# Difference in the odor concentrations measured by the triangle odor bag method and dynamic olfactometry

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**Abstract** 'The triangle odor bag method', which has been adopted for the offensive odor control law in Japan, and the dynamic olfactometry defined by EN 13725 have been compared. The odor concentration measured by the triangle odor bag method tends to be higher than that of the dynamic olfactometry in the forced choice mode, while well agreed in the Yes/No mode olfactometry when the panel is the same. The difference can be minimized by applying the panel selection criterion of EN13725 to the triangle odor bag method. The European panel selection test is useful to negate the difference in the measurement equipments although the criteria seem to be strict considering the individual threshold data of n-butanol.

Keywords Odor concentration, Olfactometry, Triangle odor bag method

#### **INTRODUCTION**

Odor measurement is essential for odor regulation and control. The triangle odor bag method (Iwasaki et.al.,1972) is an olfactory method to measure odor concentration, which has been adopted for the offensive odor control law in Japan. On the other hand, dynamic olfactometry (EN 13725) is widely used in Europe and North America (e.g., Sironi et al.2005; Dincer and Muezzinouglu, 2006). Both methods are to determine odor concentration by sniffing diluted air samples. However the dilution equipment, estimation methods of the threshold and panel selection procedures are different.

In this report, the odor thresholds measured by 'the triangle odor bag method' and two dynamic olfactometers are compared. The panel selection procedure was then considered to make the odor concentrations measured by both methods be corresponding.

#### **METHODS**

### **Triangle Odor Bag Method**

The panel consisted of at least six members who passed the selection test mentioned below. Three-liter plastic bags are used for presentation of the diluted odor samples. A certain amount of an odor sample is injected into one bag of three bags. Assessors sniff and chose the one bag having the odor out of the three bags. The presentation is performed in a descending series of odor. The procedure is repeated at the ratio of a three-times higher dilution ratio until the assessors make a wrong answer. The geometric mean of the dilution factors of the last correct answer and of the first wrong answer is calculated as the individual threshold. The odor concentration is then calculated as the geometric mean of individual thresholds excluding the minimum and maximum individual thresholds.

### **Japanese Panel Selection Test**

In Japan, five standard odors ( $\beta$ -phenylethyl alcohol, methylcyclopentenolone, isovaleric acid,  $\gamma$ -undecalactone, skatole) are used for the panel selection. This standard odor set was developed and manufactured as the "T&T Olfactometer"in 1975 (Takagi,1989). The aim of the test is to find the smell disturbance. The test is performed by the 5-2 method. Two sniffing papers out of five are soaked with the standard odor solution, and then an assessor sniffs and choses the two papers. If the assessors cannot distinguish one of the five standard odors at certain concentrations, they cannot be panel members. This method is easy and takes only fifteen minutes. Normally 95 % of ordinary people pass the test.

# Measurements of Thresholds by Japanese and European Methods

*Triangle odor bag method and dynamic olfactometry (Forced choice mode).* The olfactometer used was the olfaktomat-n2 olfactometer <u>http://www.odournet.com/tools\_analysis.html</u> made by Odournet which uses the forced choice mode. The panel members consisted of twelve Japanese who passed the Japanese panel selection test. Odor concentrations of five odorants were measured by both methods with the same panel. Each odorant was measured three or four times by both methods on the same day to prevent the effect of variability in sensitivity of the individuals.

The odorants used were n-butanol, ethyl acetate, hexanal, hydrogen sulfide and iso-butyric acid. The samples were prepared by injection of the odorants into PET bags filled with fifty liters of odor-free air. The concentrations were confirmed by gas chromatography using standard cylinder gas except for the iso-butyric acid. For the iso-butyric acid, a standard solution was used for confirmation of the concentration by gas chromatography. The odor threshold values were calculated from the odor concentration and the concentration in the sample bags.

'*Triangle odor bag method*' and 'Dynamic olfactometry' (Yes/No mode). This experiment was performed at the Szczecin University of Technology. The olfactometer in the Yes/No mode used was TO-7 made by ECOMA. The panel members were composed of twelve Polish students. Three odorants were used for measurement of the thresholds and sample preparation was done as described above.

### **Consideration of Panel Selection Criteria**

*Distribution of threshold of n-butanol.* The odor thresholds of fifty-one Japanese assessors for n-butanol were measured by dynamic the olfactometer in the forced choice mode. The individual thresholds were measured more than ten times.

The thresholds of twenty-two Japanese assessors for n-butanol were measured by the triangle odor bag method. Measurements were made more than ten times for each assessor as well as the olfactometer measurements.

*Application of EN 13725 criterion to triangle odor bag method.* Panel selection was made by the triangle odor bag method using the EN 13725 criterion. Assessors whose average thresholds for n-butanol measured by the triangle odor bag method were within 20-80 ppb were selected. The odor thresholds of the odorants measured by the triangle odor bag method with the panel were then compared to the thresholds measured by dynamic olfactometry with other panels that comply with the EN 13725 criteria. Six odorants were used and the measurements were made 4-8 times for each odorant.

# **RESULT AND DISCUSSION**

### Measurement of Threshold

The measurement results of the triangle odor bag method and dynamic olfactometer in the forced choice mode are shown in Table 1. Values of the olfactometer in the forced choice mode tend to be higher by 0.5 on the logarithm scale of concentration than that of the triangle odor bag method. This difference corresponds to 3 times the concentration.

