CHEMICAL ENGINEERING TRANSACTIONS
VOL. 40, 2014
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at: www.aidic.it/cet
Guest Editors: Renato Del Rosso

ISBN 978-88-95608-31-1; **ISSN** 2283-9216

Progress in the review of EN13725: focus on sampling and uncertainty

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The widely used EN13725 standard for dynamic olfactometry is currently being revised by a working group of experts, CEN/TC264/WG2 ‚Olfactometry'. The work is progressing steadily, with seven meetings held. A revised draft standard is expected to become available for the review process in the course of 2016. The revised standard will contain comprehensive guidance on the sampling of odour sources, an enhanced treatment of measurement uncertainty and clear general guidance on health and safety for sampling technicians, laboratory staff and assessors. This paper provides a preview of topics that may be revised in the standard, as well as a final reflection on the relation between sensory tasks and the associated measurement uncertainty.

1. Introduction

The widely used international standard for olfactometry *EN13725 Air Quality – Measurement of odour concentration using dynamic olfactometry* [CEN 2003] is currently being revised.
This revision is part of the periodic revision process common to all standards of the Committee Europeen de Normalisation (CEN) [CEN 2013]. The CEN Technical Committee *TC264 Air Quality* took a resolution to review the standard in early summer 2012.

CEN/TC 264 'Air quality'

Resolution 758 (London 31)
CEN/TC 264 accepts the proposal for the following preliminary work item dealing with the revision of EN 13725. CEN/TC 264 reactivates WG 2 with the convenor and secretary provided by NEN to perform this preliminary work item.

As a result, the Working Group 2: Olfactometry was brought to life again and I had the privilege to be invited as a convenor.
International experts volunteered to participate, representing European Accreditation member organisations from 9 countries. The current membership is listed in Table 1.

Table 1: Membership of the working group CEN/TC264/WG2 2Olfactometry", as of September 2014

Expert	Employed by	Representing Country
Nicolet Baas	NEN	Netherlands (secretariat)
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Ton van Harreveld	Odournet	Netherlands (convenor)
Toon van Elst	Olfascan	Belgium
Ilse Bilsen	VITO - Flemish Institute for Techn. Research	Belgium
Olivier Noiset	NBN (Belgium) - CERTECH	Belgium
Anne-Claude Romain	University of Liège	Belgium
Benjamin Bergmans	ISSEP	Belgium (observer)
Arne OxbØl	FORCE Technology	Denmark
Jesper Lauridsen	Dansk Standard	Denmark
Lone Skjerning	Dansk Standard	Denmark (observer)
Jean-Michel Guillot	AFNOR	France
Amelie Louvat	GDF Suez	France
Celia Sanchez	INERIS	France
Peter Wenzel	Uppenkamp + Partners GmbH	Germany
Dietmar Mannebeck	Odournet GmbH	Germany
Claus-Jürgen Richter	iMA Richter & Röckle	Germany
Isabelle Franzen-Reuter	VDI	Germany
Ralf Both	Landesamt fur Nordrhein Westphalen	Germany
Selena Sironi	Politecnico Milano	Italy
Andrea Rossi	PROGRESS S.r.i.	Italy
Frans de Bree	Buro Blauw	Netherlands
André van Boheemen	Witteveen + Bos	Netherlands
Nico Ogink	Wageningen UR Livestock Research	Netherlands
Hugo van Belois	Van Belois Environmental Services	Netherlands
Joao Paulo Vaz	Eurofins	Portugal (observer)
Carlos Diaz Jiménez	www.olores.com	Spain
Robert Sneath	Silsoe Odours Ltd	United Kingdom

The members of WG2 participate on a voluntary basis.
The work of WG2 is not mandated by its parent committee CEN/TC264 Air Quality currently, which implies there is no allocated budget and no contractual deadline. Currently the options to obtain a mandate are being explored. Such a mandate may become necessary in case more substantial validation investigations are required.

