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MARKET REVIEW ON AVAILABLE INSTRUMENTS FOR ODOUR MEASUREMENT

DELIVERABLE 1.2

Project acronym: ODOCO-ARTNOSE

by
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Title
Market Review on Available Instruments for Odour Measurement

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Abstract

Continuous and quasi-continuous odour monitoring solutions have the potential to provide essential tools to support the whole odour control procedure in sewer networks. Hence, there is a need for continuous measurement and supervision of odour emissions with technical measurement systems. Objective of this investigation is the identification of instruments on the market which have the potential to be applied for odour monitoring from wastewater collection systems or wastewater treatment works.

Generally one can distinguish between following methods of odour measurement:

- Sensory methods:

  Measurement of odour concentration by olfactometry (evaluation by human noses)

- Analytical methods:

  a. Selected sensors: Measurement of specific single odorants or surrogate parameters (e.g. H_2S-measurement)

  b. Gas chromatography, mass spectrometry, optical sensors: Measurement or quantification of a spectrum of several gas components

  c. Multigas-sensor arrays: Measurement of overall odour parameters by means of unspecific, broadband multigas-sensor arrays

Only the mentioned analytical methods provide the possibility of continuous measurements. They however do not all consider the sensory component of odour (perceived effect).

Within this report methods b. and c. will be covered. The report provides an introduction to the principle of measurement, briefly discussing examples of sampling methods and data analysis methods and gives lists of collected odour monitoring systems, tabulary providing specifications from literature, manufactures and vendors.

Table of Contents

List of Figures

List of Tables

List of abbreviations

amu	Atomic mass unit
ANN	Artificial neural networks
BAW	Bulk acoustic wave sensor
BTEX-analysis	Acronym for benzene, toluene, ethyl benzene, and xylenes
CCD	Charge-coupled device
CMOS	Complementary metal oxide semiconductor
CO	Carbon monoxide
CPS	Conducting polymer sensor
DFA	Discriminant function analysis
DMS	Differential mobility spectrometry
EC	Electrochemical sensor
FAIMS	Field asymmetric ion mobility spectrometry
FID	Flame Ionisation Detector
GC	Gas Chromatography
GC-MS-O	Olfactory Gas Chromatography
GC-MS-Sniffer	Olfactory Gas Chromatography
GPRS	General Packet Radio Service
GPS	Global positioning system
H_2	Hydrogen
H_2S	Hydrogen sulphide
He	Helium
IMS	Ion-mobility spectrometry
IR	Infrared sensor
k€	Thousand €
LDA	Linear discriminant analysis
LOD	Limit of detection
MOS	Metal oxide semiconductor
MS	Mass Spectrometry
N_2	Nitrogen
NH_3	Ammonia
NN	Neural networks
NOx	Nitrogen oxides
ou/m³	Odour unit per cubic meter
PAS	Photo acoustic Spectroscopy
PCA	Principle Component Analysis
PID	Photoionization detector
PLS	Partial Least Squares

ppb .... Parts per billion

ppm .... Parts per million

QMB .... Quartz crystal microbalance

RBFN .... Radial basis function network (neural networks)

rH .... Relative humidity

R-SH .... Thiol, mercaptan (organosulfur compound)

SAW .... Surface acoustic wave sensor

SO_2 .... Sulphur dioxide

SVM .... Support Vector Machine (for Classification)

VOC .... Volatile organic compounds

WWTP .... Wastewater treatment plant

Chapter 1

Introduction

1.1 Background

Developments during the last 10 years, such as a demographic changes, decrease in specific and industrial water consumption, novel sanitary systems, or the renovation of leaky drainage channels to avoid infiltration are connected with a decline in dry weather flow by trend within sewer systems. This decline can be observed mainly in densely populated areas, in areas with high migration into cities or where the sewer channels were dimensioned for a higher wastewater flow (e.g. shut down of a high number of industries in the 90s in Germany) (Lohse, 2010). Additionally different effects of climate change, like a shift in precipitation pattern, sea level rise, and especially the increase in temperature, have impacts on in-sewer processes. Generally all catchment areas (rural/urban areas; new/old constructions) can be affected by odour formation particularly during the summer months and when there are unfavourable conditions of the sewer system geometry such as long flow paths or low declines of channels (Frey, 2011). The consequences are long retention times of the wastewater, anaerobic conditions, corrosion of concrete pipes and shafts and increased odour formation in the sewer system which can result in intensive odour emissions to be released into the streets. Together with a decreasing public tolerance and growing public concern over odours from wastewater treatment works, odour releases from these facilities have led to increasing numbers of odour complaints. Operation costs for permanent odour countermeasures cause elevated financial burden for utilities (Stuetz & Frechen, 2001; ATV-DVWK-M 154, 2003; Barjenbruch, 2003; Weismann & Lohse, 2007).

There are a variety of methods to avoid, reduce or control odour problems from sewer systems. Measures can generally be applied in a preventive manner (e.g. constructional measures for ventilation, cleaning of channels, preventive additive dosing) or reactive after complaints (e.g. local additive dosing, air treatment). A catalogue of odour control measures has been compiled within the project ODOCO-ARTNOSE (Deliverable 1.1) (Schwarzböck, 2012). A successful odour abatement measure however requires optimal odour management including the identification of the odour origin, identification of the problem (e.g. problem of hydrogen sulphide or other dominating odorants) or the control of the efficiency of the measures.

An optimum odour management requires reliable data relating to the specific character and intensity of emerging odours. Hence, there is a need for continuous or quasi-continuous measurements and supervision of odour emissions with technical measurement systems. Continuous and quasi-continuous odour monitoring solutions have the potential to provide essential tools to support the whole odour management procedure in sewer networks. This procedure comprises identification and argumentation of odour origins, planning of abatement measures, control of dosages and the efficiency thereof, to the documentation of the whole process and, as the case maybe, demonstration of legal compliance.

Currently odour measurements rely on the use of olfactometric analysis (human panels) or on measuring surrogate parameters such as hydrogen sulphide (H_2S). However,

major drawbacks of these methodologies are the partly subjective interpretation of odour (olfactometry), the unsuitability for continuous measurements (olfactometry) or the incapability of representing the full odour spectrum (H_2S measurements) (Stuetz *et al.*, 2000).

Potentials for delivering dynamic data directly related to the parameter odour are technical measurement systems based on multigas-sensor arrays (so called electronic noses). These types of systems are already applied in the food industry, for safety control or for medical purposes. Multigas-sensor systems are gaining in importance for the online measurement of odours – compared to the discontinuous and time-consuming olfactometric analysis. Hence, they give the possibility to establish a continuous monitoring at points of interests (e.g. for the documentation of the odour dispersion and complaint management at wastewater treatment plants or at industrial sites). Practical applications of electronic noses in environmental monitoring have been described by Boeker & Horner, 2004; Guy *et al.*, 2004; Münchmeyer & Walte, 2004; Romain *et al.*, 2005; Franke *et al.*, 2009; Bootsma & Milan, 2010. Electronic noses are the main focus within this report.

The detection of single air constituent by analytical methods like gas chromatography also are considered for online odour measurements, they however do not consider the odour perception or interaction of odorants in the gas complex and can be very cost-intensive.

1.2 Objectives

- Analytical methods for odour measurements are subject of this report that aims at providing a compilation of identified systems on the market for odour monitoring from wastewater systems which are able to deliver continuous or quasi-continuous data and

- giving their specifications; e.g. on their measurement principles, target application fields, sample preparation methods and possible new innovations

Special focus of the report are multigas-sensor systems as they are gaining in importance for online monitoring of odours and are subject of the research project ODOCO-ARTNOSE at the Kompetenzzentrum Wasser Berlin (KWB).

The compilation is based on literature reviews, internet research, talks with vendors or personal experiences.

Specifications on the systems have been primarily provided by the vendors by data sheets or personal messages.

Based on this market review multigas-sensor systems have been selected within the KWB-project ODOCO-ARTNOSE and are tested for 6 months by means of a sewer research plant of Berliner Wasserbetriebe (large scale pilot).

1.3 Odour formation in sewers

Odorous emissions from sewers and wastewater treatment plants are a complex mixture with a large number of chemical compounds, including a variety of sulphur-compounds, nitrogen-compounds, acids, aldehydes, ketones, alkanes, chlorinated and aromatic hydrocarbons (examples see *Table 1-1*).

Generally one can distinguish between primary and secondary odours/osmogenes from wastewater (Frey, 2008; Koppe & Stozek, 1999):

- Primary osmogenes: are discharged into the sewer system directly and are present as dissolved molecules, such as

  - ammoniac, fatty acids, skatoles in municipal wastewater (e.g. from feces, urine, chemicals) and

  - amine, aldehyde, hydrogen sulphide, butanoic acid, etc. in industrial wastewater (e.g. from food industry)

- Secondary osmogenes: are formed by chemical and biochemical transformation processes within the sewer system along the flow path. They can emerge in aerobic or anaerobic conditions.
  Sources in wastewater works:

  - Fatty acids, alcohols, aldehyde, etc. (formed under aerobic conditions)

  - Hydrogen sulphide, mercaptane, methylsulfide, etc. (formed under anaerobic conditions)

In-sewer processes are manifold and complex and a detailed description of a general odour emerge process is not available. However, it is known that the dominant odorous substances in sewers generally emerge under anaerobic conditions.

