

# Performance Test of a Sensor Array - Based Odor Detection Instrument

Arief Sabdo Yuwono<sup>1</sup> and Peter Schulze Lammers<sup>2</sup>

<sup>1</sup> Dept. of Agricultural Engineering, Bogor Agricultural University (IPB), PO Box 220  
Bogor 16002, Indonesia. E-mail: [ayuwono@ipb.ac.id](mailto:ayuwono@ipb.ac.id)

<sup>2</sup> Dept. of Agricultural Engineering, University of Bonn, Nussallee 5, 53115 Bonn,  
Germany. E-mail: [lammers@uni-bonn.de](mailto:lammers@uni-bonn.de)

## ABSTRACT

A laboratory performance test of a new odor detection instrument based on a quartz crystal microbalance (QCM) sensor array was conducted in the Department of Agricultural Engineering, University of Bonn. The performance test includes the sensor's response time to the presence of odor, sensor sensitivity and detection limit, sensor frequency stability, the influence of water content on the sensor frequency change and the response pattern of the sensor array to the presence of odor substances at various concentrations. In the test, nine odor substances were used as odor samples, i.e., acetone, acetic acid, 2-butanone, 1-butanol, ethanol, ethyl acetate, hexane, limonene, and methanol. The results showed that the QCM sensor array was responsive to all nine odor compounds in the gas phase. The frequency of the sensor array was stable. The sensitivity and detection limit of the sensor array were improved after the installation of a thermal desorber preconcentration unit.

**Keywords:** performance test, odor, detection, instrument, quartz crystal microbalance.

## INTRODUCTION

The development of odor detection instruments is still a challenge because of a lack of technical solutions. The odor problem continuous to persists due to agricultural sources. Therefore, the existing methods of detection need to be improved. Sensor array - based odor detection instrumentation is an example of the advanced achievements. To develop an excellent and reliable instrument, however, it is necessary to conduct a series of performance tests in the laboratory as well as in the field. This paper deals with the performance test of an odor detection instrument based on a quartz crystal microbalance (QCM) sensor array which was developed in the Department of Agricultural Engineering, University of Bonn. The QCM sensor array - based instrument was tested in the laboratory before being implemented in the field to detect odor emissions under natural conditions. The results of the test were assessed and will be used as a guide for the field test. The results of the performance test can also be used to evaluate the instrument, so that its implementation in the field will result in reliable data.

The objectives of the laboratory test were as follows:

1. Test the performance of a QCM sensor array including sensor response time and recovery time, sensitivity and detection limit with regard to single odor compound emissions, and influence of water content on the sensor frequency change

2. Implement the QCM sensor array - based instrument to detect single odor compound emissions
3. Integrate a thermal desorber preconcentration unit into the QCM sensor array - based instrument and assess its performance.

## MATERIALS AND METHOD

Nine odor substances in liquid state were used as source of odor samples in the test, i.e., acetone, acetic acid, 2-butanone, 1-butanol, ethanol, ethyl acetate, hexane, limonene, and methanol. These substances are identified by an analysis of the raw gas of compost facilities and some are typical odorous compounds. The set-up of the odor detection instrument based on the QCM sensor array is presented in Figure 1. Basically, it consists of an array of six sensor elements in a temperature-controlled environment, a carrier (purge) gas source, a magnetic valve, an odor sample bubbler, mass flow controllers (MFCs), a computer and the accompanying software. After a series of measurements on various odor substances, the sensor array was improved by means of an integrated thermal desorber preconcentration unit installed to enhance its sensitivity and detection limit.

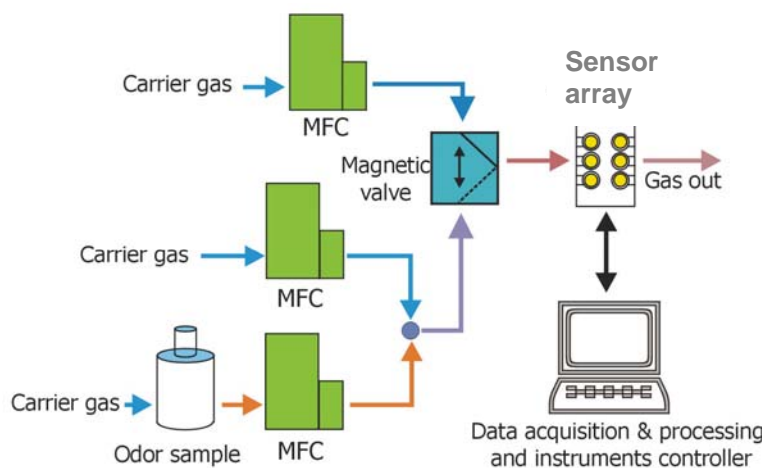


Figure 1. Set up of the QCM sensor array - based instrument for the detection of odor emissions. MFC = mass flow controller, carrier gas = nitrogen

Typically, odor molecules are volatile organic molecules with relative molar masses between 30 and 300 Dalton (Gardner and Bartlett, 1999). In the laboratory test, the molecular masses of the odor substances were between 32.04 (methanol) and 136.23 (limonene). At a composting facility near Bonn it was observed that the 22 odor substances, identified using gas chromatography - mass spectrometry (GC-MS), have molecular masses between 46.07 (ethanol) and 136.23 (limonene). The QCM sensor array was responsive to all of these compounds which make up the complex odor of the composting facility.

