Odour-sensor-assisted olfactometry

- Continuous on-line odour measurements -

Priv.-Doz. Dr. rer.nat. P. Boeker, Bonn, Dr.-Ing. G. Horner, München;

Abstract:
A new measurement system, the OdourVector™, combined with an appropriate methodology allows for real on-line odour measurements over periods of month. The ‘Odour-sensor-assisted olfactometry’ is based on a highly stable chemosensory measurement system. The integrated sensor array is adapted to odorous substances and very sensitive by means of a combined pre-concentration unit. The disadvantages of former approaches to odour measurements, e.g. sensor drift and degradation, are minimised because of a physical measurement principle. No potential irreversible and degrading sensor reactions are incorporated in the process. Three long term measurement projects from different areas are presented. The measurement of sewage odours is important for the control of counter measures and can also help to reduce the dosing of odour suppressing chemicals. The supervision of odour removing filters of an refuse incinerator is important for the environment and the inhabitants in the vicinity of the incinerator. Odours from the degradation of oils in the metalworking industry have been measured to tune and suppress the processes of formation of odorous substances.

Introduction
Olfactometry is the usual method for the measurement of odours. Unfortunately the inherent measurement error of the method is very high. The main cause lies in the certified range of the odour sensitivity of the panellists which is allowed to vary between 0.5 and 2 OU [1] of n-butanol. Other influences are the precision the olfactometer dilutes odour samples or bad management of the sampling process. As olfactometry uses the human nose as a detector this method cannot be used for continuous odour monitoring. On the other hand the inherent error of the method leads to ambiguous results when monitoring varying odour emissions.
Odour emissions frequently show high time variable dynamics, caused by the underlying processes, which can be complex for biological systems and depend on many external conditions. The intensity of the emissions is determined not only by the formation of odour substances, but also by their release. Changes of the fluid dynamics or the phase exchange conditions can lead to an increased release. This has serious consequences for the measurement of odour emissions by means of olfactometry. Olfactometric measurements are expensive and time consuming and can only be done at a discrete sampling interval. Fig. 1 shows hypothetical emission characteristics and the effect of the sampling time on the odour measurement. The olfactometry is dependent on samples. The timing of the sampling is crucial in order to get significant emission peaks. Small shifts in the sampling time can lead to completely different results, as shown in Fig. 1.

Fig. 1: Distortion of olfactometric odour measurements via the sampling process

The left bottom graph of Fig. 1 shows that high emissions can only be found at the beginning of the measurement period, whereas in the right graph only one high emission is found at the end of the measurement period. The low sampling rates in olfactometry lead to completely random results.

For the operator of an odour emitting plant this situation is not very satisfying. The information derived from olfactometry is determined by eventualities of the sampling. An analysis of the causes of odour problems cannot be based on the results of a measurement as presented in Fig. 1.

For these reasons we suggest a sensor assisted measurement of the odour emissions named ‘Odour-sensor-assisted olfactometry’.

Method of the ‘odour-sensor-assisted olfactometry’

To overcome the problems of discrete olfactometric measurements an on-line odour measuring system is proposed in combination with few olfactometric measurements. As an odour measuring system a gas measuring system is used, which has been optimised for the target gases and conditions of the respective application (see [2] for details). ‘Chemical imaging’ is used to identify different atmospheric conditions of the gas phase. The measurement system OdourVector [3] employs optimised, highly stable chemosensor arrays in combination with a gas pre-concentration unit.

As shown in Fig. 1, odour emissions of a time variable source should be measured continuously. OdourVector is an on-line measuring system, which measures the gaseous emissions continuously. The emission level can be assessed on the basis of the sensor signals. Quartz microbalance sensors with sensitive coatings for the target odorous gases in combination with thermal desorption provide a good indicator for the course of the odour emissions.

Now the sampling for the olfactometry can be synchronised with the emission peaks occurring at the time of the most relevant process conditions. The synchronisation of the olfactometric sampling with the OdourVector output highly reduces the number of olfactometric measurements necessary to recognise relevant emission peaks.

In Fig. 2 this alternative proceeding is shown. The emission process is measured on-line. Olfactometric samples are taken at times of high emissions, ideally, if the measuring system indicates an emission plateau. Some calibration measurements with low emissions are supplemented. A calibration method is then calculated using the entire sensory-chemical information of the sensors. The result is a (multi-dimensional) calibration function, which leads to a detailed reconstruction of the emission process.

The information gained by sensor assisted olfactometry goes far beyond that gained by classical olfactometry.

- The temporal gaps between individual measurements are supplemented by the continuous sensor data in connection with the odour calibration function.
- The temporal dynamics of the process induced emissions can be monitored clearly and without distortions, thus well-founded conclusions on the formation and release of odours are possible.
- The calibrated measuring system can log afterwards during an extended period the process of the odour emissions without further olfactometric measurements.
- The pattern of the sensor signals supplies further information, which indicates changes of the emission composition. Thus process conditions or different odour sources can be differentiated.