Table 1-1: Examples of osmogenes in sewer air, odour descriptor and odour threshold ranges (after Thistlethwayte & Goleb, 1972; Stuetz & Frechen, 2001; Weismann & Lohse, 2007; Frey, 2008; Munoz et al., 2010)

Substance group	Osmogene	Odour descriptor	Odour threshold [ppm]
Sulphur-compounds	Hydrogen sulphide	Rotten eggs	0,0005 - 0,1
	Methyl mercaptan	Decayed cabbage, garlic	$0,14*10^{-5}$-0,04
	Ethylmercaptan	Decayed cabbage	$1 - 50*10^{-5}$
	Amylmercaptan	Rotten, garlic	$2,4 - 12*10^{-5}$
	Dimethylsulfide	Decayed cabbage, horseradish	0,0001 - 0,0004
	Dimethyldisulfide	Rotten cabbage	$2,6*10^{-5}$ - 0,011
	Diphenyl sulfide	Unpleasant	0,0003
	Thiophenol	Rotten waste	$27 - 31*10^{-5}$
	Sulphur dioxide	Pungent, acidic	0,71
Nitrogen-compounds	Ammonia	Sharp, pungent	0,04 - 15,3
	Methylamine	Fishy, rotten	0,0009-0,053
	Ethylamine	Ammonical	2,4
	Further see "Aromatic hydrocarbons"	Further see "Aromatic hydrocarbons"	

Substance group	Osmogene	Odour descriptor	Odour threshold [ppm]
Acids	Butyric acid	Sour, perspiration, rancid	$9*10^{-5}$ - 0,020
	Acetic acid	Vinegar, pungent	0,145
	Propanoic acid	Upleasant	0,035
	Valeric acid	Sweet	0,005
Aldehydes and Ketones	Butyraldehyde	Rancid, sweaty	0,0046-0,0089
	Formaldehyde	Arid, suffocating	0,37 -0,87
	Acetaldehyde	Pungent, fruity, apple	$5*10^{-5}$ - 0,19
	Valeraldehyde	Fruit, apple	$70*10^{-5}$-0,009
	Heptanal	Saur milk, rancid, sweet, unpleasant	
Alkanes	Hexan	Faint, fuel-like	21,9
	Heptan	Faint, fuel-like	9,8
	n-Octan	Fuel-like	5,8
Highly volatile chlorinated hydrocarbons	Chloromethane	Ethereal, sweet	10,2
	Chlorethane	Ethereal, pungent	4,1
Aromatic hydrocarbons	Cumol	Glue, moth balls, aromatic, harsh	0,024
	Phenol	Penetrant, aromatic	0,110
	Benzene	Sweet, solventy	3,6 - 1,4
	Indole	Faecal, nauseating	$32*10^{-5}$ - 0,0014
	Scatole	Faecal, nauseating	$0,2*10^{-5}$-0,001
	Ethylbenzol	Pungent, synthetic	0,003
Terpene	Limonen	Citrus	0,437

Chapter 2

Odour measurement

Odour is caused by a mixture of volatile gas compounds. Usually not single compounds are responsible for the odour but a combination of the substances and their interactions. As odours have a sensorial (odour) and chemical (odorant) component the characterisation of odour emission is a challenging task. Especially, odorous emissions from sewers and wastewater treatment plants are a complex mixture with a large number of chemical compounds (see *Table 1-1* for examples). Typically very low concentrations (often in ppb and ppt levels) make them very difficult to be detected with analytical methods. But already minimal amounts of substances can be detected by human beings (see odour thresholds in Table 1-1). The smell of e.g. hydrogen sulphide can already be perceived at concentrations between 0,002 - 0.15 ppm (Weismann & Lohse, 2007). In odour measurement the distinction between odour and odorant is important (Gostelow *et al.*, 2001; Munoz *et al.*, 2010).

Odour measurement methods can be divided into following categories:

1. <u>Sensory methods</u>: Olfactometry; determination of odour concentration by presenting a sample to a panel of trained persons (human noses) (e.g. dilution olfactometry according to DIN EN 13725; expressed in ou/m^3)

2. <u>Analytical methods</u>:

    a. Selected sensors: Measurement of specific single odorants or surrogate parameters with selected sensors (gas or water phase); e.g. specific measurement of H_2S, sulphide (concentration generally expressed in ppb or ppm)

    b. Gas chromatography, mass spectrometry, optical sensors: Measurement and quantification of a spectrum of several gas components (concentration generally expressed in ppb or ppm)

    Combination with sniffing port possible where each component is led to a human nose for evaluation of odour type and intensity (GC-MS-O)

    c. Multigas-sensor arrays: Measurement of overall odour parameters by means of unspecific, broadband multigas-sensor arrays

    Training with olfactometric measurements is usually possible. Statistical data analysis tools can be used for the correlation to odour. The measured concentrations are in this case expressed in ou/m^3)

Method 2.a. (selected sensors for single compounds) is the **simplest method** and is already established in practical applications. The parameter H_2S is often used as surrogate parameter as it is relatively easy to measure with electrochemical sensors. However, this method **does not take into account other prevailing substances**.

Method 2.b. (GC, MS, etc.) is very versatile as it can quantify a **wide range of single compounds**. The disadvantages of these technologies are that they **tell very little about the perceived effect of odour**, interactions between different substances are not

considered and they are usually connected with **high costs**. Additionally they are mainly lab-based and often not feasible for online-measurements.

Multigas-sensor arrays (method 2.c.) can cover a **wide spectrum of substances** due to the unspecific sensors. They have the potential to continuously provide information on the global composition of air samples. After training (referencing) with olfactometric analysis they can be able to deliver **information about the odour concentration** (Bourgeois *et al.*, 2003).

Today, no method is universally applicable for the assessment of odour emissions. Depending on the specific application and objective of odour measurement the specific problem needs to be considered on a case by case basis (target analytes, operating conditions etc.). Besides others the selection of a method depends on the components of the odorous gas which should be covered by the measurement and on the required mode of measurement (continuous, quasi-continuous, or discontinuous measurement). If the main components are known, the measurement can be focused especially on them (e.g. H_2S) - using method 2.a.

Table 2-1 provides a summary of the basic features which need to be taken into consideration in the first stage of method selection.

Table 2-1: General odour measurement methods and their limits regarding sensory detection and continuous appraisal

| Measurement method | Consideration of | | Continuous measurement | Discontinuous measurement |
	chemical component	sensory component		
Sensory methods: Olfactometry		x		x
Analytical methods: Selected sensors for single compounds (e.g. H_2S sensors)	x		x	x
Analytical methods: Gas chromatography, mass spectrometry, optical sensors	x		x	x
- with sniffing port	x	x	(x)	x
Analytical methods: Multigas sensor arrays (electronic noses)	x		x	x
- Training with olfactometric measurements	x	(x)	x	x

Within this report analytical odour monitoring methods (multigas-sensor systems, GC, MS, etc.) are covered which are capable of delivering continuous or quasi-continuous data.

The principles are discussed below and examples of instruments and specifications are listed in chapter 3.7 and 4.1.

Chapter 3

Electronic noses (multigas-sensor systems)

Electronic noses were in its basics already developed in the 1980s to mimic the functional principle of the biological sense of smell. But too high expectations on performance and transferability led to a downturn in interest. However they could be established in fields like food or medical industry and recent developments make them also available for applications in environmental monitoring. Special adaptations of the systems like preconcentration units, employment of only specially selected sensors, or adaptations for humidity and temperature control increase their sensitivity, selectivity and raise their potential to be used under field conditions.

The aim of electronic noses is not the detection of single chemical gas components but the broadband measurement of the gas constituents to deduce overall odour parameters (such as odour concentration or odour margin levels).

Most electronic noses consist of multigas-sensor systems which are based on cost-efficient non-specific gas sensors arranged in a sensor array. The unspecific sensors are capable of responding to a wide range of volatile compounds. The signals of the sensors are processed into signal pattern information to retrieve information on odour intensity and composition. A sophisticated and adapted interpretation method of the obtained data is crucial (Munoz *et al.*, 2010).

3.1 General principle

Multigas-sensor systems (electronic noses) are based on a chemical-sensory measuring principle. Five principle components of this technical-sensory odour measurement can be identified (see *Figure 3-1)* (Boeker, 2004a):

1. Sampling and sample preparation

2. Properties of the sensor measuring system

3. Sample gas classification; including possible pattern recognition

4. Calibration for the correlation with the parameter "odour"; including statistical correlation methods (training step)

5. Olfactometric reference measuring method

Each of these components occupies certain problems which need to be overcome to allow for stable and reproducible measurements. A diligent standardization and documentation of sampling conditions and procedure as well as a proper gas transfer to the sensor array are necessary.

