2. Application and Quality Control of Odor Measurement

A Comparative Study on Odor Regulation in Japan and Korea

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Abstract

Odor control policy in Japan has been based on the Offensive Odor Control Law, which regulates the emission of offensive odors generated from business to prevent a nuisance occurring. In Korea, odor control has been under the Air Conservation Law at present. But the Korean Offensive Odor Control Law, newly prepared to be enacted this year, strengthening the regulation of emission of odor, have common aspects of the Japanese law. Both laws describe the standards of maximum permissible concentration of specified offensive odor substances and a multiple of dilution of odor at border of business or emission source in regulation area. The Japanese rule has been promulgated since early 1970’s and developed to be more ideal but complicated. In other hand, the current Korean rule is very simple, but new regulation is expected to be similar as that of Japan.

1. Introduction

In Korea, the offensive odor is defined as the odor of such as hydrogen sulfide, mercaptans, amines and other irritant gas that cause unpleasantness and repugnance, and it has been treated as the sensory pollution like noise and vibration and categorized in air pollution (1). The early industrialized nation, Japan, coped with this problem and promulgated Offensive Odor Control Law in 1973 to abate odor nuisance. Later than Japan, Korea had been industrialized in 1990’s and public complaints like offensive odor have become a social problem in heavily industrialized area, such as Ulsan and Yeocheon located in the southern coast of the Korean peninsula. In 1993, Korea enacted the limit of concentrations of the offensive odor substances at the boundary of the site, that was very similar to the Japanese Offensive Odor Control Law amended in 1978, and the limit of dilution factor of the offensive odor at the point of emission from facilities, similar to the state or city ordinance for the prevention of offensive odor of many countries. Measurement and regulation of odor in Korea and in Japan have very similarity but differences in many points. In here, difference and similarity of odor policy in two countries will be introduced.

2. Measurement method and regulation

6 stages of odor strength are the standard criteria for regulation of odor at the boundary of factories or other places of business. Table 1 shows the current Korean law and the Japanese amended in 1976 for offensive odor. Both countries use the sensory method for offensive odor and instrumental analysis for odorous compounds to estimate the odor strength. In Korea, direct sensory measurement at the boundary

of business has been used for judgment of illegality of odor emission till now. Therefore a person establishing a place of business shall observe the permissible limit of odor, meaning maximum emission of odor or odorous substances from the boundary of business or point source. In Japan, direct sensory method is not used for regulation but the primary investigation of offensive odor.

Table 1 Comparison of the current Korean permissible limit for offensive odor and the Japanese offensive odor control law promulgated in 1976

Measurement Method			Korean permissible limit for odor		Japanese offensive odor law in 1976	
Direct Sensory Method			Less than 2(odor strength)		None	
Sensory Method	Gas releasing port	Industrial	Maximum dilution factor	1000	Range of Concentration	500~1800
		Residential		500		300~500
	Border line	Industrial		20		20~30
		Residential		15		10~15
Instrumental Method	Specified offensive odor substance (ppm)		Residential Area	Industrial Area	Residential Area	Industrial Area
	Ammonia		2	1	2~5	1~2
	Methyl mercaptan		0.004	0.002	0.004~0.01	0.002~0.004
	Hydrogen Sulfide		0.06	0.02	0.06~0.2	0.02~0.06
	Methyl Sulfide		0.05	0.01	0.05~0.2	0.01~0.05
	Dimethyl Sulfide		0.03	0.009	0.03~0.1	0.009~0.03
	Trimethyl amine		0.02	0.005	0.02~0.07	0.005~0.02
	Acetaldehyde		0.1	0.05	0.1~0.5	0.05~0.1
	Styrene		0.8	0.4	0.8~2	0.4~0.8

A multiple of dilution (called as “odor concentration” in Japan and “dilution factor” in Korea), where the gas has been diluted until an offensive odor is no longer detectable to the human sense of smell, is described as the standards for odor regulation of the boundary and the emission point. Both countries differentiate the level of regulation according to the population and usage of land. In Korea, 1,000, the allowable highest dilution factor at gas releasing port of the factory established in industrial area is the substantial regulation of offensive odor. In Japan, each prefecture has his regulation standards for each regulation area within the range stipulated by the government. 8 specified offensive odor substances declared in the permissible odor emission standard enacted in the Korean air conservation law in 1993 were equal to those of the Japanese Offensive Control Law amended in 1978. The current Japanese Offensive Control Law (2) describes 22 specified substances for the regulation at boundary of a business. Although the Japanese law states the range of concentrations of specified odor substances, each prefecture applies the most severe standard to each regulation area. Regulation standards at the border of business in industrial area are equal to 3.5 in 6 stages of odor strength and in residential area 2.5 in 6 stages.

3. Standard odors for panelist and manipulation of data

Table 2 shows the standard odor liquid for selection of the panel recognized to have normal smell function. Chemicals used in Korea are easily obtainable but the Japanese are used for otolaryngology.

Table 2 The standard odors of sensory test for panelist in Korea and Japan

In Korea		In Japan	
The Standard odors	Concentration(wt%)	The Standard odors	Concentration(ppm)
Acetic acid	1.0	i-Valeric acid	30
		Skatol	10
Trimethyl amine	1.0	Methyl cyclopentenolone	30
		β-Phenyl ethyl alcohol	100
Phenol	0.1	γ-Undecalactone	30

Calculation method of a multiple of dilution called as the odor concentration in Japan and also the dilution factor in Korea differentiates the Offensive Odor Control Law of Japan and the permissible odor limit of Korea. Table 3 shows each calculation method of a dilution multiple for the odor sample obtained from an emission point. In Japan, at least 6 members of panelists are needed for judgment of odor concentration but in Korea at minimum 5 panelists are needed. In both countries, data of maximum and minimum must be excluded from calculation. Judgment of existence of smell by each panel is done with triangle comparison method of odor bag in both countries. The odor concentration calculated by the Japanese method is 500 but the dilution factor by the Korean is 448 in this table.

