European versus United States Odour / Odor Standards of Evaluation

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Presented as a Poster Paper at the
Water Environment Federation 71st Annual Conference.
Orlando, FL: 3-7 October 1998.
[Updated: 1 November 1999]

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Abstract

With the global increase of environmental regulations in the 1970's, European countries, Australia, and the United States began to develop odor regulations. These regulations created the need to standardize the methods of odor measurement. Some examples of these standards include: US - ASTM D-1391 (1978) and ASTM E679-91(1991), Germany - VDI 3881 (1980), France - AFNOR - X-43-101 (1986), Netherlands - NVN2820 (1996).

More recently, in 1990 the European Committee for Standardization (CEN) formed a technical committee (TC264) which is expected to release a final standard in 1999, entitled "Air quality-Determination of odour concentration by dynamic olfactometry", which will unify the olfactometry standards of 18 countries (Austria, Belgium, Denmark, Finland, France, Greece, Germany, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom). This standard follows ISO protocols. Australia is expected to base their practices on this standard.

DRAFT prEN 13725 "Air quality – Determination of odour concentration by dynamic olfactometry" is released for Public Comment (three official languages: English, French, German). Latest date for receipt of comments is the end of January 2000. In accordance with the CEN/CENELEC Internal regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom; note Australia will adopt 1 November 1999.

Meanwhile, since 1991, the EE-6 "Odor" committee of the Air & Waste Management Association has been developing a guideline document entitled "Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry". The final product may be submitted to ASTM to revise or supplement ASTM E679-91.

This paper presents the similarities and differences between the USA and European standards being developed. Figure 1 summarizes the elements of the most often sited national standards along with the prEN 13725 draft standard and the EE-6 draft guidelines. The question that will remain is: How will these differences be reconciled in order to achieve global standardization of odor measurement?

Presentation Statistics

The prEN 13725 draft standard and the EE-6 committee draft guidelines agree that test odors should be presented to human assessors (panelists) utilizing dynamic dilution olfactometry following a "forced-choice" ascending concentration series method. The assessors are presented with a diluted odor sample and one or two blank samples of odor free air. The assessor must choose which sample contains the odor, even if they must guess. This approach is called the "forced-choice" presentation method. After the assessor makes a selection, they are presented with the next odor sample and blank(s). However, this next odor sample is at a higher concentration (e.g. two times higher). The assessor continues to additional higher levels of sample presentation following these methods. This statistical approach of increasing levels of sample presentation is called "ascending concentration series."

There are two minor differences between the prEN 13725 and US methods regarding presentation statistics. The first difference is that the EE-6 committee stands behind the original ASTM method, which requires two blank presentations per diluted odor presentation. This approach is called a triangular presentation. The prEN 13725 standard agrees with the triangular approach, but also allows only one blank per dilute odor presentation (binary presentation). The second difference is in the dilution increase between dilution presentation levels. While the EE-6 committee recommends a constant increase of a factor of two, the prEN 13725 standard is slightly more lenient, specifying a step factor between 1.4 and 2.4.

The European Standard also allows the Yes/No Presentation Method and the Forced-Choice Probability Method. The Yes/No method is similar to audiometry protocol where dilute odors and blanks are randomly presented and the assessor identifies when they detect the odor. The Forced-Choice Method is a complex Forced-Choice Probability Method derived from the French standard AFNOR NF 43-101.

Presentation Method

Olfactometer Design. All standards developed to date have specified "odor free" dilution air. Further, the most recent drafts released by the EE-6 committee and the prEN13725 have specified the olfactometer must be constructed of components made of either glass, stainless steel, or polytetrafluoroethylene (PTFE). These standards also come relatively close to agreeing on an acceptable range of dilutions. The prEN 13725 has specified a minimum upper limit of 2^{14} and a maximum lower limit of 2^{7}. The EE-6 committee recommends a minimum upper limit of 10,000 ($\sim 2^{13}$) and a maximum lower limit of 10 ($\sim 2^{3}$).

