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A Comparative Study of Japanese and European Olfactometry Standards

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Abstract This report provides a comparative study between the odor measurement method by olfaction, which has been adopted as the Offensive Odor Control Law in Japan and the dynamic olfactometry, which has been standardized in Europe. Dilution accuracy, panel selection and odor measurements have been compared in this study. As a result of the dilution test with three standard odorants, a 46% decrease in the concentration of hydrogen sulfide at a high dilution ratio has been observed using the olfactometer, while the Japanese method has shown good performance. Twenty people have been given both screening tests. Eighteen people have passed the Japanese test, while only seven people have passed the European test. In the odor measurement of three standard odors and six actual source samples, if the panel is the same, the results of both methods have corresponded well.

1. Introduction

The method of measuring odor concentration by sniffing samples diluted with odor-free air is common worldwide. The triangular odor bag method is adopted as an olfactometry standard in the Offensive Odor Control Law in Japan. However, the dynamic olfactometry[1)] has been standardized in Europe and there is a possibility it might become the international standard in the future. Therefore, it is necessary to conduct a proper comparative study of both methods in view of such progress. The following differences exist between the Japanese and the European methods, although the aim of both methods is the same, which is to determine the olfactory threshold by sniffing diluted odor samples.

Dilution Method: Although odor samples are diluted with odor-free air using bags and syringes in the triangular odor bag method, a dynamic olfactometer continuously dilutes samples using a compressor and flow controllers, etc.

Presentation of Samples to Assessors: A diluted series is presented in descending order of stimuli in the triangular odor bag method and the step factor is 3. In the European method, the ascending method is used and the step factor is 2. The sniffing conditions are different; one sniffs the air in the bag and the other sniffs the air that emanates from a port.

The Panel Screening Test: In the Japanese method, the test is performed with five standard odorants to exclude hyposmias. In the European method, panel members who have sensitivity to n-butanol within a certain range are selected.

In this report, we describe the results of comparative experiments on these points.

2. Comparison of both methods

(1) Dilution accuracy

The European Method recommends CO as a tracer gas for calibrating the diluting apparatus. However, some actual odorants tend to be adsorbed on the surface of certain materials, therefore the dilution accuracy might not be the same as that of CO. Three odorants, m-xylene, 100ppm; n-butanol, 100ppm; and hydrogen sulfide, 10ppm, were then diluted to concentrated levels of the olfactory thresholds by each method and the concentrations of the odorants in the diluted gases were analyzed.

The dilution system of the olfactometer depends on the device. The olfactometer used in this study (Olfactomat-n2, Project Research Amsterdam B.V Netherlands) dilutes the sample gas with odor-free air by controlling the gas flow with mass flow controllers and fixed valves. The diluted sample gas emanates at 20L per minute from the sniffing port.

In the dilution procedure of the triangular odor bag method, first, odor-free air is filled in a 3L odor bag. A certain amount of an original sample is then injected into the bag with a glass syringe.

0.5-2L of the diluted gas is concentrated with liquid oxygen and then injected into a gas chromatograph (HP5890). The coefficient of variation of the analysis ranged from 1 to 4%, when 0.5L of the three standard gases was concentrated and analyzed five times.

The error (%) shown in Table 1 indicates the bias of the actual dilution factor relative to the theoretical dilution factor. In the case of the olfactometer, it was not more than 13% for m-xylene. However it was -23% after being diluted 3,543 times for n-butanol, and -46% after being diluted 10,467 times for hydrogen sulfide. The error for hydrogen sulfide tends to increase as the dilution factor becomes higher. It was also observed that the actual concentration of diluted gas tends to be lower during the first dilution operation for n-butanol and hydrogen sulfide. The results indicate a possibility of the actual concentration being lower than the setting value at a higher dilution factor for some odorants. On the other hand, the error for the same three odorants was 12% less in the odor bag method. Incidentally, the odor bags used were manufactured larger than the original size; therefore some checks are necessary on the product of each manufacture.

Table 1 Dilution test results (n=3)

	Olfactometer				Odor bag		
m-Xylene							
Dilution factor	3543	1672	870	492	3430	1140	343
Theoretical concentration (ppb)	30.8	65.2	125	222	31.8	95.3	318
Actual concentration (ppb)	33.8	67.8	142	232	29.4	87.1	306
Error (%)	10	4.0	13	4.5	-7.6	-8.7	-3.6
n-Butanol							
Dilution factor	3543	1672	870	492	3430	1140	343
Theoretical concentration (ppb)	16.5	34.9	67.1	119	17.0	51.1	170
Actual concentration ppb)	12.6	32.4	67.8	125	15.3	47.8	151
Error (%)	-23	-7	1	5	-10	-6	-12
Hydrogen sulfide							
Dilution factor	10467	6494	3543	1672	11400	3430	1140
Theoretical concentration (ppb)	0.955	1.54	2.82	5.98	0.875	2.92	8.75
Actual concentration (ppb)	0.513	1.06	2.29	5.57	0.899	2.91	8.34
Error (%)	-46	-31	-19	-6.9	2.8	-0.4	-5

Note: Volume of odor bag used was 3.43L.

