

Instability of conducting polymer sensors in an electronic nose system

E. Schaller¹, J. O. Bosset^{1*} and F. Escher²

¹ Federal Dairy Research Station, CH-3003 Bern, Switzerland

² Swiss Federal Institute of Technology (ETH), Institute of Food Science, CH-8092 Zürich, Switzerland

Abstract. A solution of 1 mL/L ethanol in water was used to study the repeatability of measurements performed with an electronic nose equipped with 12 conducting polymers based on polypyrrole. The sensor responses exhibited a poor repeatability independently of whether synthetic air or nitrogen was used as carrier gas. The responses varied from one day to the next as well as from one hour to the next. The variations in the baseline resistances, which were also examined, did not help to explain the observed instability. The variations in sensor response could however, be partly explained by a temperature fluctuation of the sensor module. A correction for the temperature changes halved the scatter of the measurements.

Keywords. Electronic nose – polypyrrole – conducting polymer – ethanol.

Introduction

Most studies on electronic nose systems published so far are based on metal oxide semiconductor (MOS), and organic conducting polymer (CP) sensors [1]. These two technologies represent the oldest used in the field of electronic noses, and both rely on changes in resistance due to adsorption of gases. When a voltage is applied across the electrodes, a current passes through the metal oxide or polymer surface. The interaction of volatile compounds with the surface alters the electron flow in the system, and hence, the resistance of the sensor [2]. The mechanisms are however very different. The MOS sensors, working at high temperature, typically 200–600 °C, are based on a combustion principle. The mechanism of CP sensors, which work at room temperature, is more complex and is not yet completely understood.

CP sensors comprise a substrate, e.g. fibre-glass or silicon, a pair of gold-plated electrodes, and a conducting organic polymer such as polypyrrole, polyaniline or polythiophene as sensing element. The polymer film is deposited by electrochemical deposition between the two electrodes which have been previously fixed to the substrate [3,4]. As the conducting polymer is grown from a solution, the film contains cation sites balanced by anions from the electrolyte as well as solvent residue [5–7]. The cation sites probably consist of polarons or bipolarons which are small regions of positive charge in the polymer chain providing mobile holes for electron transport. The volatile compounds can interact with at least 3 components within the polymer coating: (i) the polymer itself, (ii) the counterion, or (iii) the solvent [6]. Slater *et al.* [8] studied the response mechanism of polypyrrole sensors in more detail and showed that the changes in resistance are due to a mixed response involving physical

and electronic effects. Certain volatiles have a solvent type action on the polymer causing it to swell, and thus hinder the electron mobility within the coating. The electronic transport process in polypyrrole films can be divided into transport of charge along the polymer chains caused by mobile carriers, such as polarons and bipolarons, and the hopping of charge between the chains. The choice of one process over the other is determined by the conductivity of the film, which in turn is determined by “doping concentrations”, the conjugation length of the polymer chain, the chain arrangement, and the position of the counter ions relative to the chains [9,10].

A few studies with electronic noses equipped with CP sensors concluded that the responses were well reproducible [11–13]. However, most researchers found two main drawbacks for CP sensor: i) a high sensitivity to moisture, and ii) a rapid drift of their responses over time [3,13–20]. The drift could be partly explained by the oxidation of the polymer coating in contact with volatile compounds and air [21]. Most studies reporting an instability of CP sensors do not give any precise data. They only mention problems of repeatability or a drift of the sensors over time. Few studies [13,17] report in more detail the nature and the amplitude of the changes in the sensor responses and baseline resistances. The instability was noticed over a period of several days, but none of these studies mentioned a instability within one day.

The aim of the present investigation was to measure the repeatability of an electronic nose equipped with 12 CP sensors based on polypyrrole in the course of the day and over a 3-month period. A solution of 1 mL/L ethanol in water was

*Correspondence and reprints.

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used to follow the changes in the sensor response as well as in the baseline resistance.

Experimental part

An eNose5000 (EEV Limited, Essex, UK) equipped with 12 organic conducting polymer sensors based on polypyrrole was used. The system comprises a CTC Combi PAL autosampler containing 2 trays of 32 vials each.

The headspace is generated statically. The sample to be analysed is placed in an oven at a defined temperature and shaken for a user-defined time. A 5 mL syringe takes a portion of the headspace and injects it into a 5 mL Teflon loop where it is trapped by a 6 port valve. After injection of the syringe content, the valve is switched and the volatiles are mixed with a carrier gas at a defined flow rate. The sample is transferred to a control module, which measures the temperature, flow rate and percentage relative humidity of the mixture of volatiles and carrier gas. From this chamber, the gas mixture flows to the sensor module where it is kept for a user-defined time period. The volatile compounds are then purged out through the rear of the instrument. Finally, the sensors are washed with the carrier gas to allow them to return to their baseline resistance values. During the “trap” period, the volatiles interact with the sensors which alters their resistance values. These changes in resistance are converted into a normalised pattern of responses. The data are acquired and displayed by the eNose 5000 software where they can be further analysed.

The baseline is defined as the last raw resistance value before the transfer of volatiles to the sensor array. The sensor responses were defined as the maximum of each curve, and reported as relative value

$$\frac{dR}{R_b} = \frac{R - R_{\text{baseline}}}{R_{\text{baseline}}},$$

where R = raw resistance of the sensor and $R_b = R_{\text{baseline}}$ = baseline resistance.

Glass vials of 20 mL were filled with 1 mL of a solution of 1 mL/L ethanol (Merck) in distilled water. The vials were sealed with silicon/ Teflon septa embedded in magnetic caps. Both trays were fully occupied so that each series contained 64 samples. Measurements were carried out each 13 min for 87 days with breaks between measurement series varying from a few minutes to 10 days. A total of 3174 measurements were performed using two different carrier gases: first with synthetic air (N_2/O_2 , 80 % / 20 %, technical), and then with nitrogen (N_2 , technical).