Table 3 Example of sensory test on odor sample at gas releasing port

Multiple of dilution		30	100	300	1000	3000	10000	Threshold value of each panel		Exception of Max. and Min.
Algebraic value		1.48	2.00	2.48	3.00	3.48	4.00	Japan	Korea	
Panel	A	/	○	X				2.24	100	Exception
	B	/	○	○	X			2.74	300	
	C	/	○	○	○	○	○	3.74	10000	Exception
	D	/	○	○	X			2.74	300	
	E	/	○	X				2.24	100	(Exception)
	F	/	○	○	○	X		3.24	1000	

(Data of A and C were excluded from the Japanese calculation and data A, C and E from the Korean.)

- Permissible dilution factor of Korea : $\sqrt[3]{300 \times 300 \times 1000} = 448$
- Odor concentration of Japan : $10^{2.74} = 550$

448 is the geometric average of each threshold value and 2.74 is the arithmetic mean of threshold values of each panel.

4. Offensive Odor Control Law in Japan and Korea

Current Japanese regulation on odor consists of maximum permissible concentration of specified offensive odor substances and odor concentration. Table 4 shows the 22 specified substances regulated as odorous compounds at the border of a business defined in the Japanese law. Among them, 13 substances at a gas releasing port have their permissible concentrations and also maximum allowable concentrations of 4 substances in water are defined therein. 13 substances discharged from smoke stacks or other gas emission facilities are shown in table 5. This rule means higher stack can emit more odor in Japan. But in Korea, odor from all stacks in industrial area must be under 1000 of dilution factor. The Japanese odor policy has been developed since 1973, to be very complicated now. In Korea, the concept of offensive odor has not been prevailing and common and it makes the regulation not to be complicated.

Table 4 Specified odor substances regulated in the Japanese Offensive Odor Control Law

Subastance	Boundary	Gas emission	Water	Substance	Boundary	Gas emission	Water
Ammonia	○	○		i-Valeraldehyde	○	○	
Methyl mercaptan	○		○	i-Buthanol	○	○	
Hydrogen sulfide	○	○	○	Ethyl Acetate	○	○	
Methyl sulfide	○		○	MIBK	○	○	
Dimethyl sulfide	○		○	Toluene	○	○	
Trimethyl amine	○	○		Styrene	○		
Acetaldehyde	○			Xylene	○	○	
Propionaldehyde	○	○		Propionic acid	○		
n-Butyl aldehyde	○	○		n-Butyric acid	○		
i-Butyl aldehyde	○	○		n-Valeric acid	○		
n-Valeraldehyde	○	○		i-Valeric acid	○		

Table 5 Regulation standard for the flow rate or concentration of the specified odor substances at the point of emission from facilities at Chiba prefecture in Japan

Odor substance	Maximum flow rate at point source(N m^3/h)	Odor substance	Maximum flow rate at point source(N m^3/h)
Ammonia	0.108 × 1 × He^2	i-Valeraldehyde	0.108 × 0.003 × He^2
Hydrogen sulfide	0.108 × 0.02 × He^2	i-Butanol	0.108 × 0.9 × He^2
Trimethyl amine	0.108 × 0.05 × He^2	Ethyl Acetate	0.108 × 3 × He^2
Propion aldehyde	0.108 × 0.05 × He^2	MIBK	0.108 × 1 × He^2
n-Butyl aldehyde	0.108 × 0.009 × He^2	Toluene	0.108 × 10 × He^2
i-Butyl aldehyde	0.108 × 0.02 × He^2	×ylene	0.108 × 1 × He^2
n-Valeraldehyde	0.108 × 0.009 × He^2		

(He: Corrected height of the gas emission point)

Recently, the Ministry of Environment of Korea is planning the new policy for offensive odor and e×pected to enact the Korean Offensive Odor Control Law, involving more kinds of specified odor compounds at border and severe regulation of odor emission from point sources.

5. Conclusion

The Japanese Offensive Odor Control Law has been enacted in 1973 and developed to be ideal but complicated today. In late of 1900's Korea, an odor nuisance has increased drastically to be a social problem. Recently, Korean government have been considering a new policy for odor, and the Korean Offensive Odor Control Law, planned to be promulgated in this summer, will reduce the odor nuisance of Korea. Various odor control policies of industrialized countries (especially, the Offensive Odor

Control Law of Japan) have been under the investigation for establishment of the new Korean odor law.

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Quality Control of Olfactometry at SRI and in Europe

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Keywords
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Abstract

Quality of odour measurements is assured in the UK laboratories by following the European standard for “Determination of odour concentration using dynamic olfactometry”. The EN13725 is now the standard followed by all European countries for odour concentration measurements. The accuracy and repeatability of the measurements is assured by conforming to the quality criteria for accurate dilution equipment, compliance to the panel selection criteria and standardised data collection and data analysis methods.

1. Introduction

Until 1997 when a draft of the European standard for olfactometry was issued to the working group there was no single method for objectively measuring odour concentration. After an inter-laboratory comparison, committee members accepted that the draft method was suitable and most European laboratories adopted the use of the prEN13725 (now BSEN 13725 in the UK)(1). The European standard, EN13725, was ratified in late 2002 and Silsoe Research Institute Odour Laboratory has obtained Laboratory Accreditation to ISO17025 for “Determination of odour concentration using dynamic olfactometry” following the BSEN13725 (2).