The core of an electronic nose system is an array of broad-ranged and non-specific sensors. These sensors are not identical but show different response characteristics for substances or/and substance groups. The types and number of employed sensors vary from one e-nose to the other (see *chapter 3.3*). The gas is delivered to the sensor array which consists out of a certain amount of sensors. The system can include further elements such as gas preconcentration units, which can increase the sensitivity, can serve as filter and can adapt the selectivity of certain gas components. The stability of

the sensors is crucial to provide a stable detection of patterns (classification) and to avoid drift effects. The potential for a proper classification depends on a reasonable limitation of the number of sensors. To establish the correlation to odour by means of olfactometry certain pitfalls need to overcome (margin of error of olfactometry, high number of reference values, etc.).
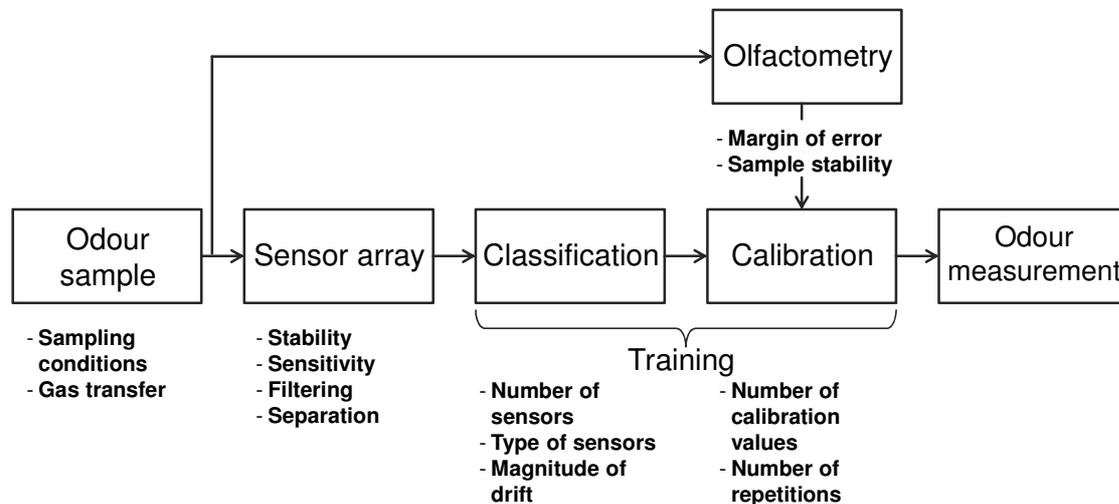
Figure 3-1: General process sequence and related problem areas of technical-sensory odour measurements (after Boeker, 2004a)

Configuration of e-nose systems

Every e-nose system needs to be adapted to the certain application field (like odours from municipal wastewater). Hence it is relevant to thoroughly select

- the sensors (types and amount)
- the sampling system (location, active, passive, etc.)
- the sample conditioning (drying, dilution, enrichment, etc.)
- the measuring cycle and data acquisition (interval)
- the parameter setting (sample flow rate, sensor temperature)
- the data evaluation method (classification methods, multivariate methods, etc.)
- etc.

Basically these properties are selected by the manufacturer based on his experiences and the target application field of the e-nose. Some internal parameter can usually be adapted to the respective needs (flow rate, sampling time, measuring cycle, etc.).

The specific problem needs to be considered on a case by case basis (target analytes, operating conditions etc.) in order to obtain a chemical fingerprint which correlates with the relevant properties of the sample (Röck *et al.*, 2008).

3.2 Odour sample and sample preparation

Sample preparation and sample handling are crucial steps in terms of reliability of results from odour measurements. One has to consider a representative selection of samples, an appropriate pretreatment, possible preconcentration or separation steps as well as a reproducible sample delivery to the sensor system.

Sampling preparation includes

- Sampling method: method of feeding the gas to the sensors. Common methods are briefly described in *Table 3-1*.

  Headspace sampling is usually limited to solid and liquid samples and laboratory applications. Field applications usually use a direct gas sampling from the source by the analyser equipment. A storage mixture chamber can be included to inject homogenous gas. A quasi-continuous sampling by means of thermal desorption can reduce the influence of temperature and humidity of the sample and therefore can already take over parts of the gas conditioning (e.g. filtering, heating). Additionally it can increase the selectivity and sensitivity of the detection device.

- Gas conditioning: Can include the filtering, lowering of the dew point, condensate trap, separation units, heated transfer line, heating of sample gas at the sampling point or the pre-dilution of the gas sample.

The general objectives of sample preparation are to separate aerosols or particles, to minimize the influence of temperature and humidity and other background interferences (such as pressure and background from applied materials), to increase the selectivity and sensitivity or to wash out interfering substances (Heining, 1998; Boeker, 2007). Provision to protect the sensors can also be stipulated (e.g. automatic dilution).

Table 3-1: Typical sampling methods for gas detection devices (after Heining, 1998; supplemented by Münchmeyer & Walte, 2004; Boeker, 2007; Röck et al., 2008)

Sampling method	Description	Pros/Cons
Static Headspace	Sample air is collected from a closed vessel where the sample is assumed to be in equilibrium with its vapours at a specific temperature and pressure. Other static technique: gas is not let to the sensor array but the sensor array is lowered into the vessel.	✓ Interfering factors can be disabled × Practicable mainly for liquid and solid samples × Limited to lab application × Applied for evaluation of single samples × High effort to stabilize temperature and humidity (esp. for gas samples)
Dynamic Headspace	Gas flow through or across the sample. Gas is then led to the continuous measuring device. Temperature and humidity adjusted in some cases.	✓ Sample can be stripped continuously × Practicable mainly for liquid and solid samples × Limited to lab applications × Applied for evaluation of single samples
Direct sampling	Gas samples are directly transferred to the measuring device by means of an internal pump from the source or from ambient air. A storage mixture chamber can be included to inject homogenous gas. Some cases: Periodic purging of the sensors with inert gas, ambient air or cleaned air.	✓ Comparably simple method × High effort to stabilize temperature and humidity (e.g. with purge gas) ✓ Relief of sensors (by purging gas) × Interruption in measurements due to purging phases
Quasi-continuous sampling	Thermal desorption (preconcentration unit): Adsorption of the gas sample onto an adsorbent (like Tenax, Chromosorb, or Silicone). After desorption with a defined gas stream and temperature, the sample is led to the sensor array.	× Prolongs measurement cycle ✓ Increase of sensitivity ✓ Less-volatile substances and low concentrated substances can be detected ✓ Improvement of selectivity ✓ Reduction of influence of humidity and temperature

Stripping unit

A special type of sampling was recently developed by the University of Kassel (2007) where water phase samples are drawn. Odorants present in that liquid phase are then stripped from this liquid under standardized conditions (see *Figure 3-2 a)*). The set-up of a compact version of this sulphide and OEC measurement is illustrated in *Figure 3-2 b)*.
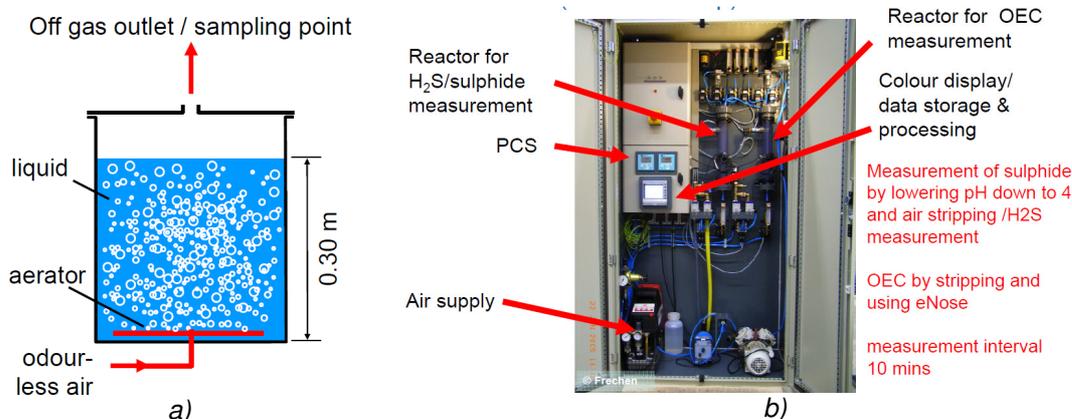
Figure 3-2: a) Stripping reactor (Frechen, 2008); b) Sulphide and OEC meaurement unit (Frechen, 2008) Vorschlag: Nummerierung in der Abbildung 3-2 b integrieren

The objectives and advantage of this method are (Frechen, 2008; Frechen, 2011):

- The possibility to measure sulphide and odour in an near-online manner
- The possibility to overcome problems with interferences of air stream from ventilation or high and low pressure areas
- The possibility to overcome problems with unsteady/inhomogeneous air flow in the headspace (different densities of odour substances)
- The Improvement of sampling via feeding of e-noses with odour concentrated air
- The possibility to interfere in the odour formation at an earlier stage (before the emission occurs); e.g. control dosing activities for odour abatement
- The possibility to quantify the concentration of all present odorants
- The determination of the OEC (Odour Emission Capacity):

    The Odour Emission Capacity OEC of a liquid is the total amount of odorants present in that liquid that can be stripped from this liquid under standardized conditions (Frechen and Köster, 1998). It is given in odour units per cubic meter of liquid. Its unit is ou/m^3_{liquid}

The stripped air can be fed to H_2S measurement devices and/or electronic noses. Hence, this method represents a batch-mode sampling including following steps (see *Figure 3-2 b)*):

1. Fill reactors with wastewater
2. Stripping of the wastewater (see also *Figure 3-2 a)*)
3. Feeding of stripped air to measurement device(s)
4. Empty reactors
5. Clean reactors with fresh water

Drawbacks of this type of sampling are the possibly longer measurement cycles (≥ 5 minutes) and the "addition of complexity" to the measurement/sampling method.

A guideline (VDI- guideline 3885) from the Association of German Engineers is currently in preparation dealing with this OEC-determination method (Frechen, 2011).

3.3 Sensor array

Usually sensor arrays are based on the employment of up to 30 non-specific sensors. In specifically adapted arrays, however, manufacturers tend to use less sensors (4 - 16) to avoid redundancies and facilitate the classification and data interpretation. A variety of different sensor types can be deployed in sensor array systems. *Table 3-2* gives a selection and description on deployed sensor types. The most common sensor types in environmental applications are metal oxide semiconductors (MOS), conducting polymer sensors (CPS), quartz microbalance (QMB), and surface acoustic waves (SAW). The two former are based on the change of conductivity by adsorption whereas the latter ones rely on the measurement of the gravimetric changes on a substrate. Also photionization detectors (PID) are sometimes deployed for measuring the sum parameter of volatile organic carbons (VOCs). Other sensors are not yet established in environmental applications. Electrochemical sensors (EC) are more and more deployed additionally to the common ones in order to enable the detection of certain specific substances (like H_2S, or NH_3).