To measure the frequency change as the response by the sensor to the presence of odor substances in the measuring chamber, pure liquid odor substances were placed in the bubbler and kept in a controlled chamber by using a water bath (*Lauda Ecoline Refrigerating Circulator Model RE 204*). The water bath was set at a constant temperature of 20 °C. The gas concentration was determined by saturated atmosphere generated by a bubbler set at a constant temperature. The odor gas was diluted with the carrier gas by using a mass flow controller (MFC). The MFCs working in a serial arrangement cause an error of less than 5 %. The concentration of the odor vapor flowing out from the bubbler can be kept by controlling air flow rate in each MFC (*Brooks Instruments*). The magnetic valve makes possible an automatic switch between sample gas and carrier gas. The carrier gas was needed not only to clean the sensor chamber but also to dilute the odor vapor. Entering the sensor array module, the odor molecules of the sample gas interact with the sensitive coating materials, which results in a change of sensor frequency,  $\Delta f$ , from its basic frequency (see Figure 2).

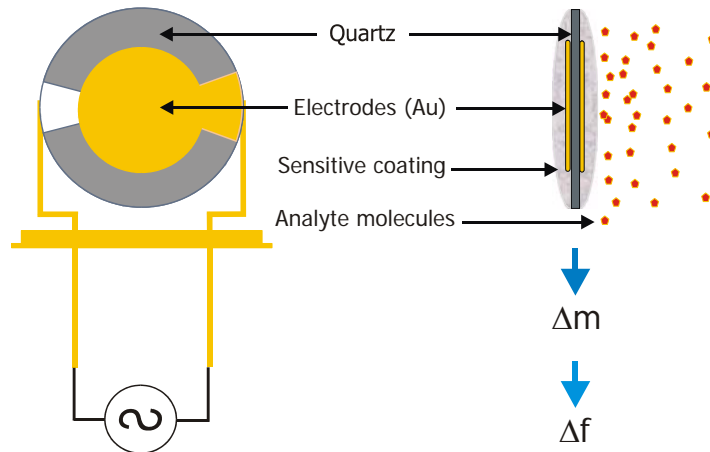


Figure 2. Schematic illustration of a quartz crystal microbalance (QCM) sensor

According to the Sauerbrey equation (Sauerbrey, 1959; Cattrall, 1997) the change of sensor frequency can be expressed as follows:

$$\Delta f = -2.3 \times 10^6 f_0^2 \left( \frac{\Delta m}{A} \right)$$

where:

- $\Delta f$  = the frequency change (Hz),
- $-2.3 \times 10^6$  = a constant ( $\text{Hz cm}^2 \text{g}^{-1} \text{MHz}^{-2}$ ),
- $f_0$  = the oscillating frequency of the quartz crystal (MHz) (for a typical AT-Quartz,  $f_0 = 10 \text{ MHz}$ ),
- $\Delta m$  = the mass change of the adsorbed analyte, i.e. odor substance (g), and
- $A$  = the area coated by the film ( $\text{cm}^2$ ).

The equation states that there will be a sensor frequency change proportional to the mass of the material (i.e., odor substances) deposited on the sensor surface. The frequency change

was monitored and recorded by means of a personal computer which was used for data acquisition and processing as well as for controlling all devices involved.

Water and odor substances were calibrated to illustrate the response of the sensor array to the presence of odor substances and water vapor in the measuring chamber. Calibration curves were drawn by plotting the frequency change of the sensor versus the ascending concentration level of odor or water vapor. The sensitivity of the sensor array was measured by introducing the odor samples to a series of descending concentrations. The sensitivity of the sensor array is defined as the ratio between the sensor frequency shift and the associated odor concentration change. In other words, the sensitivity of a sensor is the frequency change due to every unit of odor concentration change. Therefore, the measurement unit is (Hz/ppm) or (Hz mg<sup>-1</sup> m<sup>3</sup>).

The detection limit of the sensor array was measured by introducing a series of odor samples with descending concentrations until the sensed signal was about 3 times the signal noise. The odor concentration at this level is defined as the detection limit of the sensor array. In other words, the detection limit of a sensor array is the lowest concentration of an odor substance that can still be detected by the sensor when the sensor frequency change is not lower than 3 times the noise. The measurement unit is (mg/m<sup>3</sup>). The sensor detection limit and sensitivity were used as basic information on whether it would be possible to measure odor concentrations in the field with this instrument.