In the forced choice mode, presentation of the diluted samples is performed in an ascending series, and the threshold is calculated as the geometric mean of the dilution ratios of the last correct answer with the probability of inkling and of the first correct answer with the probability of certain, while in the triangle method, samples are presented in a descending series, and the threshold is the geometric mean of the dilution ratio of the last correct answer and the first wrong answer. The former tends to be higher concentration because assessors may perceive if they answer inkling. But the latter tends to be lower concentration because assessors never perceive when they answer wrong. And the latter has one-third the probability to be correct if they do not perceive. The difference in the calculation procedures causes disagreement in the odor concentration.

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	Triangle odor bag method		Dynamic olfactometer forced choice mode	
n-butanol	4.2	(17ppb)	4.7	(53ppb)
Ethyl acetate	5.6	(0.36ppm)	6.1	(1.3ppm)
hexanal	2.3	(0.20ppb)	3.0	(0.94ppb)
Hydrogen sulfide	2.5	(0.32ppb)	3.1	(1.3ppb)
Iso-butyric acid	3.6	(3.6ppb)	4.0	(11ppb)

**Table 1.** Thresholds of odorants measured by triangle odor bag method and dynamic olfactometry in the forced choice mode (log ppt)

The result of the triangle odor bag method and dynamic olfactometer in the Yes/No mode is shown in Table 2. The values measured by both methods were almost the same. The Yes/No mode calculates the threshold as the geometric mean of the dilution ratios of no perception (No) and perception (Yes). This calculation tends to be lower in concentration compare to forced choice mode. It can be concluded that if the panel is the same, the triangle odor bag method yields the same level of odor concentration as the Yes/No mode olfactometer.

**Table 2.** Thresholds of odorants by triangle odor bag method and dynamic olfactometry in the Yes/No mode (log ppt)

	Triangle odor bag method	Dynamic olfactometer Yes/No mode	
n-butanol	4.9 (88ppb)	5.1 (140ppb)	
Ethyl acetate	6.2 (1.7ppm)	6.1 (1.2ppm)	
hexanal	2.8 (0.68ppb)	2.7 (0.48ppb)	

### Distribution of threshold of n-butanol

The distribution of the average threshold values of 51 individuals for n-butanol measured by dynamic olfactometer in the forced choice mode is shown in Figure.1 (a). The average is 1.7(55ppb), and standard deviation is 0.32. It ranges from 1.2 to 2.4 (16-240ppb), the concentration ratio of the minimum and the maximum is 15. 31 assessors comply with the EN criterion of a threshold of 20-80ppb. For another criterion of the standard deviation, 31 assessors passed. 20 assessors, which correspond to 40% of total the assessors, passed both criteria.

The distribution of the average threshold values of 22 individuals for n-butanol measured by the triangle odor bag method is shown in Figure 1(b). The average is 1.4 (27ppb), which is lower than that of the olfactometry in the forced choice mode. The standard deviation is 0.41, which is higher than that of the olfactometry. The range is from 0.6 to 2.1 (4-130ppb), and the ratio is 30.

These results indicate that the criterion of the threshold of 20-80ppb is appropriate as the average level of the human sense of smell, but the range is too small, therfore we have to test many people.



**Figure1.** Distribution of average individual thresholds for n-butanol. White bar indicates complying with EN criterion.

### Application of EN criterion to triangle odor bag method

Results of the thresholds measured by the triangle odor bag method and dynamic olfactometry in the forced choice mode are shown in Table 3. In these measurements, the panels were different and they were selected by each method to comply with the EN criterion of n-butanol. The values well agreed for not only n-butanol but the other odorants. If the panel selection criteria are the same, the odor concentration measured by the triangle odor bag method is comparable to the dynamic olfactometry.

	Triangle odor bag method		Dynamic olfactometer forced choice mode	
n-butanol	4.4	(28ppb)	4.6	(37ppb)
Ethyl acetate	5.9	(0.77ppm)	5.7	(0.46ppm)
Hexanal	2.7	(0.42ppb)	2.6	(0.41ppb)
Hydrogen sulfide	2.9	(0.77ppb)	3.0	(0.96ppb)
Iso-butyric acid	3.5	(3.7ppb)	3.6	(3.8ppb)
Butyl amine	4.7	(45ppb)	4.9	(88ppb)

**Table 3.** Thresholds of odorants measured by triangle odor bag method and dynamic olfactometry in forced choice mode with each panel complying to the EN13725 criterion (log ppt)

Figure 1 shows that the standard deviation of the dynamic olfactometry seems to be lower than that of the triangle odor bag method. The triangle method in descending presentation has one third the probability that an assessor make an incidental correct answer. It should causes scatter in the distribution of threshold in Figure 1(b). To improve the repeatability, assessors were made to answer the odor intensity as well as the number of bags they perceived the odor, and the threshold was calculated regarding their answer as wrong when the odor intensity is zero (no odor) even if the answer is correct. It is called a correction by the odor intensity in this report.

Figure 2 shows that the standard deviation of the threshold measured by the triangle odor bag method with panel members whose thresholds for n-butanol are within 20-80ppb. Panel selection with the EN criterion was not enough to decrease the standard deviation of the triangle odor bag method. However, the correction by the odor intensity has a good effect of reducing the standard deviation and the value became the same as the dynamic olfactometry.

Triangle odor bag method





**Figure2.** Effect of correction by odor intensity on standard deviation of logarithm of threshold measured by triangle odor bag method

### CONCLUSION

The triangle odor bag method yield higher odor concentration than dynamic olfactometry in the forced choice mode, but the same level of odor concentration of dynamic olfactometry in the Yes/No mode if the panel is the same. The difference can be cancelled by applying the same panel selection criteria. The repeatability will also be the same by improving the methodology of determining the threshold of the triangle odor bag method. However, the panel selection criterion is strict considering the distribution of the threshold of n-butanol.

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