Presentation Flow Rate. There continues to be a strong debate surrounding the appropriate presentation flow rate. An early olfactometer developed in the US in the 1970's was designed to simulate the static "syringe method" (ASTM D-1391) of dilution olfactometry with a dynamic method. This olfactometer operated at 0.5-lpm. In the Netherlands, a simulation study used tracer gas and an anatomical model of the nose to study the effects of presentation flow rate on the determined threshold. This study showed that at flow rates of 20-lpm and higher; there is only a minor decrease in the threshold value compared to higher flows.[1]

In the mid 1980's, Dr. David C. Laing of the CSIRO Division of Food Research, N.S.W., Australia, studied the variables of sniffing and concluded that the optimum operating flow rate for an olfactometer is between 30-lpm to 40-lpm.[2]

There have been two published studies since Dr. Laing's work. Konosuke Nishida, Yasuo Yanagibashi and Marahiro Osako of the Laboratory for Control of Environmental Micro Pollutants, Kyoto University, Otsu, Japan studied flow rates of 0.5-lpm to 5.0-lpm and concluded that "the optimum flow rate of test odor by dynamic sniffing test is 4-lpm."[3]

In 1995, Martha O'Brien, Richard Duffee, and Ned Ostojic of Odor Science & Engineering (OSE), Inc., Bloomfield, Connecticut, published their research which studied the flow rate phenomena over the flow range of 1-lpm to 20-lpm and concluded "that a flow rate between 5-lpm and 10-lpm…will produce repeatable measurements most characteristic of odor perception in the ambient air."[4] The OSE study utilized two mask sizes and three test odorants (hydrogen sulfide, p-xylene, and n-butanol).

Research conducted by other olfactometry laboratories has yielded similar as well as different results from Kyoto University and OSE. Researchers in the European community and Australia have developed olfactometers with flow rates as high as 50-lpm.

The prEN 13725 has specified in their draft standard that the olfactometer must operate at a presentation flow rate of 20-lpm or higher. The draft guideline of the EE-6 committee currently specifies an odor presentation flow rate of 8-lpm. Additional flow rate studies are in progress in the US that may help resolve the flow rate issue.[5]

Presentation Face Velocity. The presentation face velocity also continues to be a discrepancy between drafts. The EE-6 committee recommends a face velocity of between 0.02 - 0.05 m/s (6-10cm mask @ 8-lpm). The prEN 13725 has specified a face velocity of no less than 0.2 m/s and recommends that it be set less than 0.5 m/s (3-5cm mask @ 20-lpm). See Figure 2 for a comparison of these parameters.

Performance Criteria

Beyond the operating parameters, prEN 13725 and EE-6 discuss performance criteria and instrument calibration. The EE-6 guidelines only outline these topics generally, leaving specifics to the laboratory. The prEN 13725 standard goes one step further by creating the groundwork for a laboratory quality assurance plan. This standard specifies laboratory accuracy and repeatability performance criteria (including instructions on how to test the criteria) for the olfactometer following the international standard ISO5725 *Accuracy (Trueness and Precision) of Measurement Methods and Results Parts 1-4*. The prEN13725 standard also lists strict criteria for qualifying an assessor for olfactometry panels.

The prEN 13725 standard requires the olfactometer must be periodically calibrated at each dilution level with a suitable tracer gas. Each level must be accurate to within 20%. For instrument performance, instability is used to represent repeatability. During simulated operation of the instrument with a tracer gas as the odorant, multiple chemical measurements are taken to determine the instrument's stability around each dilution ratio.

The laboratory must periodically test its performance using a reference gas at a defined concentration. The prEN 13725 standard gives procedures and example calculations for testing and defining a laboratory's accuracy. For example, the standard contains specific criteria for confirming that a laboratory can accurately assess standard reference odorants. For repeatability, the standard requires that "the difference between two single measurements, performed on the same testing material [gas] in one laboratory…will not be larger than a factor of 3 in 95% of the cases." If the laboratory meets these criteria, it is assumed that the quality level is transferable to other, environmental odorants.

The EE-6 guidelines state that individuals "representing 'normal' sensitivity are selected to serve as panelists." The guidelines do not go any further to define "normal sensitivity." Since odor sensitivity in the general population is normally distributed, it is implied that a laboratory should aim for assessors who lie near the center of the bell curve (i.e. within 1 to 2 standard deviations of the mean).

The European standard takes a different approach altogether. This standard gives very strict criteria for assessor selection based on accuracy and repeatability to a standard, reference odorant. The standard states that "assessors with a specific sensitivity to the reference odorant n-butanol are selected to be panel members." Each potential assessor must be tested to n-butanol on the olfactometer a minimum of 10 times. The individual's average threshold measurement of n-butanol must be in the range of 20 - 80 ppb. Further, the antilog of the standard deviation must be less than 2.3. Once the assessor is accepted as a panelist they must be continually checked to this n-butanol reference with a rolling average of 20 measurements compared to the above criteria.

