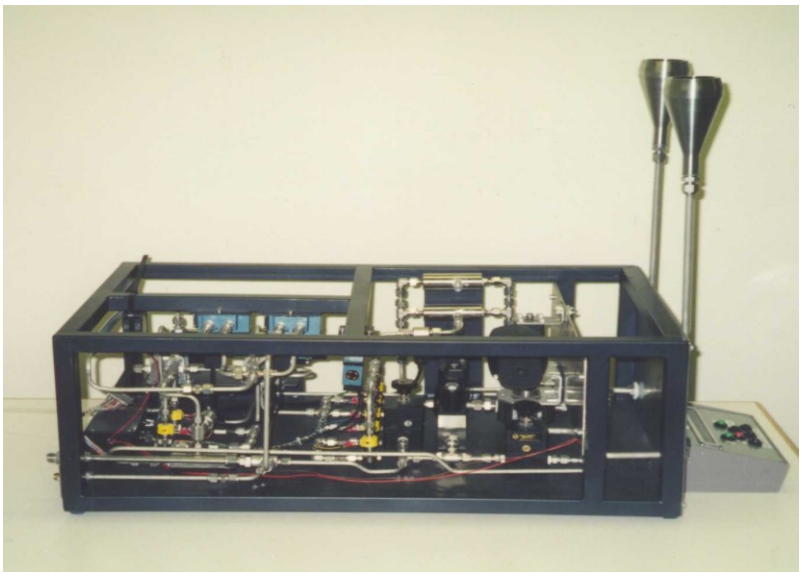

Figure 1: Olfactometer.

Two modes of presentation were used: the yes/no method and a two-alternative forced-choice method (2AFC). The latter is used routinely as part of the company undertaking. In either mode, presentation of dilutions was in descending order (ascending concentration). Presentation of dilutions to panellists was started by selecting an appropriate dilution based on prior assessment of the odour sample by the olfactometer operator. After stabilization of the diluted gas flow, panellists were invited, one after the other, to make a judgement (Figure 2). Presentation of dilutions was halted when all panellists attained an pre-set response criterion. In all experiments, two series of dilutions were presented in order to complete one experimental session. Minimum odour concentration to be established is 20 ou_E/m^3.

2.1 Yes/No mode

Panellists were asked to evaluate a diluted gas flow presented from a specific sniffing port and to indicate if an odour is perceived (yes/no). Presentation of dilutions was halted when all panellists obtained at least two consecutive true responses (yes) for the presentations with the highest odorant(s) concentration following a false response (no). Throughout all presentations, the odd port was made available to panellists holding neutral gas as reference. Inclusion in every dilution series of one or more

blanks at random position, as prescribed by EN 13725, was omitted since addition of blanks in dilution series is not practised at Witteveen+Bos. It was expected that this would adversely affects the panellists' performance and may bias the outcome of the experiments.

2.2 AFC mode

Panellists were presented with two ports, of which one holds the stimulus and the other holds neutral gas. Stimuli in consecutive presentations were randomly distributed over both ports. Panellists were asked to indicate which of the ports was holding the stimulus. When in doubt, they were asked to indicate a port 'at random'. To reduce variability and achieve convergence with the yes/no mode, panellists were asked whether their choice was a guess, inkling or certain. Presentation of dilutions was halted when all panellists obtained at least two consecutive true responses (correct and certain) for the presentations with the highest odorant(s) concentration following a false response (incorrect in combination with guess or inkling). The 2AFC method performed was in accordance with EN 13725.

2.3 Panellists

A total of 15 panellists (8 female, 7 male, age range: 18-35 y, mean age: around 22 y) participated in the experiments, all of which were qualified in accordance with EN 13725 and were in good health. Panellists were recruited using viva voce techniques and received financial remuneration for their participation. Panellists were asked to participate in all experiments that were foreseen on a particular day. Most of them participated more than one day.

Prior to experimentation, procedures were explained in detail to all panellists. Among others, they were instructed to refrain from communicating with each other about the results of their judgements as well as to avoid interference with their own perception and/or that of others. All panellists provided oral consent.

Experiments were carried out on eight different days. Each day 4 to 6 panellists participated in the experiments. Typically six panellists were selected from the laboratory's database on qualified panellists. However, as a result of unforeseen obligations elsewhere and/or retrospective screening, non-starters and drop-outs occurred. In compliance with EN 13725, the minimum panel size after retrospective screening was 4 in all experiments.

Figure 2: Presentation of a dilution to a panellist.