The results of the measurements of electronic noses are highly affected by the very varying and complex gas mixtures (different nature, polarity, volatileness), low concentration levels, high sensitivity of the sensors towards change in temperature and humidity or the cross-sensitivity to odourless substances.

Table 3-2: Types of sensors typically used for electronic nose systems for environmental applications (Heining, 1998; Stuetz & Frechen, 2001; Mannino et al., 2006; Röck et al., 2008; Munoz et al., 2010; Frey, 2011)

Type	Functionality	✓Pros/ ×Cons
Metal oxide semiconductor (MOS)	Adsorption of gases and surface reactions (oxidation of gas molecules via electron transfer from the gas to the metal oxide). This leads to a **change in conductivity** of the semiconductor.	× High humidity sensitivity × Availability of oxygen in the gas sample is necessary × less selective (sensitive to ethanol and methane) × Sensor poisoning × Long term drift ✓ High sensitivity ✓ Fast response ✓ Low recovery times ✓ Low production costs ✓ Applicable for flammable substances (200-650°C) ✓ Low temperature sensitivity
Conducting polymer sensor (CPS)	Principle similar to MOS. Adsorption of substances onto polymer at low temperatures (ambient temperature). Measurement of **change in conductivity.**	× High humidity and temperature sensitivity × Lifetime only 9-18 months ✓ Good sensitivity for polar compounds ✓ High reproducibility ✓ High stability (Resistance to poisoning)

Type	Functionality	✓Pros/ ×Cons
Quartz crystal microbalances (QMB)	Quartz crystal coated with Sorbent. Stimulated to swing at resonant frequency in an electrical oscillating circuit. Measurement of **change in frequency** and determination of **mass change** of the sorbent.	× Problems of reproducibility in commercial production (complex process) × High temperature sensitivity × Medium humidity sensitivity (dependent on coating) ✓ Measure preferable high-molecular materials ✓ Fast response time (<10s) ✓ Good selectivity
Surface acoustic wave sensors (SAW) Bulk acoustic wave sensors (BAW)	Similar to QMB but operate at much higher frequencies. Sorbent on carrier material. Measurement of **runtime and change in amplitude** SAW: 2-dimensional BAW: 3-dimensional waves travelling through the crystal	× High temperature sensitivity × Medium humidity sensitivity (dependent on sorbent) ✓ Higher frequencies allow for higher sensitivities (better for nitrogen and sulphur compounds compared to MOS)
Photoionization detector (PID)	Measurement of several inorganic and primarily volatile organic compounds (VOC) that have ionization potentials equal to or less than the energy of the UV radiation. e.g. detection of amines, benzene, aromatic compounds, ammonia, ethanol, acetone	× Humidity sensitivity ✓ Easy to handle
Electrochemical sensors (EC)	Target gas is either oxidized or reduced and determines the current between the sensing and the counter electrode. The current is proportional to the target gas concentrations. Designed to detect one special gas (often not completely specific)	✓Reliable detection of a wide range of single gas compounds (e.g. H_2S, NH_3)
Fibreoptic sensors		In development stage
Bimetal sensors		In development stage
Optical sensor systems	The modulation of light properties is measured; Operation modes: e.g. changes in absorbance, fluorescence, optical layer thickness, polarization Applied technologies: e.g. diverse light sources, optical fibres, photodiodes, CCD, CMOS cameras	× More complex ✓ Wide range of applicable technologies

3.4 Classification

In order to give information on odours, substances or changes in the sample the system has to be trained. Training means that the system needs to "learn" a set of different response patterns depending on the problem faced. For example to train sewer air, different air conditions need to be trained (different odour ranges/odour types). The applied samples need to be characteristic for the specific site, the occurring odours and ranges. Then a pattern recognition method (usually principle component analysis or similar) can be applied to establish a **classification model** (see *Figure 3-3*).

Once the training is done, the system can recognize the patterns continuously when "unknown" air is applied.

To establish a correlation to the parameter odour, a number of reference values for "odour" is necessary for each class. The reference values are defined by human observations (Gostelow *et al.*, 2001).
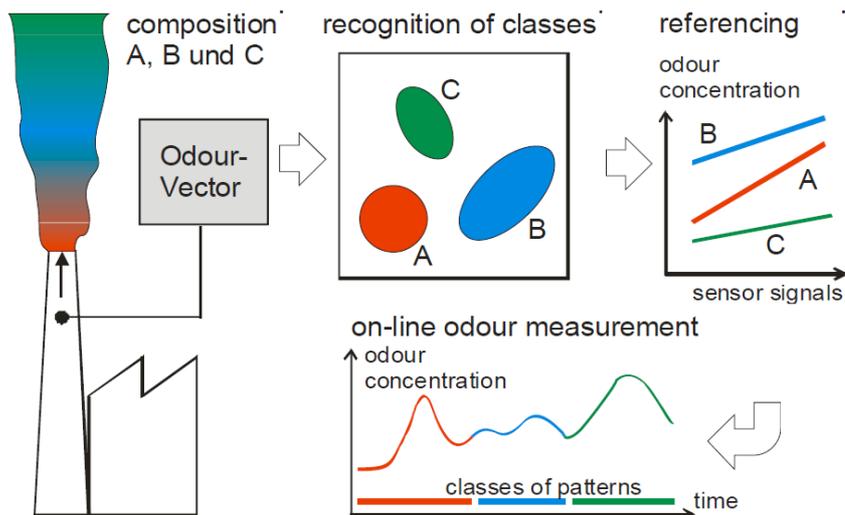
Figure 3-3: General odour classification procedure for an E-Nose, example of the system "OdourVector" (Boeker & Haas, 2009)

3.5 Calibration/Referencing

For odour the reference method is the measurement of the odour concentration by means of olfactometry (e.g. EN 13725). A sophisticated calibration model should be developed to describe the function between the sensor signal and the olfactometric values (see *Figure 3-3* top right).

Currently no other method than the deployment of human noses (olfactometry) exists to conclude on the perceived effect of odour.

Olfactometric measurements:

Olfactometric measurements employ the human nose to directly relate the properties of odours as experienced by humans. The total effect of the overall odour is measured regardless of how complex the mixtures are and how the components interact among each other. Olfactometry is a time-intensive method and inherits a high degree of uncertainty of measurement and errors. Many factors other than the properties of the odour sample itself may influence the perception (e.g. variability of the sense of smell, sample presentation to the observer). Hence the usage of this referencing method for the determination of the odour concentration introduces a high degree of uncertainties into the odour measurement (Heining, 1998; Gostelow *et al.*, 2001). An uncertainty of 25 % to 400 % was concluded by Boeker & Haas (2007) after conducting a reference simulation.

Also the sampling methodology (location, timing, choice of material, storage, etc.) is of highest importance for the quality of the calibration model for the e-nose.

The European Standard DIN EN 13725 and the VDI-guideline 3881 of the Association of German Engineers describe the measuring procedure and test conditions for olfactometric measurements.

Setting up a calibration model:

For setting up a calibration model for the parameter odour with e-noses measurements, parallel measurements with the e-nose and olfactometer are necessary. Then the

different sensor readings need to be converted into one odour concentration. Different data analysis methods ranging from linear and non-linear regression to artificial neural networks can be applied to set up a model (see *chapter 3.6)*. Then the odour concentration (in ou/m³) can be calculated and online-odour measurements are possible (Heining, 1998; Gostelow *et al.*, 2001; Boeker, 2007).

For setting up a calibration model the whole expected range of the gas matrix needs to be covered by olfactometric measurements. For example samples need to be taken at high, as well as at medium and low concentration levels. This needs to be done within each detected class. The amount of necessary parallel measurements with olfactometry depends on the quality and usability of the measurements. Generally valid is that the more parallel reading, the higher the possibility to create a reliable mathematical model (Frechen & Giebel, 2010). As stated by some e-nose vendors, a small amount of parallel readings (1-10) per class (or type of air) can be enough to create a model. But one needs to consider that the smaller the mathematical data sample size, the bigger the possibility to get a deterministic model. The model itself can have an error as high as the olfactometric measurements (Frechen & Giebel, 2010). Frechen & Giebel point out that it is crucial not only to set up an explanation model, but also to determine the prediction capabilities. This is, to determine the correlation coefficient with unknown data (e.g. by data splitting).

Setting up a threshold model:

In case it is requested to detect if there is a new odour compared to a "base odour", one can set up a threshold model. Then the system can store the pattern of the base odour and can detect if the incoming odour is different from the "normal" one (Della Torre, 2010).

3.6 Data analysis methods for e-nose signals

E-nose measurements are often provided as raw signals (resistance values, frequency values). Hence these signals need further analysis to deliver useful information on odour. Data evaluation of e-nose signals is a complex task as it is not limited to pattern recognition but also needs to include factors like the choice of appropriate sensors, feature selection, scaling, and normalization. A variety of different approaches is available which need to be adapted for a specific application. There is no general guideline for the choice of a strategy.

The high variation of the gas matrix and complex gas compositions require that the training is done with real experimental data and must be specific to the application and the electronic nose used (Röck *et al.*, 2008).

Most e-nose vendors provide also tools for data analysis (software), where training data can be integrated and different statistical methods can be used. Common methods are briefly described in *Table 3-3*.

