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Development of Odour Intensity Reference Solutions for Environmental Odour Evaluation

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Abstract

For the appropriate evaluation of environmental odours, it is necessary to develop a reliable odour measurement scale. Odour intensity is one of the main odour characterisation parameters and a remarkably common and important sensory indicator of environmental odours. In this study, the odour intensity level between two and four of the six-point odour intensity scale was focused on, and odour intensity reference solutions of representative odorants for environmental odour evaluation were developed. As a result, propionic acid, propylamine, ethyl acetate, and isobutyraldehyde were selected as representative odorants, and three concentration steps of each odorant were determined to cover the odour intensity of two, three, and four of the six-point odour intensity scale. These reference odour solutions will be applicable to the training of inexperienced panel members and reliable on-site investigations of environmental odours.

Keywords: odour intensity; reference odour solution; propionic acid; propylamine; ethyl acetate; isobutyraldehyde; environmental odour evaluation

1. Introduction

Odours discharged from various human activities may cause severe damage to residents. Odour measurement and evaluation are fundamental elements for the solution of odour issues. Since environmental odours are perceived by the human sense of smell, sensory odour measurement and evaluation as well as instrumental analysis of individual odorants are indispensable. For appropriate evaluation of environmental odours by using the human sense of smell, it is necessary to develop a reliable sensory odour measurement scale.

Odour intensity is one of the main odour characterisation parameters [1] and a remarkably common and important sensory indicator of environmental odours. Odour intensity directly reflects people's perception of environmental odours and contributes to effective odour management.

Several odour intensity scales have been developed and used for decades in the world [1–3]. According to VDI 3882 Part 1 [4] developed in Germany in 1992, odour intensity measurements are carried out with dynamically diluting olfactometers. The category scale of odour intensity is primarily an ordinal number scale, and a specified ranking is assigned to its categories. ASTM E544-24 [5] was developed in the U.S.A. in 2024. In this standard, both dynamic and static scales are designed to compare the odour intensity of the sample with the odour intensities of a series of 1-butanol concentrations. Higuchi et al. [6] developed a new series of six dilution steps of 1-butanol aqueous solutions

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for environmental odour evaluation. The scale consists of a concentration series of 0, 10, 600, 2600, 9000, and 22,500 ppm (vol/vol), and each dilution step has an explanatory label for odour intensity level to ensure equal intervals between odour intensity levels.

In Japan, the six-point odour intensity scale shown in Table 1 was developed more than 50 years ago. This scale is very important in Japan because the regulation standards based on the Offensive Odour Control Law [7] were set equivalent to the odour intensity that ranges from 2.5 to 3.5 on this scale [8]. In the measurement, six or more panel members sniff a testing odour directly and classify their impressions in accordance with the scale in 0.5 segments. After discarding the maximum and minimum values, the remaining values are averaged [9]. This scale is very easy-to-use, understandable to residents, and applicable to any field at any time without any equipment. On the other hand, the independent judgments of the panel members are subjective, and equal intervals between intensity levels are not necessarily ensured. Therefore, how to maintain the reliability of odour intensity measurement is a key factor for appropriate odour evaluation.

Table 1. Six-point odour intensity scale.

Intensity	Description
0	No odour
1	Barely perceivable (Detection threshold)
2	Faint but identifiable (Recognition threshold)
3	Easily perceivable
4	Strong
5	Extremely strong

Considering principles, ideas, and applications of several odour intensity scales, the disadvantages of the conventional Japanese six-point odour intensity scale need to be improved. In this study, the odour intensity level between two and four, which covers the range of odour regulation standards in Japan, was focused on, and odour intensity reference solutions of representative odorants for environmental odour evaluation were developed.

2. Materials and Methods

2.1. Selection of Representative Odorants Used for Reference Solutions

Odorants used for odour intensity reference solutions representing typical odour emission sources were selected.