Each day of experimentation, panel response was tested using n-butanol as reference odour. Butanol samples were withdrawn from a cylinder containing a mixture of n-butanol in nitrogen. The concentration, (60 ± 1.2) µmol/mol, was certified by NPL (UK). The concentration was traceable to (inter)national standards. Panel responses to n-butanol observed varied between 32.1 and 56.4 µmol/mol. Panellists sensitivity and variability to n-butanol was tested individually using procedures as

pointed out in EN 13725. Sensitivity of participating panellists, expressed as individual threshold estimates, varied between 20.9 and 97.5 µmol/mol. The variability was between 1.21 and 2.12.

2.4 Samples

Samples of which the odour concentration was established, were obtained within the framework of the company undertaking. Most of them were part of odour emission surveys whilst others were part of schemes on quality control and proficiency testing. A description of all samples involved is included in Table 1.

Samples were collected and stored in 50 L. ®Nalophan bags (Figure 3). Bags were pre-conditioned with odour sample by filling approximately 25 % of the volume, emptying the bag and re-introducing the odour sample until approximately 90 % of the volume was filled out. Storage time was always less than 30 h. With the exception of n-butanol, general storage time was 20 - 28 h. n-Butanol samples were stored for approximately 1 hour. Sampling bags were used once. Prior to use, bags were tested for leakage. All batches of bag materials were tested on the presence of residual odours prior to use.

Figure 3: Sample bag.

3. Results

Basic experimental results are included in Table 1 and shown by Figure 4.

Since odour thresholds are not normally distributed all data points were subjected to logarithmic transformation (^{10}log). Effect of method was tested using a paired t-test ($\alpha = 0.05$, two sided). No main effect of method was established ($p > 0.10$). The mean of differences between pairs and standard error of mean were calculated to be -0.054 and 0.039 respectively. The coefficient of correlation between both methods was 0.85. A scatter plot with trend line displaying the values recorded for both methods is shown by Figure 5.

4. Discussion

Referring to the claim that analyses of odour concentration in should yield equal results, the following applies. In the present study no main effect between method was established ($p > 0.10$). On a logarithmic scale, the coefficient of correlation between both modes was 0.85. This suggests a certain correlation. Furthermore, the mean of differences on a logarithmic scale between pairs was -0.054. This suggests that odour concentrations measured in 2AFC mode tends to be lower than in yes/no

mode. The trend that odour concentrations measured in yes/no mode are somewhat higher than those measured in 2AFC mode coincides with data published by Ueno et al. (2009).

Table 1: Basic Exerimental Results

session	sample	odorant	2AFC [ou_E/m^3]	yes/no [ou_E/m^3]	2AFC [$\log(ou_E/m^3)$]	yes/no [$\log(ou_E/m^3)$]	difference factor
1	1	n-butanol	1,810	1,350	3.26	3.13	0.13
2	2	n-butanol	1,430	1,790	3.16	3.25	-0.10
	3	rendering plant	488	1,510	2.69	3.18	-0.49
	4	rendering plant	459	510	2.66	2.71	-0.05
	5	rendering plant	443	1,140	2.65	3.06	-0.41
	6	rendering plant	443	345	2.65	2.54	0.11
	7	rendering plant	382	394	2.58	2.60	-0.01
	8	rendering plant	569	452	2.76	2.66	0.10
3	9	saw dust incineration	157	218	2.20	2.34	-0.14
	10	saw dust incineration	452	268	2.66	2.43	0.23
	11	saw dust incineration	82	95	1.91	1.98	-0.06
	12	saw dust incineration	245	131	2.39	2.12	0.27
	13	saw dust incineration	192	290	2.28	2.46	-0.18
4	14	n-butanol	1,940	1,800	3.29	3.26	0.03
	15	sludge processing	2,890	2,260	3.46	3.35	0.11
	16	sludge processing	3,100	2,020	3.49	3.31	0.19
	17	sludge processing	3,080	2,690	3.49	3.43	0.06
5	18	n-butanol	1,880	2,510	3.27	3.40	-0.13
6	19	n-butanol	1,790	2,510	3.25	3.40	-0.15
7	20	n-butanol	1,130	514	3.05	2.71	0.34
	21	incinerator at oil refinery	66	150	1.82	2.18	-0.36
	22	incinerator at oil refinery	49	56	1.69	1.75	-0.06
	23	incinerator at oil refinery	70	100	1.85	2.00	-0.15
8	24	n-butanol	1,830	2,170	3.26	3.34	-0.07
9	25	n-butanol	,966	2,720	2.98	3.43	-0.45
	26	composting plant	1,300	1,360	3.11	3.13	-0.02
	27	composting plant	922	976	2.96	2.99	-0.02
	28	composting plant	1,680	2,720	3.23	3.43	-0.21
		average			2.79	2.84	-0.05