This standard is different from the EE-6 guidelines in two respects. First, the criteria is specifying an assessor who is accurate and repeatable to one specific odorant (n-butanol). It is assumed that each assessor's accuracy and repeatability will be the same for all odors measured in the lab (livestock, wastewater, compost, MSW landfill, etc.). Second, the prEN 13725 criteria does not necessarily select assessors who are "normal" in the population. It is not known if these assessors actually lie above, below, or on the average in the general population.

Summary

There are two major differences between the new European prEN 13725 Draft Standard and the A&WMA EE-6 Odor Committee Draft Guidelines:

1. Odor presentation parameters including volumetric flow rate and face velocity of the air stream; and

2. Assessor selection criteria for panel participation.

The prEN 13725 standard requires a minimum volumetric flow rate of 20-lpm and the EE-6 guidelines specify only 8-lpm. Furthermore, the documents require two different mask sizes, which are dependent on the different presentation face velocities and the different presentation volumetric flow rates. The lower flow rate (8-lpm vs. 20-lpm) of the EE-6 guidelines leads to a face velocity one tenth that of the prEN 13725 standard (0.02 to 0.05 m/s vs. 0.2 to 0.5 m/s) (See Figure 2).

Selection of the sensor (human assessors) for olfactometry continues to be a point of controversy in comparing standards. The EE-6 committee recommends selecting panelists which represent the normal population by excluding only those who are very insensitive and those who are very sensitive. The prEN 13725 standard has a very strict set of criteria which requires a laboratory to continually test their assessors to be sure they stay within a set of accuracy and repeatability criteria for a standard odorant (n-butanol). The prEN 13725 standard is striving to create a repeatable and accurate instrument, however it is uncertain if the criteria set for n-butanol will transfer to other odorants.

The United States, the European Community, and Australia have come a long way in the development of olfactometry standardization. However, there still remain critical differences, which must be reconciled, before international standardization can be achieved. Everyone involved in the odor industry must take the responsibility to take a long look at these differences and work towards a unified standard.

Standard References

ASTM D-1391. *Standard Test Method for Measurement of Odor in Atmospheres (dilution method)*. American Society for Testing and Materials, Philadelphia, PA, USA, 1978.

ASTM E679-91. *Standard Practice for Determination of Odor and Taste Thresholds by a Forced-Choice Ascending Concentration Series Method of Limits*. American Society for Testing and Materials, Philadelphia, PA, USA, 1991.

VDI 3881 Part 1-4. *Richtlinien, (draft) Olfactometry, Odour Threshold Determination, Fundamentals*. Verein Deutsche Ingenieure Verlag, Düsseldorf, Germany, 1980.

AFNOR X-43-101. *Method of the Measurement of the Odor of a Gaseous Effluent*. Bureau de Normalisation, Paris, France, 1981, 1986.

NVN 2820. *Provisional Standard: Air Quality. Sensory Odour Measurement using an Olfactometer*. Netherlands Normalization Institute, The Netherlands, March 1996.

CEN TC264/WG2. *Draft: Air Quality - Determination of Odour Concentration by Dynamic Olfactometry*. European Committee for Standardization (CEN), May 1997.

Draft: Guidelines for Odor Sampling and Measurement by Dynamic Dilution Olfactometry. Air & Waste Management Association, EE-6 Odor Committee, Chairman: Martha A. O'Brien, May 1993.

ISO5725. *Precision of Test Methods - Determination of Repeatability and Reproducibility for a Standard Test Method by Inter-laboratory tests*. International Organization for Standardization, 1986.

References

1. Feenstra, L., *Onderzoek verdunning ruikbekers. Intern reprot MT-TNO. No. 80-04274*. TNO, Apeldoorn, The Netherlands, 1980.

2. Laing, D.C., *Optimum Perception of Odours by Humans*. The Seventh World Clean Air Congress, Sydney, NSW, Australia, 1986.

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4. O'Brien, M.A., Duffee, R.A., and Ostojic, N., *Effect of Sample Flow Rate in the Determination of Odor Thresholds*. Air & Waste Management Association International Specialty Conference, Odors: Indoor and Environmental Air, Bloomington, MN, Sept. 1995.