The gas flow rate was kept constant at 100 mL/min. The samples were incubated for 10 min at 30 °C and agitated, during incubation, at 500 rpm every 5 s for 15 s. After the incubation period, 5 mL of the headspace was injected within 10 s using a 5 mL syringe heated at 35 °C with the following injection parameters: filling speed: 500, pull-up delay: 500 ms, injection speed: 500, pre-injection delay:

500 ms, post-injection delay: 500 ms. After a transfer period of 4 s, the volatile compounds were trapped for 2 min in the sensor chamber for measurement. Before and after each measurement, the sensors were cleaned for 5 min with the carrier gas, which gave a total cleanup time of 10 min. At the same time, the syringe was flushed with the carrier gas for 85 s in order to avoid cross-contamination between samples.

Results and discussion

Sensor responses to the ethanol solution

Figure 1 represents the maximum responses to the ethanol solution of all 12 CP sensors over the entire 3-month period. The left side shows the measurements performed with synthetic air as carrier gas (1076 measurements), and the right side the measurements carried out with nitrogen (2098 measurements). The 1 mL/L aqueous ethanol solution, which is recommended by the manufacturer for calibration, is well-defined, homogeneous and stable compared to other samples such as real food products. However, even after elimination of the outlying responses, the sensor responses obtained were rather unrepeatable. All sensors followed the same trend, *i.e.* when the response of a given sensor was low or high, all other sensors responded the same way.

The sensors had slightly different responses depending on the carrier gas used. All sensors gave generally higher responses with nitrogen, especially sensor types 458, 297 and 298. However, both carrier gases gave similar response variations, and hence, the same poor repeatability of the measurements. This finding may or may not be in agreement with the results of Bartlett and Ling-Chung [13] who claimed that their laboratory-made CP sensors gave “similar responses” to saturated methanol vapour whether air or oxygen-free nitrogen was used. As no data are given, it is difficult to interpret the exact meaning of “similar responses”. If “similar” means the same type of response qualitatively, *i.e.* curve shape, then our finding agrees with theirs. However, if “similar” means quantitatively, *i.e.* curve height, then we cannot completely agree with their assertion. As the type of variations observed in our measurements is qualitatively similar for all sensors, it could mean that the sample preparation is responsible for the lack of repeatability. However, it will be shown later that this explanation is not satisfactory.

As all sensors showed the same trend in their response variability, subsequent discussion is based on results obtained with sensor type 458. This arbitrary choice does not influence the discussion or the validity of the conclusions drawn.

Figure 2 presents the relative maximum response values over several days of sensor 458 versus time of measurement. The bottom of the graph corresponds to measurements performed with synthetic air and the top of the graph to measurements carried out with nitrogen. This figure clearly

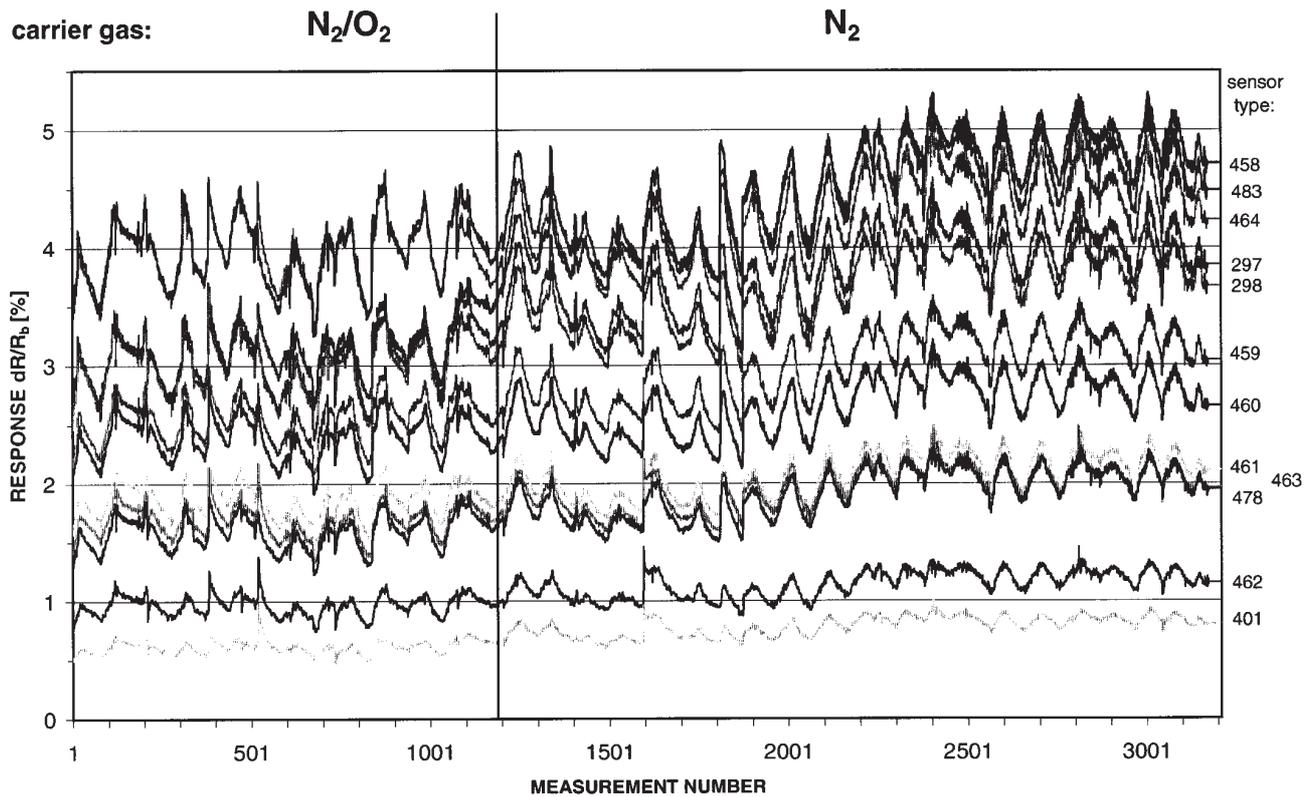


Fig. 1 Maximum response (dR/R_0) of 12 conducting polymer sensors based on polypyrrole to a 1 mL/L-ethanol solution over a 3-month period. **Caption:** Each curve represents one sensor.

