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Comparing the Accuracy of Three Odour Analysis Techniques Used in Europe, North America, Australia, New Zealand and Asia

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There are currently several different methods for dynamic olfactometry analysis that are universally used. In Europe, Australia, New Zealand and North America, the Triangular Forced Choice Method, the Binary Forced Choice Method, and the Yes/No Choice Method are commonly used for dynamic olfactometry analysis. They are supported by the European Standard: EN13725: 2003, the USA ASTM E679-04: 2011 standard, and the Australian/ New Zealand AS/NZS4323: 2001 Standard. All of these methods use a decreasing dilution series designed to determine an odour detection threshold value. In China, Japan, and much of South East Asia, an increasing dilution series is used for odour evaluations that are described in the "Odor Index Regulation and Triangular Odor Bag Method" and the document: GB/T14675-93 guideline. The Triangular Forced Choice Method is believed to be statistically the most accurate method, however the downside of this method is the length of time it takes to perform analysis. The long analyses time may incur possible fatigue from panelists who are evaluating samples. Over time, this may result in less accurate results. Also, with this method, there are usually less samples analyzed per odour panel session or day. The objective of this paper is to conduct a comprehensive quantitative comparison of the three most commonly used methods for dynamic olfactometry odour analysis. The results obtained from this study are based on analysis using the same type of olfactometer and the same group of panelists, in order to minimize any deviation caused by other factors than the test methods. This study compared a decreasing dilution series to the increasing dilution series methods. A modified Japanese method was used for this study where standard Triangular Forced Choice Method was conducted in the increasing dilution series.

1. Introduction

1.1 Dynamic Olfactometry

Typical odour analyses are performed by introducing an odour sample to screened panel members using a dynamic olfactometry. There are several commonly used types of olfactometer: anywhere from one station, to four stations, to six stations to eight stations, where one person at one time, performs evaluations or four, six or eight people perform the analysis at the same time, respectively.

1.2 The Different Methods for Odour Analysis

There are also different methods for odour analysis. These include: the Triangular Forced Choice Method, the Binary Forced Choice Method, and the Yes/No Choice Method. The European EN13725:2003[1], Australian/New Zealand AS/NZS4323 [2] or ASTM 679-04[3] standards recognize these methods.

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For the Forced Choice Method the panellist is presented with two or three ports, of which one presents diluted sample and the other(s) neutral gas. The sample is presented randomly over the two or three ports. The panellist is asked to indicate the port with the sample. The panellist is also asked if his/her choice was a guess, inkling or certain. From the combination of the choice result and the indicated level of certainty the response is classified as false or true. The odour evaluation procedure requires the first sample to be presented to the panellist at the dilution that will be below the detection threshold. Therefore, the first several presentations to the panellists require the panellist to select one port by guessing. Each panellist observes an odour sample in the ascending concentration series (increasing concentration).

In the Yes/No Mode the panellist is asked to evaluate the sample presented from the specific port and to indicate if an odour is perceived (Yes/No). A second port with blank is always provided as a reference.

In Japan, China and some South East Asian countries, a very simple method is still used for odour evaluations – the Direct Triangular Bag Method, where samples are introduced to the panel members directly, by sniffing the contents of the bag. Each sample bag is analyzed at different levels, starting at a low dilution level. The method is described in the “Odor Index Regulation and Triangular Odor Bag Method” [4] and document GB/T14675-93 [5]. A sensory test is usually conducted by at least six members of the panel. Each panel member is given three bags with one being a sample and two blanks of clean air. Panellists are asked to sniff the bag directly. If the panel can tell the correct bag, then the odour is diluted and the test is continued until it becomes impossible to identify the bag with odour. This method uses increasing dilution (producing a weaker bag) and cumulates at the point where the panellists are not able to detect the odour in the bag. However, if panellists are exposed to high odours in the beginning of evaluations, there is possibility of “overexpose of the human nose” which may result in not only fatigue, but also in error within the results.

1.3 Decreasing Versus Increasing Dilution Series

European, Australian and New Zealand standards use decreasing dilution between analysis, and a factor of two is usually used between dilutions. The odour detection threshold value (ODTV) for an evaluated sample is based on fifty per cent of panellists detecting odour. Most countries follow these two mentioned standards. However, in Japan, China and some of Eastern Asia, the Triangular Odour Bag Method with increased dilution is commonly used. This method first calculates the odour detection threshold of each panellist, then the average threshold of the panel. Later on, the odour index is established for the sample. For samples with low odour, the odour index is calculated based on the averaging of scores of panel members. China and other Asian countries adapted the Japanese method as a simple method for odour evaluations. However, this method is time consuming due to the fact that the samples need to be prepared and diluted in the laboratory during actual evaluations.

Some countries in Asia would like to shorten their evaluation process by using dynamic olfactometry. On the other hand, with this approach, even with an available olfactometry, they would like to follow an evaluation with decreasing odour concentration (increasing dilution factor). Therefore, this study was necessary to demonstrate any similarities and differences between evaluations with increased dilution versus decreased dilution for the same type of analysis – the Triangular Forced Choice Method. This study also covers aspects of evaluation using the Binary Forced Choice Method versus the Triangular Forced Choice Method for the same decreasing dilutions.

2. Case Study 1: Determination of Odour Detection Threshold Values for Selected Pure Compounds using Three Varied Methods

2.1 Methodology

For this study six compounds were chosen for odour evaluations. For each compound, one gaseous sample was prepared in an odour test facility by injecting a known amount of pure compound into the known amount of nitrogen. The following pure compounds were used for this study: ammonia, hexane, n-butanol, acetone, ethanol, butyric acid. Each gaseous sample was analysed three times using a dynamic olfactometry with the same panellists and the same type of olfactometer.