2. Work programme

During the first meeting, which was held 20-21 November 2012, a list of the key issues that require revision was drawn up, based on the outcomes of an earlier formal enquiry among CEN member states and the input from national technical experts. For each key issue that was identified a small task group was formed by volunteer members of WG2:
The following task groups were formed to review the publications and relevant unpublished material and draft initial recommendations regarding the following key issues relevant to the revision:

- Task group 1: Sample storage and materials for olfactometry
- Task group 2: Reference material for panel selection and panel management procedures
- Task group 3: Sampling of passive area sources (without flow)
- Task group 4: Sampling of active area sources (biofilters)
- Task group 5: Dynamic dilution during stack sampling
- Task group 6: Implications of EN 15259 air quality - measurement of stationary source emissions and other relevant sampling standards
- Task group 7: Calculation of uncertainty
- Task group 8: Compatibility of Yes/No and Forced Choice methods
- Task group 9: Health and safety issues

The task groups have identified a large number of relevant documents and data, including main national documents and research reports that will be used as input in the revision process, such as:

- VDI/DIN3880 – Sampling of odours
- VDI/DIN3884 – Olfactometry - Determination of odour concentration by dynamic olfactometry - Supplementary instructions for application of DIN EN 13725
- EN 15259 air quality - measurement of stationary source emissions
- Netherland's guidance on measurement and calculation of odours NTA9065
- etc.

So far 104 new documents have been distributed within the revision working group, which have been discussed in the course of 6 meetings. Working groups 1, 2, 4, 6, 7, 8 and 9 have delivered detailed reviews of their topics which are currently being used to as a starting point for writing revised normative text in the relevant clauses of EN13725.

The revision process is expected to require between 10 and 12 two day meetings, held about twice a year. The next planned meeting is meeting nº. 7, in Barcelona, Spain, on 25-26 November 2014.
The final draft text for revision is expected to become available in the course of 2016.

Once the endpoint of the revision has been reached a release of an ISO standard based on EN13725 is envisaged, under the Vienna agreement [CEN (2014)] that sets mechanisms aimed at avoiding duplication between CEN and ISO.

3. Main topics and expected outcomes

The Working Group has identified a few key needs:

- To improve the uncertainty of the measurement, in particular where precision under reproducibility conditions is concerned.
- To include guidance on risk assessment for Health and Safety
- To include technical guidance on sampling of ducted sources, passive area sources and active area sources
- To review guidance on sample storage

These needs are form the 'mission' of the task groups. Many of the task groups have finalized collecting and reviewing relevant data, but are still in the process of drafting revised clauses for the

standard. It is therefore not possible to provide a full overview of expected outcomes. At present I can only indicate in general terms which topics are under consideration and an impression, on my personal title, of the possible outcomes. In the following sections, these expected outcomes are outlined, for the topics covered by each task group.

Task group 1: Sample storage and materials for olfactometry

An extensive review of published and ongoing research in this area has been conducted. No major new breakthrough solutions have been identified. However, it has become clear that some compounds are much more vulnerable in storage than others [Boeker et al (2014), Hansen (2010)]. Other compounds show losses in 30 hours, in the order of 20-30% in mass concentration, which are not very significant in the context of odour concentration. Some practical options may be included, such as double bags, to reduce losses during storage [Sironi et al (2014)].

Another issue is the recovery of odorants in dilution systems, as used in olfactometers. Potentially meaningful losses of specific odorant compounds have been observed [Hansen (2013)], which may require revised criteria for materials used in olfactometer systems. A progress report of ongoing research into this topic will be presented at this conference [Kasper et al, 2014]

Task group 2: Reference material for panel selection and panel management procedures

A wide range of improvement options has been reviewed for feasibility and cost/benefit in terms of improved uncertainty. An extensive statistical analysis has indicated that the reproducibility, between laboratories, may add significant uncertainty to the measurement results in those applications where results from more than one laboratory are used [Klarenbeek (2014)]. To improve reproducibility an increase in the number of panel members required for a measurement seems to provide the best opportunity for improvement.

A procedure for experimentally establishing a value for the EROM value for other reference odorants, traceable to the agreed reference value for the EROM of n-butanol of 123µg will be included in the revised standard, to open possibilities of using multiple reference odorants or even defined odorant mixtures for improved panel selection and quality assurance.

