# Chemical Imaging:
# Trends in Multiparameter Sensor Systems

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## 1 Abstract:

The traditional use of chemical sensors focuses at the output of an individual chemical sensor with its single sensor signal (feature), e.g. a specific current at a fixed potential of an electrochemical cell or a resistance change of a metal oxide semiconductor, etc. Chemical imaging now aims at the electronic recording of time and spatially resolved chemical species and odours. This work describes concepts for the necessary increase in the number of features in order to perform such advanced chemical sensing. This will also allow a deeper understanding of elementary processes in chemical sensing or in odour perception.
The case studies presented in this context are organised according to the following strategies to produce features:

- Similar sensitive layers investigated as coatings on different transducers
- The same sensitive layer investigated with a single transducer, recording of different physical properties
- The same sensitive layer and transducer, same physical property investigated by different modes of operation

## 2 Introduction:

In principle available and easily measurable transducer properties of chemical sensors are changes of:

- resistance, $\Delta R$ e.g. in semiconductor gas sensors,
- current, $\Delta I$ e.g. in electrochemical cells,
- capacitance, $\Delta C$ e.g. in humidity sensors,
- work function, $\Delta\phi$ e.g. in Gas FETs,
- mass, $\Delta m$ e.g. in BAW and SAW sensors,
- temperature, $\Delta T$ e.g. in pellistors and thermopiles, and
- optical properties, $\Delta n \cdot d$ e.g. in optical sensors.

One or several of these transducer properties may be measured for a large variety of chemically sensitive materials starting from inorganic oxides up to biological function units. For example, during the last two decades extensive work was done using metal oxides (e.g. $SnO_2$) and varying the gas sensing properties by doping, adding catalysts, modifying film structures and electrodes etc. [1]. Another example concerns polymers with a recent overview given in [2]. Results on supramolecular structures including combinatorial chemistry, biomimetic structures and biological function units are reported in [3]. Further examples may e.g. be found in the proceedings of the last EUROSENSORS conferences and references given there.