2.1.1. Extraction of Major Odour Emission Sources

Using odorants emitted from major odour emission sources, which cause a number of odour complaints, is important to ensure effective odour evaluation. Major odour emission sources were narrowed down based on the statistical data on odour complaints from FY2019 to FY2021 compiled by the Japanese Ministry of the Environment. Annual survey reports on the enforcement status of the Offensive Odour Control Law from FY2019 to FY2021 [10–12] were used as the odour complaints data source. In these reports, the number of annual odour complaints registered in Japan was aggregated by 13 categories and 102 subcategories of odour emission sources. Based on the odour complaints data from FY2019 to FY2021, major odour emission sources with 100 or more annual odour complaints on average were extracted.

2.1.2. Investigation of Odorants Emitted from Major Odour Emission Sources

Odorants emitted from major odour emission sources were investigated comprehensively by the literature review, including academic articles and survey reports by the local authorities [13–21]. The literature reporting odorants and their concentrations at odour emission sources or fencelines was compiled. After scrutinising a list of them, odorants that fulfilled the following requirements remained as candidates:

- Odour activity value (OAV) is 10 or more;
- No carcinogenicity is reported;
- Not solid nor gas at room temperature;
- Chemically stable;
- Water-soluble;
- Volatility is moderate enough to keep a constant odour intensity.
- Odour intensity of the undiluted solution is not extremely strong.

OAV of 10 or more was adopted as one of the requirements because the lowest value of the odour index regulation standard at the fenceline is 10 in Japan. If the odour sample consists of a single odorant, an OAV of 10 is equivalent to an odour index of 10. Therefore, odorants with an OAV of 10 or more need to be considered as a potential cause of odour complaints.

Odour wheels have been developed as a useful method of combining olfactory and chemical data at various odour emission sources [22]. In this study, however, the aforementioned measurement data obtained in Japan [13–21] were prioritised to ensure appropriate selection of representative odorants for local odour emission sources.

2.1.3. Determination of Representative Odorants Used for Reference Solutions

Selecting odorants that have similar perceptual characteristics or that are emitted from similar sources should be avoided to prevent unnecessary overlap of odour evaluation. Therefore, the appropriateness of candidate odorants was investigated by the semantic differential (SD) method and cluster analysis.

First, the odour impression of aqueous solutions of five candidates for representative odorants (propionic acid, propylamine, ethyl acetate, isobutyraldehyde, and isobutyl alcohol), which fulfilled the requirements in Section 2.1.2, was experimentally evaluated by the SD method. Wide-mouth Erlenmeyer flasks with a capacity of 500 mL were prepared, and aqueous solutions of candidate odorants that correspond to an odour intensity of around three on the six-point odour intensity scale were placed into them. The volume of the solution was 200 mL, and the amount of odorants pipetted into the flasks was determined through trial and error by experimenters with sufficient odour measurement experience. The mouth of each flask was covered with aluminium foil tightly. The panel members gently shook a flask prior to each sniffing to ensure equilibrium, and odour impression was evaluated by the seven-point SD scale with 10 adjective pairs shown in Figure 1. Odour intensity of each aqueous solution was also evaluated by the six-point odour intensity scale to confirm that each solution corresponded to an odour intensity of around three, as intended. A total of 40 panel members who ranged from 19 to 24 years of age and passed the Japanese panel screening test [23] joined the experiment.

Then, cluster analysis was applied to the results of odour impression evaluation to narrow down the candidate odorants. In the analysis, the mean values of evaluation results on the seven-point SD scale were used. Euclidean distance of candidate odorants was calculated based on Ward’s method. A statistical programme, HAD [24], was executed to conduct the analysis. Finally, representative odorants used for reference solutions were determined, taking the similarity of perceptual characteristics and emission sources into consideration.

