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Odour Measurement in Wastewater Treatment Plant by a New Prototype of e.Nose: Correlation and Comparison Study With Reference to both European and Japanese Approaches

Vincenzo Naddeo[a], Tiziano Zarra[a], Giusy Oliva[a], Akira Kubo[b], Naoia Ukida[b], Takaia Higuchi[b]

[a]Sanitary Environmental Engineering Division (SEED), Department of Civil Engineering of the University of Salerno, via Giovanni Paolo II,132, 84084, Fisciano (SA), Italy
[b]Graduate School of Science and Engineering, Yamaguchi University. 2-16-1, Tokiwadai, Ube, Yamaguchi 755-8611, Japan
uO18vn@yamaguchi-u.ac.jp

In the modern world of wastewater treatment, control of odors has moved from an afterthought to a primary design consideration for most collection and treatment facilities. As development encroaches on our facilities and our new neighbours become less tolerant of nuisance odors, wastewater professionals have found the need to address odor as a primary concern in the design and operation of collection and treatment facilities.
When facility odors affect air quality and cause citizen complaints, an investigation of those odors may require that specific odorants be measured and that odorous air be measured using standardized scientific methods. Odour intensity is one of main odour characterization parameters, and remarkably common and important sensory indicator of environmental odours. Odour intensity reflects people's perception of odours and contributes to effective odour management.
The "triangle odors bag method', which has been adopted for the offensive odors control law in Japan, and the dynamic olfactometry defined by EN 13725 has been adopted in Europe. These methods are the worldwide used for as reference methods for the sensorial measurement of odours. On these methods are also based the training of electronic nose (e.Nose).
In this study odour samples were collected on municipal wastewater treatment plant located in south Italy at four different treatment units to determine the relationship between odour intensity assessed by Japanese "triangle odors bag method', odour concentration measured with dynamic olfactometry according to European standard EN13725:2003 and with odour concentration measured by a novel prototype of e.Nose patented by University of Salerno. A monthly sampling and relative measurements were carried out for consecutive 12 months at Laboratory of Sanitary Environmental Engineering Division (SEED) at University of Salerno (Italy).
Results show when high odours were detected by Japanese standard, the odour concentrations measured according European standard were also high (e.g. emission from sludge line). Concentrations measured by e.Nose are strongly correlated with the measurements carried out by standard methods.

1. Introduction

Wastewater treatment plants (WWTPs) are considered one of the main odour sources in urbanized area (Naddeo et al., 2012; Ueno et al., 2009; Zarra et al., 2008). In recent years, there have been new odour assessment tools available to help municipal WWTPs understand their off-site odour impact, such as dispersion modelling, portable odour measurement panel equipment, electronic noses, air tracer compounds, and odour sensors that can be used to measure ambient odours at the plant fenceline. However, perception of odours is highly subjective, individuals might have different responses to an odour at various concentration and duration.
In Europe, odour analyses are performed by introducing an odour sample to screened panel members using dynamic olfactometry (Zarra et al., 2012). There are currently several different methods for dynamic olfactometry analysis that are universally used. In several countries from Europe (EN13725: 2003) to North