The response time of the sensor is here defined as the time required by the sensor to reach 90% of the final response. It is obtained from the graph that describes the relationship between sensor frequency change and elapsed time. The unit of the response time is seconds. The recovery time is the time required by the sensor to reach a normal basic frequency after it has shown the final response frequency of a measurement cycle.

## RESULTS AND DISCUSSION

The performance test showed that the QCM sensor array was responsive to all 9 odor compounds in the gas phase. The response by the QCM sensor array to the presence of the odor substances in the measuring chamber was obtained in the form of a frequency change. The QCM sensor array also showed that it is responsive to odor substances at various concentration levels. From the calibration curves it is clear that the dynamic response is logarithmic rather than linear. Furthermore, no saturation was observed for the concentration range in the test.

The QCM sensor array was designed as a non-specific or non-selective sensor. It was responsive not only to one specific odor compound but to all odor compounds in the test. This is due to the sensitive coating materials on the sensor surface. In further stages of development, the sensor array can be a basic part of an electronic nose since the design of electronic noses is based on an array of non-specific chemical sensors (Vlasov and Legin 1998). A pattern recognition system will then be necessary to mimic the working principle of a human nose.

## Response Time and Recovery Time

The definition of response time ( $t_{on}$ ) varies. Some authors state that it is the time required by a sensor to reach 80% of a full-scale reading, others refer to 90% (Gardner and Bartlett, 1999) or 95% of a full-scale reading (Cattrall, 1997). In general, however, response time is ideal if it is only a few seconds. A combined response and recovery time ( $t_{on} + t_{off}$ ) of the order of one or two minutes is adequate (Gardner and Bartlett, 1999).

A schematic calculation of the sensor response time ( $t_{on}$ ) and recovery time ( $t_{off}$ ) for single odor emissions is presented in Figure 3. This is an illustration of the response time of the QCM sensor array to acetic acid. The response time is here defined as the time required by the sensor to read 90% of the full-scale reading (full response) after being exposed to an odor substance. The full response value is ca. 40 Hz, i.e., 90% equals about 36 Hz. In the test, the time required by the sensor to reach the full value was ca. 6 sec, and recovery time ca. 27 sec.

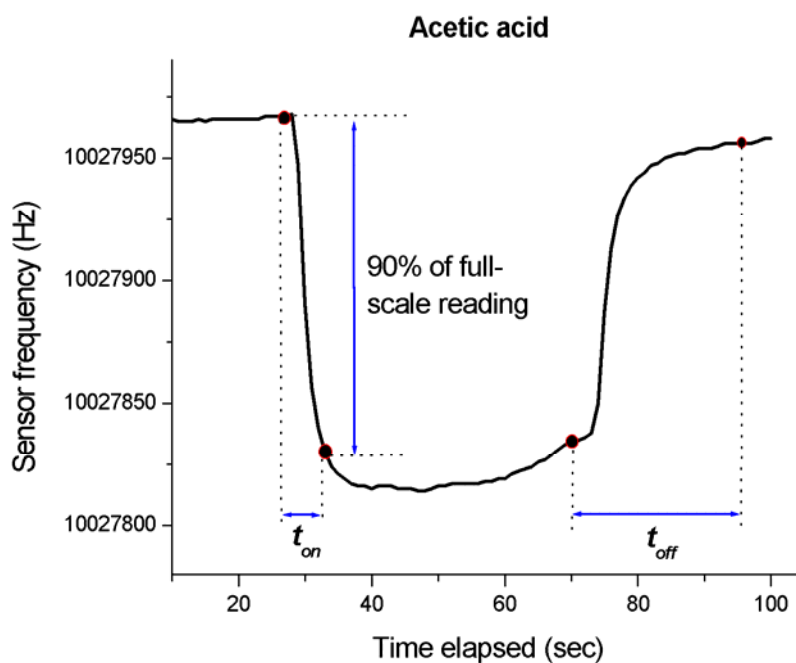


Figure 3. Response time of a single sensor ( $t_{on} \cong 6$  sec) in accordance with 90% of full-scale reading and recovery time ( $t_{off} \cong 27$  sec)

## Sensitivity and Detection Limit

Figure 4 shows the sensitivity of the sensor array in a test on various odor compounds. The highest sensitivity,  $36 \text{ Hz g}^{-1} \text{ m}^3$ , is reached by sensor 4 on limonene, the lowest with hexane, ca.  $0.1 \text{ Hz g}^{-1} \text{ m}^3$ . Hexane always leads to a minor frequency change on all sensors. In contrast, limonene causes high frequency changes on all sensors. This sensitivity variation is due to the different sensitive coating materials on the sensor surfaces. A number of polymers can be used on QCM sensors as sensitive coating materials.

The QCM sensors obtain their sensitivity and selectivity from a chemically active coating, which interacts with the surrounding environment (Lucklum and Hauptmann, 2000). Basically the QCM sensor sensitivity can be enhanced by raising its resonant frequency (Dickert et al., 1998). The sensor module is equipped with 10 MHz oscillators.