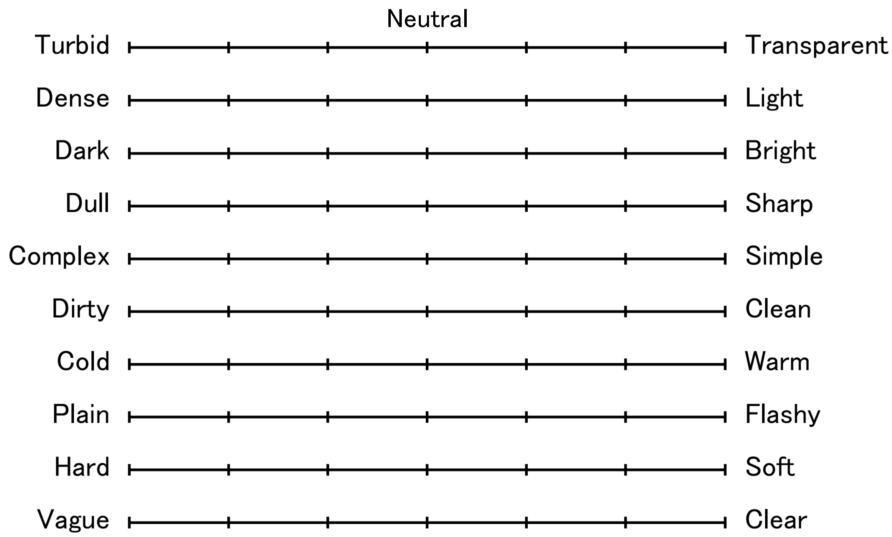

Figure 1. Seven-point SD scale with adjective pairs.

2.2. Determination of Concentrations of Reference Solutions

Olfactory measurement data for representative odorants selected in Section 2.1. were obtained to determine the concentrations of aqueous solutions that correspond to odour intensities of two, three, and four on the six-point odour intensity scale. First, provisional concentrations of representative odorants that were likely to cover the odour intensity range from one to four were determined through trial and error by experimenters with sufficient odour measurement experience. Then, wide-mouth polypropylene (PP) bottles with a capacity of 500 mL were prepared, and aqueous solutions of four representative odorants (propionic acid, propylamine, ethyl acetate, and isobutyraldehyde) were placed into them. The volume of the solution was 200 mL, and the mouth of each PP bottle was capped tightly. The panel members gently shook a PP bottle prior to each sniffing to ensure equilibrium, and odour intensity was evaluated by the six-point odour intensity scale. A total of 42 panel members who ranged from 19 to 24 years of age and passed the Japanese panel screening test [23] joined the experiment. Then, regression analysis was applied to the results to determine the concentrations of reference solutions that correspond to odour intensities of two, three, and four on the six-point odour intensity scale.

3. Results and Discussion

3.1. Selection of Representative Odorants Used for Reference Solutions

3.1.1. Extraction of Major Odour Emission Sources

Among 13 categories of odour emission sources, five categories (open burning, construction sites, mobile sources, garbage collection points, and unknown) were excluded because open burning, construction sites, and mobile sources are temporary sources, and garbage collection points caused many fewer odour complaints than the others. Major odour emission sources were narrowed down by extracting categories or subcategories with 100 or more annual odour complaints on average. Table 2 shows 15 major odour emission sources extracted based on the statistical data on odour complaints in Japan.

Table 2. Major odour emission sources extracted based on the statistical data on odour complaints in Japan.

Odour Emission Source	Number of Odour Complaints			
	FY2019	FY2020	FY2021	Average
1. Livestock yards	961	1181	1164	1102
Pig farming	149	212	250	204
Cattle farming	113	154	166	144
Poultry farming	121	134	144	133
Farm	164	255	282	234
Compost	188	303	271	254
2. Feed and fertiliser factories	259	211	190	220
3. Food factories	630	608	538	592
Seafood products factories	85	108	116	103
Oil/fat-based food products factories	145	87	75	102
Prepared foods factories	94	128	116	113
4. Chemical plants	147	169	144	153
5. Miscellaneous factories	875	930	901	902
6. Services and other businesses	1842	2025	1909	1925
Restaurants	669	736	770	725
Automobile repair shops	108	110	115	111
7. Drainage	402	503	491	465
8. Houses and apartment houses	1474	1936	1731	1714

3.1.2. Investigation of Odorants Emitted from Major Odour Emission Sources

Although the number of annual odour complaints was over 700 on average, as shown in Table 2, restaurants were excluded from further consideration because there is a wide variety of types, and odour measurement data reported in the literature were limited. Odorants emitted from major odour emission sources were confirmed to be 63 by a literature review, as shown in Table 3. Among them, five odorants (propionic acid, propylamine, ethyl acetate, isobutyraldehyde, and isobutyl alcohol), which fulfilled the requirements in Section 2.1.2, remained as candidates.