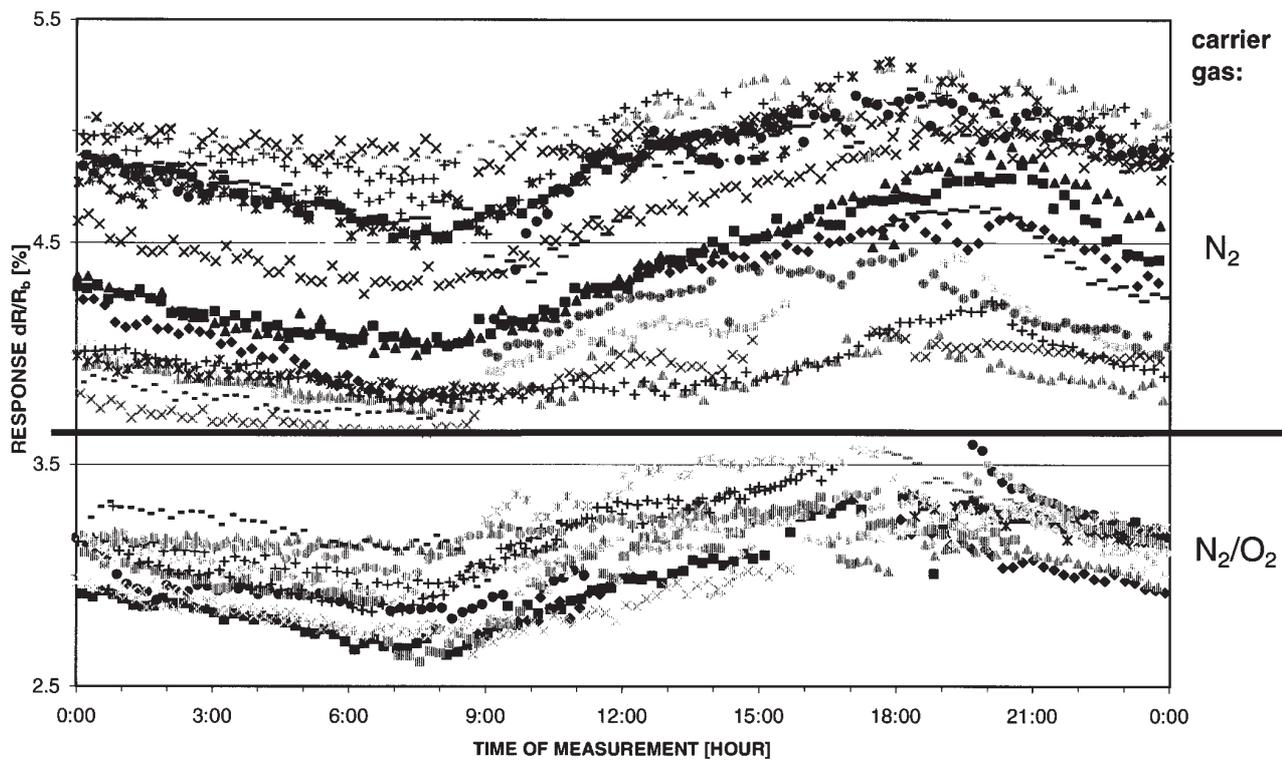


Fig. 2 Maximum response (dR/R_0) of sensor 458 versus time of measurement (hour). **Caption:** Each symbol represents a day.

shows that the sensor responses depended on the time of day when the measurements were performed. For both carrier gases, the responses were always the lowest in the morning (around 8 h), then rose to reach a maximum in the evening (around 18 h), and finally declined again until morning. The same cycle was recorded each day, although the magnitude of the difference between morning and evening measurements can differ significantly.

Table I summarises the sensor fluctuations observed within the same day as well as over the entire period of measurements, *i.e.* 3 months. The relative fluctuations, $\Delta(dR/R_b)$, represent the difference between the highest and the lowest value of the response (dR/R_b) as related to the average, and were calculated as

$$\Delta\left(\frac{dR}{R_b}\right) = \frac{\left(\frac{dR}{R_b}\right)_{\max} - \left(\frac{dR}{R_b}\right)_{\min}}{\left(\frac{dR}{R_b}\right)_{\text{average}}} * 100.$$

When synthetic air was used as carrier gas, the fluctuations in sensor response within the same day ranged between 44 % (sensor 401) and 7 % (sensor 458, 463 and 298). When nitrogen was used as carrier gas, the fluctuations were between 33 % (sensor 401) and 4 % (sensor 458), and hence were slightly smaller than with synthetic air. The instability observed by us seems however to be much smaller than the one reported by Bartlett and Ling-Chung [13] who noticed a change of 3.6 to 5.0 k Ω on responses of 3 to 30 k Ω , which gives a variation of 12 to 167 % of the sensor responses.

The hypothesis that the sample preparation alone was responsible for the poor repeatability is shown not to hold because of the fact that the shape of the sensor response in

the course of the day was repeatable. However, figure 2 suggests the variation in temperature as another explanation for the trend observed. The measurements were performed during the summer, and there were large temperature differences between morning and evening in the west oriented laboratory. This external temperature significantly affected the temperature of the sensor module. The room temperature was not recorded during the measurements. However, due to the location of the laboratory, the room temperature was always lower in the morning and higher in the evening. The current software does not make possible an automatic recording of the sensor chamber temperature. Therefore, a complete survey of this parameter was not possible. However, it was possible to record the temperature manually as the software always shows the temperature of the module on the screen. This temperature was recorded for several hours and several days.

The sensor chamber is supposed to be kept at a constant temperature of 30 °C, although a variation of 3 °C was observed during this study. Figure 3 clearly shows that the sensor responses were greatly influenced by an elevation of the sensor chamber temperature. Although data for the highest temperatures are missing, it seems that a small temperature difference between 30.3 and 31.0 °C has a greater effect on the sensor response than the same difference at higher temperatures. This observation confirms what Yueqiang *et al.* [9] reported for polypyrrole films. They found that the temperature dependency of the polymer coating was related to the conductivity of the film: the lower its conductivity, the higher the temperature dependency. This observation may explain the greater stability of the sensor response at higher temperatures.