The choice of a new type of sensitive coating material (sensing reagent) is of considerable importance if sensors are to be provided with good sensitivity properties (Yang et al., 2000). The sensitivity of the QCM sensor array should be considered when selecting and preparing coating materials. As an additional measure, a preconcentrator can also improve sensor sensitivity (Nakamoto et al., 2000).

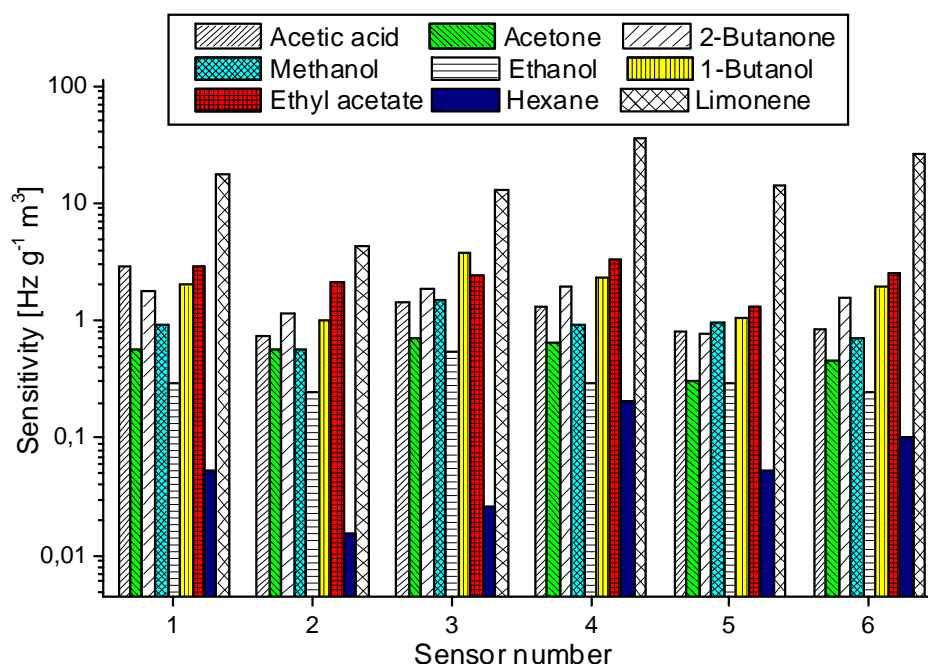


Figure 4. Sensitivity of the sensor array without preconcentration unit to various single odor compounds

Table 1 shows the detection limit of the sensor array after a series of laboratory tests as compared with odor threshold values. As indicated in the table, the average detection limits of the sensor array of odor substances under test conditions are about 400 to 500 times as

high as the odor threshold values. This indicates that, in terms of detection limits, the sensor array is not better than the human olfactory system. The performance of the system needs to be improved so that the detection limits close to or below the odor threshold values.

Without a preconcentration unit, the average sensitivity of the sensor array was merely 0.0175 Hz/ppm. This denotes theoretically that an odor concentration change of 1-ppm in the air results in a 0.0175 Hz frequency change, which is relatively difficult to detect. On the other hand, an odor concentration change of 1-ppm can be significant for odor substances with low threshold values, e.g., 1-butanol, acetic acid, and methanol (see Table 1).

The low sensitivity of the instrument can lead to a situation where a high concentration change of an odor substance in the environment does not lead to a visible signal. Therefore, a marked concentration change can go undetected by instruments with a low sensitivity. A poor detection limit results in an undetected odor even though its concentration is high. An improvement of the measuring system is, therefore, necessary in order to enhance the sensitivity and the detection limit.

Table 1. Odor threshold values and detection limits of the sensor array without preconcentration unit

Odor substance		Detection limit of sensor array		Odor threshold value
Name	Formula	(ppm)	(mg/m <sup>3</sup> )	(mg/m <sup>3</sup> )
Acetone	C <sub>3</sub> H <sub>6</sub> O	700	1663	48 <sup>(1)</sup>
Acetic acid	C <sub>2</sub> H <sub>4</sub> O <sub>2</sub>	160	393	2.5 <sup>(1)</sup>
2-Butanone	C <sub>4</sub> H <sub>8</sub> O	300	885	6 <sup>(2)</sup>
1-Butanol	C <sub>4</sub> H <sub>10</sub> O	100	303	0.123 <sup>(1)</sup>
Ethanol	C <sub>2</sub> H <sub>6</sub> O	3000	5652	19.1 <sup>(1)</sup>
Ethyl acetate	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>	300	1081	22 <sup>(1)</sup>
Hexane	C <sub>6</sub> H <sub>14</sub>	470	1657	458 <sup>(3)</sup>
Limonene	C <sub>10</sub> H <sub>16</sub>	10	63	100 <sup>(4)</sup>
Methanol	CH <sub>4</sub> O	4000	5242	5.3 <sup>(1)</sup>

(1) LAI (2000); (2) Burright (1990); (3) EPA (2001<sup>b</sup>); (4) Gardner and Bartlett (1999)

There are a number of methods for improving the detection limit of this sensor system. The method chosen in our research project is the use of a preconcentration unit. Here, odor molecules in the sample are preconcentrated by means of a thermal desorption mechanism. The concentrated target odor molecules are then released gradually and detected by the sensor. A review on adsorbent materials commonly used in air analysis and thermal desorption of volatile organic compounds can be found in Dettmer and Engewald (2002) and Baltussen et al. (2002).

