CHEMICAL ENGINEERING TRANSACTIONS
VOL. 68, 2018
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet
Guest Editors: Selena Sironi, Laura Capelli

ISBN 978-88-95608-65-5; **ISSN** 2283-9216
DOI: 10.3303/CET1868055

Testing Performances of a Newly Designed Olfactometer

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Dynamic dilution olfactometry as regulated by EN 13725 requires instrumentation of adequate technology and in order to spread the use of Dynamic Olfactometry high usability of the device is a must. A new dynamic dilution olfactometer has been designed and manufactured after the experience gained in previous prototype development and performance studies as well as from experimental applications. Materials have been selected in order to be compliant with the expected next-to-come updates of the EN13725 technical norm and checks on pneumatic steps required by the odour concentration analysis procedure have been implemented. In order to generate specific dilutions in a wide range, a high precision stepper motor is used, instead of the more common calibrated orifices. The instrument comes with option of incrementing dilution with a factor of $\sqrt{2}$ (instead of more usual 2), so to increase resolution of the odour measurements. The consumption of neutral compressed air has been highly reduced in comparison with previous prototypes. The new features of the instrument as well as the available dilution steps will be presented. A careful check of accuracy and operative speed at high dilutions has been performed. Standard n-butanol, and samples from ambient air collected in close proximity from odour emitting sources have been collected for testing the olfactometer and the panel response. A market top instrument has been considered for sake of comparison.

1. Introduction

The European technical norm EN 13725:2003 standardizes the sensorial method of dynamic olfactometry, for the determination of odour concentration in gas samples (EN 13725, 2003). Various technologies have been applied for olfactometers by instrument producers, aiming at effectively generate the needed dilution of the air samples to be presented to the olfactory panel members; for example calibrated orifices are used in TO8 or TO- Evolution olfactometers from Olfasense, or mass flow controllers in SS600 from Scentroid have been used. Another option for controlling the amount of the air sample that is distributed to the sniffing ports, is the use of calibrated needle valves, described in (Brattoli, 2014).
Years of experimental applications of the norm have revealed aspects of the norm that could be improved. Adsorption of odorants on materials of the olfactometer are a possible critical point (Hansen, 2013; Kasper, 2017). In 2012 a revision process of the norm has been started (van Harreveld, 2014). The revision is intended to address several issues, among them there are the materials used to build the parts of the instrument in direct contact with the sample and a new, detailed and comprehensive approach to uncertainity assessment. Updates on the revision process were communicated recently (van Harreveld, 2018). The olfactometers are expected to adequate to the novelties that emerge from research and from the normative revision. Dynamic dilution olfactometry requires instrumentation of adequate technology and in order to spread the use of dynamic olfactometry high usability of the devices both for analysis and sampling is a must.
The experience gained in previous olfactometer development (Brattoli et al., 2014) and instrument performance studies as well as in field applications (Licen et al. 2018) have driven us to design and manufacture a new dynamic dilution olfactometer able to foster the experimental resolution of the analyses.

Please cite this article as: Cozzutto S., Pettarin N., Barbieri G., Licen S., Barbieri P., 2018, Testing performances of a newly designed olfactometer, Chemical Engineering Transactions, 68, 325-330 DOI: 10.3303/CET1868055

Materials have been selected in order to be compliant with the expected next-to-come updates of the EN13725 technical norm and checks on pneumatic steps required by the Odour Concentration analysis procedure have been implemented.
In order to generate specific dilutions in a wide range, a high precision stepper motor is used, instead of the more common calibrated orifices. The instrument comes with option of incrementing dilution with a factor of 1.41 (i.e. √2 instead of the more usual 2), so to increase resolution of the odour measurements. A careful check of accuracy and operative speed at high dilutions has been performed. Standard n-butanol, thiols and samples from ambient air from the close proximity to odour emitting sources have been collected for testing the olfactometer and panel response. The consumption of neutral compressed air has been highly reduced in comparison with previous prototypes. A market top instrument has been considered for supporting the check of the instrument performances.