Task group 3: Sampling of passive area sources (without flow)

The task group has conducted an extensive review of published papers and investigations known by its experts. This again confirmed that the type of device used, and its operational parameters, both strongly influence the numeric results in terms of specific odour emission rate (SOER) in $ou_E \cdot m^{-2} \cdot s^{-1}$.

The task group is considering stating a benchmark for SOER for a chemical component, soluble in water, under standard conditions, which can serve to test various devices for compliance with that benchmark. That would avoid a prescriptive approach for the most suitable device, leaving as much space for innovation as possible. The benchmark is likely to seek alignment with the approach stated in the German VDI3880 standard.

Task group 4: Sampling of active area sources (biofilters)

The approach of the German VDI3880 standard will serve as the starting point for this topic, taking into account other good practice documents as well.

Task group 5: Dynamic dilution during stack sampling

This task group has not reported in detail until now

Task group 6: Implications of EN 15259 air quality - measurement of stationary source emissions and other relevant sampling standards

The application of EN15259 has led to an incisive change in the structure of EN13725, which has been agreed in WG2. Also a number of terms and definitions need adaptation to align the revised EN13725 with the EN15259 standard.

Task group 7: Calculation of uncertainty

The existing set of statistical performance metrics for n-butanol will be maintained. However, WG2 has identified a clear need to provide a defined approach to not only consider precision under repeatability conditions but also under reproducibility conditions, with a view to assigning uncertainty to measurements carried out by different laboratories. This approach will not be normative for those elements where more than one laboratory is required for implementation. It is after all a requirement of a technical standard: to be applicable in one single laboratory. However, the informative clause will provide clear guidance on the treatment of measurement uncertainty, also when multiple laboratories are involved. Task group 1 is currently finalizing a choice between two alternative methods, which are aligned except for the treatment of measurement bias.

Task group 8: Compatibility of Yes/No and Forced Choice methods

The task group has considered available data, from interlaboratory comparisons and internal parallel measurements in organisations where both modalities are applied. The conclusion is that the Yes/No modality and the forced choice modality can produce EN13725 compliant results that are comparable. There are no grounds to consider either method to be performing better than the other. There are some indications that the effort of panel training and panel supervision during the measurement process is higher in the Yes/No modality. Also, WG2 reached the conclusion that panel selection and training are specific to the presentation modality used. This implies that a panel member qualified in one modality cannot be assumed to be qualified for the other modality. The qualification is not transferrable across presentation modalities.

Task group 9: Health and safety issues

Very practical advice on health and safety for sampling technicians, laboratory staff and panel members/asessors will be included in the revised standard. By necessity the advice identifies general principles. The health and safety regulations and the duty of care required by national legislation and standard remains the primary guide for health and safety practice in olfactometry.

In addition to the work of the task groups, the consistency and general clarity of the standard is being reviewed. This includes review of terms and definitions, symbols used, and also the delineation of the scope of the standard. The scope will be made more specific in that field olfactometry, as practiced in the USA and Canada, will be outside the scope of the revised EN13725 standard. For direct impact measurement in using field measurements reference will be made to the new CEN standards, currently in CEN enquiry phase, titled *Ambient air — Determination of odour in ambient air by using field inspection*
Part 1: Grid method and *Part 2: Plume method*. Sampling of odours will be added to the scope of EN13725.

4. Final thoughts on the how the methodology of sensory measurement can determine measurement uncertainty

Discussions on measurement uncertainty in olfactometry, for some reason, do not come easy. Sometimes the impression exists that it is all so ‘subjective’ that the issue is somehow beyond the domain of statistical analysis. To remove that impression perhaps the following contemplation can be helpful. Instead of the ethereal area of odours, let’s consider a more tangible sensory measurement challenge. The simple sensory task is:

How heavy is that bag?

This measurement can of course be performed measuring mass with scales. But it can also be done using sensory methods, in a variety of ways.

The task can be made more specific:

- Is the bag perceived as *light* or *heavy*?
- What is the *weight* of the bag in kilo's
- Is the bag *lighter* or *heavier* than a standard bag?