America (USA ASTM 679-04: 2011), including Australia and New Zealand (AS/NZS4323:2001), there are standardised methods that are commonly used for dynamic olfactometry (DO) analysis (Bokowa et al., 2014; Dincer et al., 2006; Zarra et al, 2008). All of these methods use a decreasing dilution series to determine an odour detection threshold value (Bokowa et al., 2014).
On the other hand, in several Asian countries, an increasing dilution series is used for odour evaluations. The triangle odour bag method (TOBM) is an olfactory method to measure odour concentration, which has been adopted for the offensive odour control law in Japan described in the “Odour Index Regulation and Triangular Odour Bag Method” and the document: GB/T14675-93 guideline.
Mixed techniques use sensorial/analytic approach and propose new instrument that are able to simulate human sense (electronic nose - eNose). eNose has the potentiality to combine “the odour perception” and the continuous “field monitoring”. The instrument, based on non-specific gas chemical sensor arrays combined with a chemometric processing tool, provides a suitable technique for in site monitoring of odours (Nicolas et al., 2004; Capelli et al., 2008; Giuliani et al., 2012).
The electronic nose has probably the best potentialities to answer to the expectations of the various actors of the environmental issues in relation with the odours annoyance (Belgiorno et al., 2012). eNoses has long been used for a number of different applications from food to cosmetic industries to quality control (Barsan et al., 1999). However, a number of limitations in environmental field are associated with the properties of chemical sensors, the signal processing performances and the real operating conditions of the environmental field (Lannto et al., 1988).
These methods have the potentiality to combine “the odour perception” and the “field monitoring”. The instrument, based on non-specific gas chemical sensor arrays combined with a chemometric processing tool (Gardner et al., 1994) are nowadays the only applicable technique to measure odours continuously at sources and/or at the receptors (Romain et al., 2008). It has probably the best potentialities to answer to the expectations of the various actors of the environmental problems in relation to odours annoyance (Romain et al., 2008). The classification of the odours is based on the comparison of the e-nose signals with a database of patterns acquired by the instrument in a previous training phase. The training of the electronic nose is one of the most important phases (Capelli et al., 2008, Giuliani et al., 2012). In addition, a number of limitations in environmental sector application are associated with the properties of chemical sensors adopted for the e-nose function (Barsan et al., 2007), the signal processing performances (Dutta et al., 2013) and the standardization of the real operating conditions (Giuliani et al., 2012, Giuliani et al, 2013).
The classification of the odours is based on the comparison of the e-nose signals with a database of patterns acquired by the instrument in a previous training phase. The training of the electronic nose is a very important and delicate phase (Capelli et al., 2008, Giuliani et al., 2012).
In this paper, the relationship between odour concentrations emitted by WWTP assessed by Japanese standard methods and odour concentration measured with dynamic olfactometry according to European standard EN13725:2003 are discussed and compared to the measurement carried out by novel prototip of e.Nose patented by University of Salerno.

2. Materials and methods

2.1 Sampling program

Research studies were carried out at conventional WWTP designed for 700.000 PE, located in the industrial area of the municipality of Salerno (Italy). To investigate the correlation between the three methods were selected four treatment units (Figure 1) of the plant which present the most significant odour emissions according to previous studies (Zarra et al., 2008): grit channel (P1), primary sedimentation (P2), aeration tank (P3), sludge conditioning (P4).
Air samples were collected one a month for eight consecutive months at each sampling point. A total of 32 samples were collected over the research period. For each month, all four samples were taken during the same day in stable meteorological conditions with not significant wind speed. During the sampling program the WWTP was operating with an average daily flow of 8000 m3/h.
Air samples were collected according to the methods recognized by the technical-scientific literature and 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. Nalophan® sampling bags with 20 liters volume are used for the sampling.

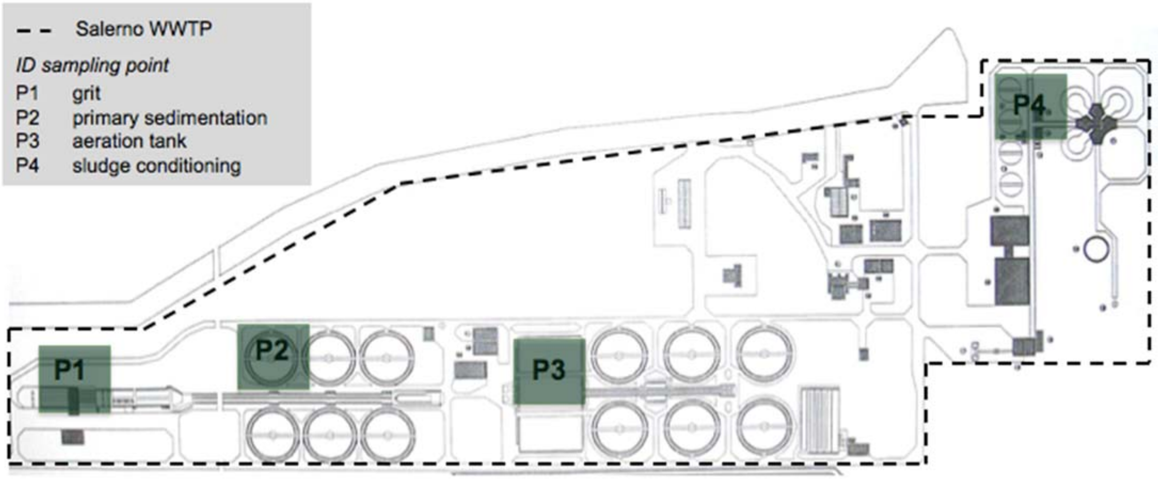

Figure 1: Wastewater treatment plant of Salerno (Italy) with localization of sampling points.