Table I. Fluctuations in the maximum response with synthetic air or nitrogen for each of 12 sensors observed within the same day as well as within the entire period of measurement, *i.e.* 3 months.

Measurements with synthetic air		458	459	460	461	462	Sensor type		478	483	297	298	401
Response fluctuation	MIN	7	8	8	9	13	463	464	10	8	8	7	10
within one day [%]	MAX	26	28	28	29	42	36	26	34	25	31	31	44
Response fluctuation within 3 months [%]		31	36	39	42	66	51	34	42	34	44	43	84
Measurements with nitrogen		458	459	460	461	462	Sensor type		478	483	297	298	401
Response fluctuation	MIN	4	6	7	7	8	463	464	7	5	6	6	6
within one day [%]	MAX	21	26	27	30	31	28	25	30	24	27	26	33
Response fluctuation within 3 months [%]		42	39	41	43	52	46	36	42	35	39	40	51

Caption: The fluctuations, $\Delta(dR/R_b)$, represent the difference between the highest and the lowest value of the response (dR/R_b), and were calculated as follow:

$$\Delta\left(\frac{dR}{R_b}\right) = \frac{\left(\frac{dR}{R_b}\right)_{\max} - \left(\frac{dR}{R_b}\right)_{\min}}{\left(\frac{dR}{R_b}\right)_{\text{average}}} * 100, \text{ where } R = \text{raw resistance of the sensor, and } dR = R - R_b \text{ (} R_b = \text{baseline resistance).}$$

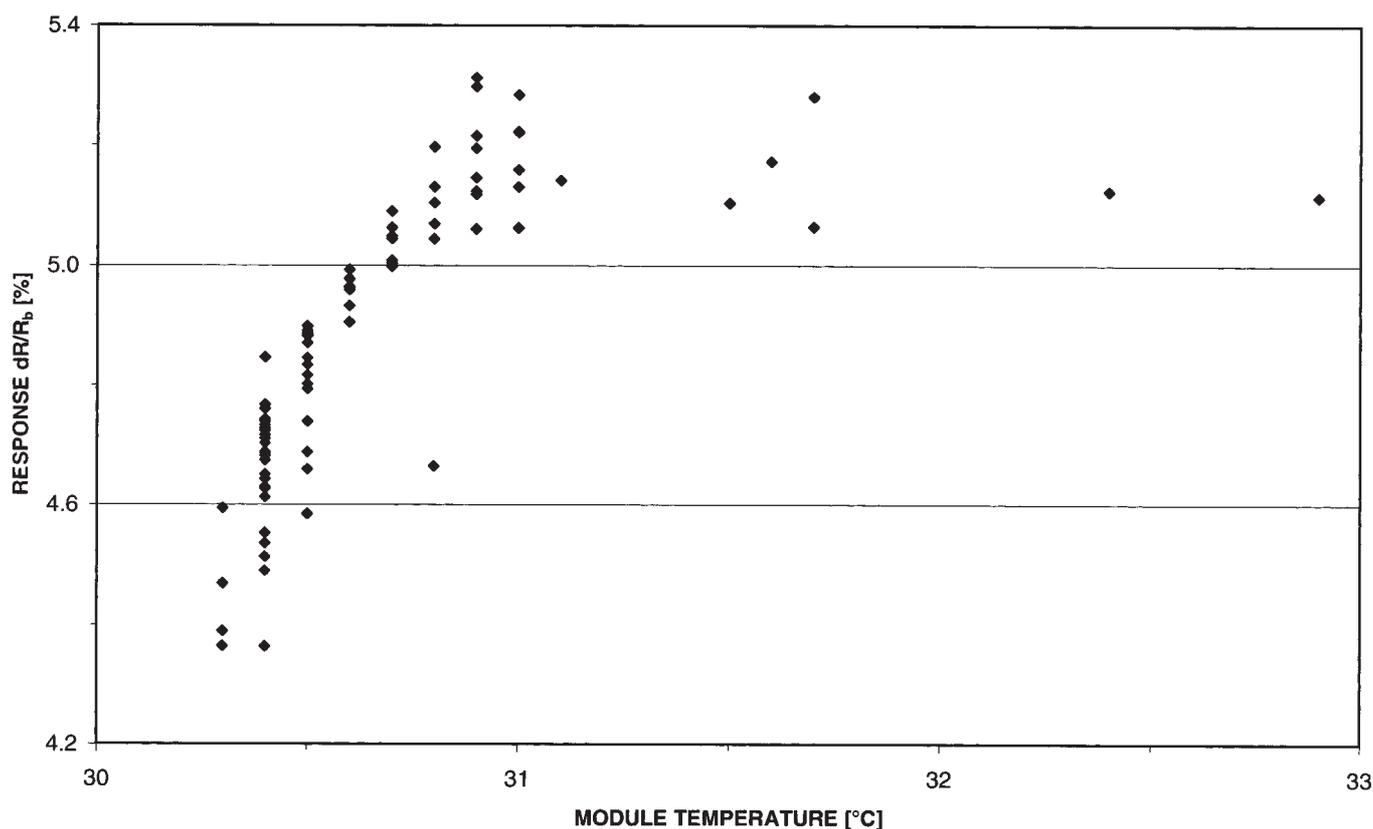


Fig. 3 Maximum response (dR/R_b) of sensor 458 versus temperature ($^{\circ}\text{C}$) of the sensor module as displayed on the computer screen.

The variation in the module temperature could partly explain the poor repeatability of the results as well as the cyclic shape of the response over 24 hours (Fig. 2). However, measurements performed during days where the room temperature was nearly constant did not show a much better repeatability than measurements carried out when there were large variations in the external temperature between morning and evening. Therefore, this parameter alone can not fully explain the observed instability.