The second method is an improvement of the sensor itself, i.e., by selecting specific coating materials. The coating is the limiting factor in the development of mass sensitive sensors (Dickert et al., 1996). The coating issue includes the choice of coating materials and coating techniques as well as the study of the host-guest mechanism between odor molecules and

the sensitive coating materials. A number of studies and researches related to coating issues have been carried out, e.g., on coating materials (Buhlmann et al., 1995; Cao et al., 1996; Dickert et al., 1999; Jarrett and Finklea, 1999) and guest-host chemistry (Dickert et al., 1996; Dickert et al., 2000<sup>a</sup>; Dickert et al., 2000<sup>b</sup>).

### **Improving QMC Sensor Array Performance with a Thermal Desorber Preconcentration Unit**

The results from the previous tests showed that the detection limits of the sensor array for various odor substances, except for limonene, were far above the odor threshold values. In terms of the detection limit, the instrument is not better than the human nose. The sensitivity of the sensor array (average value = 0.0175 Hz/ppm) is also low. Therefore, a thermal desorber preconcentration unit (Figure 5) was installed in an attempt to improve the sensitivity and the detection limit. Findings on the use of a preconcentration unit to improve the sensitivity and detection limit of any measurement process can be found in, for example, Valcarcel (2000), Nakamoto et al. (2000), Briand (2002) and Dettmer and Engewald (2002).

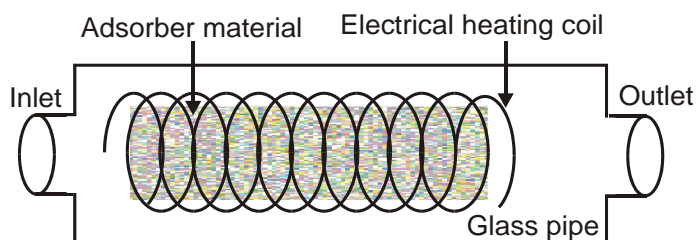


Figure 5. Thermal desorber preconcentration unit

The integrated thermal desorber preconcentration unit in the sensor system was made up principally of 35 mg Tenax GR as adsorbent and a heating system to enable the adsorption-desorption mechanism (Niess, 2002; Yuwono, 2003). By regulating the inner temperature of the preconcentration unit and the flow rate of the preconcentrated sample, the odor substances from the sample were first preconcentrated and detained in the adsorbent and then discharged into the measuring chamber of the sensor system. Thus, non-target components can be removed from the sample and the target components (i.e. odor molecules) can flow through the measuring chamber. The integration of the thermal desorber preconcentration unit into the sensor system resulted in an improvement of the sensitivity and the detection limit of the sensor array by average factors of 42 and 23, respectively (Yuwono et al, 2002).

### **Sensor Frequency Stability**

The QCM sensor array was tested continuously for 44 hrs in the laboratory to observe its frequency stability. A total of 15686 frequencies were recorded (each record lasting 10



sec). The test results are presented in Table 2. In general, the sensor frequencies are sufficiently stable. Only a slight frequency change, known as a *drift*, which is a normal phenomenon of sensor behavior, was observed during the test. A drift is a temporal change of the sensor response under constant physical and chemical conditions (Holmberg et al., 1997). The low standard deviation values (Table 2) show that the sensor frequencies were at an average level.

Table 2. Average, maximum, minimum and standard deviation (SD) of sensor frequency during continuous test (n = 15686)

Frequency	Sensor number					
	1	2	3	4	5	6
Average	10027844	10048681	10066638	10038312	10058225	10077418
Maximum	10027855	10048685	10066655	10038326	10058238	10077432
Minimum	10027606	10048563	10066491	10037504	10057875	10077033
SD	6.4	4.4	7.7	11.2	8.2	8.6

Sensor drift can occur with time and can be due to aging effects or slow morphological changes of the sensor materials (Gardner and Bartlett, 1999). According to Holmberg et al. (1997), the reasons for drift are unknown dynamic processes in the sensor system, e.g., poisoning or aging of the sensors or environmental changes such as temperature and pressure variations. Roth et al. (1996) proposed a method for reducing long-term drift effects by temperature modulation together with adapted signal processing. Holmberg et al. (1996) proposed two methods to counteract the drift in sensors used in the electronic nose. The first method is based on reference patterns that are updated when new measurements are made; the other uses models known from the system identification theory. Both were suited for different experimental situations.