Table 3-3: Common methods for data analysis of signals from multigas-sensor systems (Stuetz et al., 1998; Mitrovics, 2004; Boeker et al., 2009)

Analysis method	Description	Type
Principle Component Analysis (PCA)	Reduction of a large number of variables to a smaller number of components. Extract maximum variance in the data. Proportional relation of odours to each other; classification of emission situations	Multivariate method
Partial Least Squares (PLS)	Describe dependence between 2 variable blocks (e.g. sensor response, time variables)	Multiple linear regression
Discriminant function analysis (DFA)	Training data is used with known properties. Maximises the variance between categories and minimizes the variance within categories. Simple: the act of distributing things into classes or categories of the same type	Multivariate method
Canonical correlation analysis (CCA)	Explores linear relationships between sets of variables and independent sets of variables by maximising the correlation in the data	Multivariate method, Regression
Cluster analysis	Find natural grouping or clusters of individual observations within a data-set Clustering based on distance measures of dissimilarity or similarity	Multivariate method
Feature weighting	Assumes that all features are of equal value for the classification. Approximates the optimal degree of influence of individual features using a training set. Relevant features are weighted high, irrelevant features weighted close to zero	
Neural networks (NN)	Consists of a set of interconnected network of neurons. Input layer has one neuron for each sensor signal, output layer has one neuron for each of different sample properties. Parallel- interconnected processing elements ("neurones") are weighted mathematically Classification due to chemical composition. "learning phase" , high number of samples necessary	Multivariate method
Linear discriminant analysis (LDA)	Used to find a linear combination of features which characterize or separate two or more classes of objects or events. Resulting combinations are used for classification. In regression analysis: dependent variable is a numerical quantity, while for LDA it is a categorical variable. LDA explicitly attempts to model the difference between the classes of data. PCA on the other hand does not take into account any difference in class.	Multivariate method; Closely related to PCA

3.7 List of electronic nose systems and specifications

Table 3-4 to *Table 4-4* provides a list of identified vendors or developers of electronic noses (multigas-sensor systems). 18 different companies and research institutes were identified who provide this kind of systems.

A couple of e-nose systems regularly appear in literature listing artificial olfaction systems but are no longer on the market. Therefore Appendix B provides a list of these non-available instruments.

Application fields and scale

Listed systems are either designed for wastewater monitoring applications or were identified to have the potential to be used within this field. Most of the mentioned systems are stipulated for environmental monitoring purposes although some have their original application field in food or medical industry. Additional development work might then be necessary for entering the field of wastewater. *Table 3-4* to *Table 4-4* gives specifications for the instruments as available (from vendors and literature).

Some multigas-sensor systems are already in full-scale operation for the mentioned target application field or are subject of large scale case studies (e.g. systems from Odotech, Alpha MOS, Altrasens, Comon Invent). Practical applications in environmental monitoring have also been described in literature, e.g. by Boeker & Horner, 2004; Guy *et al.*, 2004; Münchmeyer & Walte, 2004; Romain *et al.*, 2005; Franke *et al.*, 2009; Bootsma & Milan, 2010.

Sensors used

Most dominantly used sensors in the field of environmental monitoring are the metal oxide semiconductors (MOS). They are preferably applied due to their low production costs and high sensitivity. Some of the systems are configured with additional specific sensors (like electrochemical cells for detecting H_2S or NH_3).

Sampling method and gas conditioning

The most comon method of transferring the gas to the sensor arrays in electronic noses is the direct sampling. Quasi-continuous sampling methods (like thermal desorption) are sometimes included or offered on optional basis. Some vendors employ thermal desorption (preconcentration units) to overcome problems with low concentrated compounds and to adapt the selectivity of the sensors. Static dilution or automatic dilution are more and more implemented to protect the sensors from being exposed to high concentrations (esp. H_2S) which could have a poisoning effect or decrease the sensitivity.

To handle the sensitivity of the sensors to humidity and temperature, vendors increasingly consider additional gas conditioning steps (such as heated transfer lines or condensate traps) to create standard conditions for each measurement.

Customized systems (type and number of sensors, sample preparation) are sometimes offered (e.g. by 3S gas sensing solutions).

Training

Most of the vendors provide software tools in order to evaluate the e-nose signals. They usually provide a variety of methods to analyse the data (examples are provided in *Table 3-5*). Some software tools can set up models to provide an indication of odour concentration in ou/m³ once calibrated with olfactometric analyses. Prediction capabilities of the created models however are rarely considered (i.a. Frechen & Giebel 2010).

Costs

Costs for a multigas-sensor systems range from 12 k€ to 48 k€, depending on the configuration and additional devices (such as thermal desorption unit). A control and data acquisition software is usually included. For programs allowing data analysis sometimes additional costs have to be considered (up to ~10 k€ or annual fees). Simpler systems are also available from 4,5 k€ or are provided on a full operational lease for ~2 k€/year.

Appendix A provides contact details of the listed vendors and products.

Table 3-4: Specifications of identified electronic nose systems for environmental monitoring and/or wastewater applications 1/2

	Vendor	Product(s)	Sensor type/method	Application field	Sampling/gas conditioning	Measuring cycle	Comments
1	**Airsense Analytics**	PEN (Portable Electronic Nose) GDA	PEN: 10 MOS; GDA: IMS, PID, EC, MOS	Environment and security: Compost works; WWTPs; control of filters; leakage detection, fire early warning; Food; materials, medical; GDA 2: hazardous gases, chemical warfare agents	Gas stream; Dynamic headspace; Automated predilution; Optional: Thermal desorption; Flushing of sensors with filtered ambient air	Several secs to 5 - 10 minutes (with preconcentration)	Control of concentration variations at inlet (to avoid distortion of results and poisoning of sensors)
2	**Alpha M.O.S.**	RQ BOX	3 MOS + PID + EC (H_2S, NH_3, R-SH)	Waste management, wastewater, food, beverage, plastic and packaging, pharmaceutics and nutraceutics, health, cosmetics, flavors and fragrances, chemicals, paper & pulp	Gas stream Optional: Dry and dilute system; PTFE-filter for particle separation	1 sec	Atmospheric dispersion modelling (weather tower)
3	**AltraSens**	OdourVector	6 QMB; Optional: EC (H_2S, NH_3)	Emissions from paper and metal processing factories; waste disposal; sewer system; control of filters; enamelling lines; odour complaint management; reporting to authorities	Thermal desorption unit with partial separation of gas components; Industrial gas conditioning to control humidity and for particle separation	5 – 10 minutes	Prediction of odour emissions; process control possible
		Triple-Sensor	3 MOS elements on one common heater; 3 temperature levels	Food industry (up to now)	Direct sampling	1s	Newly developed system; virtually 9 sensors; Sensor from UST- Umweltsensor-technik
4	**Applied Sensor**	VOCmeter VOCcheck VOCvario	4 -12 MOS, QMB	Pharmaceutical, chemical industry, filter monitoring, food, plastic, textile	Measurement direct in the process; Optional: Dynamic headspace; autosampler, purge&trap unit, thermal desorption	1 sec. to unlimited	up to 8 measuring points
5	**Comon Invent**		8 MOS + EC, optical gas sensors	Oil and gas companies, petrochemical refineries, soil pollution, WWTPs, sewage wells	Continuous gas stream sensing	1 - 60 secs.	Onboard GPRS for data processing and data management
6	**CSIC - Instituto Física Aplicada**	WiNOSE	4 - 6 MOS	Food, beverage, medical engineering, environmental	Gas stream with internal pump	5 -15 minutes	Wireless (up to1 km) Battery operated using solar cells; Not commercialized yet

	Vendor	Product(s)	Sensor type/method	Application field	Sampling/gas conditioning	Measuring cycle	Comments
7	**Ezent**	iNOSE	MOS (primary sensor); + peripheral sensors	Environmental pollution, fire hazard, smoke control, process control, Pipelines and leak detection, electronic devices; Any kind of mixed gas classification, suitable for teaching process	Temperature swing, various modulation strategies according to range and focus of application	From 10ms up to 10min	Newly developed; Sensitivity and selectivity Adjustable; GSM data transfer
8	**GenMark Dx** (Osmotech PLC)	Aromascanner	CPS	Healthcare diagnostics and medical devices	NA	NA	
9	**KIT - Karlsruher Forschungszentrum für Technologie**	SAGAS	8 SAW	Indoor air monitoring (quality control, fire alarm), natural gas analysis, storehouse monitoring for chemicals	Purge and trap unit	5 -10 min	Especially sensitive to hydrocarbons
10	**Multisensoric GmbH**	Multigas-SENSORiCCARD	MOS	Food, medical engineering, environment, technique	Head space T-socket on a pipe String reactor (new method!) 3 measuring channels	NA	Tin dioxide sensors
11	**Odometric**	FIDOR	6 - 8 MOS + IR	Outdoor (compost, landfill areas, biogas, e-noses network); indoor environment	Continuous gas stream Optional: Predilution	Continuous (no cycling)	Optional: Several e-noses exchanging information through a data bus
12	**Odotech**	OdoWatch	16 MOS	Composting, landfills, WWTPs, food processing, breweries, petrochemicals, refineries, biofuel, chemical plants, pulp & paper mills	Automated predilution	NA	Atmospheric dispersion modelling (weather tower)
13	**RST Rostock System Technik GmbH (EADS RST)**	SamDetect FF2 SamDetect FF2D SamDetect GFD1	6 MOS	Pollution and hazards control, leakage detection, fire early warning (tunnels, IT-Server , power plants, paper industry, indoor)	Continuous data acquisition Static headspace	1 sec.	- Detection of major, abrupt changes of gas spectrum in the atmosphere - Event storage in the detector