In this paper the quality controls required are described and data from the measurement of environmental odours illustrate the benefits of the method in producing good repeatability.

2. Principle of measurement

The odour concentration of a gaseous sample of odorants is determined by presenting a panel of selected and screened human subjects with that sample, changing the concentration by diluting with neutral (odourless) gas, in order to determine the dilution factor at the 50% detection threshold ($Z_{50} \equiv \overline{Z}_{\text{ITE,pan}}$).
At that dilution factor the odour concentration is 1 ou_E/m^3 by definition. The odour concentration of the examined sample is then expressed as a multiple (equal to the dilution factor at Z_{50}) of one European Odour Unit per cubic metre [ou_E/m^3] at standard conditions for olfactometry (20C, 1013mbar).

2.1 The forced choice mode

The Silsoe Research Institute laboratory operate a forced choice dynamic olfactometer, (model OdourNet olf-n6) Fig. 1, it has two outlet ports from one of which the diluted odour flows and clean odour-free air (neutral gas) flows from the other(s).
The other choice mode allowed under the Standard is “Yes/No” mode where panel members respond “yes” to and odour and “no” when the odour is not detected in the air stream flowing from a single port. Odours of random dilution are presented interspersed with blanks.

In the forced choice mode, measurement starts with a dilution of the sample large enough to make the odour concentration below the panel members’ thresholds, the concentration is increased by an equal factor in each successive presentation, and factor at SRI is between 1.7 and 1.8. The port carrying the odorous flow is chosen randomly by the control sequence on each presentation. The assessors sniff and indicate from which of the ports the diluted odour sample is flowing using a personal keypad. They also indicate whether their choice was a guess, whether they had an “inkling” or whether they were certain they chose the correct port. Only when the correct port is chosen and the panel member is certain that the choice was correct is it taken as a TRUE response. At least two consecutive TRUE responses must be obtained for each panel member. The geometric mean of the dilution factors of the last FALSE and the first of at least two consecutive TRUE presentations determines the individual threshold estimate (ITE) for a panel member. The odour concentration for a sample is calculated from the geometric mean of at least two ITEs for each panel member.

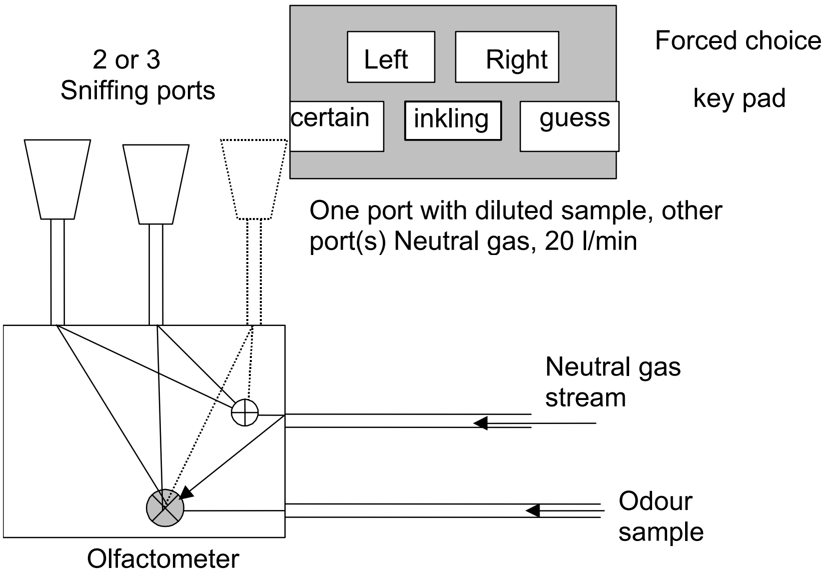

Fig 1 Schematic diagram of a forced choice olfactometer

For measurements on reference odorants this value can be converted to an individual threshold estimate expressed as a mass concentration using the known concentration of the reference gas divided by the ITE.

3. Laboratory Quality Control

3.1 Instrument quality criteria

The accuracy and repeatability of the dilution apparatus, the olfactometer, is the first step in quality control of olfactometry. The first important criterion is the accuracy of the dilution equipment. The criteria set out in the standard BSEN13725 are that the accuracy A_d of the dilution setting should deviate less than 20% from the required setting and the repeatability on setting that dilution ratio must be better than 5%. The ratio or step factor between the set points must be between 1.4 and 2.4. At SRI it is between 1.7 and 1.8

The Standard stipulates that a calibration should be done at least annually, however as a precaution the SRI quality assurance system ensures that a check on the calibration settings is carried out monthly. This is done using a Brηel & Kjaer 1302 gas analyser and sulphur hexaflouride as the tracer gas. Values recorded in these checks are compared with the values on the calibration certificate, if the values are outside the standard's criteria adjustments are made and previous results are checked and recalculated if necessary.

3.2 Assessor selection

The second key part of accurate odour measurement is the selection of the odour assessors who make up the panel. In order to select odour assessors *n*-butanol (butan-1-ol) has been chosen as the reference material. (It is recognised that a single component reference gas is not the ideal but no representative odorant mixture has yet been formulated.) Only people with a mean ITE for n-butanol in neutral gas of between 20 ppb and 80 ppb and a log standard deviation of less than 2.3, are acceptable, calculated from the previous 10 to 20 ITEs. These assessors are checked after no more than 12 regular odour measurements, the equivalent to calibrating a gas analyser, for their detection threshold and have to remain within these limits to be a panel member.