2.2 Odour Measurement

Air samples, collected during the sampling program at WWTP, were characterized by both dynamic olfactometry (DO) and triangular odour bag methods (TOBM) at the SEED Laboratory of the University of Salerno.

DO analyses were conducted using the dynamic olfactometer TO8 (ECOMA, GmbH) with the ‘‘yes/no’’ method for the measurement of Odour Concentrations (Cod).

Odour concentrations (Cod) were also measured by the triangle odour bag method according to Japanese offensive odour control law.

Odour measurements were carried out by the same group of panelists, in order to minimize any deviation caused by other factors than the test methods.

Odour measurements were also performed with the multisensor array system seedOA (Sanitary Environmental Electronic Device for Odour Application) according to procedure proposed by Giuliani et al (2012). The seedOA consists in a set of 12 metal oxides non-specific gas sensors (MOS), 2 specific gas sensors and 2 internal conditions sensors (humidity and temperature), placed on two different levels of a innovative measurement chamber (CODE®) (Viccione et al. 2012) patented by the SEED research group of the University of Salerno. All the used sensors (TGS, Figaro, F) were chosen according to their suitability for sanitary environmental engineering applications. Working flow rate was settled equal to 300 ml/min. A specific software controls the hardware and allows the acquisition of the sensor signals.

To train the seedOA and create the quantitative model, were collected from each of the investigated sources, before starting the monitoring program, five samples. Each sample was diluted according to the ratio 1:2, 1:10, 1:25 and analyzed by both seedOA and dynamic olfactometry. All the acquired data are then processed by the Linear Discriminant Analysis (LDA) and the Partial Least Squares regression (PLS) statistical mathematical method to define the quantitative model.

The results were compared in terms of Odour Index (OI), calculated with the following equation:

$$OI = 10 \, Log(Cod)$$

3. Results and discussion

Variability of odour index measured with both methods at each investigated sampling point of WWTP is represented with a Box-Whisker Plots (Figure 2). Considering all measurement carried out at WWTP, the results shown greater variability of DO versus TOBM respectively with odour index ranged from 13,8 to 40,0 for DO and from to 18,4 to 40,1 for TOBM. eNose resultrs are in the middle.

According to the literature, the results show that odour concentrations emitted by units of the sludge line (P4) are significantly highest of the odour emitted from the unit in the wastewater line. In detail the highest concentration was detected at the P4 while the lowest in the oxidation unit (P3).