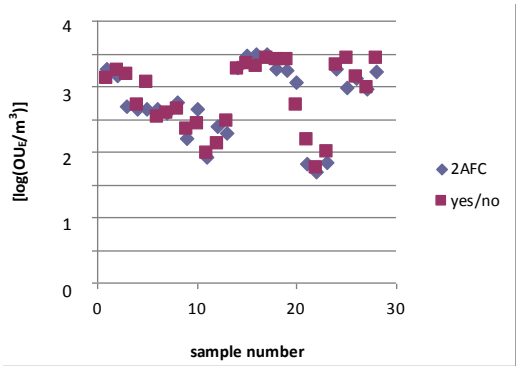

Figure 4: Graph of Odour Concentrations

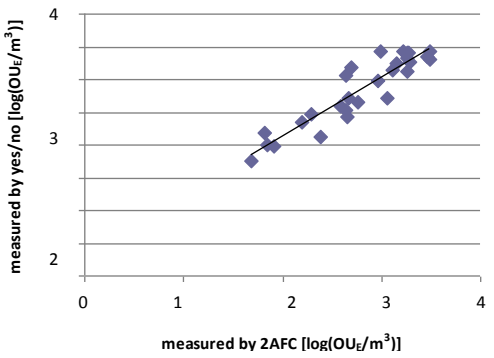

Figure 5: Scatter Plot of Odour Concentrations

It is estimated from their work that the mean of pairs was -0.033 on a logarithmic scale (n=3). However, the spread in the present study between the standard deviation of pairs (0.28) and the standard error of pairs (0.032) cannot be ignored. This points in the direction of insufficient power to surpass the effect of nominal high uncertainty in measurement which is associated with analyses of odour concentration.
In this study, the 95 %-confidence interval on a linear scale of yes/no and 2AFC mode was 1.62 and 1.60 respectively. It is generally accepted that a 95 %-CI coincides with the expanded uncertainty of measurement (k=2). This implies that the ratio between two single measurements, performed in this laboratory under reproducibility conditions (different samples, operators and panels), will not be larger than 1.62 in 95 % of cases. Both values compare favourably with data published by Boeker et al (2008). In their study, the expanded uncertainty of measurement was calculated to be 4. Furthermore, both values are well within the repeatability limit set by EN 13725. In this standard, the limit of repeatability (one laboratory, sample, operator and panel) is ≤ 3 on a linear scale.

5. Conclusions

In the present study no main effect between analyses of odour concentration in yes/no mode and 2AFC mode was established. Results in the yes/no mode tend to be somewhat higher than in 2AFC mode. This coincides with data published by Ueno et al. (2009). These findings indicate that odour concentration analyses in yes/no mode and 2AFC yield equal results.
In the present study the 95 %-CI of analysis of odour concentration in yes/no mode and in 2AFC mode was well within the limits set by EN 13725. These findings indicate that as far as uncertainty of measurement is concerned both modes of odour presentation will yield equal results.
However, the spread between the standard deviation of pairs (0.28) and the standard error of pairs (0.032) cannot be ignored. This points in the direction of the sample size that may have been insufficient to surpass the effect of uncertainty in measurement. In order to produce decisive answers on the topic that odour concentration analyses in yes/no mode and in 2AFC mode yield equal results, a lager sample size then is used in the present study may be needed.

Acknowledgements

The author wishes to thank operators Bas Bessling, Jeroen W. Melcherts and Yanaika M. Quist for their assistance in performing the experiments, and Sjoerd Veenstra for adapting the olfactometer.

References

Boeker P., Haas T., Diekmann B., Schulze Lammers P. 2008. A Monte-Carlo simulation of the measurement uncertainty of olfactometry. Chem Eng Trans, 15, 109-114.

CEN 2003. Air quality - Determination of odour concentration by dynamic olfactometry (EN 13725). Brussels, B: European Committee for Standardization.

Ueno H., Amano S., Merecke B., Kosmider J. 2009. Difference in the odor concentrations measured by the triangle odor bag method and dynamic olfactometry. Water Sci Technol, 59, 1339-42.