

## Odour Emission from Liquid and Solid Area Sources: a Large Intercomparison of Sampling Devices

Jean-Michel Guillot<sup>a</sup>, Anne-Sophie Clincke<sup>b</sup>, Maud Guilleman<sup>c</sup>

<sup>a</sup> Ecole des Mines d'Alès, LGEI, 6 av. de Clavieres, 30319 Alès cedex, France

<sup>b</sup> INERIS, Parc technologique ALATA, BP 2, 60550 Verneuil-en-Halatte, France

<sup>c</sup> IRSN, PSN-RES/SCA/LEMAC, BP 68, 91192 Gif-sur-Yvette cedex, France

[jean-michel.guillot@mines-ales.fr](mailto:jean-michel.guillot@mines-ales.fr)

In this study, the objective was to compare different sampling devices used, on solid or liquid sources, to measure odour emission from passive sources. Experiments were firstly carried out on two real sources: a solid source made of compost and a liquid source corresponding to a basin containing leachates from the composting piles. In a second time, experiments with some selected sampling devices were also carried out with pilot units. For experiments on real sources, samples were coded before analysis in order to be anonymous for olfactometric measurement, based on EN13725 standard and performed by laboratories under French accreditation (COFRAC).

For laboratory test, some devices and their efficiency were tested on a liquid source and/or a solid source. The pilot unit for liquid source (at the site of Ecole des Mines d'Alès) is a 150L tank containing a solution of n-Butanol used as tracer. The pilot unit for solid source (at the site of INERIS) is sand square where the porosity of such a ground is used to diffuse Methane used as tracer. For both cases, direct analysis by FID was carried out joining the outlet of the device to the detector.

This study shows the high complexity of such a sampling because devices (chambers) give results in a large range with odor flow that are 10 times, 100 times higher or even more for some chambers comparatively to the lower result. Because, the source was the same, the Nalophan bags came from the same production, the period of sampling was identical and the same laboratory analyzed samples, the major factor of difference was typically the chamber itself because Olfactometry uncertainty cannot explain so huge differences.

The difference can be limited excluding chambers with high flow rate. In that case, because the odor flow is the factor of odor intensity and flow rate, this last one is very influent on the result. If a higher limit of flow rate seems obvious, a lower limit appears too, because low flow chamber give high concentration due to accumulation inside, and then low odor flow due to lower impact of the flow rate in calculation.

### 1. Introduction

Even if a source doesn't present an air flow, potential odorous compounds can be emitted typically when emissions are linked to a large area, the global impact can lead to odor annoyance. So, to characterize such area sources (opened tanks, evaporation lagoons, landfills, composting piles...), sampling devices were built to isolate a small part of the surface and then collect an air sample linked to the isolate part and the forced or extracted flow in the device. Area sources are typically difficult to characterize and sampling must be developed for such sources as for other critic points (Guillot, 2012). It is easy to see that the representativeness of the emission includes sampling on different points and the use of a device allowing the sample collection with the minimal perturbation of the emissive area. In the French Committee of Standardization (AFNOR), a group has worked on this topic in the frame of "Olfactometry commission". In order to increase the knowledge on the subject and to compare the different devices used, an inter-comparison study was decided. It was carried out with financial support of ADEME (French Agency for Environment and Energy Control) and with the participation and the investment of several teams

(laboratories, companies...). This aspect presents a large interest because few standardized protocols are available around the world for area source sampling. And, for standardized protocols, devices and their use present a lot of differences. This study was also guided by the fact that some previous study had shown high differences in measured odor flow with two different types of sampling devices. The originality of the present study is firstly to combine laboratory tests and measurement on industrial sources and secondly to compare approximately ten devices on a real source at the same time.