Table 3. Odorants emitted from major odour emission sources.

Acetaldehyde	Geosmin	Pentylamine
Acetic acid	Hexylamine	Phenol
Acetoin	Hydrogen chloride	Propionaldehyde
Acrolein	Hydrogen cyanide	Propionic acid
Ammonia	Hydrogen sulfide	Propylamine
1-Butanol	Indole	Propylbenzene
Butyl acetate	Isobutyl alcohol	Styrene
Butyl cellosolve	Isobutylamine	Sulfur dioxide
Butyraldehyde	Isobutyraldehyde	Toluene
Butyric acid	Isobutyric acid	2,4,6-Trichloroanisole
m-Cresol	Isopropanol	Trimethylamine
p-Cresol	Isovaleraldehyde	1,2,3-Trimethylbenzene
Diethylamine	Isovaleric acid	1,2,4-Trimethylbenzene
Dimethyl disulfide	Methanol	1,3,5-Trimethylbenzene
Dimethyl sulfide	Methyl ethyl ketone	Undecanal
Ethyl acetate	3-Methylindole	Valeraldehyde
Ethylbenzene	2-Methylisoborneol	Valeric acid
o-Ethyltoluene	Methyl isobutyl ketone	Vanillin
m-Ethyltoluene	Methyl mercaptan	*o*-Xylene
p-Ethyltoluene	Methylamine	*m*-Xylene
Formaldehyde	Nitrogen dioxide	*p*-Xylene

3.1.3. Determination of Representative Odorants Used for Reference Solutions

Table 4 shows concentrations of aqueous solutions of candidate odorants used for odour impression evaluation by the SD method. The mean values and standard deviations of odour intensity are also represented in Table 4. After evaluating the odour impression of aqueous solutions of five odorants by the SD method and applying cluster analysis to the results, the dendrogram shown in Figure 2 was obtained. Odorants connected by shorter lines in Figure 2 represent more similar perceptual characteristics. Although propylamine and isobutyraldehyde were connected by the shortest lines, both odorants remained because their emission sources were different. Isobutyraldehyde and isobutyl alcohol were connected by the second-shortest lines, and they were emitted from the same category of odour emission sources, i.e., miscellaneous factories, services, and other businesses. Considering the similarity of odour impression and odour emission sources between isobutyraldehyde and isobutyl alcohol, isobutyl alcohol was excluded because a larger standard deviation of odour intensity was observed, as shown in Table 4. As a result, propionic acid, propylamine, ethyl acetate, and isobutyraldehyde were selected as representative odorants used for odour intensity reference solutions. Table 5 shows representative odorants and examples of corresponding odour emission sources.

Table 4. Concentrations of aqueous solutions, mean values, and standard deviations of odour intensity of candidate odorants used for odour impression evaluation by the SD method.

Candidate Odorant	Concentration of Aqueous Solution (ppm (vol/vol))	Odour Intensity	
		Mean Value	Standard Deviation
Propionic acid	400	4.0	0.99
Propylamine	400	2.9	0.88
Ethyl acetate	550	4.0	0.79
Isobutyraldehyde	25	3.2	0.70
Isobutyl alcohol	1000	3.1	1.00

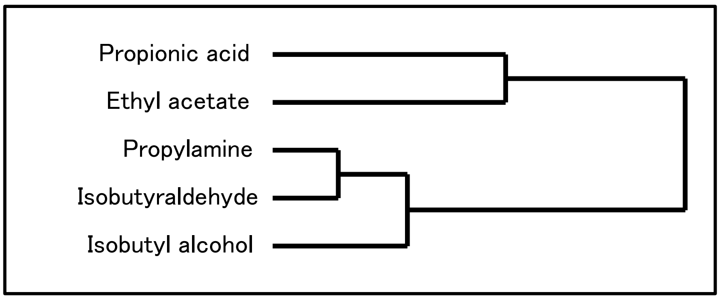

Figure 2. Dendrogram obtained by cluster analysis of odour impression evaluation data.