Baseline study

Pearce *et al.* [17] suggested that “the change in sensor conductance is a function of the baseline conductance”. Therefore, in order to explain the poor repeatability of the sensor responses, the baseline resistance values of the sensors were examined.

Unlike the sensor responses represented in figure 2, figure 4 shows that the baseline values varied more from day to day than within a day; only a small cyclic variation, *i.e.* maximum of the baseline in the morning and minimum in the evening, could be observed. This statement is confirmed by table II which summarises the baseline fluctuations of each sensor in the course of the day as well as during the

entire period of measurement. The fluctuations were calculated in the same way as for the sensor response but by using $R_{baseline}$ instead of dR/R_b . Over the entire 3-month period, measurements performed using nitrogen as carrier gas generally showed smaller baseline fluctuations than measurements performed using synthetic air. Whereas the minimum fluctuation within one day for all sensors was much smaller with nitrogen than with synthetic air, the maximum fluctuation was, generally much bigger (except sensor 458). Therefore, the carrier gas used does not seem to influence the baseline stability over a short period of time (one day). However, the baseline values were more stable over a relatively long period of time (3 months) when nitrogen was used as carrier gas.

Contrary to other papers [13,17] which report sensor responses 1.5 to 6 times more stable than the baseline resistances, we observed that, independently of the carrier gas used, the baselines were much more stable than the sensor responses. The baseline fluctuation over 3 months was on average only one tenth of the response fluctuation. The maximum baseline fluctuation with nitrogen over one day for sensor 458 was less than one two hundredth of the maximum value of the corresponding sensor response. Thus, although the value of the sensor baseline may be an

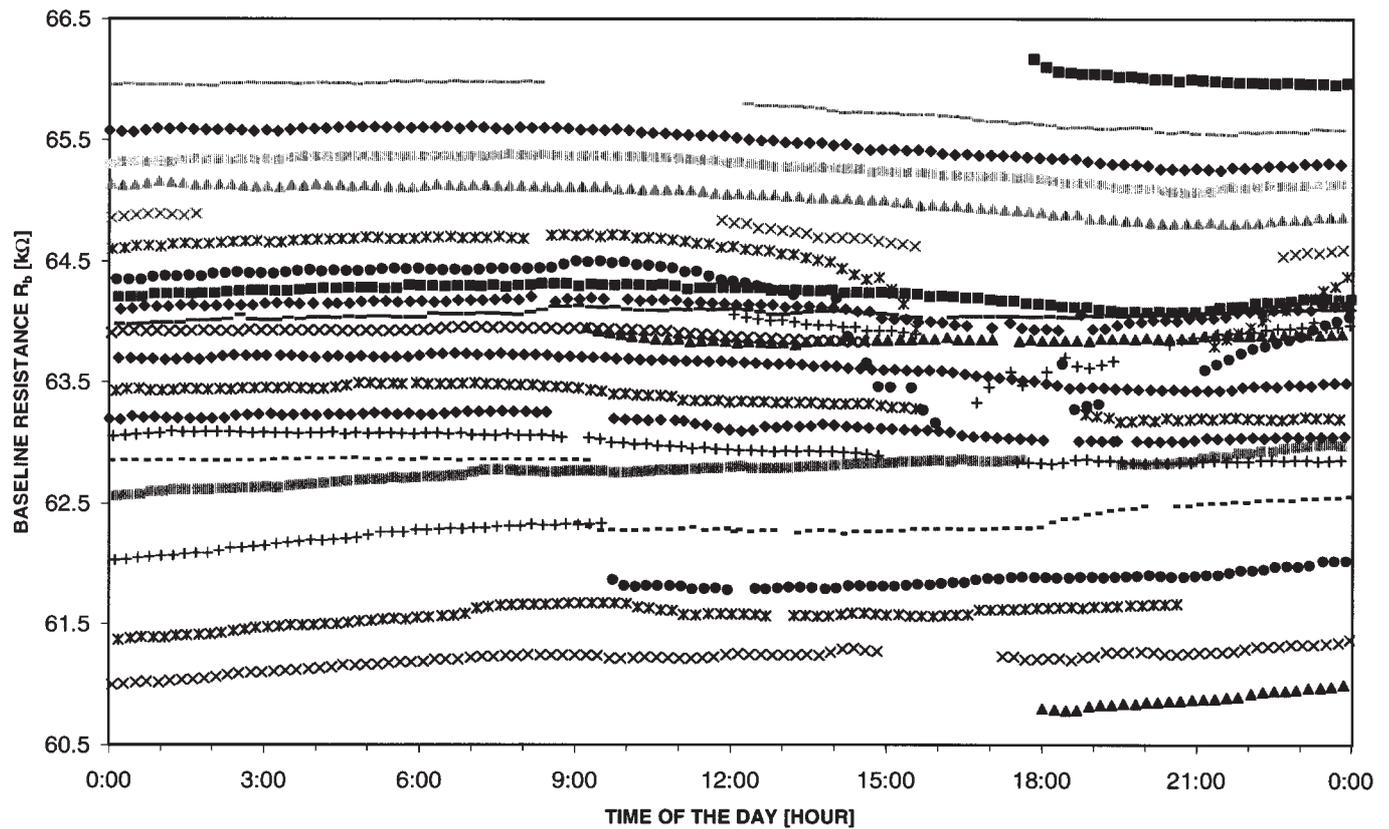


Fig. 4 Baseline resistance ($k\Omega$) of sensor 458 *versus* time of measurement (hour). **Caption:** Each symbol represents a day. There was no difference between measurements performed with synthetic air or those carried out with nitrogen as carrier gas.

Table II. Fluctuations in the baseline resistance with synthetic air or nitrogen for each 12 sensors observed within the same day as well as within the entire period of measurements, *i.e.* 3 months.