### Response of the QCM Sensor Array to Water Vapor

The QCM sensor array is found to be responsive to water vapor. The water content of air can be expressed in forms of ppm<sub>v</sub> (parts per million, volume based) or gram water vapor per liter of air. The water content of air can be read in a psychrometric chart by providing data of the dew point temperature or two of the following parameters: relative humidity, dew point temperature, wet bulb or dry bulb temperature. Here, the response of the QCM sensor array to water vapor is expressed as the relationship between the dew point temperature with associated water content and the frequency change, as described in Figure 6. However, for measurements of odor emissions in the field, the influence of water vapor in the sample can be eliminated. This is due to the calculation technique that was based only on the difference between “signal with and without odor”, regardless of whether water vapor was present. Furthermore, the sample can be kept at a constant temperature by using a cooling unit as well.

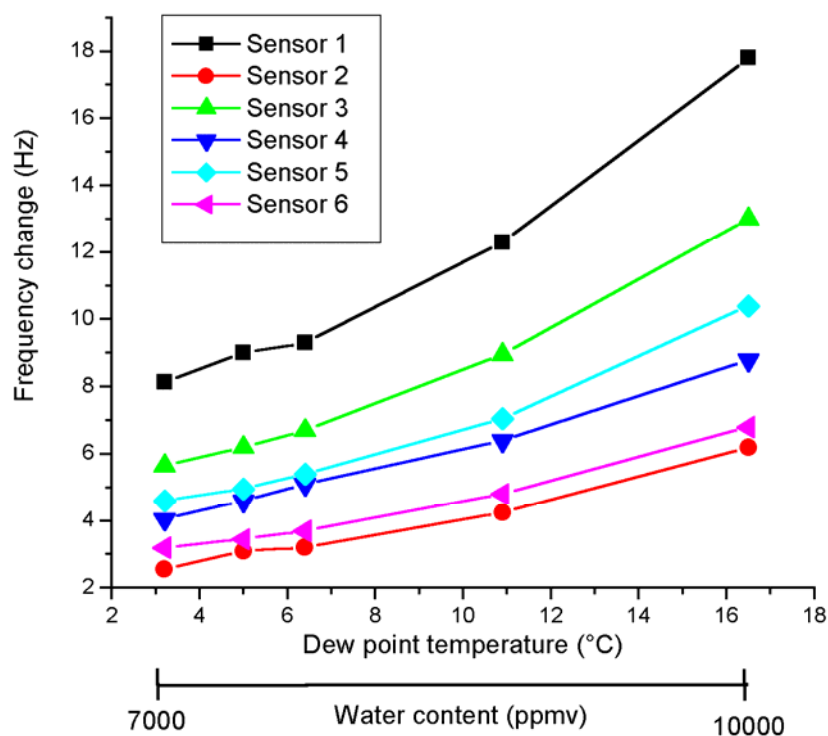


Figure 6. Influence of water content on sensor frequency change

### Response of the QCM Sensor Array to Different Odor Substances

Figure 7 shows the polar plots of the single odor compound emissions to be detected. Odor substances from a certain chemical group, for example alcohols (i.e. butanol and ethanol) show an almost similar pattern; here the highest response was obtained from sensor 3 (see 2-butanone, acetone, ethanol, methanol and butanol).

Generally, the responses of the QCM sensor array to different single odor emissions are not linear. At low concentrations it has a linear form, whereas when the concentration becomes higher, responses are no longer linear. Most of these relationships (Figure 8) seem to be logarithmic functions, except for those relating to acetic acid.