	Vendor	Product(s)	Sensor type/method	Application field	Sampling/gas conditioning	Measuring cycle	Comments
14	**SACMI**	EOS Ambiente	6 MOS	Any outdoor odour monitoring (e.g. landfills, biogas plants, composting plants, WWTPs, livestock, refineries, chemical plants, rendering plants)	Continuous gas stream sensing; internal concentration controller	Continuous, 1point/sec	Weather tower included; Neutral air generator (filter system) → no external gas cylinder necessary
15	**Scensive Technologies**	Bloodhound ST306 Bloodhound Odourspace	6 CPS	Bacteria	Manual sampling – (automated sampling capability expected in 2012)	2 - 3 minutes	Designed for lab use; no automatic sampling capability yet; not ruggedized
16	**Scientec Lab Center Co., Ltd**	OdorCatch	7 - 10 MOS + EC	Pollution level in electronics and interior, food industry	Vibrating Armature pump	1 - 5 min	Odour Analyzer, network type, handheld type; Electronic tongue
17	**University of Extremadura**	UEX E-nose	6 MOS	Food, beverage, wastewater monitoring, odours	Optional: Purge & trap unit; Static headspace with effluent transfer	2 - 10 min	Optional: GPS; not commercialized; predecessor of WiNOSE (see 6)
18	**3S Gas Sensing Solutions**	OdourChecker	1-10 MOS, EC, IR, Pellistor (depending on application)	Product quality: Food & Beverage, plastics; Process control: Biologically active filter control	Full adaptable headspace or direct sampling, full automated pumped system	< 5s up to 20 min. (depending on specific application)	System is available as stand-alone system or with integrated PC; data storage by SD mini Card; mobile system also available; customized adaptation of system

Table 3-5: Specifications of identified electronic nose systems for environmental monitoring and/or wastewater applications *2/2*

	Vendor	Product(s)	Data analysis methods	Measurement of odour concentration	Measurement range; Accuracy	Temperature, humidity sensitivity	Stability	Price	References
1	**Airsense Analytics**	PEN (Portable Electronic Nose); GDA 2	PCA, DFA, LDA, PLS, Mahalanobis, etc.	ou/m³; calibration with olfactometry	LOD: 0.1- 5 ppm for gases; e.g. H_2S: 0.1ppm, Benzol: 1ppm; ppb range with thermal desorption	Adaptation by variable sorption material, variable duration, and temp; 5- 95 % rH non condensing	Long durability (concentration control at inlet)	18 k€ + 10 k€ for preconcentrator (incl. analysis software)	Münchemeyer & Walter (2004); Franke *et al.* (2009)
2	**Alpha M.O.S.**	RQ BOX	PCA, DFA, PLS, NN, Fuzzy, SIMCA	ou/m³; calibration with olfactometry	ppm-range	Optional: Dry and dilute system (DnD)	MOS: 2-3 years	15 k€ + 6 k€ for DnD (+ 10k€ for software)	
3	**AltraSens**	OdourVector	PCA, Mahalanobis	ou/m³; calibration with olfactometry	Lower ppm range; H_2S, NH_4: 1–100 ppm	Control of humidity by gas conditioning	> 5 years (up to now - filter control)	28 k€ (excl. analysis software)	Boeker (2004a); Boeker (2004b) ; Boeker & Horner (2004)
		Triple-Sensor	Feature extraction	NA	NA	NA	NA	< 4.5 k€	
4	**Applied Sensor**	VOCmeter VOCcheck VOCvario	PCA, PCR, LDA, Mahalanobis, ANN, etc.	Internal calibration [ppb]	0.1-1000ppm for all molecules, e.g. CO_2 up to 3000 ppm, some down to 10 ppb	Variable sorption material, temperature control, QMB 1°C steps, MOS 200 °C	10 years minimum	~ 6 k€ - 24 k€ (excl. analysis software?)	Mitrovics (2004)
5	**Comon Invent**	E-nose	PCA, etc.	Odour classification	Odour threshold limit: ~1 ou/m³	No gas conditioning; Humidity and temperature compensation; 15-98 % rH	> 1 year; Remote compensation of zero drift	Full operational lease: non-recurring: 2 k€; Recurring costs (server licence): 750€ annually	Immission monitoring in the port of Rotterdam (NL) (Bootsma, 2010); Bootsma & Milan (2010)
6	**CSIC - Instituto Física Aplicada**	WiNOSE	PCA, neural networks	NA	ppb range	Humidity and temperature	2 years	Expected less than 10 k€	

	Vendor	Product(s)	Data analysis methods	Measurement of odour concentration	Measurement range; Accuracy	Temperature, humidity sensitivity	Stability	Price	References
7	**Ezent**	iNOSE	Neural networks	Detection of preteached samples; advanced incident correlation libraries	Sensitivity Adjustable; starting at ppb. optimal working point at 10...500 ppm	Temperature and humidity compensation; Teaching patterns for applications to compensate cross-impacts	> 5 years	Online lab systems 10 k€, autonomous mass sensors 100 €	www.ezent.com
8	**GenMark Dx** (Osmotech PLC)	Aromascanner	NA	NA	NA	NA	NA	NA	
9	**KIT - Karlsruher Forschungszen trum für Technologie**	SAGAS	NN, PLS, LDA, Cluster, PCA	NA	ppb range; mainly aliphatic, aromatic and halogenated hydrocarbons	Preconcentration, Dehumidifier possible (down to 10-15 %)	NA	12 k€ - 15 k€ (excl. analysis software)	
10	**Multisensoric GmbH**	Multigas-SENSORiCCARD	Pattern recognition	Classification by means of lab analytics	NA	NA	NA	NA	Ahlers et al. (2010)
11	**Odometric**	FIDOR	DFA, PLS, Mahalanob is, etc.	ou/m³, odour emission rate, max. perception distance (coupled with weather data), calibration with olfactometry	Depends on the application: 10…50 ou/m³ for compost	Precondensation unit or predilution; Temperature regulation by PID; Humidity and temperature compensation	5 - 6 years with the same sensors	~ 15 k€ (incl. analysis software)	Romain et al. (2002); Nicolas & Romain (2004); Romain et al. (2005); Nicolas et al. (2006); Kuske et al. (2006); Romain & Nicolas (2010)
12	**Odotech**	OdoWatch	NN, cluster analysis	ou/m³; calibration with olfactometry	± 5 ou/m³	NA	2 - 3 years (depends on operating conditions)	NA	Guy et al. (2004)
13	**RST Rostock System Technik GmbH (EADS RST)**	SamDetect FF2 SamDetect FF2D SamDetect GFD1	Pattern recognition , NN	Not exactly concentration measurement,; alarm classes by neural network (NN)	e.g.: CO: < 10 ppm H$_2$: < 10 ppm NO$_x$: < 5 ppm Ozone: < 1 ppm SO$_2$: < 10 ppm	Suppressing cross-sensitivities by adaptive system; Temperature and humidity compensation Max 97 % rH, non-condensing	3 - 5 years (depends on operating conditions)	~ 3 k€ - 10 k€ (excl. analysis software)	

	Vendor	Product(s)	Data analysis methods	Measurement of odour concentration	Measurement range; Accuracy	Temperature, humidity sensitivity	Stability	Price	References
14	**SACMI**	EOS Ambiente	PCA, DFA, LDA, ANN, RBFN, LDQD, PLS, SVM, Linear-Regression	ou/m³; calibration with olfactometry	Typically ppm range Repeatability: ±10% odour quantitative measurement; 0-1000 ou/m³	Not sensitive to humidity or temperature variation; humidity compensation; 5-95 % rH	18 - 24 months	48 k€ (incl. analysis software)	Oil refinery (Italy) (Demattè, 2010)
15	**Scensive Technologies**	Bloodhound ST306 Bloodhound Odourspace	PCA, NN	Bloodhound Odourspace: only pass/fail out	ppm to ppt (sulphur compounds to 4 ppb)	50-80 % rH non condensing	NA	7 k€ - 20 k€ (incl. analysis software?)	
16	**Scientec Lab Center Co., Ltd**	OdorCatch	NA	ou/m³, OI (odour intensity), odour pattern	High, e.g. 0.1 ppb benzene in water	NA	NA	NA	
17	**University of Extremadura**	UEX E-nose	PCA, NN	ou/m³; calibration with olfactometry	ppm range	Optional: dry & dilution unit	MOS: 2 - 3 years	< 6 k€	Aleixandre *et al.* (2008); Lozano *et al.* (2010)
18	**3S Gas Sensing Solutions**	OdourChecker	Multivariate data analysis, pattern recognition	ou/m³; ppm; calibration with olfactometry	ppb range by long measuring cycle, ppm-range calibration	Pressure, temperature and humidity compensation	NA	12 k€ - 15 k€ (OC Standard) + customized adaption costs	

Chapter 4

Further analytical methods for odour analysis

Analytical measurement methods such as gas-chromatography (GC), or mass-spectrometry (MS) allow for objective identification and quantification of specific gaseous substances. The detection of these substances can then be used in the frame of odour measurement but do not deliver information on the odour perception. The methods comprise the technology of gas chromatography, mass spectrometry, photo-ionization detector, or photo acoustic spectroscopy (see *Table 4-1* for further examples and brief descriptions).

The advantages are

- the objectivity
- the repeatability
- the accuracy

However drawbacks are that

- they cannot provide information on odour or regarding the potential for odour nuisance. Though the concentrations can be compared to odour threshold values for each substance (expressed in ppm or ppb).
- they do not consider the odour perception and
- they do not consider interaction of odorants (synergy, inhibition, masking) in the gas complex. Hence, the sensory component of emissions is not considered.