It is difficult to think that one chamber can be adapted to all situations and the application fields can be distinguished for low (isolation chamber type) or high flow rate (wind tunnel type). For both types, different designs have been developed even if an isolation chamber referenced by US-EPA is often used as flux chamber. For the case of wind tunnels, it's easy to find different devices depending of the laboratory and/or the country (Capelli, 2009; Leyris, 2005; Sohn, 2005). Typically, low flow rate chambers must be used for low emissions (low concentrations) like some cases of polluted soils or diluted tanks. With a low flow rate, pollutants can accumulate inside to have significant and measurable odor. In case of low emission, a high flow rate can dilute the odorous gas to values lower than 50-70 OU, cut off range to use dynamic olfactometry. Such chambers need longer equilibrium time than dynamic chambers and the sampling procedure must include this time. High flow chambers (wind tunnel types) are generally designed to simulate the wind action over a surface and then to represent real conditions. But due to the relative little size of wind tunnel (more or less 1m long), external conditions cannot be represented inside for high speed values (>3-5 m/s). So, such simulation is limited and the flow rate (or wind speed) must be fixed to be compatible with the chamber and its geometry. Studies have shown than results obtained from sampling with a flux chamber (low flow - isolation chamber type) and wind tunnel (high flow) could be very different (Bokowa, 2010; Hudson and Ayoko, 2009; Jiang and Kaye, 1996).

For low or high flow rate chambers, the emission rate and the odour flow are only estimated results because in both case, no warranty can be given for strictly equivalent emission conditions. When the same chamber is used and in the same conditions, area sources can be compared and their evolution too with relative value of odour flows. The comparison of results of low with high flow rate chamber must be prohibited because sampling conditions are too different. This study gives limitations linked to area sampling and restrictions to precise for analytical reports that include such sampling. Even for devices that are mentioned in a standard, it is necessary to precise that the results are not absolute values but relative ones and therefore emission values are always estimations highly dependent on sampling conditions.

## 2. Materials and methods

### 2.1 Participants and sampling devices

The standardization AFNOR olfactometry commission is composed by companies, laboratories, consultancies... A work group on area sampling is composed by some participants of the commission. These participants are (by alphabetic order): APAVE, AROMACONSULT, BURGEAP, CAP ENVIRONNEMENT, COVAIR, ECOLE DES MINES-ALES, EGIS, INERIS, IRH, IRSN, IRSTEA, KTT-IMA, SUEZ, TOTAL, VEOLIA.

The main characteristics of sampling devices are listed in Table 1 and Table 2 for chambers dedicated to sampling on solid and liquid sources respectively. All these sampling devices are shown on solid source (Figure 1) and liquid source (Figure 2). For all devices, floats are added around devices for liquid tests.

Table 1: Sampling devices for solid source (compost)

Number	Flow	Type	Air control
1	High flow	Wind tunnel with chicanes	Push
2	High flow	Linear wind tunnel	Push and pull
3	High flow	Linear wind tunnel	Pull
4	High flow	Ventilated box	Pull with pressure monitoring
5	High flow	Linear wind tunnel	Pull
6	Low flow	Parallelepipedic box	Pull
7	Low flow	Parallelepipedic box	Pull
8	Low flow	Cylindrical box	Pull

Table 2: Sampling devices for liquid source (leachates)

Number	Flow	Type	Air control
1	High flow	Wind tunnel with chicanes	Push
2	High flow	Linear wind tunnel	Push and pull
3	High flow	Linear wind tunnel	Pull
4	High flow	Box with chicanes	Pull
5	High flow	Linear wind tunnel	Pull
6	Low flow	Linear wind tunnel	Pull
7	Low flow	Parallelepipedic box	Pull
8	Low flow	Parallelepipedic box	Pull
9	Low flow	Flux chamber	Push

Devices number 1 and 2 are strictly identical for both solid and liquid tests. Devices number 3 and 5 are identical in size for both solid and liquid tests but materials are different.

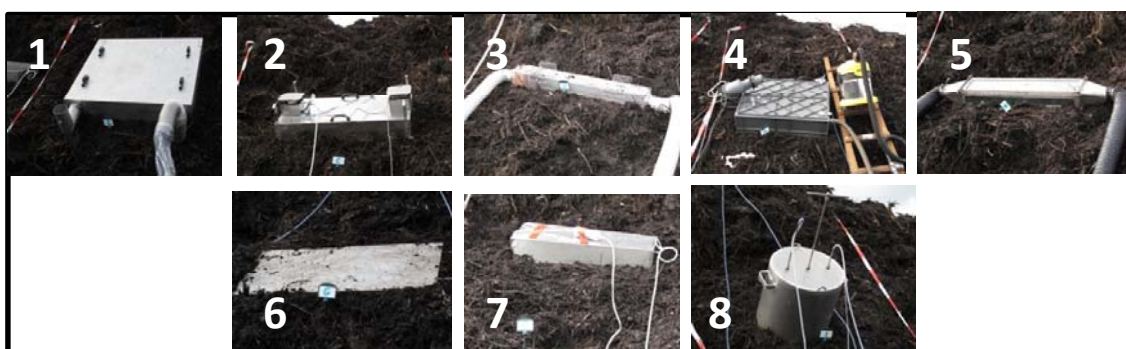


Figure 1: Pictures of the eight sampling devices used on solid source (composting pile). High flow tunnels are numbered from 1 to 5 and low flow chambers from 6 to 8.