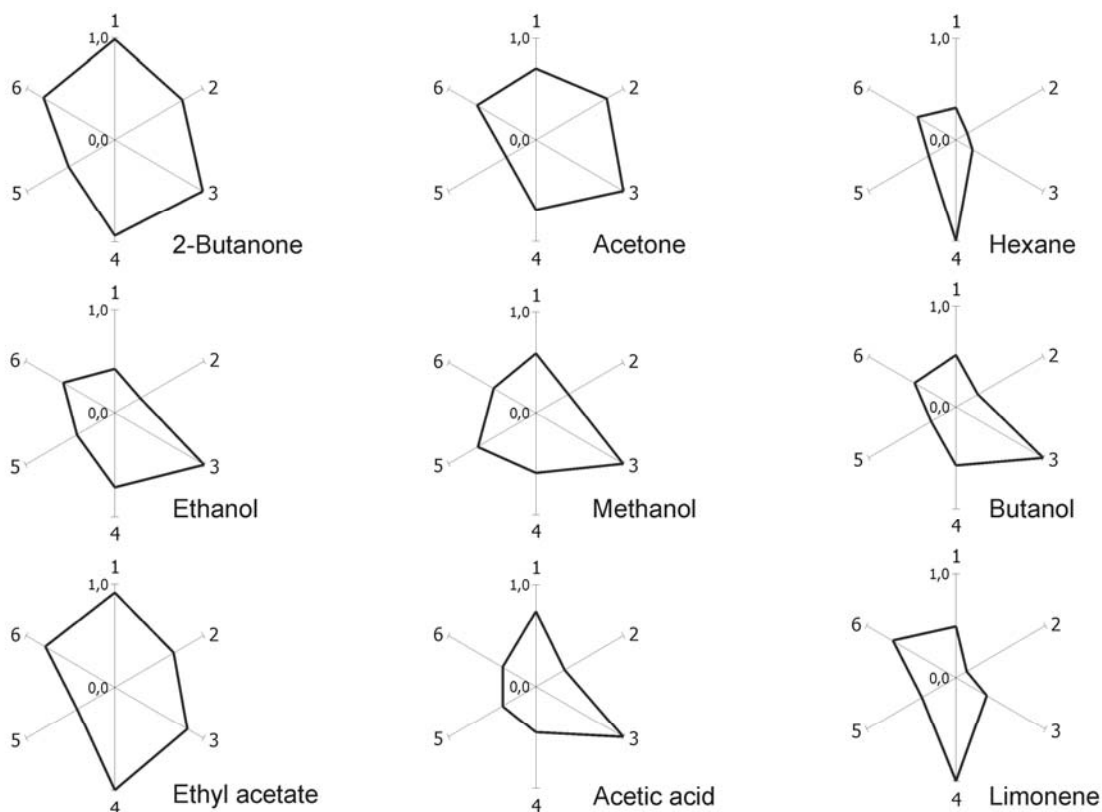


Figure 7. Polar plots of single odor compound emissions detected by the QCM sensor array - based instruments. Numbers 1 to 6 are sensor numbers