These are very different measurement tasks, and we will see that the specific way of setting the tasks has significant implications for the measurement uncertainty.

When we set the task *Is the bag perceived as light or heavy?* we aim to determine an affective attribute. How is the weight of the bag perceived?
To measure this, we can simply ask a panel of assessors to pick up the bag and ask: *Is the handbag light or heavy?* In this setup the context can be expected to determine the result to a significant degree. Say the bag actually weighs 1 kg. If we offer the bag in the shape of a sailors kit bag filled with plastic foam, to reach 1 kg, most assessors will assign the *'light'* category .A small gold bar in an elegant ladies purse will lead most to the category *'heavy'*. But even if the physical shape is the same, say a regular computer bag, the way in which we ask the question can be very relevant.
'This is a new business laptop. If you need to travel extensively with it, in planes and trains, would you say it is light or heavy?' is likely to lead to a very different set of responses than the question: '*This is a new maxi smartphone, to take on your next biking holiday to stay connected. Would you say it is light or heavy?'*.
Also random factors, uncontrolled, can influence the outcome. If an assessor enters the lab for the assessment with a computer bag weighing 2,7 kg, this individual recent experience will influence the assessor's responses. The bag under study will almost certainly be perceived to be *'light'* affected by a factor completely alien to the measurement setup.

Measuring affective attributes is not easy. The context of the test, including the phrasing of the question and the short and long term memory of experiences will all be determining the result. Even when the test conditions and context are strictly controlled, a large number of assessors will be required to determine of the new product bag will be perceived to be light or heavy by a significant slice of the potential consumer base. This is mainly caused by the large variation in the 'sensory memory' and experience history of our sensors/asessors.

Another sensory challenge is to replace scales to determine bag mass, using sensory measurement.

We can simply ask: *State the weight of the bag in kilograms*. Our assessors will delve into their individual sensory memory database and give us an estimate, which will include an uncertainty. My guess would be that most people, say 95%, would indicate a value between 0,5 and 2,0 kg for our 1,0 kg bag. The more assessors we use, the smaller uncertainty of the average result. At n =100 we are perhaps 100 grams off. At n =1000 the mean should not be further than 20 grams from the truth.

If we train our assessors with standard reference weights, they will get a lot better at estimating weight. Maybe we could reduce the n in our assessor pool to half? Maybe a quarter if we use only those who are best at the task?

In a more elaborate measurement setup we can ask our assessors to fill the water bottle inside the unknown bag in such a way that its weight matches a reference bag weighing 1,2 kg, allowing them as many iterations as needed. Knowing that water has a specific mass of 1 g/ml we can use the volume scale on the water bottle to measure the difference or 'distance' between the standard bag of 1,2 kg and our unknown bag. Simple calculation will provide the weight of our bag. Using this method, we

can reach a very small uncertainty even using a low number of assessors. I would guess we could measure the weight, still without scales, with an uncertainty of less than 0,050 kg using n = 10 untrained assessors.

This last approach leads us to the simplest sensory task: Is the bag lighter or heavier than a reference bag?

When we apply forced choice, the answers are correct or incorrect. If we allow 'dunno' as an answer we get *green*, *orange* and *red* results. The fraction of *red* and *orange* responses will increase as the difference between the bag and the reference bag gets smaller. What is the smallest noticeable difference for most? Is 0,100 kg the difference 95% of assessors get right? Or can you train this and select the best to reduce to 0,020 kg at 95% correct answers?

This story illustrates that the uncertainty of the results depends highly on the mechanism of our sensory scales. In EN13725 olfactometry we ask a slightly different task, if we translate to touch and weight. We put a small spherical ball of lead on the outstretched hand of the assessor. Using smaller and smaller balls, we determine the minimum weight that 95% of people can detect as 'present'. Then we move to wood balls. Then to foam. Then to fluffy feathers. Each will have its own value.
And so it is with our olfactory sense, with different odorants.

And also for olfaction, the sensory task and the associated number of assessors required for an acceptable level of uncertainty are closely linked. And the same statistics apply, mark my words!

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