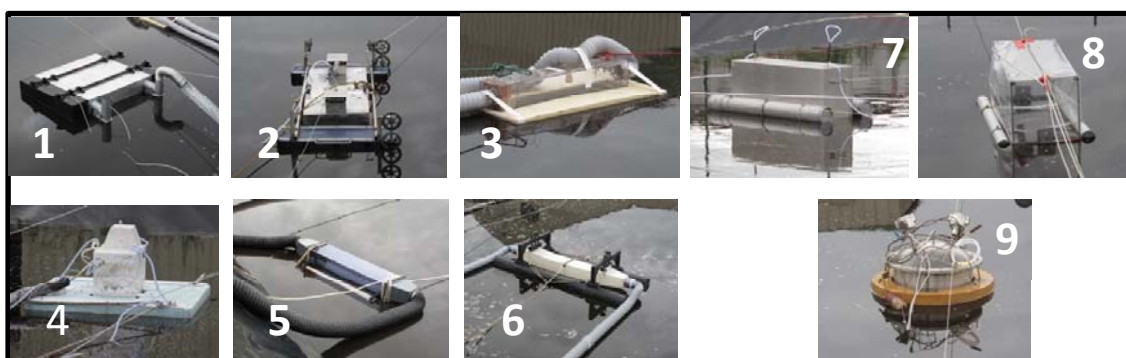


Figure 2: Pictures of the nine sampling devices used on liquid source (leachates). High flow tunnels/chambers are numbered from 1 to 6 and low flow chambers from 7 to 9.

## 2.2 Experiments on site

Experiments on site were carried out in October in the north of France with the participation of AROMACONSULT, BURGEAP, ECOLE DES MINES-ALES, EGIS, INERIS, IRH, IRSN, IRSTEA, TOTAL, VEOLIA. All teams used their own sampling device with their own equipment (pump, vacuum box...). For sampling on real conditions, experiments were carried out three times for both sources; In order to warranty the compost homogeneity, the composting pile was previously characterized and analyzed to

define equivalent location for all devices. Because liquid heterogeneity is more limited, such a procedure of characterization was not developed for the liquid source.

Nalophan bags (from the same supplier) were given to all participants by the laboratory in charge of olfactometric measurements. Sampling duration time for bags was fixed at 10 minutes with the same start for all devices. The experiments were carried during two consecutive days and the three different samples (3 tests) for each source were chosen to cover different period of a day (morning, mid-day and afternoon) as show in Table 3. Collected bags were coded before olfactometric measurement in order to have anonymous samples without potential link between results and sampling device for people in charge of olfactometry. Two accredited companies were selected for olfactometric measurement. One carried out transport measurements on solid source samples when the second carried out transport and measurements on liquid source samples. All samples were analyzed according to the maximum time storage of 30h fixed by EN13725.

Table 3: Sampling period on solid and liquid sources

Day	Sampling hour	Solid source	Liquid source
1	10 am	Test S1	
1	12 am	Test S2	
1	4 pm		Test L1
2	10 am		Test L2
2	12 am		Test L3
2	3 pm	Test S3	

During sampling, some parameters were analysed by INERIS by analysers placed in a mobile laboratory. It concerned Total hydrocarbons, ammonia and total reduced sulphur compounds.

### 2.3 Experiments on synthetic source at pilot scale

For experiments at pilot scale in laboratories, sources were as follows :

- The solid source (INERIS site) was made with a tank filled with sand and a methane flow through the sand simulates the gas emission.
- The liquid source (Ecole des Mines d'Alès site) was made with a 150L tank containing a solution of n-butanol in water. Some chambers cannot be tested on this pilot because of the size limitation.

For both pilot units, measurements were carried out with direct FID after calibration of analytical equipment. Such analysis avoids a sampling step and gives directly the concentration of tracer (methane or n-butanol).