Measurements with synthetic air		458	459	460	461	462	Sensor type						
		463	464	478	483	297	298	401					
Baseline fluctuation	MIN	0.32	0.18	0.14	0.20	0.24	0.19	0.27	0.17	0.23	0.36	0.35	0.13
within one day [%]	MAX	0.76	0.47	0.45	0.41	0.44	0.50	0.77	0.39	0.63	1.40	1.32	0.30
Baseline fluctuation within 3 months [%]		10.95	7.58	6.75	4.52	5.67	6.40	10.39	4.03	8.33	12.05	11.29	3.19
Measurements with nitrogen		458	459	460	461	462	Sensor type						
		463	464	478	483	297	298	401					
Baseline fluctuation	MIN	0.04	0.09	0.09	0.09	0.11	0.11	0.08	0.05	0.05	0.04	0.07	0.06
within one day [%]	MAX	0.09	2.06	2.21	2.25	2.55	1.95	2.06	1.86	1.64	2.22	2.40	1.64
Baseline fluctuation within 3 months [%]		5.21	3.36	3.45	3.06	4.32	3.94	3.34	4.08	3.48	6.69	6.91	3.22

Caption: The fluctuations, ΔR_b , represent the difference between the highest and the lowest value of the baseline resistance, R_b , and were calculated as follow:

$$\Delta R_b = \frac{(R_b)_{\max} - (R_b)_{\min}}{(R_b)_{\text{average}}} * 100.$$

important factor in the repeatability of the sensor response, it is not the only one. In our measurements, the baseline factor played rather a small role in the poor repeatability of the sensor responses, and a direct correlation between the sensor baseline and the sensor response was not apparent. A closer investigation of the baseline variation in the course of the day revealed that the baseline variation showed an opposite trend to the sensor responses, *i.e.* when the sensor baseline was low, the response to the ethanol solution for the corresponding sensor was high, and vice versa. Figure 5 shows that, although the cycle is clearly repeated, it is difficult to define a mathematical function for the relation between the sensor response and the baseline value.

Figure 6 shows the average baseline resistances for sensor 458 versus the measurement period. When synthetic air was used, the average baseline shows an upward trend. This observation is in accordance with several studies which have reported that CP sensors based on polypyrrole increase their resistance with time when exposed to air [21,22]. This change in resistance could be explained by an oxidation of the polymer coating. This explanation could also account for the decline of the average baseline values when nitrogen was used. Nitrogen seemed to “regenerate” or reduce the polymer coating to some extent after its exposure to oxidising

gases. The baseline increase or decrease was not linear versus time either with synthetic air or with nitrogen and for any of the 12 CP sensors used. When synthetic air was used, the drift was independent of whether the instrument was operating or not, *i.e.* system completely shut down or withdrawal of the CP module from the system. When nitrogen was used, the sensor baseline resistance increased when the instrument was not on, *i.e.* the sensors were no longer exposed to nitrogen. This fact supports the hypothesis that the CP sensors are easily oxidised by air even by a very small concentration [21].

The investigation of the sensor responses revealed a correlation between the sensor response and the temperature of the sensor module (Fig. 3). As expected, a variation of the sensor baseline value was also observed when the chamber temperature increased (Fig. 7). Temperature had opposite effects on the sensor baseline resistance and on the sensor response. Both observations could be related to an increase in the conductivity of the sensor when the temperature increases. Therefore at high temperatures, the sensor conductivity is high, which means a lower baseline resistance value and a higher sensor response. This observation is in agreement with other works [14,22], which report an inverse relation between temperature and baseline resistance of