These selection criteria used at the Silsoe Research Institute laboratory leads to us rejecting about 43% of those tested because they are not sensitive enough and about 12% because they are too sensitive.
Selection of the panel members using the above method leads to the accuracy and precision to enable the laboratory to comply with the criteria set in the standard (EN 13725)

3.3 Laboratory conditions

The third feature that ensures quality measurement is the laboratory conditions and assessors behaviour. For laboratories to conform to the required standard, they must be guaranteed to be free from odour, at Silsoe we have an air-conditioned laboratory with activated charcoal filtration to ensure an odour free atmosphere. We also have a source of odour free air, neutral gas, again cleaned with an activated charcoal filter, with which to dilute the odour sample. The olfactometer, which is a dilution device, is made entirely from approved materials, glass, FEP, or stainless steel. Samples are processed the next day, within 30 hours of collection as dictated by the BSEN13725.

3.4 Quality criteria

The Standard is based on the following accepted reference value and shall be used when assessing trueness and precision:

$1\ ou_E \equiv 1\ \text{EROM} = 123\ \mu g$ *n*-butanol

When 123 µg *n*-butanol is evaporated in one m³ of neutral gas at standard conditions for olfactometry the concentration is 0,040 µmol/mol (40 ppb or a $\log_{10}$ value of 1.6)

Two quality criteria below are specified to measure the performance of the laboratory in terms of the standard accuracy and precision, respectively.

Accuracy reflects the trueness or closeness to the correct value, in this case the true value for the reference material is 40 ppb and the precision is the random error. The standard specifies how these two quality criteria are calculated.

The criterion for accuracy A_{od} (closeness to the accepted reference value) is

$A_{od} = \leq 0{,}217$, at Silsoe this statistic is currently $A_{od} = 0.152$

In addition to the overall accuracy criterion, the precision, expressed as repeatability, r, shall comply with

$r \leq 0.477$, currently $r = 0.283$

This criterion for repeatability can also be expressed as:

$10^r \leq 3.0$, currently $10^r = 1.92$

This laboratory repeatability implies that the factor that expresses the difference between two consecutive single measurements, performed on the same testing material in the laboratory will not be larger than 1.92 in 95% of cases.

3.5 Laboratory History

Data for establishing the above criteria are collected at least once per day of measurement; the laboratory history is displayed in Fig 2. The mean and standard deviation are plotted and all lie within the criteria. Minor variations can be seen these are caused by variations in the make up of the panel as well as day to day variations in individual's sensitivity. These records provide us with a continuous record of the quality status of our measurements.

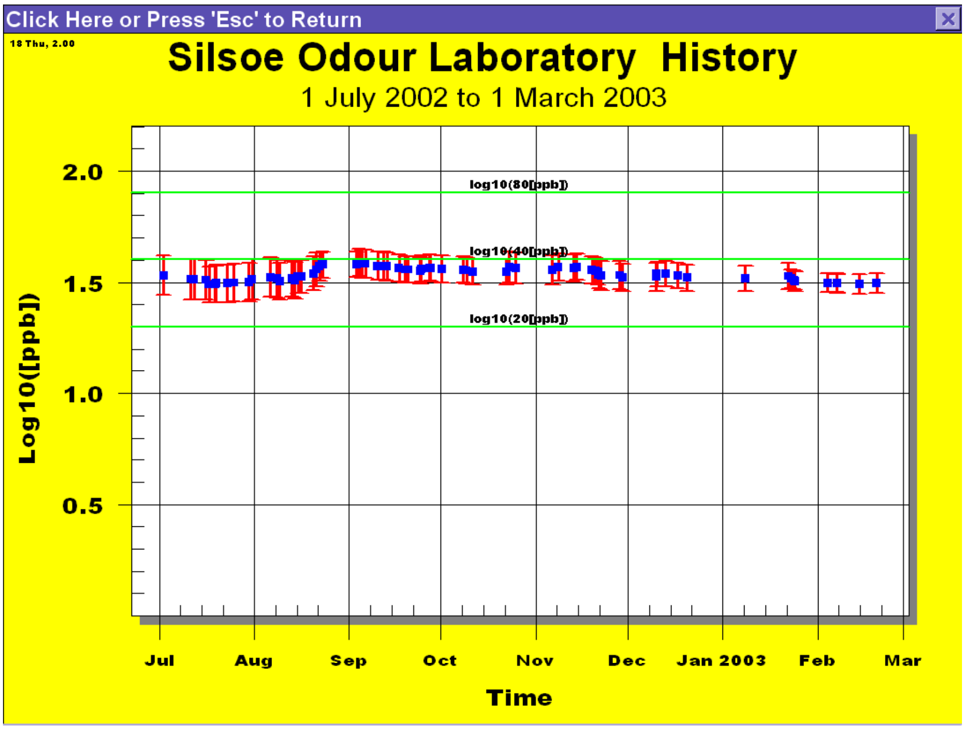

Fig 2 Record of the laboratory accuracy and precision with n-butanol.

4. Quality of measurements of environmental odours when the EN quality criteria are met

SRI laboratory repeatability calculated in 3.4 above implies that the difference between two consecutive single measurements, performed on the same testing material in the laboratory will not be larger than a factor 1.92 in 95% of cases.

However there are always questions about the applicability of this single reference odorant when the primary use of olfactometry is measurement of environmental odours that contain scores of odorants. Early this year we briefly investigated the repeatability by replicating measurements on samples reaching the lab; these are shown in Table 1. Although there may be questions about the transferability of assessor’s perception of *n*-butanol to their perception of real odours the results do show that the repeatability of measurements on real odours is better than predicted from the n-butanol data.

In order to comply with all the requirements of ISO17025 an accredited laboratory is required to participate in a “Round Robin” interlaboratory comparison, we have arranged for this to take place in spring 2003. The samples used in the comparison will be both the reference material, *n*-butanol, and environmental odour samples.