The sensory component can only be considered by the employment of a human nose at a sniffing port after the separation of gas components (GC-MS-O). This is only feasible in discontinuous mode and interactions of odorants are not considered with this method either.

The identification of odorants present in wastewater and sewer emissions can however help to identify odour sources and to understand formation processes along the sewer. Enhanced peak resolutions (e.g. combination of gas chromatography with mass spectrometer detector) can be an ideal technique for the measurement of complex mixtures at trace levels concentrations. Recent developments allow for shorter analysis times (down to a few seconds) and make these methods able to provide online-data (Munoz *et al.*, 2010).

Sampling methods as described in *Table 3-1* are also applied for these analytical methods.

Table 4-1: Types of analytical methods for analysis of gaseous substances (selection) (Stuetz & Frechen, 2001; Gostelow et al., 2001; Röck et al., 2008; Frey, 2011)

Type	Functionality	Pros/Cons/Examples
Gas Chromatography (GC)	**Separation of complex volatile components**. Carrier gas passes over a stationary phase for which the volatile components have a different affinity (according to their physical, chemical properties). This determines the order of elution of the components. New developments for e-noses: Fast or ultrafast mode (e.g. increase of flow rate, temperature)	× No detection of odour ✓ Detection of full spectrum of gas constituents
Mass Spectrometry (MS)	**Electron or chemical ionization of compounds and determination of their molecular mass**. Then the molecule ions and their fragment ions are separated according to their mass-to-charge ratio (m/z) with an electric and/or magnetic field. Mass analyzer: e.g. static fields, quadrupole mass analyzer	× No detection of odour × Sensitivity increases with lower gas flows × Possible inflow of oxygen into the ion source from the sample →reduces sensitivity × Operation requires vacuum ✓ Capacity to identify unknown compounds
Flame Ionisation Detector (FID)	**Predominantly detection of the sum of volatile organic carbon (VOC).** Measures ionisation current of the substance after combustion. For nonspecific determination of flammable compounds.	× No detection of odour × Lower sensitivity to sulphides (compared to MS) × Humidity sensitivity ✓ Detection of all carbon ions which can be detected by ionisation ✓ Easy to handle
Olfactory Gas Chromatography (GC-MS-Sniffer)	**Determination of several odour substances.** Some of the chromatographic eluent is diverted to a sniffing port	× No online measurement × Cost intensive, time consuming ✓ Detection of gas constituents as odorous substances
Infrared spectroscopy (IR)	**Detection of single gaseous substances.** A beam of infrared light is passed through the sample. Examination of the transmitted light reveals how much energy was absorbed at each wavelength. A transmittance or absorbance spectrum can be produced. Analysis of these absorption characteristics reveals details about the molecular structure of the sample.	× No detection of odour × Complex gas mixtures
Photoionization detector (PID)	**Measurement of several inorganic and primarily volatile organic compounds (VOC)** that have ionization potentials equal to or less than the energy of the UV radiation. e.g. detection of amines, benzene, aromatic compounds, ammonia, ethanol, acetone	× Humidity sensitivity ✓ Easy to handle
Photo acoustic Spectroscopy (PAS)	**Detection of gaseous substances.** Gas is irradiated with a light, which corresponds to a resonant vibration frequency. Some light will be absorbed. This cause excitation of some molecules. The relaxation process results in an increased heat energy of the gas molecules. This modulation of temperature and pressure results in an acoustic wave which can be detected by a sound-measuring device.	× No detection of odour Innova Airtech Instuments (2010): e.g. LumaSense
Field asymmetric ion mobility spectrometry (FAMS); also: Differential mobility spectrometry (DMS)	**Separation and identification of chemical ions** based on their mobility under a varying electric field.	× No detection of odour Schuhmann analytics (2010): e.g. Owlstone

4.1 List and specifications of instruments based on analytical methods other than electronic noses

Table 4-2 and *Table 4-3* provide a list of identified vendors or developers of systems for analytical instruments which can be used in the frame of odour measurement. Specifications are given for the instruments as available (from vendors and literature).

7 instruments were identified to have the potential to be employed for the monitoring of gaseous substance from wastewater systems. *Table 4-4* additionally lists 14 products where the availability is unclear (no response from vendor) and no specifications were available by the time of writing.

Application fields

Instruments based on gas chromatography or mass spectrometry which are suitable for online measurements have recently been developed. However, most of them are lab applications and have no implementation for wastewater works. Hence, the applicability has still to be proven for this field and actual special implementations for the monitoring of gaseous substances from sewers or wastewater treatment plants are rare. This is due to only recent market entries, new developments and ongoing adaptations of the systems. Ongoing developments mainly try to reduce the analysis time in order to provide online solutions.

Sampling method and gas conditioning

For the listed instruments headspace sampling is the most common. Each applied method requires certain sample preparation or gas conditioning.

Training

A training with olfactometry is usually not considered, hence there is no information on the odour concentration (in ou/m³) provided by the systems. Output parameters are usually atomic mass units (amu) or concentrations in ppm or ppb.

Costs

Compared to electronic noses the systems are more cost-intensive, ranging between 12 k€ and 150 k€. These more complex technologies however provide the possibilities to detect compounds down to the ppb-level without being highly sensitive to temperature or humidity.

Table 4-2: Specifications on identified analytical instruments for environmental odour monitoring 1/2

	Vendor	Product(s)	Sensor type/method	Application field	Sampling/gas conditioning	Measuring cycle	Comments
21	**Alpha M.O.S.**	FOX, GEMINI, HERACLES, KRONOS, PROMETHEUS, AIRSENSE Analyzer	MS, GC, FID	Waste management, wastewater, food, beverage, plastic and packaging, pharmaceutics and nutraceutics, health, cosmetics, flavors and fragrances, chemicals, paper & pulp, WWTP, landfill sites, composting plant	Dynamic. and static headspace Autosampler	1-2 to 20 minutes; 200 samples/day	Lab applications
22	**Electronic Sensor Technology**	zNose	Ultra-Fast GC; SAW Detector	Petro-chemical and Chemical, Food and beverages, environmental, research (chemical, biosciences), military, security	Internal sample pump; Thermal desorption	30 secs.	Purge gas: Helium
23	**LumaSense Technologies**	Innova 1412, 1316, 1314, 1313, 1309, 1303	Photoacustic infrared detection	Indoor air quality, health & safety, hospitals, greenhouse gas monitoring, chemical processes	Gas stream, internal pump	13-150 secs.	
24	**Next Dimension Technology**	Sensor array systems	Chemiresistive films; Custom-designed			5 min (adjustable)	no automated sampling capability (at the moment)
25	**Owlstone Nanotech**	LoneStar	FAIMS	Safety, industrial (petrochemical, etc.), automotive, Environmental (air quality, water quality), medical	Headspace, ambient air	1 sec ; with GC-column : 5-10 min	
26	**SLS Mikro Technology**	GCM 5000, GCM Micro Box	GC	Application specific adaptations (e.g. Biogas-analysis, sulphur-analysis, pyrolysis-gas analysis, BTX-analysis, methane-analysis, C5-C11, C3-C8, alcohol mixtures)	Headspace, injector	35-240 secs. (depends on application)	Different columns (depends on application & substances); carrier gas: H_2, He, N_2 or air; process control possible
27	**Smart Nose**	Smart NOSE-300	MS	Food, materials, polymers, paper, wood, chemicals, pharmaceutical, cosmetics, perfumes, polymers; environment, medical, research; microbiology	Headspace, Autosampler, Optional preconcentration, optional inlet device for online analysis	200 samples/day	Purge gas: N_2

Table 4-3: Specifications on identified analytical instruments for environmental odour monitoring 2/2

	Vendor	Product(s)	Data analysis methods	Output parameter	Measurement range; Accuracy	Temperature, humidity sensitivity	Stability	Price	References
21	**Alpha M.O.S.**	FOX, GEMINI, HERACLES, KRONOS, PROMETHEUS, AIRSENSE Analyzer	PCA, DFA, PLS, NN, Fuzzy, SIMCA	MS: Mass range: 1-200 amu	ppm range (MS, GC) <5% and <1 % in quantitative determination; MS: 1-200 amu	Humidity and temperature control	NA	20 k€ - 150k€	Dewettinck *et al.* (2001) Pardo *et al.* (2000) Röck *et al.* (2008)
22	**Electronic Sensor Technology**	zNose	NA	NA	in ppb for many compounds	Optional humidity filter (e.g. Anhydron); 0-95 % RH, non-condensing	NA	26 k€ (Model 4300)	
23	**LumaSense Technologies**	Innova 1412, 1316, 1314, 1313, 1309, 1303	NA	NA	ppb range; gas-dependent	Temperature influence: ±10% of detection limit®/°C Pressure influence: ±0,5 % of detection limit®/mbar	NA	NA	
24	**Next Dimension Technology**	Sensor array systems	NA	Signal strength, noise level	ppb range	NA	NA	12 k€ - 15 k€	
25	**Owlstone Nanotech**	LoneStar	NA	NA	ppb range	Temperature and humidity control, flow and pressure sensors	NA	30 k€	
26	**SLS Mikro Technology**	GCM 5000, GCM Micro Box		Internal calibration	< 80 % humidity	Adaption by variable columns (thin-layer columns, packed columns); thermal conductivity detector (temperature independent); No humidity control	No drift, very low maintenanc e	8 k€ - 19 k€	
27	**Smart Nose**	Smart NOSE-300	PCA, DFA	Mass range: 1-200 amu	1-200 amu	NA	Several thousands of analysis	> 80 k€	