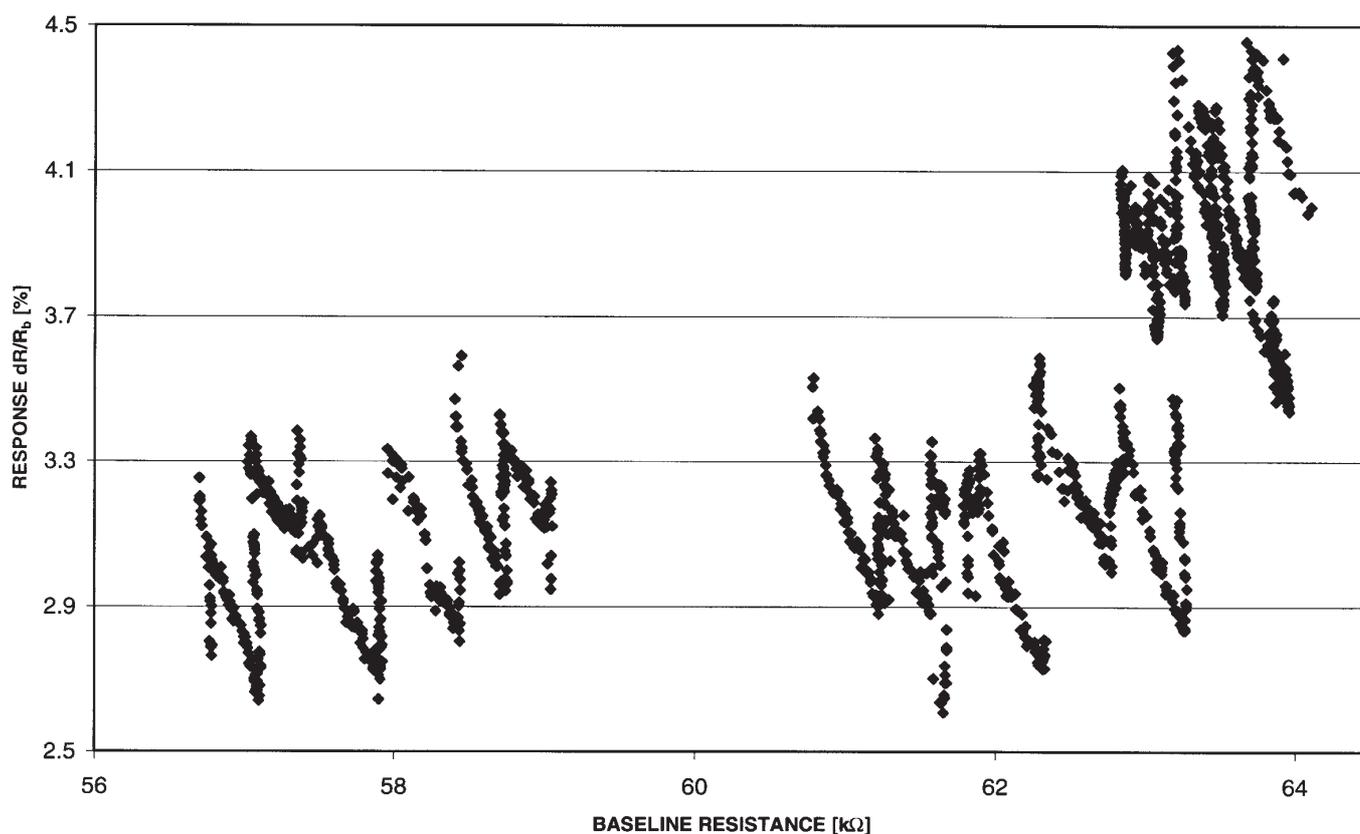


Fig. 5 Maximum response (dR/R_b) of sensor 458 versus baseline resistance ($k\Omega$).

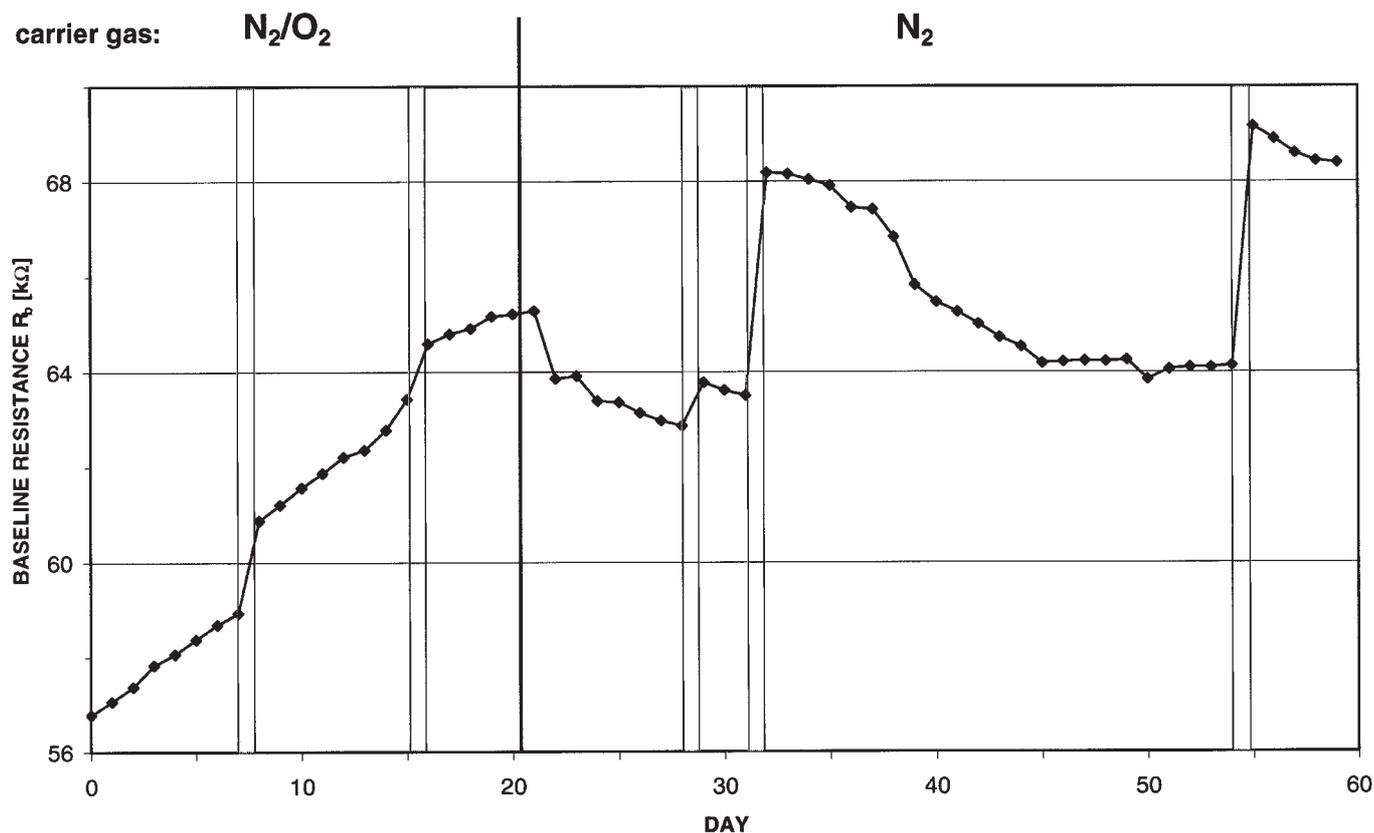


Fig. 6 Daily average baseline resistance (kΩ) of sensor 458 *versus* day of measurement. **Caption:** The shaded zones correspond to time periods when the sensors were not used.

polymer sensors based on polypyrrole. In the study of Neaves and Hatfield [22], the sensor resistance suffered from a change of $1.25 \Omega/^\circ\text{C}$ for an absolute baseline of approximately 100Ω . The sensors from Meijerink [14] were affected of a difference of $5 \Omega/^\circ\text{C}$ for an absolute baseline of approximately 500Ω . In our case, the sensors showed a variation of $540 \Omega/^\circ\text{C}$ for an absolute baseline of approximately $640 \cdot 1000 \Omega$. The ratio of baseline variation to absolute baseline value was 10 times smaller in our case due to a very high absolute resistance.

Attempts at correcting the sensor instability

A linear parametric drift compensation was applied to the data set corresponding to the sensor responses when the module temperature was registered. This correction was based on the model proposed by Pearce and Gardner [23] where the systematic drift is approximated linearly. In our study, two linear functions were calculated: i) sensor response (dR/R_b) *versus* time of day, and ii) sensor response (dR/R_b) *versus* temperature of the sensor module. The slopes of the linear functions obtained were then used to correct the sensor responses by using the equation

$$(dR/R_b)_{\text{corr}} = (dR/R_b)_{\text{uncorr}} - a * t, \quad (1)$$

where $(dR/R_b)_{\text{corr}}$ = corrected sensor response, $(dR/R_b)_{\text{uncorr}}$ = uncorrected sensor response, a = slope of the linear function, and t = time or temperature of measurement.