Table 5. Representative odorants and examples of corresponding odour emission sources (∨).

Odour Emission Source	Representative Odorant			
	Propionic Acid	Propylamine	Ethyl Acetate	Isobutyraldehyde
Livestock yards	∨			
Feed and fertiliser factories		∨		
Food factories	∨			
Chemical plants			∨	
Miscellaneous factories			∨	∨
Services and other businesses			∨	∨

3.2. Determination of Concentrations of Reference Solutions

Table 6 shows provisional concentrations of four representative odorants determined through trial and error by experimenters with sufficient odour measurement experience. Prior to the data analysis, panel members' screening was conducted to ensure the reliability of measurement results. For each representative odorant, panel members who gave any of the following answers were excluded:

- Odour intensity for the lowest concentration was equal to or stronger than that for the highest concentration;
- Odour intensity for the lowest concentration was four or stronger;
- Odour intensity for the second-lowest concentration was five;
- Odour intensity for the second-highest concentration was one or weaker;
- Odour intensity for the highest concentration was 1.5 or weaker;
- Odour intensity of zero was given besides the lowest concentration.

Table 6. Provisional concentrations of representative odorants.

Representative Odorant	Concentration of Aqueous Solution (ppm (vol/vol))
Propionic acid	170, 550, 1900, 6200
Propylamine	200, 350, 650, 1100
Ethyl acetate	90, 180, 360, 700
Isobutyraldehyde	5, 40, 200, 1000

The number of panel members after the screening was 33 for propionic acid, ethyl acetate, and isobutyraldehyde, and 34 for propylamine.

The mean values and standard deviations of odour intensity for aqueous solutions of representative odorants are depicted in Figure 3. Table 7 shows regression equations, coefficients of determination, and probability values (p-values) for representative odorants. All representative odorants have strong linear relationships between the logarithm of concentration and perceived odour intensity. The mean odour intensity values for four aqueous solutions of propionic acid, propylamine, ethyl acetate, and isobutyraldehyde, shown in Table 6, ranged from 1.5 to 4.0, 1.9 to 3.9, 2.1 to 3.9, and 1.9 to 3.8, respectively. These results imply that the provisional concentrations of representative odorants shown in Table 6 were appropriate for the experimental purpose. The standard deviations for four aqueous solutions of propionic acid, propylamine, ethyl acetate, and isobutyraldehyde, shown in Table 6, ranged from 0.74 to 0.99, 0.81 to 1.05, 0.58 to 0.88, and 0.75 to 0.98, respectively. The regression lines are also drawn in Figure 3. As shown in Table 7, the slope of the regression line for isobutyraldehyde (Figure 3d) was the smallest, and that for propylamine (Figure 3b) was the largest. These results represent the difference in slope of the Weber–Fechner Law. Using representative odorants with different slopes of the regression line is valuable to understand the psychophysical characteristics of a wide variety of odours.

Table 7. Regression equations, coefficients of determination, and p-values for representative odorants.

Representative Odorant	Regression Equation	Coefficient of Determination	p-Value
Propionic acid	$y = 1.58 \log x - 1.93$	0.994	0.0029
Propylamine	$y = 2.71 \log x - 4.39$	0.989	0.0054
Ethyl acetate	$y = 1.88 \log x - 1.55$	0.986	0.0069
Isobutyraldehyde	$y = 0.818 \log x + 1.26$	0.962	0.0193