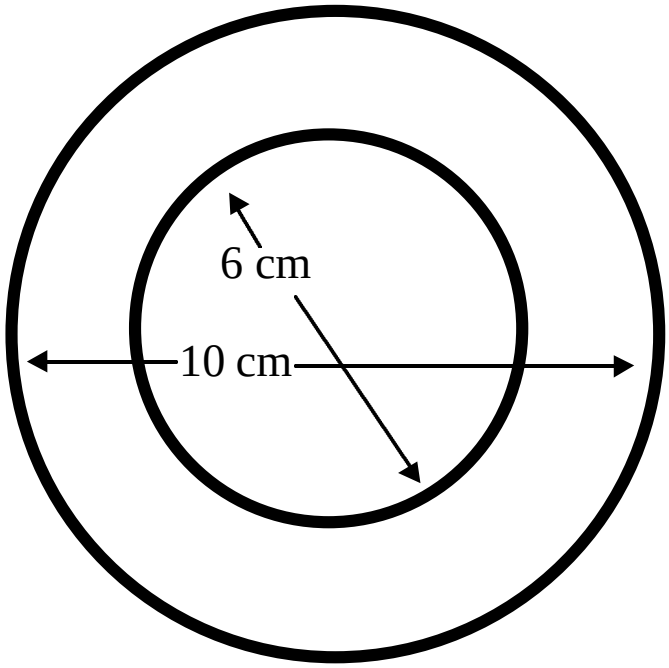
5. McGinley, C.M., and McGinley, M.A. *Olfactometry Flow Rate Criteria - A Multiple Laboratory Study, Part I*. Water Environment Federation Specialty Conference: Control of Emissions of Odors and Volatile Organic Compounds, Houston, TX, 20-23 April 1997.

Figure 1. International Odour/Odor Testing Elements

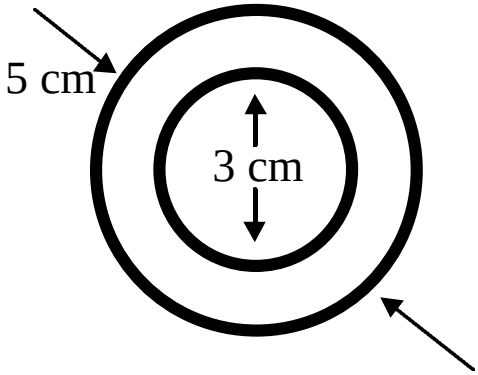
Elements	ASTM E679-91 United States	NVN 2820 Netherlands	VDI 3881 Germany	A&WMA EE-6 (DRAFT)	DRAFT prEN 13725 1999
Dynamic Dilution	X	X	X	X	X
Static Dilution		X			
Triangular Forced Choice Method	X	X	X	X	X
Binary Forced Choice Method		X	X		X
Yes / No Method			X		X
Ascending Concentration Series	X	X	X	X	X
Integral Odor Free Dilution Air	X	X	X	X	X
Integral Dry Dilution Air		X	X		X
Presentation Flow Rate (lpm)	- - -	20	20	8	20
Presentation Face Velocity (m/s)*	- - -	(0.09-0.26)	- - -	0.02-0.05	0.2-0.5
Presentation Mask Diameter (cm)*	- - -	4-7	- - -	6-10	(2.9-4.6)
Minimum Lower Dilution Ratio	- - -	- - -	2^3	2^3	2^7
Minimum Upper Dilution Ratio	- - -	2^14	- - -	2^13	2^14
Dilution Ratio Range	- - -	- - -	- - -	- - -	2^13
Dilution Ratio Factor Increase	2-3	1.4-3.0	2	2	1.4-2.4

* Item in parenthesis indicates calculated values.

Figure 2. Olfactometer Presentation Parameters of the U.S. AWMA EE-6 Odor Committee Guidelines and the European prEN 13725 Draft Olfactometry Standard.



U.S.: EE-6 Committee Guidelines
Mask Dimension: 6 - 10 cm
Face Velocity: 0.02 - 0.05 m/s
Flow Rate: 8-lpm



European: **prEN 13725 Draft** Std.
Mask Dimension: 3 - 5 cm
Face Velocity: 0. 2 - 0. 5 m/s
Flow Rate: 20-lpm