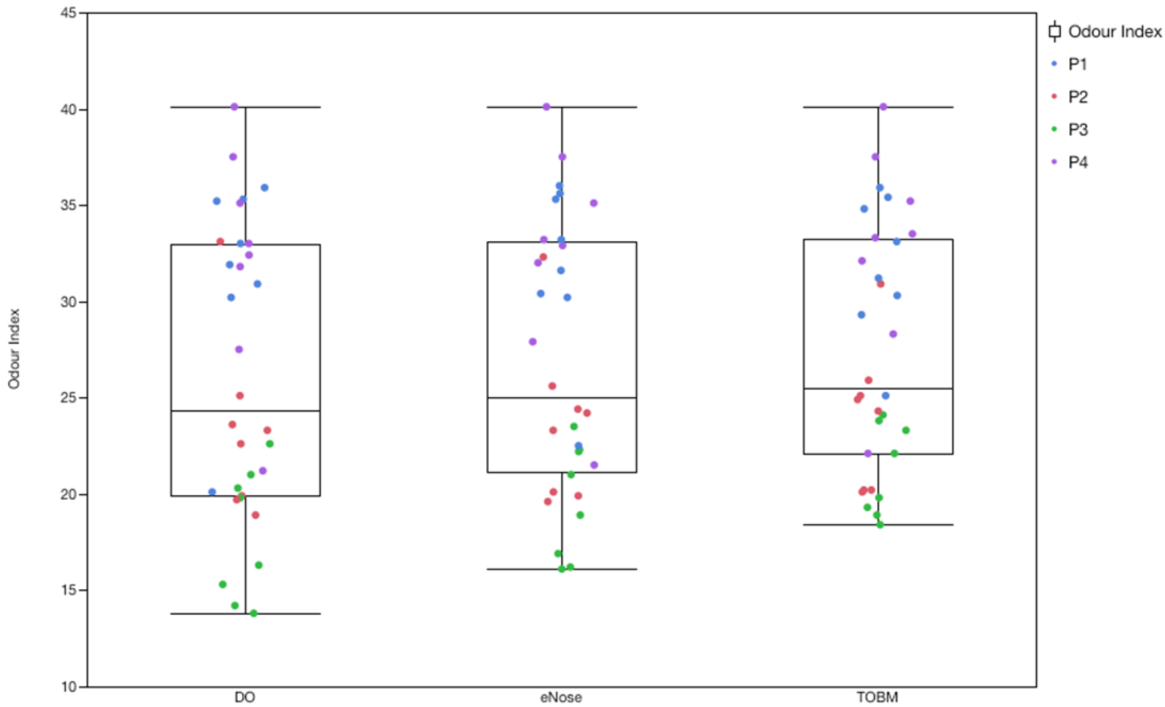

Figure 2: Box-Whisker diagrams of odour index measured at WWTP with all investigated methods.

P2 and P3 are characterized by lower concentration of odour in all methods and for these investigated units the results highlight a major divergence between the methods. The same methods show a better match of the measures for the samples from sources P1 and P4, characterized with an odour index generally greater than 25 (Figure 3).

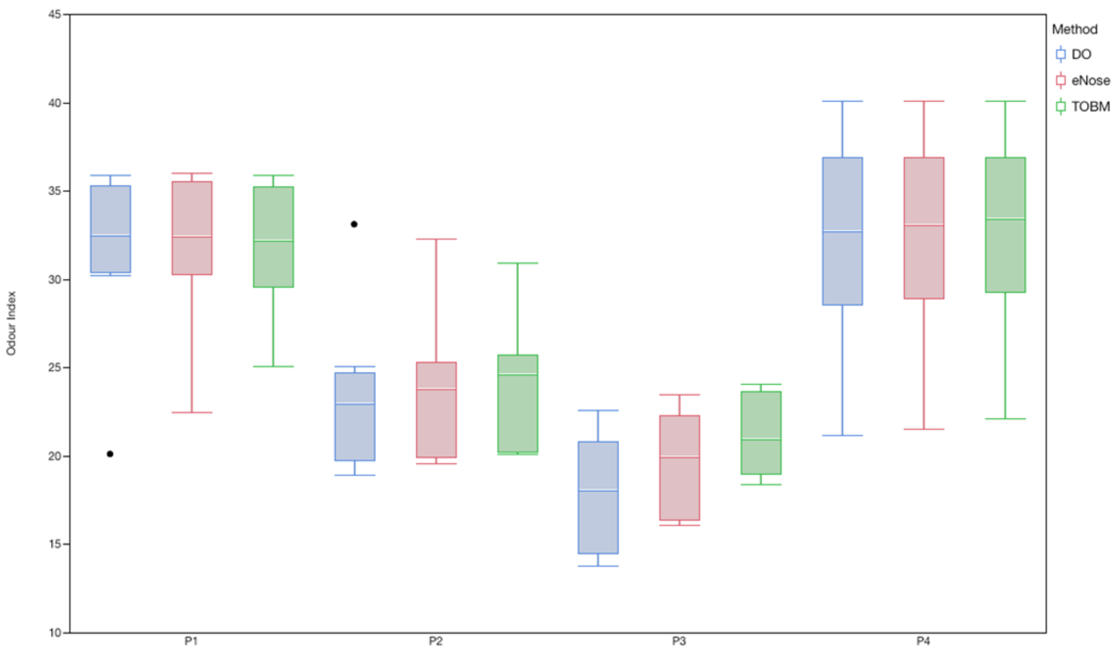

Figure 3: Box-Whisker diagrams of odour index for each sampling point detected by all investigated methods.

Comparing the results obtained by both methods (Figure 3), a strong correlation (R^2>0,987) was observed. However, as observed by analysing the individual sampling points, the correlation between the TOBM and the DO is lower for samples with concentrations less than 100 OU/m^3 (corresponding to 20 odour index).