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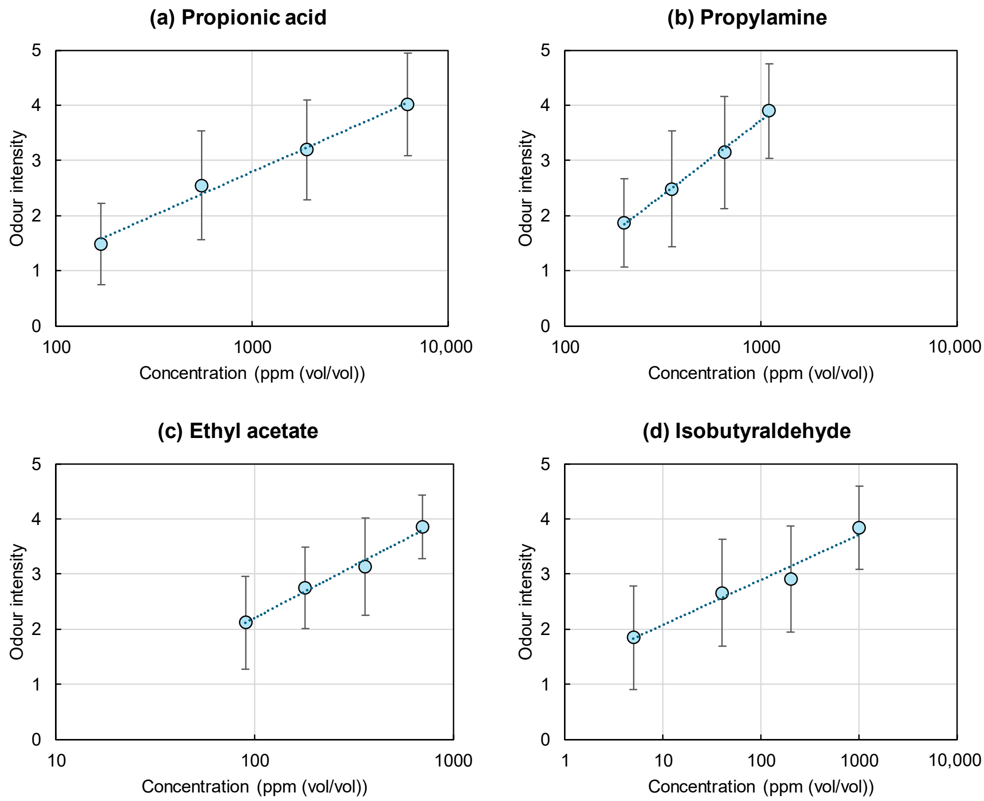

Figure 3. Mean values and standard deviations of odour intensity for representative odorants.

Based on the regression lines drawn in Figure 3, the concentrations of aqueous solutions that correspond to odour intensities of two, three, and four were calculated. These values were reconsidered and rounded from the practical point of view. As a result, the concentrations of reference solutions for odour intensity evaluation were determined as shown in Table 8. Three concentration steps of each representative odorant were determined to cover the odour intensity of two, three, and four of the six-point odour intensity scale. The concentrations (ppm (vol/vol)) of reference solutions using propionic acid, propylamine, ethyl acetate, and isobutyraldehyde range from 310 to 5820, 230 to 1260, 80 to 900, and 8.0 to 2240, respectively. These reference odour solutions will be applicable to the training of inexperienced panel members and reliable on-site investigations of environmental odours.

Table 8. Concentrations of reference solutions for odour intensity evaluation.

Representative Odorant	Concentration of Aqueous Solution (ppm (vol/vol))		
	Odour Intensity 2	Odour Intensity 3	Odour Intensity 4
Propionic acid	310	1350	5820
Propylamine	230	540	1260
Ethyl acetate	80	260	900
Isobutyraldehyde	8.0	130	2240

4. Conclusions

In this study, odour intensity reference solutions using four representative odorants (propionic acid, propylamine, ethyl acetate, and isobutyraldehyde) for environmental odour evaluation were developed. Three concentration steps of each representative odorant were determined to cover the odour intensity of two, three, and four of the six-point odour intensity scale.

These reference solutions can be used appropriately depending on the odour emission source concerned because they correspond to some of the major odour emission sources. To ensure the reliability of odour intensity evaluation using reference solutions, a comparative study on the effectiveness of odour intensity measurement with and without reference solutions developed in this study would be necessary in the future.

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