Error (%) = (Actual concentration–Theoretical concentration)/ Theoretical concentration・100

(2) Panel Screening Test

The same assessors were examined by each panel screening test on the same day, and the results were compared.

The outline of each screening procedure is as follows:

The Japanese method: Five standard odor solutions, which are prepared by dissolving β-phenylethyl alcohol, methyl cyclopentenolone, isovaleric acid, γ-undecalactone, and Scatorl in odor-free liquid paraffin, are used for the screening. The test is carried out using odor-free paper by a 5-2 method. Assessors who can distinguish two of the papers which were soaked in the standard solution form the other three papers soaked in the odor-free solution for all of the five odorants can be a panel member. The concentrations of the standard solutions are set at the point of 1.5 times the standard deviation from the mean value based on the olfactory threshold distribution of Japanese people. In this study, the individual threshold values for the five odorants were measured using lower concentration solutions.

The European method: Assessor selection is based on their individual sensitivities and variability for n-butanol. At least ten individual threshold values for each assessor are measured in at least three sessions on separate days with a pause of at least one day between sessions. The antilog of the standard deviation expressed as log (ppb) should be less than 2.3, while the geometric mean should be between 20 and 80 (ppb).

In this study, measurement of the threshold by the olfactometer has been carried out in conformity with the European method. Assessors are presented with two ports and choose which of the ports with stimulus, and indicate their certainty: certain, guess, or inkling. Presentations are done in ascending order and continued until at least two consecutive TRUE responses (correct and certain) are collected. The individual threshold is determined by the geometric mean of the dilutions at which odor is detected and the preceding higher dilution.

There were twenty assessors between the ages of 18 and 63 years old examined in this experiment. Each assessor participated in the tests for three non-consecutive days. The individual threshold measurement for each assessor was carried out twice for five Japanese standard odorants and six to eight times for n-butanol by the olfactometer on each day. The number of individual data for the threshold is six for five Japanese standard odorants, and about twenty for n-butanol.

Table 2 shows the result of the panel screening tests. In the Japanese method, two people did not pass the β-phenylethyl alcohol test. In the European method, ten people did not meet the sensitivity criterion and four people did not meet the variability criterion. In total, fourteen people did not pass the test. The selection criteria in the European method are considerably stricter than those in the Japanese method.

Table 2　Results of Panel screening tests

Assessor	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
Japanese method	○	○	○	○	○	○	○	×	○	○	○	○	○	○	○	○	○	○	○	×
European method Sensitivity	×	×	×	○	×	○	○	×	○	×	×	×	○	○	○	×	○	×	○	○
Variability	○	○	×	○	○	○	○	○	×	○	○	○	○	×	○	○	○	○	○	×

Note: Odorant for which assessors did not meet the criteria in the Japanese method was β-phenylethyl alcohol

Assuming that the distribution of the logarithm of the individual threshold becomes a normal distribution, the distance of each selection criterion of sensitivities from the mean value were calculated. In addition, the ratio of the group, which does not meet the criterion, was obtained from the normal distribution table. The results are shown in Table 3.

Table 3 Comparison of criteria selection in view of the distribution of the individual threshold

	β-phenylethyl alcohol	methyl cyclopentenolone	isovaleric acid	γ-undecalactone	Scatorl	n-butanol	
Mean (m)	5.37	5.97	6.14	5.60	6.99	1.91	
Standard Deviation(s)	0.98	0.33	0.28	0.50	0.40	0.38	
Selection Criteria	4 m-1.4s	4.5 m-4.5s	5 m-4.1s	4.5 m-2.2s	5 m-5.0s	1.3 m-1.6s	1.9 m+0.04s
Ratio of disqualification (%)	8.1	0.1<	0.1<	1.4	0.1<	5.5	48

Note: The values for five Japanese standard odorants are n of concentration 10^{-n} (w/w). For n-butanol, the values are the logarithm of the concentration (ppb). Therefore, in the case of five odorants, if the individual threshold is smaller than the value of the criterion, it does not meet the criteria as hyposmia. In the case of n-butanol, if the individual threshold is smaller than 1.3, it means the assessor has a super-nose, and if it is larger than 1.9, the assessor's sensitivity is weak.

The selection criteria for the five standard odorants became at the point of 1.4 - 5 times the standard deviation from the mean value. Ratios of disqualification were 1.4% or less except for β-phenylethyl alcohol, the ratio of which was 8.1%.