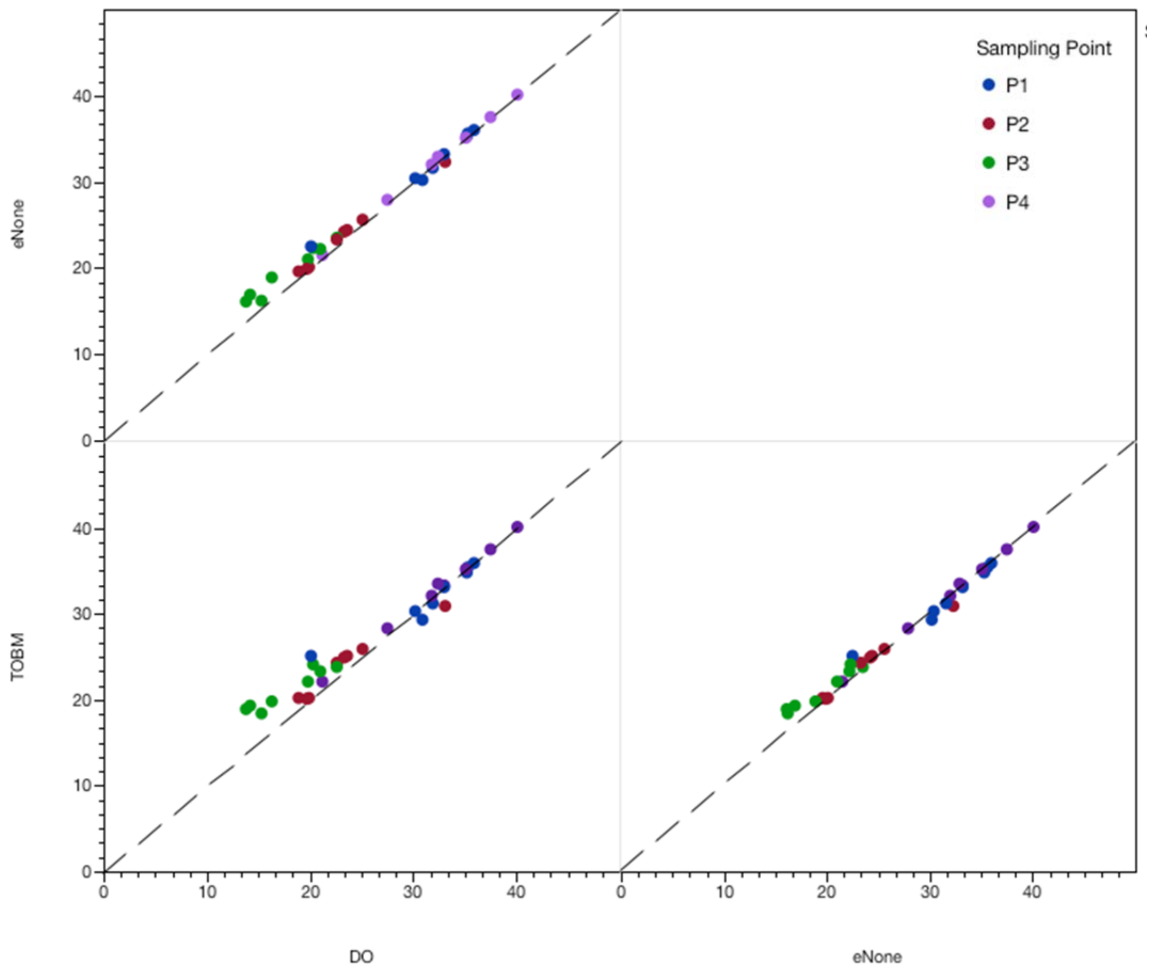

Figure 3: Correlation between the Odour Index (OI) measured by Dinamic Olfactometry (DO), Triangular Odour Beg Method (TOBM) and electronic Nose (e.Nose).

For samples with lower concentration, the dynamic olfactometry highlight less affability and repeatability of measurements. While there are not significant differences between the DO and the TOBM with odour concentration greater than 30 odour index (corresponding to 1000 OU/m3). On other way e.Nose measurement are perfectly in line with sensorial measurements.

4. Conclusions

Odour concentrations detected from air samples analyzed by the triangle odour bag method (TOBM) show comparable results respect of samples measured by the dynamic olfactometry (DO) for the sources with higher concentration.

Dynamic olfactometry highlight less affability and repeatability of measurements for samples with lower concentrations, versus the TOBM method.

However the implementation of the TOBM method require longer analysis times for the preparation and for the number of the samples to analyse. On other way the results show that electronic nose (e.Nose) is valid alternative for a quickly and affidable measurement of odours.

The novel e.Nose device (seedOA) proposed for the control of odour emission in WWTPs, showed hight performances compared to standards methods.

The results of the laboratory investigation have shown the great efficiency of the multisensor array system seedOA to detect odours emitted by different treatment units.