2. Materials and methods

2.1 LEO: general description

The new olfactometer named Light Evolution Olfactometer (LEO) is light and compact (~30 kg, body of (100 x 30 x 50) cm), easy to transport, and requires oil free air compressor providing 40 l/min during the analysis in comparison to 160 l/min requested in the previous series of 8 panelist instrument (Wolf).

The olfactometric apparatus is composed of:

- a mechanical section constituted by a central unit and the user's positions;
- a pneumatic section including the diluter and the air distributor;
- the electric and electronic section for all the connections;
- the software.

Figure 1 shows a picture of the new olfactometer.

Figure 1: A picture of the Light Evolution Olfactometer (LEO)

All the instrumental components and used materials are in compliance with the specific requests of the European technical norm EN 13725.

Teflon is the only material used to build the central internal part of the olfactometer, LEO's core. All the pipes which link the sample bag connection to all the mask for odour assessment by the panelists are built using Teflon, as well. In this newly designed olfactometer the use of stainless steel for the pipes has been avoided to produce an olfactometer in compliance with the expected next-to-come updates of the EN13725. Glass has been used for the masks. The instrument implements zero-air valves to avoid odour dragging between a sample submission step and a blank step.
The core of the instrument is a high precision stepper motor which activates a needle which acts on the Venturi system allowing to perform 29 different dilution steps with √2 pitch. The dilution range starts from $(\sqrt{2})^5$ (1:5,7) and ends at $(\sqrt{2})^{33}$ (1:92682). The 29 available dilution steps are shown in Table 1.

Table 1: The 29 available instrumental dilution steps

Dilution steps:					
1:92,682	1:16,384	1:2,896	1:512	1:91	1:16
1:65,536	1:11,585	1:2,048	1:362	1:64	1:11
1:46,341	1:8,192	1:1,448	1:256	1:45	1:8
1:32,768	1:5,793	1:1,024	1:181	1:32	1:5.7
1:23,170	1:4,096	1:724	1:128	1:23	

Figure 2 shows the pneumatic section of the olfactometer. LEO adopts the yes/no method for the presentation of odour samples and the dilution steps are produced automatically.