In the case of n-butanol, the criterion to exclude the super-nose was at the point of 1.6 times the standard deviation from the mean value. The criterion concerning weak sensitivity was at the point of 0.04 times the standard deviation. The latter value is almost the same as the mean value. The ratio of disqualification as a super-nose was 5.5%, while that due to weak sensitivity was 48%. In total, more than half of the people might not qualify to be a panel member.

Whether a panel member who passed the screening test using one standard odorant has adequate sensitivity for any actual odor is a difficult question to answer, because individual sensitivity might vary significantly depending on the odor substances. The results in table 2 show that one out of two assessors who did not have sufficient sensitivity for β-phenylethyl alcohol met the sensitivity criteria for n-butanol. Although this result suggests that some mixture is needed as a standard odor, it might be realistic to exclude the outlier by discarding the data after measurement.

(3) Result of olfactory measurement

To grasp the difference in the odor concentration values determined by both methods, various odor samples were measured.

Twelve people who passed the Japanese screening test were selected as panel members.

They were divided into 2 groups of 6 people. Group A consisted of 6 people who passed the European screening test, in contrast to group B which consisted of the other 6 people who did not pass the European test. In each group, the same samples were measured by the both methods on the same day.

Three standard odorants were measured as samples, m-xylene, 35.6ppm; n-butanol, 31.9ppm and hydrogen sulfide, 0.299ppm, and then six actual source samples were also measured. All samples were prepared in 50L polyester bags.

In the case of source samples, the original gas samples were left untouched for two weeks after sampling to stabilize their odor concentration. They were then diluted and the odor was measured. There was a one-day gap between measuring group A and group B.

An individual threshold was measured five or more times for standard odor samples and three times for source samples. The first measurement data taken by the olfactometer were discarded in conformity with the European method.

The measurement results for the standard odor samples are shown in Table 4. The mean values in this table are indicated in the logarithm of the olfactory threshold. The measured results of both methods in each group were generally the same. A difference outcome was expected from the dilution accuracy test for hydrogen sulfide, however none was apparent. That is, there was a possibility that the logarithm of the threshold determined by the olfactometer would raise, due to a decrease of the diluted gas concentration. The corresponding data for m-xylene, which is diluted very accurately, indicate that a difference in the methods such as descending or ascending was not apparent.

Table 4 Olfactory measurement results for standard odor samples (threshold logarithm)

		Group A		Group B	
		Triangular odor bag method	Dynamic olfactometry	Triangular odor bag method	Dynamic olfactometry
m-xylene	Mean (ppb)	2.0 (98)	2.0 (90)	2.2 (166)	2.3 (215)
	Standard deviation	0.37	0.08	0.11	0.04
	n	7	7	8	7
n-butanol	Mean (ppb)	1.3 (20)	1.6 (40)	1.4 (23)	1.4 (24)
	Standard deviation	0.17	0.06	0.15	0.10
	n	6	5	7	6
hydrogen sulfide	Mean (ppb)	2.7 (523)	2.8 (661)	2.8 (591)	2.7 (497)
	Standard deviation	0.20	0.08	0.19	0.15
	n	7	5	7	5

Note: Threshold of m-xylene, n-butanol are indicated in ppb, hydrogen sulfide in ppb.

The panel-screening test using n-butanol was held six months before this odor measurement. The results showed the threshold values of group A members were approximately 40ppb while most of the members from group B were valued at 100-200 ppb. However, a significant difference was not observed in this measurement. This suggests that the sensitivity of some individuals may largely vary over a period of years. Provided that this is true, selecting panel members by the European method has to be performed very carefully.

The measurement results for the source samples are shown in Table 2. The value is indicated in the odor index. (odor index = 10 log [odor concentration]) The results of both methods in each group corresponded very well as well as for standard odors, though a difference of 4 was observed for the excrement odor for group B. It seems that the odor index in group A tends to be higher than that for group B. However, it cannot be concluded that the reason is whether there is a difference of sensitivity between members of each panel or differences of sample concentrations.

Table 5 Olfactory measurement result for source sample (odor index)

	Group A		Group B	
	Triangular odor bag method	Dynamic olfactometry	Triangular odor bag method	Dynamic olfactometry
Spray painting	27	25	22	22
Baking finish	28	26	26	23
Offset printing	29	29	27	26
Sewage	28	30	24	25
Excrement	32	31	28	24
Rendering	29	30	30	29

3. Conclusion

Provided that the same panel is used, the triangular odor bag method and dynamic olfactometry agree in results in spite of such differences as descending or ascending, sniffing conditions, etc. However, the selection criteria for the panel screening show a large difference. Although this may influence odor measurement results, the effect could not be observed in this present study because of variation in sensitivity. It will be necessary to acquire further data on variability over the long term, and to study the relation between measurement results and the performance of panel.

References

1) European Committee for Standardization, DRAFT prEN13725 Air quality-Determination of odor concentration by dynamic olfactometry (1999)