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Methodological Aspects of Sample Collection for Dynamic Olfactometry

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Odor assessments by dynamic olfactometry can be affected by odor background from sampling bags and by interactions of samples with the bag material during storage. We have conducted a methodological study which aimed to assess the odor background from new bags and the impact of sample storage in both Tedlar® and Nalophan® bags - focusing on odors emitted from municipal sewage, aeration basin, sludge, livestock manure and coffee. The odor background from new non-flushed Tedlar and Nalophan bags (in which fresh air was stored for 24 h) was as high as 75-317 odor units; OU (Tedlar) or 36-43 (Nalophan). For pre-flushed bags the background was reduced to 25-32 (Tedlar) or 19-22 (Nalophan). It suggests that although modern olfactometers allow very low dilution ratios, in practice, special caution would be needed before considering values in the range of several to low tens OU. The effect of sample storage was found to vary with different odor sources and type of bag. Storage had the greatest impact on coffee odor (both in Tedlar and Nalophan) with averages losses by factors of 4-5, and for manure odor in Tedlar bags with average losses by factor of around 6. Opposite effects were observed for sewage odor, with losses by factor of around 2 in Tedlar but *increase* of odor by factors around 3 in Nalophan. The performance of the jury was monitored within actual sessions along the study period. The panel response to n-butanol was found to be fairly stable (geometric mean of 37 ppb) suggesting that the effect of storage can only be partly attributed to the uncertainty that is associated with variation among panelists. For practical reasons and before more advanced sampling methodologies are available, it is recommended that the standard will include more information about the potential impact of storage, such that labs will be able to provide a better assessment of the potential uncertainties associated with odor values and dispersion models.

1. Introduction

In accordance with the European Standard "Air quality - Determination of odour concentration by dynamic olfactometry" (EN 13725; CEN, 2003), sampling bags such as Tedlar® (polyvinyl fluoride) or Nalophan® (polyethylene terephthalate) are

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considered appropriate. For practical reasons, sample storage of up to 30 h is allowed before measurement. The need to clean (flush) bags as well as to minimize storage time are stated but no details are provided as to the potential odor background that might be associated with pre-flushed and non-flushed new bags as well as the potential losses of different kinds of odors during storage.

Background levels can have serious consequences when sampling low level odors (ambient air or low-emitting sources), or when pre-dilution is required during field sampling. The latter is often recommended to prevent condensation when sampling hot and moist sources. In such cases, ignoring background odor may cause severe errors by multiplying these values by the pre-dilution factor. Reported background values in commercially available bags without pre-cleaning were in the range of 20-60 in Tedlar (Parker *et al*., 2003), 30-100 in Nalophan (Juarez-Galan *et al*., 2008) or 2-30 and 10-50 in Tedlar and Nalophane, respectively (Miller and McGinley, 2008). Flushing the bags with non-odorous air and in some cases coupled by heating were shown in these studies as efficient to reduce background levels to about 10 OU. Flushing several bags simultaneously at oven temperatures is not always practical when large bags (40-60 litres) are used.

The information regarding sample preservation is much more limited. It was shown for example that specific odorants are more or less susceptible for losses during sample storage (Koziel *et al*., 2005; Trabue *et al*., 2006). Yet, there is lack of data which can be used to evaluate the potential impact of sample storage of various odor sources. The present study aimed to assess the odor background from new bags and the impact of sample storage in both Tedlar and Nalophan bags - focusing on odors emitted from municipal sewage, aeration basin, sludge, livestock manure and coffee. This kind of information is critical for assessing the uncertainty associated with olfactometry and related odor dispersion modelling.

2. Methodology

2.1. Odor Analysis

All measurements were conducted on a dynamic olfactometer (Odile 2510, Odotech Inc., Canada; Fig 1). Odile enables dilution ratios of 3-2,000,000 (pre-dilution is rarely needed) and an unlimited step factor between successive dilution levels. Sample bags are pressurized in a 250 l vessel. Fresh air/odorous air exposures are presented simultaneously at six stations each consisting of three sniffing ports, and is designed to provide 22 liters/min from each port with air velocity of 0.2 m/s. Exposure time was set to 15 sec for all analyses (during this time the panelists sniff from the three ports and make their triangular evaluation of the perception thresholds). The selection of the odor port was done randomly. Although the step between dilutions has no limits, the algorithm default of 1.58 was used in most cases. The panel included between 4-6 people, where in most cases the panel included 5 or 6 people. Data analysis was conducted in accordance with the EN 13725. In addition, 50% dose-response analysis was performed by means of a logarithmic curve fitting ($y=a\ ln(x)+b$; where y is the % of panelists who detected the odor, x is the dilution factor, and a and b are the curve fitting coefficients). This analysis included all data points (three rounds) without retrospective screening.

