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# A comparative and Critical Evaluation of Different Sampling Materials in the Measurement of Odour Concentration by Dynamic Olfactometry

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The selection of an odour sampling device and relative materials may influence the composition of the resulting odour sample. Current literature is limited to discussing how the measurement of emission rates derived from the same odour source could be related to materials and methods. There is therefore compelling evidence that current odour sampling practices should have greater regard for the fundamental physical and chemical principles, the nature of the odour source and the conditions created by the sampling device. Additional studies are necessary to identify the most appropriate situations under which the use of these devices may or may not be correct.

The scope of this study is to investigate the influence on the determination of odour concentration with dynamic olfactometry, according to EN 13725 (2003) standards, applying different initial sampling condition, with the objective to define the optimal criteria to evaluate the odour concentration and obtain the highest repeatability and accuracy of the sensorial measure.

A critical evaluation was carried out in relation to the same odour source with the comparison of sampling bag materials (Nalophan<sup>tm</sup>, Tedlar®, Teflon<sup>TM</sup>) and intervals of time elapsed between the sampling and analysis phase (3, 7, 14, 30, 48 h).

The results obtained show that the selection of an odour sampling materials may influence the composition of the resulting odour sample. There is therefore compelling evidence that current odour sampling practices should have regard for these factors and identify the situations under which these material and methods may be used appropriately.

# 1. Introduction

Odour from wastewater treatment plants is typically measured with olfactometry, whereby odour samples are collected in sampling bags and assessed by human panelists within 30 h, according to EN 13725:2003 (Stuetz et al., 2001; Gostelow et al., 2001; Zarra et al., 2010).

Dynamic olfactometry is actually the main method used for measuring odour concentration at the source emission, so as to evaluate the odour abatement technology efficiency. It is also the only European standardized method.

Odour concentration by dynamic olfactometry presents however different type of problems. In current literature, there are different articles that analyze the problem of the subjectivity related to the use of human noses (Köster, 1986; Jiang, 1996; Stuetz et al., 2001; Zarra et al., 2008; Zarra et al., 2009a;

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Zarra et al., 2009b; Muller et al., 2010). While, there are a few items that analyze the other aspects related to the sampling phase, such as time elapsed from the sampling phase to the analyses, the type of bags material, the time necessary to full the sampling bag, the minimum volume of sampling bag, etc. (Van Harreveld, 2003; Trabue et al., 2006; Mochaliski et al., 2009; Hansen et al., 2011).

In the present study, the storage stability of odorants in three types of sampling bags that are often used for olfactometry was investigated. the influence of the intervals of time elapsed between the sampling and analysis phase on the determination of odour concentration is was also investigated.

The scope of the study was to define the optimal criteria to evaluate the odour concentration by dynamic olfactometry and obtain the highest repeatability and accuracy of the sensorial measure.

# 2. Materials and methods

#### 2.1 Sampling

The evaluation of the influence of the sampling bags materials and the intervals of time elapsed between the sampling and analysis phase in the determination of odour concentration have been carried out by the full-scale wastewater treatment plant (WWTP) LFKW localized at the Institute of Sanitary Engineering, Water Quality and Solid Waste Management (ISWA) at the Stuttgart University Campus (Baden Wuerttemberg Region, Southwestern Germany) (Figure 1).

The treatment plant has a conventional activated sludge treatment and operates with domestic sewage originating from the university and Büsnau, a nearby village, and industrial discharge coming from the university laboratories.



Figure 1. Localization of the LFKW wastewater treatment plant in the Baden Wuerttemberg Region (Germany)

For the analysis program, samples were collected from the same odour point source at the sludge treatment units implemented by the centrifuge of the plants. The centrifuge sludge treatment unit consists of a drum centrifuge type (Kemper GmbH, D), confined within a dedicated building, place in depression, with the exhaust air sent to two specific and covered biofilter through a pipe with a diameter of 150 mm (Figure 2).

Air samples are taken, from the outlet pipe conducting the exhaust air from the centrifuge unit to the biofilters unit, using the 'lung' technique, whereby the sampling bag is placed inside a rigid container, and the container evacuated using a vacuum pump in accordance with EN 13725 (2003). This method avoids contamination, which may arise from the direct use of pumps in the sampling line.



Figure 2. Centrifuge treatment unit

# 2.2 Dynamic olfactometry

The determination of the odour concentration was made by dynamic olfactometry. The olfactometric analyses were conducted at the ISWA centre of University of Stuttgart using an olfactometer model TO8 by ECOMA, based on the "yes/no" method. All the measurements were conducted, except for some concerning the analysis time within the 30 h after sampling, according to the European normative EN 13725 (2003), relying on a panel composed of 4 qualified panellists.

# 2.3 Analysis program

Three different sampling bags materials type (Nalophan<sup>TM</sup>, thickness 25  $\mu$ ; Tedlar®, thickness 50  $\mu$ ; Teflon, thickness 50  $\mu$ ) with the same air volume (30 L) are used for the sampling.

To analyze the influence of the material type in the determination of the odour concentration, the samples were taken simultaneously from the same point.

Three odour samples were taken every day for 7 days at the investigated point of the treatment units of the plant. In total, 21 samples were collected.

For each of the collected samples the odour concentration by dynamic olfactometry after 3, 7, 14, 30 and 40 h of sampling was also determined. In total, 105 odour concentrations were determined, 35 for each type of bag material.

Figure 3 summarizes the experimental program for each collected i-samples (i = day samples).