Table 1 Comparison of range of results from real odours and n-butanol.

Odour source	Measured values, $ou_E\ m^{-3}$	Mean, $ou_E\ m^{-3}$	sd	Maximum ratio between duplicate measurements
n-Butanol			(n=20)	1.92 (95%ile)
Pig feed 1	11250			
	12304			
	16633	13124	1.22	1.47
Pig feed 2	6988			
	6637			
	7659	7082	1.07	1.15
Restaurant	751			
	997	865		1.32
Restaurant	789			
	806	797		1.02
Restaurant	997			
	1324	1149		1.32

5. Conclusions

(1) The European Standard BSEN13725 provides a basis for quality assurance for measurement of odour concentration.

(2) Key quality criteria are

- Accurate dilution apparatus
- Rigorous selection of the odour assessors
- Carefully controlled laboratory conditions

(3) Measurement results on real odours show the level of repeatability to be better than with the *n*-butanol reference material

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A Comparative Study of Japanese and European Olfactometry Standards

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Keywords
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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 triangle odor bag method is adopted as an olfactometry standard in the Offensive Odor Control Law in Japan. However, the dynamic olfactometry1) 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 triangle 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 triangle 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 dysosmias. 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

2.1 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 triangle 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

2.2　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 dysosmia. 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.

2.3 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	
		Triangle odor bag method	Dynamic olfactometry	Triangle 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 5. 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	
	Triangle odor bag method	Dynamic olfactometry	Triangle 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 triangle 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.

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Quality Control of Olfactometry in Japan

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Olfactometry, quality control, triangle odor bag method

Abstract

In Japan, the necessity of developing quality control system for olfactometry and standardization of measurement procedure for the promotion of nationwide spread of olfactometry has been recognized especially in recent years. In this paper, the establishment of quality control framework for olfactometry in Japan, including selection of a reference odor, development of reference odor preparation technique, interlaboratory comparison of olfactometry, and fundamental constitution of quality control manual for laboratory use, was discussed. Ethyl acetate was selected as a reference odor for olfactometry, and four preparation methods (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were verified and confirmed to be applicable to quality control processes. In late 2000, interlaboratory comparison of olfactometry was carried out in order to collect basic data for the establishment of quality control procedure and the determination of quality criteria. Mean values, repeatability standard deviation, reproducibility standard deviation, and standard deviations under intermediate conditions of detection threshold were calculated from the measurement results. On the bases of these discussions, quality control framework for olfactometry was proposed. In early 2003, the quality control manual for laboratory use was published.

1. INTRODUCTION

In Japan, regulatory policy of offensive odor based on olfactometry (i.e., triangle odor bag method and triangle odor flask method) was first introduced into the Offensive Odor Control Law in 1995.1) However, the necessity of developing quality control system for olfactometry and standardization of measurement procedure for the promotion of nationwide spread of olfactometry in municipalities has been recognized especially in recent years.

This paper presents about the establishment of quality control framework for olfactometry in Japan, including selection of a reference odor, development of reference odor preparation technique, interlaboratory comparison of olfactometry, and proposal of fundamental constitution of quality control manual for laboratory use.

2. FUNDAMENTALS OF TRIANGLE ODOR BAG METHOD

The triangle odor bag method, the most popular olfactory sensory test in Japan, was first developed by Tokyo metropolitan government in 1972.[2)3)] This is an air dilution method in which "odor concentration" or "odor index" is measured. Odor concentration is the dilution ratio when odorous air is diluted by odorless air until the odor becomes unperceivable. Odor index is the logarithm of odor concentration, multiplied by ten. The olfactory measurement method of offensive odor was notified in 1995 (Notification No. 63 of the Japan Environment Agency, 1995). This method consists of the following parts:

- Panel selection
- Apparatus
- Sampling
- Testing procedure

Individual panelist is required to have passed the screening test of using five odorous compounds (i.e., phenethyl alcohol, methylcyclopentenolone, isovaleric acid, γ-undecanolactone, and skatole). Measurements for samples taken at odor emission sources are made in three-fold dilution descending series. In this method, three odor bags are prepared and filled with odorless air passed through the activated carbon column. Odorous sample is injected into one of these three odor bags. Each panelist sniffs these odor bags and chooses one odor bag that is likely to contain odorous air. The test is continued until all panelists make incorrect replies. Then odor concentration or odor index is calculated.[1)]

3. REFERENCE ODOR FOR OLFACTOMETRY

3.1 Selection of reference odor

Reference odor is necessary in order to conduct interlaboratory comparison of olfactometry as well as routine verification of measurement results in each olfactometry laboratory. Four odorous compounds (i.e., *n*-butanol, ethyl acetate, *m*-xylene, and dimethyl sulfide) were proposed for reference odor. In Europe, *n*-butanol is defined to be a reference odor in CEN draft standard prEN 13725.[4)] Ethyl acetate is one of "specific offensive odor substances" designated in the Offensive Odor Control Law in Japan, and *m*-xylene and dimethyl sulfide are compounds used as reference odors in previous interlaboratory comparison in Japan. Reference odor for olfactometry should fulfill the following requirements:

- Odor sample should be prepared easily and accurately.
- Odor sample should remain stable for a period of the measurement.
- Odor threshold values of panelists should not vary widely.
- Odor quality should be easily recognized.
- Low health and psychological effect on operators and panelists should be ensured.

Considering all these things, ethyl acetate was selected as a reference odor for olfactometry in Japan. Although *n*-butanol is designated as a reference odor in CEN prEN 13725, it was not selected because there is less measurement data for *n*-butanol in Japan and ethyl acetate has the advantages in sample preparation and data accumulation.