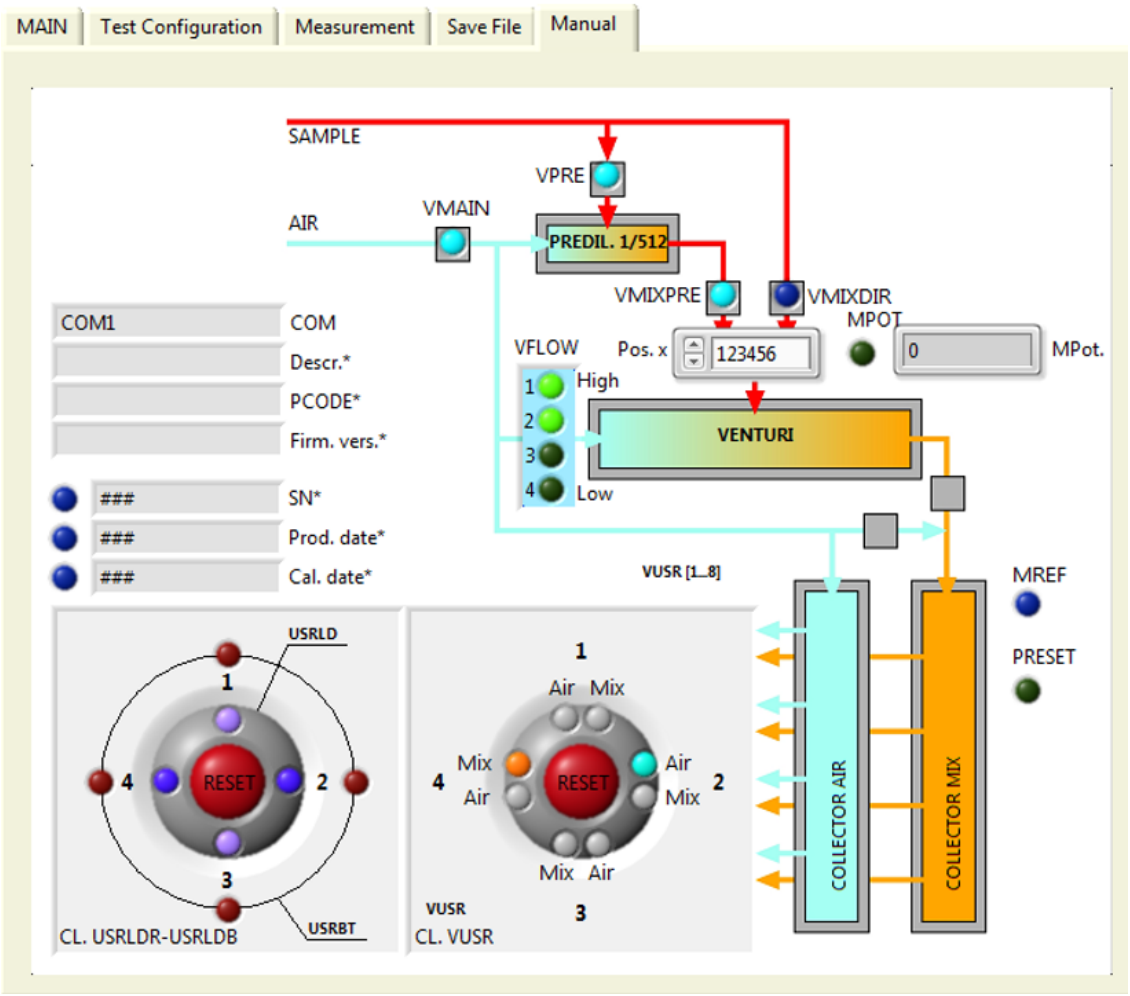

Figure 2: Scheme of the pneumatic section

Sampled air, after connection of the sample filled bag to the olfactometer, is sucked in by the pneumatic system; part of the sample is sucked directly by the (VMIXDIR) valve, and introduced in the main flow; another sample aliquot, taken by the (VPRE) valve, is prediluted and then sent to the (VMIXPRE) that introduces to the main flow. All dilutions are carefully calibrated so to obtain a constant flow at the sniffing ports, higher of the 20 L/min minimum flow established by the norm; in order to fulfill this operation four valves (VFLOW) can be activated, as needed. Depending on the logical state in which one of the four sniffing positions is, the following valves are activated: (Air) if the reference air has to be sent to the panelist; (Mix) if a dilution of the sample air has to be sent to the panelist; the (Mix) valve is activated also in case of input of "0" dilution in the sequence of analyses.

2.2 The software

The software has been designed, starting from the WOLF one, to set the different operational parameters related to the breathing time, the flushing time between rounds, the flushing time after sample, the percentage of blank samples in the dilution sequence and the order of presentations of the different step of dilution. What is new is the control of the motor stepper to obtain a wider range of dilution steps compared with the previously produced olfactometer.
Two software screenshots are presented in Figure 3, so to provide an example of the interface available to the panel leader.