Table 4-4: Further vendors of analytical instruments for air monitoring and analysis (unclear availability)

	Vendor	Sensor type/method	Website	Application field
28	**Agilent (US)**	Quadrupole fingerprint MS	www.chem.agilent.com	
29	**Avanzare (ES)**	EC		
30	**Chemsensing (US)**	Colorimetric array	www.chemsensing.com	
31	**CSIRO (AU)**	Receptor-based array Cybernose	www.csiro.au	
32	**Dräger**	Ion mobility spectrometry (Multi-IMS); EC	www.draeger-safety.com	
33	**Enviro Technology (UK)**	FID, etc.	www.et.co.uk	
34	**Environics (FI)**	Ion mobility spectrometry	www.environics.fi	
35	**Illumina (CA)**	Fluorescence sensors – bead array	www.illumina.com	Medical
36	**Ingegneria dei sistemi elettronici (IT)**		www.ise-srl.com	
37	**Leister (CH)**		www.leister.com	
38	**MSA (US)**	Infrared, PID, EC, catalytic, photoacoustic	www.msanorthamerica.com	Health and safety
39	**Proengin (FR)**	Flame spectrophotometer	www.proengin.com	Nerve Agents, Blister Agents, Blood agents
40	**RaeSystemes (US)**	Ion mobility spectrometry, GC, EC, PID	www.raesystems.com	
41	**UST – Umweltsensortechnik (DE)**	3-layered sensors	www.umweltsensortechnik.de	

Chapter 5

Conclusion

Objective of this report is to give an overview on available analytical instruments for odour measurement. Instruments have been identified which have the potential to be used for odour monitoring at wastewater collection systems or wastewater treatment plants.

Special focus is set to multigas-sensor systems, often called electronic noses, which are typically based on unspecific sensor arrays. A set of digital signals can be processed in order to extract information on odour characteristics and quantification. This needs to be done by adapted data analysis methods. If requested a link to odour can be established by correlating the data to olfactometric measurements (human nose observations). Then the systems can be capable of continuously providing information on odour concentration.

Besides multigas-sensor systems this report also provides a compilation of further analytical methods based on gas-chromatography (GC), mass spectrometry (MS). These methods are based on the detection of specific substances in the gas sample. Information on the olfactory components by GC-MS can be provided by applying a sniffing port after the separation of gas components (GC-MS-O). This however requires the deployment of a human nose, hence is not feasible in continuous mode and interactions between odorants are not considered.

18 different companies and research institutes were identified who provide electronic noses for odour measurement. They are based on the application of different sensor types (e.g. MOS, QMB, CPS, SAW, EC). 7 continuous working instruments based on other analytical methods were identified to have the potential to be employed for odour monitoring from wastewater works. Further 14 companies were identified for which no specifications or information on availability were available by the time of writing. Measurement instruments are typically connected with high purchase prices.

Appendix A

Contact details of vendors

Table A: Contact details of vendors of electronic noses

	Vendor	Product(s)	Country	Website	Email
1	**Airsense Analytics**	PEN, GDA 2	Schwerin, Germany	www.airsense.com	schmidt@airsense.com
2	**Alpha M.O.S.**	RQ BOX, lab applications	Toulouse, France	www.alpha-mos.com	aranyos@alpha-mos.com vivola@alpha-mos.com
3	**AltraSens**	OdourVector	Bonn, Germany	www.altrasens.de	boeker@uni-bonn.de
4	**Applied Sensor (sales DE: GSG Mess-und Analysegeräte)**	VOCmeter, VOCcheck, VOCvario	New Jersey, USA Sales: GSG-Bruchsal, Germany	http://www.appliedsensor.com www.gsg-analytical.com	info@appliedsensor.com application@gsg-analytical.com r.gohlke@gsg-analytical.com
5	**Comon Invent**	E-nose	Delft, Netherlands	http://www.comon-invent.com/	bootsma@comon-invent.com
6	**CSIC - Instituto Física Aplicada**	WiNOSE	Madrid, Spain	http://www.ifa.csic.es/Default.aspx?Lang=EN	josepe@ifa.cetef.csic.es
7	**Ezent**	iNOSE	Cottbus, Germany	www.ezent.com	kontakt@ezent.com
8	**GenMark Dx** (Osmotech PLC; formerly: Aromascan)	Aromascanner	California, US	http://www.genmarkdx.com/ http://www.osmetech.com/	info@genmarkdx.com
9	**KIT - Karlsruhe Institute of Technology**	SAGAS, KAMINA, SAMONA	Eggenstein-Leopoldshafen, Germany	http://www.fzk.de/ifia	michael.rapp@kit.edu
10	**Multisensoric GmbH**	Multigas-SENSORiCCARD®	Jena, Germany	http://www.multisensoric.de/1.html	hhh-ahlers@multisensoric.de
11	**Odometric**	FIDOR®	Arlon, Belgium	http://www.odometric.be	info@odometric.be
12	**Odotech**	OdoWatch®	Montreal, Canada	www.odotech.com	prenyi@odotech.com
13	**RST Rostock System Technik GmbH**	SamDetect	Rostock, Germany	www.rst-rostock.de	h.dietz@rst-rostock.de
14	**SACMI**	EOS Ambiente	Imola, Italy	http://www.sacmi.com	fabrizio.dematte@sacmi.it
15	**Scensive Technologie**	Bloodhound ST306® Bloodhound Odourspace	Leeds, UK	www.scensive.com/index.htm	member@merchab.fsnet.co.uk
16	**Scientec Lab Center Co., Ltd**	OdorCatch	Daejeon, South Korea	www.sclab.co.kr	kalhintzsunider@msn.com
17	**University of Extremadura**	UEX E-nose	Badajoz, Spain	http://eii.unex.es/profesores/jlozano/index_eng_archivos/Page888.htm	jesuslozano@unex.es
18	**3S Gas Sensing solutions**	OdourChecker SniffChecker	Saarbrücken, Germany	http://www.3S-ing.de	info@3s-ing.de

Table B: Contact Details of vendors of analytical instruments for air monitoring

	Vendor	Product(s)	Country	Website	Email
21	**Alpha M.O.S.**	RQ BOX, lab applications	Toulouse, France	www.alpha-mos.com	aranyos@alpha-mos.com vivola@alpha-mos.com
22	**Electronic Sensor Technology** (sales Europe: Brechbühler)	zNose™	California, USA Sales Europe: Brechbühler AG, Switzerland	http://www.estcal.com/index.html	sales@brechbuehler.ch peterpichler@brechbuehler.ch
23	**LumaSense**	Innova 1412	Frankfurt / Main, Germany	www.lumasenseinc.com	impac@lumasenseinc.com knauf@lumasensic.com
24	**Next Dimension Technology**	Sensor array systems	California, US	http://www.nextdimensiontech.com/index.htm	royea@nextdimensiontech.com
25	**Owlstone** Nanotech (sales DE: Schumann Analytics)	LoneStar	Cambridge, UK Sales DE: Einbeck, Germany	www.owlstonenanotech.com www.schumann-analytics.de	info@schumann-analytics.de Billy.Boyle@owlstone.co.uk
26	**SLS Micro Technology**	GCM	Hamburg, Germany	www.slsmt.com	kuelavir@slsmt.de
27	**Smart Nose**	Smart NOSE-300	Marin-Epagnier, Switzerland	www.smartnose.com	marketing@smartnose.com
28	**Agilent**	Quadrupole fingerprint MS	USA	www.chem.agilent.com	
29	**Avanzare**	EC	Spain		
30	**Chemsensing**	Colorimetric array	USA	www.chemsensing.com	
31	**CSIRO**	Receptor-based array Cybernose	Australia	www.csiro.au	
32	**Dräger**	Ion mobility spectrometry (Multi-IMS); EC	Germany	www.draeger-safety.com	
33	**Enviro Technology**	FID, etc.	UK	www.et.co.uk	
34	**Environics**	Ion mobility spectrometry	Finnland	www.environics.fi	
35	**Illumina**	Fluorescence sensors – bead array	Canada	www.illumina.com	
36	**Ingegneria dei sistemi elettronici**		Italy	www.ise-srl.com	
37	**Leister**		Switzerland	www.leister.com	
38	**MSA**	Infrared, PID, EC, catalytic, photoacoustic	USA	www.msanorthamerica.com	
39	**Proengin**	Flame spectrophotometer	France	www.proengin.com	
40	**RaeSystemes**	Ion mobility spectrometry, GC, EC, PID	USA	www.raesystems.com	
41	**UST – Umweltsensor technology**	3-layered sensors	Germany	www.umweltsensortechnik.de	

Appendix B

Outphased systems

Table C: Vendors and systems which are no longer available but often appear in literature

Vendor	Type	Product(s)	Comment on availability
e2V Technologies (formerly: Marconi Applied Technologies; Neotronics) **(UK)**	CPS	e-Nose 4000, e-Nose 5000	no e-noses anymore (only sensors)
Födisch Umweltmess-technik (DE)	MOS	OMD 1.10; OMD 98	No e-nose any more
HKR Sensorsysteme (DE)	QMB	QMB 6	No longer available
Lennartz Electronic (DE)	QMB	MOSES II	No longer available
Smith Detection (formerly: Cyrano Sciences) **(UK)**		Cyranose 320	No longer available
Sysca (DE)	MOS	ARTINOS 210, ARTINOS 510, ARTINOS 110	No longer available
Technobiochip (IT)	QMB	LibraNOSE	No longer available

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