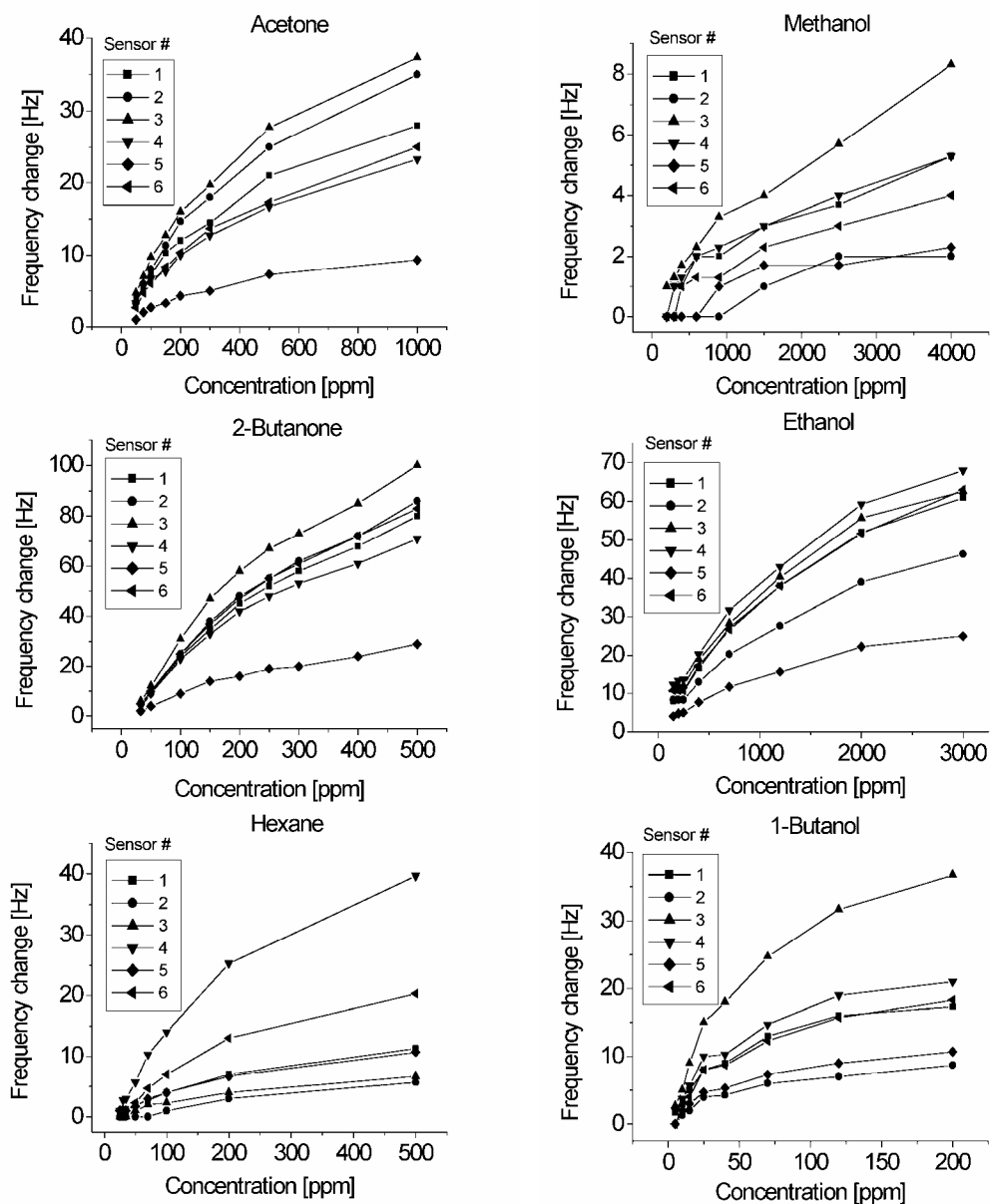


Figure 8. Response of QCM sensor array to different odor substances after installation of thermal desorber preconcentration unit

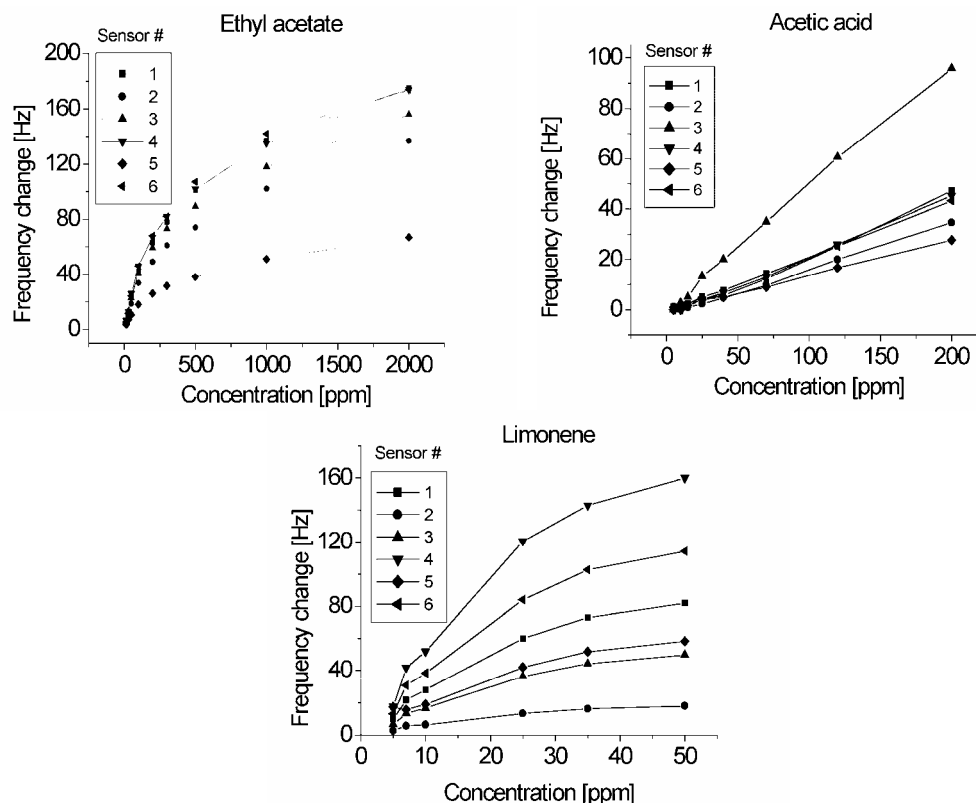


Figure 8. Response of QCM sensor array to different odor substances after installation of thermal desorber preconcentration unit (continued)

These relationships can be used as basic information on the sensor behavior. Together with odor detection threshold data, it can be determined whether this sensor can be used to detect odor at very low concentrations. For example, the detection limits of the QCM sensor array regarding acetone ( $1663 \text{ mg/m}^3$ ) and methanol ( $5242 \text{ mg/m}^3$ ) are relatively high. On the other hand, the threshold values of these substances are very low, i.e.  $48 \text{ mg/m}^3$  and  $5.3 \text{ mg/m}^3$ , respectively (see LAI, 2000). This means that the detection limits of the QCM sensor array without a preconcentration unit are far above the threshold values. This indicates, the sensor array without a preconcentration unit does not detect certain odor substances at the lowest concentration level, a level at which they can already be sensed by the human nose.

## CONCLUSIONS

The performance of a newly developed instrument for odor emission detection based on a quartz crystal microbalance (QCM) sensor array was tested under laboratory conditions to determine its ability to detect single odor compound emissions. The test results show that the frequencies of all sensors are relatively stable. The response time is ca. 6 sec, while the recovery time is about 27 sec. The QCM sensor array is responsive to all single odor

compound emissions tested. The relationship between odor concentration and the frequency change of the sensor is not linear. A series of performance tests showed that the sensitivity and the detection limit of the sensor array without a preconcentration unit are very low. The use of a thermal desorber preconcentration unit leads to improvements of the sensitivity and of the detection limit of the sensor array by factors of 42 and 23, respectively.

## ACKNOWLEDGEMENT

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