2.2. Samples preparations

Bags of 60 liters were used as typically needed for Odile olfactometers. Tedlar bags (~70×70 cm) were provided from Odotech Inc. Nalophan bags were prepared in-house from a Nalophan roll (Kalle, Germany; 30 cm diameter, 20 μm thickness). Theses bags were made by cutting the desired lengths of film, clamping both sides and inserting plastic valves (same as used for Tedlar bags). Odor background (in Tedlar and Nalophan) was measured in new bags before and after flushing with filtered air for 24 h. For testing various odor sources, Tedlar bags were always pre-flushed for 24 h before use. Based on the relatively low background levels found in new Nalophan bags (Section 3), these bags were further used without pre-flushing. Odor samples were prepared in the lab by collecting the headspace above solid/liquid samples. The following odor sources were prepared and tested:

1. "Manure odor" – prepared from the headspace of calves manure collected at the barn in Newe Ya'ar.
2. "Sewage odor" – prepared from the headspace of sewage collected at the headworks of the Afula wastewater treatment plant, northern Israel.
3. "Activated sludge odor" – prepared from the headspace of liquid collected at the aeration basin of the Afula wastewater treatment plant, northern Israel.
4. "Municipal Sludge odor" – prepared from non-stabilized sewage sludge collected at the Afula wastewater treatment plant, northern Israel.
5. "Coffee odor" – prepared from the headspace of a coffee drink.

Bags were "pre-conditioned" with the odor sample by filling half bag, emptying the bag, and re-introducing the odor sample. These bags were tested immediately after sample preparation and after storage for 24 h at 25°C. All analyses were performed at least in triplicate bags.

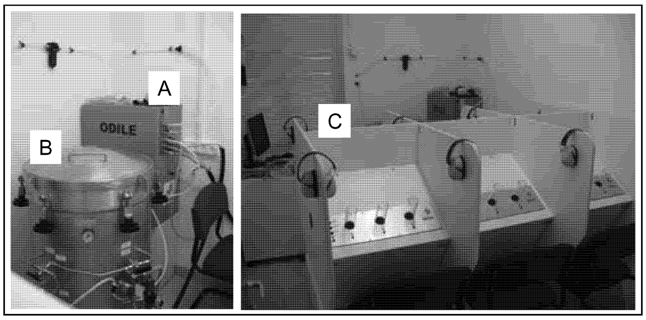

Fig. 1. *Odile 2510 (Odotech Inc., Canada).* ***A.*** *Dilution unit (computer-controlled).* ***B.*** *Pressurized vessel.* ***C.*** *Six panelists' stations.*

2.3. Jury performance

Individual panelists were certified according to the EN 13725. Additionally, the panel response to n-butanol was frequently monitored within actual sessions along the study period.

3. Results and Discussions

3.1. Assessment of bag background

If bags were tested immediately after filling them with clean reference air, the background from Tedlar and Nalophan bags (either pre-washed or non-washed) was around 20 odor units (OU). Yet, after storage of bags containing clean air for 24 h, the background from non-washed bags increased to 75-317 and to 36-43 OU for Tedlar and Nalophan, respectively. The background of stored clean air from pre-washed bags decreased to 25-32 OU (Tedlar) and 19-22 (Nalophan) (Table 1). These results suggest that although modern olfactometers allow very low dilution ratios, in practice, special caution would be needed before considering values in the range of several to low tens OU. For moderate and strong odor sources however, the background from Nalophan appears to be negligible and pre-washing is not necessary.

3.2. The impact of sample storage

The results obtained for the various odor sources are summarized in Table 2. The impact of storage was the most dominant for coffee odor (both in Tedlar and Nalophan bags) with averages losses by factors of 4-5, and for manure odor in Tedlar bag with average losses by factor of around 6. Opposite effects were observed for sewage odor, with losses by factor of around 2 in Tedlar but *increase* of odor by a factor around 3 (factor of ~0.3) in Nalophan. The impact of sample storage was similar in Tedlar and Nalophan for activated sludge (aeration basin) and sludge odors, the latter was basically non-affected by storage. In the case of manure odor, it could be that the differences observed between Tedlar and Nalophan are related to the fact that more odorous samples were introduced into the Tedlar than into the Nalophan bags. The relations between odor strength and the impact of sample storage are still under investigation.

Table 1: *Background odor (odor units; OU) from new Tedlar® and Nalophan® bags before and after flushing with clean air.*

	Time of analysis	**Tedlar**	**Nalophan**
Non-flushed	Immediate	18* (17-19)#	16 (13-21)
	After 24 h	169 (75-317)	40 (36-43)
Pre-flushed	Immediate	24 (19-27)	15 (13-19)
	After 24 h	29 (25-32)	20 (19-22)

**average value; #range of values obtained in replicate bags*

Table 2: *The impact factor of sample storage on odor measurements, defined as: [OU measured immediately] / [OU measured after 24 h].*

		Impact of sample storage	
Odor source		**Tedlar**	**Nalophan**
Manure	Average factor	**5.87**	**1.79**
	Range of factors	4.61-6.75	1.16-2.67
Sewage	Average factor	**1.99**	**0.34**
	Range of factors	1.41-2.61	0.23-0.44
Aeration basin	Average factor	**2.67**	**1.87**