3.2 Preparation of reference odor

Easy-to-operate and cost-effective technique for reference odor preparation is necessary to be employed in nationwide municipalities and olfactometry laboratories. On the assumption that reference odor sample with odor concentration of two to three thousand is appropriate to be used in quality control process, the concentration of ethyl acetate is calculated to be around 2000 ppm in consideration of odor threshold of 0.87 ppm.[5)]

Four preparation methods for reference odor (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were proposed. Steel cylinder containing ethyl acetate of 2010 ppm was specially ordered. In odor bag/vacuum bottle method, an odor bag or a glass vacuum bottle is employed to vaporize ethyl acetate reagent. These four preparation methods were verified at three olfactometry laboratories and confirmed to be applicable to quality control processes.

4. INTERLABORATORY COMPARISON OF OLFACTOMETRY

4.1 Method

In late 2000, interlaboratory comparison of olfactometry was carried out in order to collect basic data for the establishment of quality control procedure and the determination of quality criteria. A total of seven olfactometry laboratories in Japan participated in the test. A three-liter-capacity sampling bag filled with ethyl acetate of around 2000 ppm was delivered to each laboratory four times. Odor index and odor concentration of each sample were measured according to the official procedure of the triangle odor bag method. The tests were conducted six times over four days (i.e., three times for the second sample and only once for other three samples). Steel cylinder method was used to prepare reference odor, i.e., ethyl acetate of 2010 ppm. Gas concentration of each sample was analyzed with GC-FID just before the delivery.

4.2 Results

Figure 1 shows detection thresholds of ethyl acetate calculated from odor concentration and gas concentration obtained from the tests.

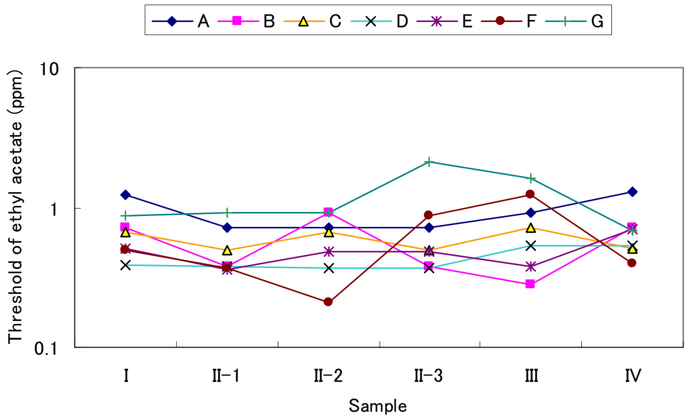

Figure 1. Detection thresholds of ethyl acetate measured in seven laboratories (A-G). The measurement results of II-1, 2, and 3 represent the repeated data of the same sample within one day.

The mean value, repeatability standard deviation, and reproducibility standard deviation of detection threshold were calculated from the results of II-1, 2, and 3 according to JIS Z 8402-2 [6), which is Japanese version of ISO 5725-2 (1994). On the other hand, the results of I, II-2, III, and IV were used for the calculation of the mean value and standard deviations of detection threshold under intermediate conditions.[7) **Table 1** shows these evaluation results. In practice, the logarithm of detection threshold was used for the calculation of these values.

Table 1. Mean values (m), repeatability standard deviation (s_r), reproducibility standard deviation (s_R), and standard deviation under intermediate conditions (s_i) of the logarithm of detection thresholds.

Repeatability and reproducibility conditions	Measurement results	m	Antilog of m (ppm)	s_r	s_R
	II-1, 2, 3	-0.26	0.56	0.17	0.22
Intermediate conditions	Measurement results	m	Antilog of m (ppm)	s_i	s_R
	I, II-2, III, IV	-0.20	0.63	0.18	0.21

5. FRAMEWORK OF QUALITY CONTROL MANUAL

On the bases of foregoing discussions about reference odor and interlaboratory comparison, quality control manual for laboratory use was published in early 2003. **Figure 2** shows quality control framework for olfactometry in a laboratory. The fundamental topics in the manual are as follows:

- Establishment of quality control system and organization in a laboratory
- Education and training of staff concerned
- Documentation of measurement processes
- Preparation of standard operating procedures (SOPs)
- Evaluation and report of measurement results
- Regular internal quality checks using reference odor
- Occasional proficiency tests using certified reference odor

Quality assessment process is depicted in **Figure 3**. On the basis of collaborative assessment experiment, accepted reference value, repeatability, and reproducibility of reference odor are obtained. Then individual olfactometry laboratory is able to carry out regular quality checks and compare the results with these values. In this research, mean value, repeatability, and reproducibility of reference odor threshold were calculated from the results of interlaboratory comparison. However, further investigation will be necessary to obtain the accepted values. Odor bag/vacuum bottle method and handy gas cylinder method seem to be appropriate for the preparation of reference odor for internal use because of the easiness of preparation technique and cost-effectiveness. Certified reference odor (CRO) is necessary for the proficiency tests. Therefore, CRO supply system should be developed in the framework of traceability concept. In this case, steel cylinder method and handy gas cylinder method have advantages.