As the sensor responses taken into account for the time of day correction were collected between 7 am and 8 pm, the instability in the course of the day could be considered as approximately linear. The slope for correcting the systematic instability caused by temperature changes was calculated between 30.3 and 31.0°C , as linear approximation cannot be done for the entire range, and only too limited a number of points were available for higher temperatures.

Figure 8 a) shows measurements without any correction, *i.e.* as they are registered by the electronic nose software. On this graph, one could see a dispersion of the data set of 1 %, *i.e.* the sensor responses range from 4.3 to 5.3 %. Figure 8 b) displays the same data set with the time of day as correcting factor, *i.e.* “ a ” in equation (1) was replaced by the slope obtained on figure 2 when only measurements between 7 am and 8 pm are taken into account, and “ t ” was replaced by the corresponding time of the day. Figure 8 c) shows always the same data set but this time the temperature of the sensor module was used as correcting factor, *i.e.* “ a ” in equation (1) was replaced by the slope obtained on

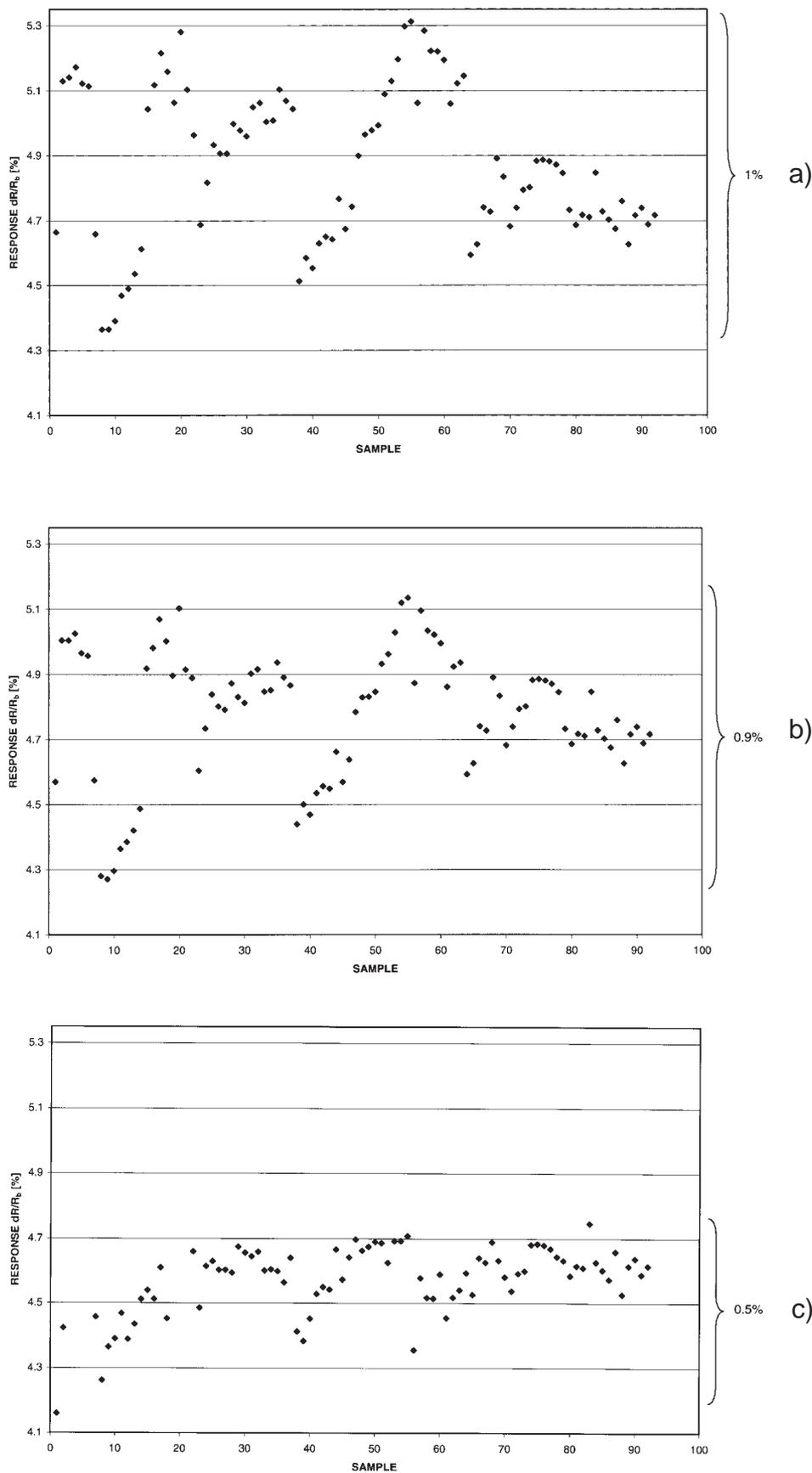


Fig. 8 Comparison of uncorrected and corrected measurements

Caption: a) Maximum response (dR/R_b) of sensor 458 without correction.

b) Maximum response (dR/R_b) of sensor 458 with a linear parametric correction to compensate for the drift observed from one hour to the next. The data for the correction were taken between 7 am and 8 pm.

c) Maximum response (dR/R_b) of sensor 458 with a linear parametric correction to compensate for the instability due to the temperature variation of the sensor module. The data for the correction were taken between 30.3 and 31.0 °C.

suitable for products having a constant relative humidity as well as presenting well-differentiated responses so that the residual response variation could be neglected. Only a few rare products correspond to the above-mentioned criteria. If the technical control as well as the understanding of the working mechanism of CP sensors are not improved, the future of this technology as sensor for volatile compounds is seriously jeopardised.

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