	Range of factors	1.03-3.96	1.00-3.68
Sludge	Average factor	**1.01**	**1.09**
	Range of factors	0.86-1.20	0.82-1.46
Coffee	Average factor	**4.94**	**4.19**
	Range of factors	1.79-9.24	2.12-8.01

3.3. Data treatment

The procedure detailed in the EN 13725 may become problematic in cases of relatively large variations among panelists. In such cases (and when using a multi-station olfactometer), presenting a large number of exposures may confuse the panelists, resulting in "never-ending" rounds. The 50% dose-response based on logarithmic curve fitting (Section 2.1) was found to be a useful alternative in such cases. The full report of this study will include all evaluations of the storage effect both by the EN 13725 procedure and by 50% dose-response. Two typical examples are presented in Fig. 3, illustrating the larger differences between the two methods in cases of more dispersed data. In most cases however, the results obtained by the two methods are not substantially different.

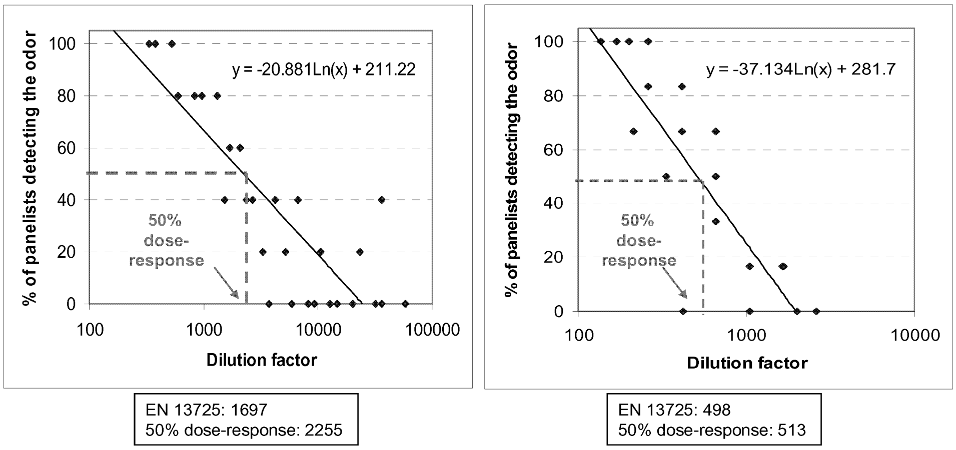

Fig. 3. *Two datasets treated by the 50% dose-response. The values obtained both by the EN 13725 method and by 50% dose-response are reported.*

3.4. Panel performance

A total of 22 n-butanol analyses were integrated within actual sessions along the study period (Fig. 4) demonstrating fairly stable panel response. The geometric mean of the panel was 37.2 (averaging the geometric means of individuals) or 29.2 (obtained by 50% dose-response). This suggests that the effect of storage reported in this study (Table 2) can only be partly attributed to the uncertainty that is associated with variations among panelists (Boeker *et al.*, 2008).

Apparently, panel response to n-butanol may not reflect its response to other odorants. Yet, as recently demonstrated by McGinley and McGinley (2010), odor assessors who conformed to the EN 13725 based on their response to n-butanol also conformed to a similar range of acceptability for hydrogen sulfide. This finding supports the assumption that panel sensitivity to a reference material can be a fairly good predictor for its sensitivity to other odorant mixtures and environmental odors.

Finally, for practical reasons and before more advanced sampling methodologies are available, it is recommended that the standard will include more information about the potential impact of storage, such that labs will be able to provide a better assessment of the potential uncertainties associated with odor values and dispersion models. An on-going work is designed to measure the types of odorants that are more susceptible to losses for the different odor types.

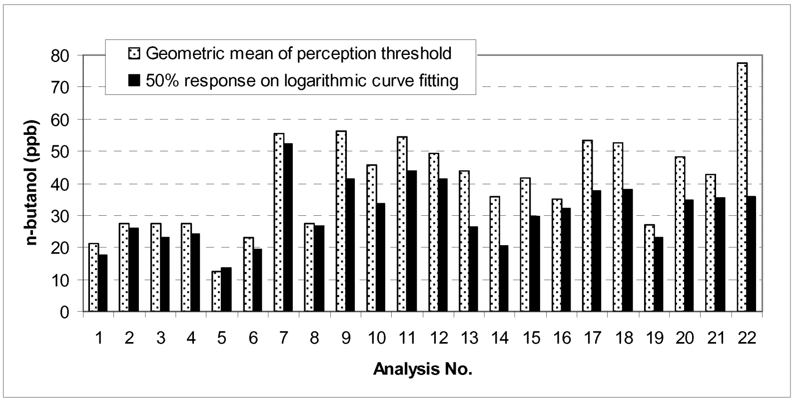

Fig. 4. *Jury performance monitored along the study period. The sensitivity to n-butanol is expressed both as the geometric mean (averaging the geometric means of individuals) and by 50% dose-response obtained from logarithmic curve fitting.*

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