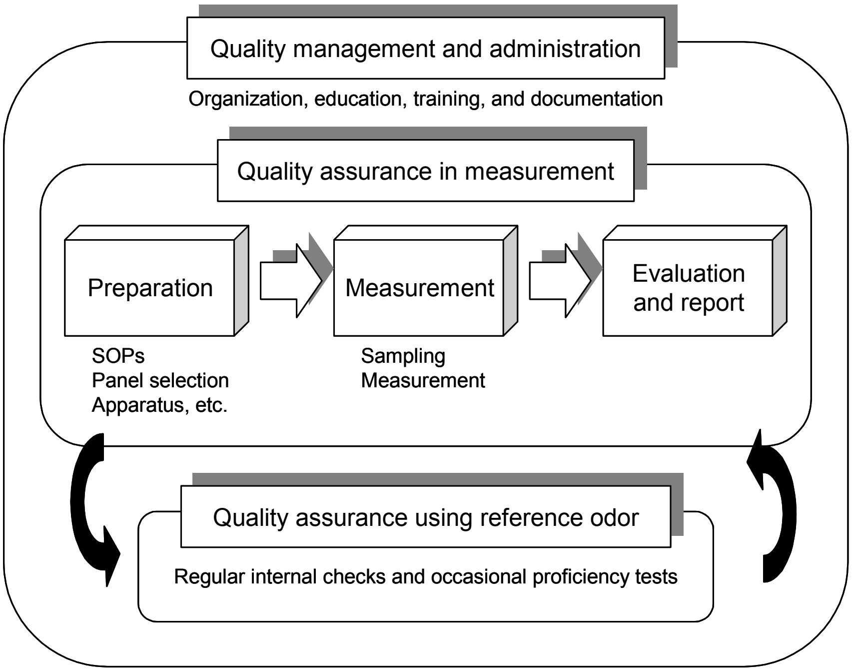

Figure 2. Quality control framework for olfactometry in a laboratory.

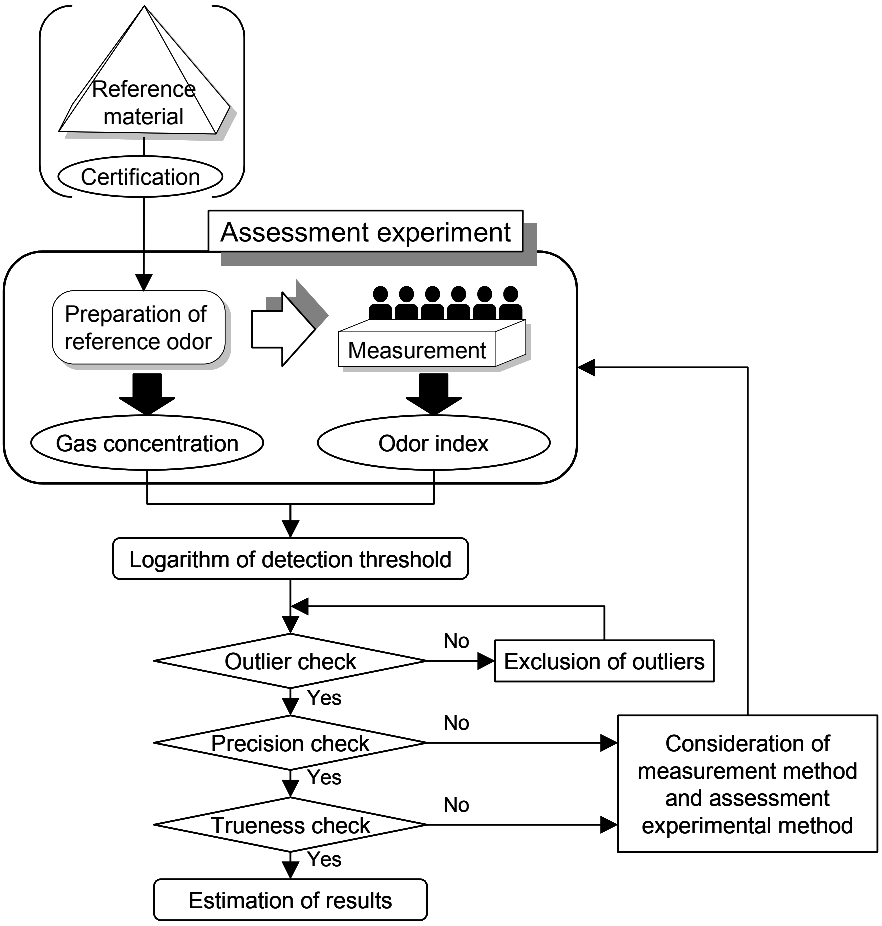

Figure 3. Outline of quality assessment using reference odor.

5. CONCLUSIONS

In this paper, the establishment of quality control framework for olfactometry in Japan was discussed. Ethyl acetate was selected as a reference odor for olfactometry, and four preparation methods (i.e., steel cylinder method, standard gas generator method, odor bag/vacuum bottle method, and handy gas cylinder method) were verified and confirmed to be applicable to quality control processes. In late 2000, interlaboratory comparison of olfactometry was carried out and mean values, repeatability standard deviation, reproducibility standard deviation, and standard deviations under intermediate conditions of detection threshold were calculated from the results. On the bases of these discussions, quality control framework for olfactometry was proposed. In early 2003, the quality control manual for laboratory use was published.

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Standard Odors for Selection of Panel Members

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Keywords
Standard Odors, sense of smell, selecting panel

“The Olfactory Measurement Method" is the method of evaluating the stink by sense of smell. The members to do this measurement are called Panel members.
It is essential to have panel members with an accurate sense of smell to check odors, particularly stink smells. However, up to now, no method existed to confirm whether panel members had an accurate sense of smell or not.
In order to confirm this, it is necessary to have standard odors, and the T & T Olfactometer (Standard Odors for Measuring Sense of Smell) was developed for this purpose. It is used in Japan with approval of the Ministry of Health and Welfare to diagnose symptoms of abnormality in sense of smell.
The standard odors for selecting panel members have been developed based on the T & T Olfactometer, and a lot of data has been collected on Environment Pollution of Offensive Odors by authorities and large organizations. These standard odors are used for selecting panel members by the "5-2 method".