Figure 3. Analysis program for each collected i-samples

#### 3. Results and discussion

#### 3.1 Influence of intervals of time after collection

Table 2 shows the influence of the different intervals of time elapsed between the sampling and analysis phase outcome over all the analysed samples, in the odour concentration determination.

DAY SAMPLE	time material	ODOUR CONCENTRATION (OU/m <sup>3</sup> )				
		3 h	7 h	14 h	30 h	40 h
ID1	nalophan	6,137	5,125	4,895	4,325	4,185
	tedlar	7,128	7,025	7,136	6,815	5,535
	teflon	6,535	6,326	6,026	6,253	6,015
ID2	nalophan	8,565	8,218	8,120	8,120	8,050
	tedlar	9,753	9,655	9,545	9,545	9,215
	teflon	9,656	9,656	9,654	9,600	9,540
ID3	nalophan	10,568	10,463	10,200	11,200	11,050
	tedlar	10,856	10,653	10,600	11,050	11,050
	teflon	10,586	10,490	10,530	10,850	10,865
ID4	nalophan	10,956	10,850	10,850	10,360	9,540
	tedlar	11,230	11,205	11,305	11,205	10,250
	teflon	11,230	11,230	11,080	11,080	11,089
ID5	nalophan	23,170	18,625	18,625	18,252	17,530
	tedlar	25,650	23,850	23,100	23,200	21,500
	teflon	24,220	23,150	23,050	23,050	22,110
ID6	nalophan	20,156	20,030	20,010	19,990	19,870
	tedlar	21,520	20,400	20,400	20,600	20,200
	teflon	21,210	21,210	21,190	21,190	20,980
ID7	nalophan	10,565	10,350	10,350	10,105	9,856
	tedlar	10,560	10,298	10,290	10,256	9,947
	teflon	10,360	10,360	10,180	10,180	9,860

Table 2: Odour concentration determination outcome over the whole analysis period.

The results show, except for the samples taken in the third day, that the odour concentration decay with the elapsed of time from the sampling phase, for any considered type of bag material. In particular, it is possible to observe that the odour concentration undergoes a slight decay in the span of 14 h after sampling, while a greater decrease if the analysis is carried out within 30 h. However, the odour concentration decays significantly after 30 h of the sampling phase for all the analyzed samples. Thus, demonstrating that the value of 30 h after sampling indicated by the European standard EN 13725 (2003) as a maximum span time for the analysis, proves to be effective as a superior limit value, but it does not appear to be the optimal time of determination.

Odour concentration decay with the passing of storage time is greater than the lower initial concentrations (such as samples taken on the day one), compared to higher initial concentrations (such as example, samples taken on the day six).

# 3.2 Influence of different gas sampling bags materials

Figure 4 shows the influence of the different gas sampling bag materials outcome over all the analysed samples, in the odour concentration determination. Odour concentration decay from 3 h to 30 h (Figure 4, *left*) and from 3 h to 40 h (Figure 4, *right*) after sampling phase, in percentage terms, in a relationship of uses of different sampling bag materials are represented with a Box-Whisker plots:

odour concentration decay (%) =  $|\Delta C|/C$ 

(1)

with:  $\Delta C$  = difference from odour concentration determined after 3 h and 30 h or 40 h from the sampling phase





Figure 4. Box-Whisker diagrams of dour concentration decay from 3 h to 30 h (left) and from 3 h to 40 h (right) after sampling in percentage terms in relationship of uses of different sampling bag materials.

The results highlight a major odour concentration stability in teflon sampling bags, with a lower reliability for nalophan sampling bags.

Nalophan bags involves a medium decay of the order of 5%, significantly higher than using Tedlar (2.9%) or Teflon bags (1.7%), by analyzing the odour concentration decrease between 3 and 30 h. When analyzing the odour concentration decay between 3 and 40 h after sampling phase, both the use of bags material of Nalophan (6.7%) and Tedlar (6.2%), show significant values; while Teflon bags (2.6%) continue to have good stability characteristics.

Odour concentration values detected at the same sampling day and elapsed time before analysis (Table 2), shown average values greater with Tedlar bags than odour concentrations values determined with the use of Teflon and/or Nalophan bags.

#### 4. Conclusions

The study shows how important olfactometric analysis is in to defining not only the detected odour concentration, but also the type of sampling bag material used as well as the time elapsed from the sampling phase to analysis. This clarification is particularly important when the odour concentration analysis should be compared.

It does not specify and take into account that these factors can result in the wrong estimation of odor emissions from environmental engineering plants and consequently lead to errors in all odour management strategies, such as in the design of odour abatement technologies as well as in the assessment of compliance and identification of possible annoyance limit.

The results obtained show that the odour concentration determined by dynamic olfactometry in air samples from environmental engineering facilities significantly decrease in time elapsed from the sampling phase, especially after having elapsed 30 h, as required by European standard EN 13725 (2003). Storage in Teflon bags are the most stable, while Nalophan bags are less reliable.

An optimal criteria to evaluate the odour concentration by dynamic olfactometry and obtain the highest repeatability and accuracy of the sensorial measure was found in this study when using Teflon bags and carrying out the analysis always at the same elapsed time after the sampling phase and specifically within 14 h, that present the minimum odour concentration decay percentage.

Further studies are needed to confirm the obtained results on other sources types and especially to investigate the possible influence of other variables in the determination of odour concentration by dynamic olfactometry, such as the sampling air volume, the samples storage and transport methods.

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