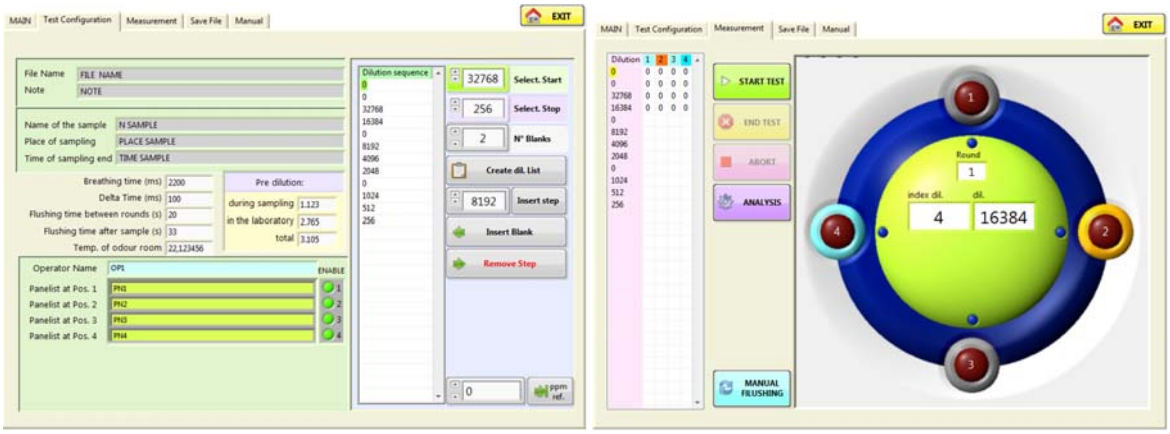

Figure 3: Screenshot of the LEO software

The software has a management section where all information required by the technical norm can be entered. During the analysis, the panel leader can check in real time the dilution level of the sample sent to the panelists, beside all the information about the correctness of the answers. At the end of the session all data are saved and they can be processed by the "Analysis" software in on-line or off-line mode.

2.3 Standard measurements

The calibration of the dilution valves has been made using propane as tracer gas and a FID as analyzer.
To cover the whole range of dilutions, three propane cylinders of different concentrations, respectively 200,000 ppm, 10,000 ppm and 1,000 ppm purchased from Air Liquide Italia Service s.r.l. have been used.
Regarding the verification of the valves conformity, analysis have been performed by a flame ionization detector Eco Control model ER600.

2.4 Comparison test and real sample analyses

A comparison between one of the market top olfactometers has been planned and conducted. Results In order to check the performances of the instrument LEO, from the analysis of the samples prepared in the same condition by TO Evolution Olfactometer (by Olfasense GmbH Germany) - from now on named as TO Evo - and by LEO were produced and they are discussed in the following paragraphs.

Figure 4: the setup of the TO evolution in one of the two olfactometric chambers in the lab performing the tests

The tests were carried out in two adjacent olfactometric chambers and the same samples were analyzed.
The two distinct types of samples analyzed are:

- the 60.4 ppm n-butanol cylinder has been purchased by SIAD S.p.A. (Italy);
- real samples collected near oil sources and with very different odour concentration.

3. .Results and discussion

In order to verify the instrumental performances 60.4 ppm n-butanol reference standard has been used. In Table 2 the results of the olfactometric analysis are presented. As it can be observed, the instrumental responses are in very good accord. The same panelists have been involved in all the analyses.

Table 2: Olfactometric results of n-butanol samples (LEO and TO Evo)

N-Butanol concentration (ppm)	C_{od} (OU_E/m^3) LEO	C_{od} (OU_E/m^3) TO Evo
60.4	1579	1351
60.4	1218	1360
60.4	1328	1624
60.4	1649	1773

Two real samples collected near oil sources and with very different odour concentration have been submitted to the panelists. In the case of the analysis with LEO the samples were analyzed twice. The Table 3 illustrates the results obtained

Table 3: Olfactometric results of real samples (LEO and TO Evo)

Real sample	C_{od} (OU_E/m^3) TO Evo	C_{od} (OU_E/m^3) LEO (analysis I)	C_{od} (OU_E/m^3) LEO (analysis II)
A	9447	9329	8933
B	258	292	318

All the results are in good agreement both between the two different olfactometers and within the LEO measurements.

4. Conclusions

The experience gained in previous prototype development and performance studies as well as from experimental applications, allowed us to design and build a new dynamic dilution olfactometer able to foster the experimental resolution of the analyses.
Experiences about criticalities arisen from the use of WOLF in a broad range of applications allowed us to understand in detail aspects of the previous instrument that could be improved. The changes introduced in the newly designed LEO instrument allowed us to:

- avoid odour dragging between a sample submission step and a blank step;
- radically reduce the compressed air consumption;
- optimize the sample consumption;
- enhance the instrumental resolution.

The comparison of the LEO analyses of both n-butanol and two real samples at different concentrations with results of a market top instrument (TO Evo) showed a good correlation in all the analyses.

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