**Technology**

The on-line measurement of odours is a complex technical and methodical task. Past attempts were disappointing, because some basic conditions could not be met [4]. In order to get a reliable and meaningful output, odour measurement systems should comply with the following requirements:

- Reliable and long-term stable sensors and sensor arrays.
- Selective response of the sensors to odorous gases.
- Low sensitivity of sensors to non odorous main components.
- Application of a special methodology for odour measurements.
- Use of robust data interpretation methods.

The central deficit of past odour measurements was the neglect of the systematic difference between the human sense of smell and the physical principle of gas measuring systems.

A novel measuring system, however, meets all above requirements. The OdourVector from AltraSens uses physical chemically working long-term stable and low-drift quartz microbalance sensors. It has an integrated pre-concentration unit for better selectivity and sensitivity increase and a methodical approach to the measurement of odours is adapted.

The measurement principle of the OdourVector is ideal for higher molecular odorous gases, which are measured with a high sensitivity. The stability of the system allows for long term...
measurements over periods of months without maintenance or recalibration. Once a calibration by olfactometry is made it is valid for a long period of time.

Applications

The method of the ‘Odour-sensor-assisted olfactometry’ has been applied on different applications. Three examples show the potential of the method and measurement technology.

1. Monitoring of sewage odours
The aim in an evaluation project was the measurement of sewage odours. The measurement system OdourVector was installed at a sewage pump station with an anti odorant dosing facility. In Fig. 3 the course of the sensor data is shown. It comprises of about 7,000 complete cycles of pre-concentration and sensor measurements. During the measurements the system was calibrated with olfactometric reference data. Fig. 4 shows the calibration characteristics. The error band of 50% includes all (but one) of the measurements, indicating that the calibration is well within the error band of olfactometric measurements. The course of the calculated odour concentration as measured with the calibrated system is presented in Fig. 5.

![Sensor signal vs. Time](image)

Fig. 3: Long term measurement of sewage emissions with the OdourVector system (6 weeks shown out of 14 weeks total measurement time), Letters A and B indicate different chemical composition and hence different odour qualities.
Fig. 4: Calibration of the OdourVector by olfactometric measurements. The error bands between +- 50% include most of the reference measurements.

Fig. 5: Calculated course of the odour emission from the sewage plant.
Monitoring of odour filters

In an infuse incinerator odour filters control the odour emissions. The efficiency of the filters is temporarily checked by olfactometric measurements. In order to further increase the absorption of odours the filter function shall be controlled by an automatic on-line monitoring system. During a period of three months the OdourVector monitored the course of the emissions. In this time two of the filters (1 and 2) lost their capacity to remove odours. The increase of odours is clearly shown in Fig. 6. The olfactometric references are indicated as dots. Fig. 7 shows the calibration between sensor signals and the odour concentration. Not only the signal intensity but also the signal pattern changes when the filters loose there ability to remove odours. Fig. 8 shows the pattern change on the discriminant plane. The measurements can be divided into three classes: clear air with well working filters, odorous air and signal patterns when the degradation process of the filter takes place.

Fig. 6: Calculated odour concentration and reference olfactometry. The actual used filter units are indicated below.
Fig. 7: Calibration between sensor signals and the odour concentration.

Fig. 8: Principal component analysis of the clean air (left class) and the odorous air with exhausted filters (right class). Between the two classes the transition of the signal patterns during the degradation of a filter can be observed.
Monitoring of odour emissions from metal processing

In a metal rolling plant oil is used in the process. The degradation of the oil leads to the release of odours. The maximum allowed odour emission is limited by technical regulations. In order to avoid the formation of odorous substances the on-line monitoring of the actual level is useful for the analysis of formation processes and influences on the release of odours.

In a first calibration phase (Fig. 9) the OdourVector measured the emissions while olfactometric reference measurements were taken. Based on this calibration the odour emissions were measured during three weeks (Fig. 10).

![Fig. 9: Calibration period for the on-line monitoring of emissions from metal processing](image-url)
Fig. 10: Long term odour measurement with process induced fluctuations.

Summary

‘Odour-sensor-assisted olfactometry’ can considerably improve the monitoring of odour emissions and help to control and counteract odour emissions in a variety of applications. The combination of quartz microbalance sensors with a pre-concentration unit proved to be very effective for the measurement of odours as the combination enhances sensitivity and selectivity for the relevant substances. The application specific calibration of the chemosensory measurement system by olfactometric measurements produces reliable and long-term stable predictions of odour emissions. Due to the short measurement interval of the sensor system odour emissions can be monitored on-line and counteractions can be taken before large quantities of odorous substances are released into the environment.