The comparison with Triangular bag methods and dynamic olfactometry confirmed that the multisensor array system is able to identify and quantify the odours emitted by the Salerno WWTP with a minimum margin of error, specially at lower concentration. This aspect is not critical due to the lower concentration not causing significant odour impacts.

Future studies on the procedure for the panellist selections, on the economic and human resources necessary for the analysis are need for a complete comparison.

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References

ASTM 679-04, Standard Practice for Determination of Odor and Taste Thresholds by a Forced Choice Ascending Concentration Series Method of Limits.

Australian/New Zealand AS/NZS4323:2001 Standard (2001), Stationary Source Emissions. Part 3: Determination of Odour Concentration by Dynamic Olfactometry.

Barsan, N., Schweizer-Berberich, M. and Gopel, W. (1999) Fundamental and practical aspects in the design of nanoscaled SnO2 gas sensors. A status report, Fresen. J. Anal. Chem., 365, 287–304.

Barsan N., Koziej D., Weimar U. (2007) Metal oxide-based gas sensor research: how to? Sensors and Actuators 121 (1) 18–35.

Belgiorno, V., Naddeo, V., Zarra, T., 2012, Odour Impact Assessment Handbook, Wiley & Son. London. ISBN: 978-1-119-96928-0

Bokowa A. H., Bokowa M. (2014), Comparing the Accuracy of Three Odour Analysis Techniques Used in Europe, North America, Australia, New Zealand and Asia, Chemical engineering transactions, 40, 1-6. doi: 10.3303/cet 1440001.

Capelli L., Sironi S., Céntola P., Del Rosso R., Il Grande M., 2008. Electronic noses for the continuous monitoring of odours from a wastewater treatment plant at specific receptors: Focus on training methods. Sensors and Actuators B 131, 53–62.

Dincer F. and Muezzinouglu A. (2006), Chemical characterization of odors due to some industrial and urban facilities in Izmir, Turkey, Atmospheric Environment 40(22), 4210-4219.

Dutta N., Bhuyan M. (2013) Instruments and Methods for Odour Sampling and Measurement in Odour Impact Assessment Handbook. Di. 63-83, John Wiley & Sons, Inc.. ISBN: 978-1-119-96928-0

Giuliani, S., Zarra, T., Nicolas, J., Naddeo, V., Belgiorno, V., Romain, A.C., 2012, An alternative approach of the e-nose training phase in odour impact assessment, Chemical Engineering Transactions, 30, pp. 139-144

Giuliani S., Zarra, T., Naddeo, V., Belgiorno, V. (2013). Measurement of odour emission capacity in wastewater treatment plants by multisensor array system. Environmental Engineering and Management Journal, vol. 12, 173-176

Lantto, V. and Romppainen, P. (1988) Response of some SnO2 gas sensors to H2S after quick cooling, J. Electrochem. Soc., 135, 2550–2556.

Naddeo V., Zarra T., Giuliani S., Belgiorno V., 2012, Odour Impact Assessment in Industrial Areas, Chemical Engineering Transactions, 30, pp. 85-90

Nicolas J., Romain A. C., 2004. Establishing the limit of detection and the resolution limits of odourous sources in the environment for an array of metal oxide gas sensors. Sensors and Actuators B, 99, 384–392

Romain A.-C., Delva J., Nicolas J. (2008) Complementary approaches to measure environmental odours emitted by landfill areas, Sensors and Actuators B: 131 (1), 18–23

Ueno H., Amano S., Merecka B., Kośmider J. (2009), Difference in the odor concentrations measured by the triangle odor bag method and dynamic olfactometry, Water Sci Technol. 59(7), 1339-1342.

Viccione, G., Zarra, T., Giuliani, S., Naddeo, V., Belgiorno, V. 2012, Performance study of e-nose measurement chamber for environmental odour monitoring. Chemical Engineering Transactions, 30, pp. 109-114.

Zarra T., Naddeo V., Belgiorno V., Reiser M. and Kranert M. (2008), Odour monitoring of small wastewater treatment plant located in sensitive environment. Water Sci. Technol. 58 (1), 89-94.

Zarra, T., Reiser, M., Naddeo, V., Belgiorno, V., Kranert, M., 2012, A comparative and critical evaluation of different sampling materials in the measurement of odour concentration by dynamic olfactometry, Chemical Engineering Transactions, 30, pp. 307-312.