The following options were chosen for analysis: the Triangular Forced Choice Method, the Binary Forced Choice Method, both with decreasing dilution series and a third option: the Triangular Forced Choice Method with increasing dilution series.

For Binary and Triangular Methods with decreasing dilutions, the odour detection threshold value (the point where 50% of panellists detect odour) was calculated for each evaluated sample and geometric mean from three evaluations was used for the final ODTV calculations. For the Triangular Method with increasing dilutions, the ODTV was also calculated based on panellist's responses.

2.2 Table Results

Table 1 summarizes the results for three types of analysis representing ODTV, when the Binary Method and the Triangular Method were used with decreasing dilutions as well as the Triangular Method with increasing dilutions.

Table 1: ODTV for selected compounds based on Binary, Triangular Methods with decreased dilution compared to the Triangular Method with increased dilution

Compound	**ODTV based on Binary Method with decreasing dilution * OU**	**ODTV based on Triangular Method with decreasing dilution* OU**	**ODTV based on Triangular Method with increasing dilution** OU**
Ammonia	489	407	272
Hexane	493	433	120
N butanol	1310	914	766
Acetone	1700	1610	855
Ethanol	1200	980	410
Butyric Acid	120	98	49

*Based on Geometric Mean of ODTV from three evaluations

** Based on ODTV from one evaluation

2.3. Case Study 1 Conclusions

Based on the results from this case study, it was determined that there is no significant difference in the ODTV if the Binary or Triangular Methods with decreasing dilution were used for odour evaluations with slightly lower values for Triangular Method. It may be attributed to the panellists fatigue due to the longer analysis time (three ports versus two ports). However, if these two methods were compared with the Triangular Method with increasing dilution there is a significant difference in results. In all cases, the ODTV obtained by the Triangular Method with increasing dilution were significantly lower, resulting in lower panellist's sensitivity for each evaluated compound.

3. Case Study 2: Determination of Odour Detection Threshold Values for Selected Samples using Three Methods: Binary and Triangular Method with Decreasing Dilution and the Triangular Method with Increasing Dilution

3.1 Methodology

This study was based on analysis of three sets of samples during nine different sampling Episodes. Each sampling Episode was designed for different types of sources ranging from very odorous sources to sources where the odour was weak.

For each Episode, three samples were collected and analysed three times: using the Binary Method with decreasing dilution, the Triangular Method with decreasing dilution, and the Triangular Method with increasing dilution series. For the first three Episodes, all collected samples were high in odour. They also had a high content of hydrogen sulphide, therefore for Episode 1, 2 and 3, samples were diluted on site with nitrogen 73, 73 and 55 times, respectively.

During three other Episodes, samples were diluted only 12 times where for Episode 7, Episode 8 and 9, samples were very weak and they were not diluted. For Episode 7, 8 and 9, samples were collected from ambient locations.

3.2 Table Results

Table 2 shows the results for Raw ODTV for samples collected during nine Episodes. The Raw ODTV represents an odour detection threshold value obtained from analyses of already diluted on site samples. However, the Net ODTV can be calculated by multiplying dilution used on site during collection of the samples (73, 73, 65, 12, 12, 12 and 1, 1 and 1 for Episodes 1, 2,3,4,5, 6,7,8,9 respectively) and corresponding Raw ODTV for the sample. For the purpose of this study there was no need to calculate Net ODTV for each sample; therefore the results are presented as Raw ODTV.

Table 2: Raw ODTV for Samples Collected During Nine Episodes Based on Evaluation of Samples using three Different Methods

Episode	**Raw ODTV based on Binary Method with decreasing dilution OU**	**Raw ODTV based on Triangular Method with decreasing dilution OU**	**Raw ODTV based on Triangular Method with Increasing dilution OU**
Episode 1	2200	2120	1200
Episode 2	1650	1570	870
Episode 3	1450	1380	790
Episode 4	120	110	54
Episode 5	167	154	83
Episode 6	194	175	89
Episode 7	22	18	15
Episode 8	36	29	24
Episode 9	25	19	14

3.3 Case Study 2 Conclusions

This study showed a good correlation between the two most commonly used methods in Europe, Australia, Canada and the USA: the Binary and Triangular Method with decreased dilutions between steps with slightly lower values for Triangular Method. However, for samples with high odour concentrations, the ODTV obtained by the Triangular Method with increasing dilution steps were significantly lower than obtained by analysis using two methods with decreasing dilution steps. It may be attributed that during analysis with increasing dilution, the system may be contaminated at the beginning of analysis, and the fact that the sample with high concentration is introduced first. Furthermore overexposing panellists to thee high odours in the beginning of evaluations may result their possible fatigue.

For samples with low odour, the results are similar for all three methods.

4. Conclusions

Based on the results from both studies, it can be determined that the Binary Method and the Triangular Methods (both with decreasing dilutions) appeared to have similar ODTV results. However, when the Triangular Method with increasing dilution between steps is compared with the Binary and Triangular Methods with decreasing dilution between steps, the results showed significant differences between these methods for samples with high odours. However, for weak samples, in the range between 20 OU to 30 OU, the ODTV are similar for all three methods. The differences between results for samples with strong odour may be attributed to the fact that when samples are analysed using the Triangular Method with increasing dilution it may contaminate the olfactometer in the beginning of evaluations resulting in false responses. Overexposing the panellists in the beginning of the evaluation and their fatigue may also play a significant role.

There is no significant difference between the Binary Method and the Triangular Method with decreasing dilutions between steps. Usually the analysis with Triangular Method require longer analysis time due to the fact that additional third port needs to be sniffed all the time, therefore the panellists may be fatigued faster than during the binary analysis.

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