## 3. Results and discussion

### 3.1 Experiments with real solid source on site

For results on solid source shown on Figure 3, odour concentrations are close for the different tests. This figure illustrates a relative stability of the source and sampling repeatability with more or less all chambers. Comparing chambers, differences are generally lower than a factor 10 and are due to the conception and sampling conditions. The odour concentration is partially correlated with some chemical data such as total hydrocarbons and ammonia but no link was observed between sulphur compounds and odour. This fact seems logical for a composting pile source.

When the flow is integrated to calculate the odour flow, differences are sometimes 100 times or even 1000 times between lower and higher result. So differences increase instead of decrease. It could confirm that low flow chambers underestimate the emissions.

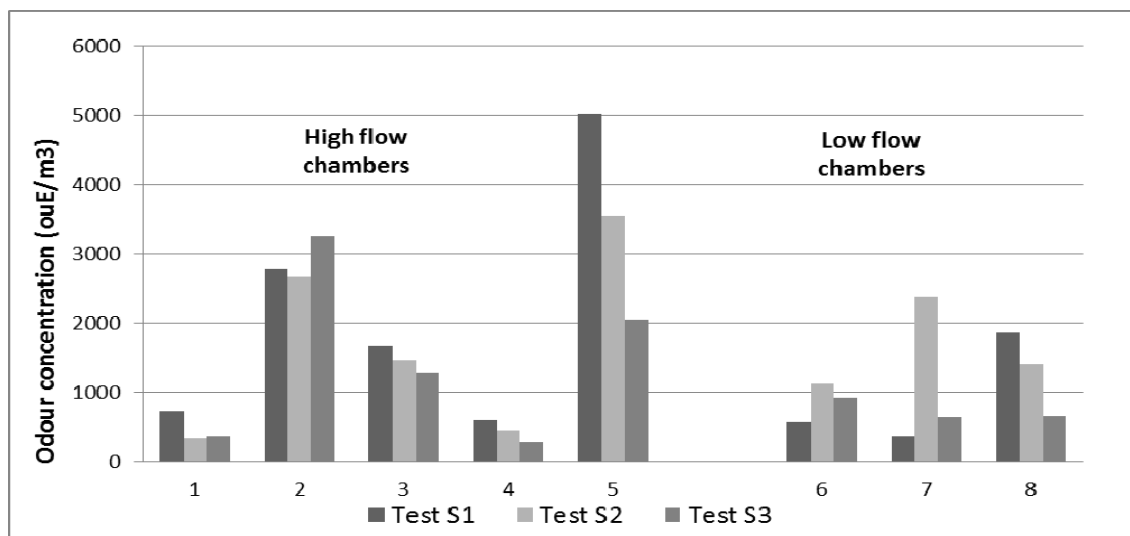


Figure 3: Odour concentration for the 3 tests on solid source and for devices 1 to 8 (described in Table 1 and Figure 1)

### 3.2 Experiments with real liquid source on site

For results on liquid source shown on Figure 4, odour concentrations are spread in a wide range and some values are not obtained due to values lower than 50 OU/m<sup>3</sup>, the limit fixed value for olfactometric measurement. It demonstrates the limit of flow increase and the resulting strong dilution of the emission. It can be noticed that low flow chambers are globally more stable than dynamic devices with lower variations between the 3 tests. Devices 1 and 2 with moderate flows seem also relatively stable.