The Offensive Odor Control Law in Japan was revised on April 1, 1996. A national qualification of 'Olfactory Measurement Operator' came to be given to the person who passed the national examination which Ministry of the Environment provided by this revision. The Standard Odors for Selection of Panel Members is used in this national examination. Moreover, The Offensive Odor Control Law in Japan was partly revised on April 1, 2001. In this revision, The Olfactory Measurement Method was added. This product is similarly adopted for selection of panel by this method.

1. Chemical Components and their Odor Quality

A ---------- β-Phenylethyl Alcohol
  * Flower odor
  * Smell of rose petals
B ---------- Methyl Cyclopentenolone
  * Sweet burning smell
  * Smell of caramel in custard pudding
C ---------- Isovaleric acid
  * Smell of sweat
  * Smell of stinking socks

D ---------- γ-Undecalactone
* Smell of ripe fruit
* Smell of canned peaches

E ---------- Skatole
* Musty smell
* Smell also found in excrement

2. Content of the Standard Odors

The densities of the standard odors are all w/w.
The control liquid is odorless liquid.
There are 500 olfaction test papers in one packet.

3. Five standard odors for selecting panel members

This set consists of 5 standard odors A, B, C, D, and E. The middle density is set at the "standard density for selection" as stated in the report by the Environment Agency.

3.1 Standard odors for measuring sense of smell:

A ($10^{-3.0}$,$10^{-3.5}$,$10^{-4.0}$,$10^{-4.5}$,$10^{-5.0}$)
B ($10^{-3.5}$,$10^{-4.0}$,$10^{-4.5}$,$10^{-5.0}$,$10^{-5.5}$)
C ($10^{-4.0}$,$10^{-4.5}$,$10^{-5.0}$,$10^{-5.5}$,$10^{-6.0}$)
D ($10^{-3.5}$,$10^{-4.0}$,$10^{-4.5}$,$10^{-5.0}$,$10^{-5.5}$)
E ($10^{-4.0}$,$10^{-4.5}$,$10^{-5.0}$,$10^{-5.5}$,$10^{-6.0}$)

	5 ml bottle each
Control liquid	50ml 3 bottles
Olfaction test papers	4,000

(note. ﹏ underlining indicates the standard odor for selection)

3.2 Application

This is used to test whether the examinees have a normal sense of smell. At the same time, it is possible to check the degree of ability to sense smells of candidates with a normal sense of smell, by checking whether they can discern densities lower than the standard density for selection.

For examinees with an abnormal sense of smell, it is possible to check how close to normal it is.

4. Preparation

Note. The operator (examiner at the sense of smell test) and the collector of test materials should have a normal sense of smell. This should be confirmed before carrying out the tests on panel candidates. Also the hands of the operator and examinees should be checked to ensure they do not carry any odor. If they do, the hands should be washed well with an odorless, liquid soap.

5. How to Use

The test is carried out using either the 3 standard odors or the 5 standard odors, by the

"5 - 2 method". In this method, the operator selects at random 2 olfaction test papers out of 5, and dips them up to 1 cm from the edge into one standard odor liquid. (Each paper has the 1 cm level marked on it). The remaining 3 olfaction test papers are then dipped into the control liquid. (Fig.1)
The examinees are given 5 test papers one by one, and sniff the tip of each one by bringing the test paper close to, but not actually touching the nose. (Fig.2)
After sniffing all 5 papers, the examinees should select 2 test papers which they think carry the odor, and answer by giving their numbers (both should be correct).(Fig.3)
The examinee passes the test by giving the correct answers for all the standard density odors.

Fig.1

Fig.2

Fig.3

Five standard odors for selecting panel members

6. Points on Usage

(1) How to sniff

Examinees should sniff lightly and briefly while concentrating. They may sniff a second time if they cannot discern the odor the first time. There should be a slight time interval before sniffing again.

(2) Using and Discarding the Olfaction Test Papers

Always wipe the test papers against the inside neck of the bottle after dipping them into the standard odor liquids, to prevent them from dripping.

Do not use olfaction test papers again once they have been dipped into the standard odor liquid. They should be discarded after each test. New olfaction test papers must be used for each examinee, dipping them into the standard odor liquid for each sense of smell test.

Since the discarded test papers would leave some odor in the examination room, they should be thrown away in a waste basket with a lid, or placed in a plastic bag and closed with a rubber band before discarding into a waste basket.

(3) Answering the Test

Answers should be written on the answer sheet provided by the operator.

(4) Letting the Examinee Check his Own Odor Sensitivity

The examination should be carried out using the "5-2 method" for each standard odor liquid.

7. Preliminary Practice to Test for Selection of Panel Members

The operator should give each examinee a preliminary practice. This is to allow the examinee to get used to the test, and to free him of anxiety. The practice should be given once or twice using a density one step higher than that used for the standard density for selection.

8. Supplementary Test for the Selection of Panel Members

If an incorrect answer is given to only one of the standard density odor liquids, the examinee may be given a supplementary test depending on the wishes of both parties.

9. Retesting

It is generally considered that the sense of smell for people who have passed the test for the selection of panel members, remains stable for a period of 5 years. Therefore panel members should be retested every 5 years. It is wiser to retest panel members over 40 years of age, since their sense of smell ability tends to deteriorate.

However, the above does not apply to people who were found to have an abnormality in their sense of smell due to disease, traffic accidents, and so on.

10. Validity Period of Standard Odors for Measuring Sense of Smell

The standard odors remain valid for one year after opening, or two years from the date of manufacture for unopened bottles. Do not use standard odors which validity period was over.

11. Storage

Please note that the standard odors for measuring sense of smell should be stored in a cool, dark place.