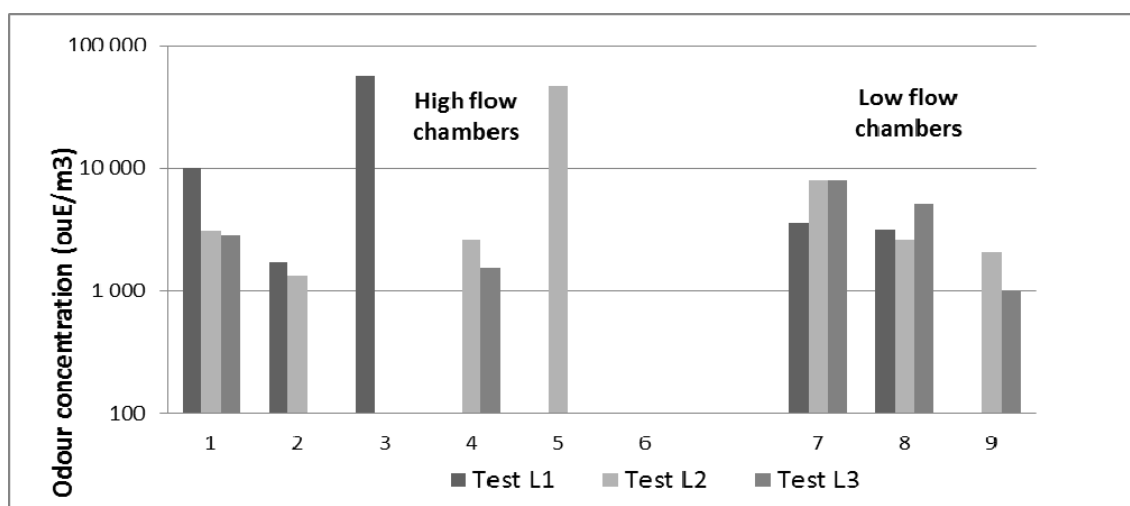


Figure 4: Odour concentration for the 3 tests on liquid source and for devices 1 to 9 (described in Table 2 and Figure 2)

### 3.3 Experiments on synthetic solid source at pilot scale

Chambers 1 to 7, previously tested on the compost, were also tested on the pilot with four new chambers numbered from 10 to 13. Results are given in table 4. Specific flows are obtained in a range from 1 to 10 showing that even in a controlled source, the choice of the sampling device induce the result. Some chambers are more "stable" with lower variation.

*Table 4: Specific flow measured with different devices on the synthetic solid source (Average and Standard deviation are based on 6 experiments.*

Chambers	Specific flow (average) mg/min/m <sup>2</sup>	Standard deviation	Standard deviation %
1	12.70	0.32	2.50
2	27	1.41	8.79
3	54.63	0.35	11.82
4	12.81	0.34	2.64
5	23.90	3.73	15.62
6	9.74	0.35	3.58
7	8.06	1.41	17.49
10	5.14	1.56	30.28
11	6.40	0.35	3.44
12	9.57	1.41	2.38
13	8.21	0.35	4.14

### 3.4 Experiments on synthetic liquid source at pilot scale

On the synthetic source, low flow chambers don't seem adapted for concentrated source (high saturation of atmosphere inside the chamber. In that case, dynamic are more adapted but concentration cannot be determined before experiment in real experiments.

## 4. Conclusions

This study confirms the great influence of the sampling device on the final results. If different studies previously reported differences between wind tunnel and flux chamber types, these new results show that the difference could be higher than expected. So, It must be considered that odour emission from a static area source cannot be compared to another if sampling conditions are not exactly the same. Of course, if two sampling devices are similar and more or less used in the same conditions, a comparison can be done with all limitations due to both sampling devices.

## Acknowledgments

The authors thank all participants of the AFNOR commission on olfactometry and especially members of the work group on sampling area for their participation to the elaboration of the inter-comparison program and their investment to test their devices. The authors and participants thank French Agency (ADEME) to financially support program by giving the possibility to organise meetings, tests and cost of analyses linked to the inter-comparison. Of course, all participants on the site thank the company that allowed the access to its composting plant for both solid and liquid sources.

## References

- Bokowa A., 2010, The effect of sampling on the measured odour concentration, *Chemical Engineering Transactions*, 23, 43-48, DOI: 10.3303/CET1023008
- Capelli L., Sironi S., Del Rosso R., Centola P., 2009, Design and validation of a wind tunnel system for odour sampling on liquid area sources, *Water Sci. Technol.* 59, 1611–1620.
- Guillot J.M., 2012, Odour measurement: Focus on main remaining limits due to sampling, *Chemical Engineering Transactions*, 30, 295-300, DOI: 10.3303/CET1230050
- Hudson N., Ayoko G.A., 2009, Comparison of emission rate values for odour and odorous chemicals derived from two sampling devices, *Atmos. Environ.* 43, 3175–3181.
- Jiang K., Kaye R., 1996, Comparison study on portable wind tunnel system and isolation chamber for determination of VOCs from areal sources, *Water Sci. Technol.* 34, 583–589.
- Leyris C., Guillot J.M., Fanlo J.L., Pourtier L., 2005, Comparison and development of dynamic flux chambers to determine odorous compound emission rates from area sources. *Chemosphere* 59, 415–421.
- Sohn, J.H., Smith, R.J., Hudson, N.A., Choi, H.L., 2005, Gas sampling efficiencies and aerodynamic characteristics of a laboratory wind tunnel for odour measurement. *Biosyst. Eng.* 92, 37–46.