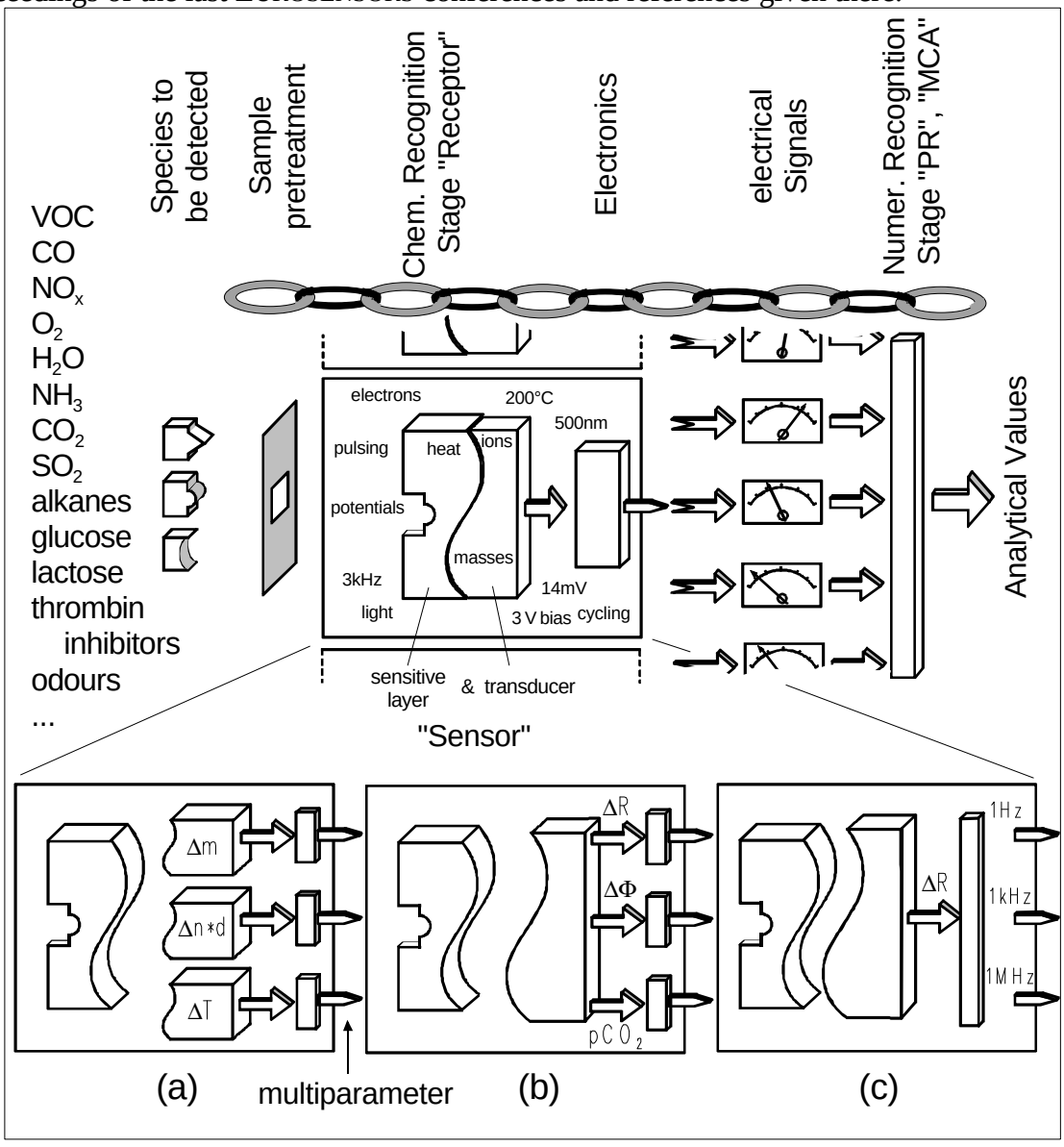



*Fig. 1: Schematic set-up of a sensor system (details are given in the text).*

In these studies several sensitive layers (which may form arrays) are often characterised simultaneously. If in such arrays a multidimensional signal (feature) space is generated by recording only one type of physical property this is -in the present paper- not considered as a multiparameter sensor system approach (this corresponds to the use of several "sensors" shown in Fig. 1, "vertical approach"). The higher dimensional feature space resulting from such arrays is usually evaluated by pattern recognition and multicomponent analysis methods. Combinations of sensor arrays, driving electronics and evaluation software are currently commercialised as electronic noses [4].

With the increased potential and reduced costs of single chip micro-controllers it is achievable and desirable for different applications to extract more than one signal (feature) from a single sensor element. This approach will open new market fields which today are partly covered by analytical instruments such as infrared or UV spectrometers or GCs. For example, sweeping of the measurement potential of an electrochemical cell allows to measure more than one substance with a single sensor. Another already commercialised approach is to pulse the heating element of a metal oxide sensor to make use from a change in the kinetics of surface chemistry between lower and higher temperatures.
The combination of measurements of different physical properties simultaneously in connection with non-stationary operation modes increases the number of features further more. If the time resolution is not a key issue also spectroscopic concepts can be applied. With a better understanding of sensing mechanisms one may optimise the coating material and derive reproducibly different physical properties or parameters.
The main topic of this paper concerns multiparameter sensor systems. The aims is to illustrate all these concepts by using only one sensitive layer as shown in Fig. 1. ("horizontal approaches" (a), (b) and (c)). The possible significant extension of the feature space by using more sensitive layers is then obvious.
The grouping into different categories in Fig. 1 is done according to the following scheme:

(a) Similar sensitive layers investigated as coatings on different transducers
(b) The same sensitive layer investigated with a single transducer, recording of different physical properties
(c) The same sensitive layer and transducer, same physical property investigated by different modes of operation

To illustrate these different concepts, selected case studies will be presented for each category.
It is obvious that an additional optimised sample pre-treatment will allow to increase the sensitivity and selectivity of the whole sensor system. The latter is maybe performed by the use of a purge and trap unit, by a pre-separation with a chromatographic column / sensor combination etc. [5]. This will enlarge the dimensionality of the feature space furthermore. The discussion of this specific topic (like the topic of choosing different materials as mentined above) is beyond the scope of this paper because of space restriction. For the same reason the following examples focus at gas sensing only.

# 3 Experiments and Discussion

## *(a) Similar sensitive layers investigated as coatings on different transducers*

Measuring different physical properties with optimised transducers (interdigital structures to measure $\Delta C$, quartz microbalances to measure $\Delta m$, refractrometric interference spectroscopy to measure $\Delta n \cdot d$, calorimetric devices to measure $\Delta T$) allows to characterise a selected non-conducting polymer which is used as identical coating on all transducers. Fig. 2 shows as an example the different transducers used by Haug et al [6] for such measurements. Results with different concentrations of $C_2Cl_4$ and poly(dimethylsiloxane) (PDMS) as sensitive coating are given in Fig. 3, 4 and 5. Similar results are also reported in [7, 8]
For the detection of $SO_2$ in nitrogen a gas sensor system comprising dielectric and mass sensors was reported by Endres et. al [9]. Fig. 6 presents comparative measurements.

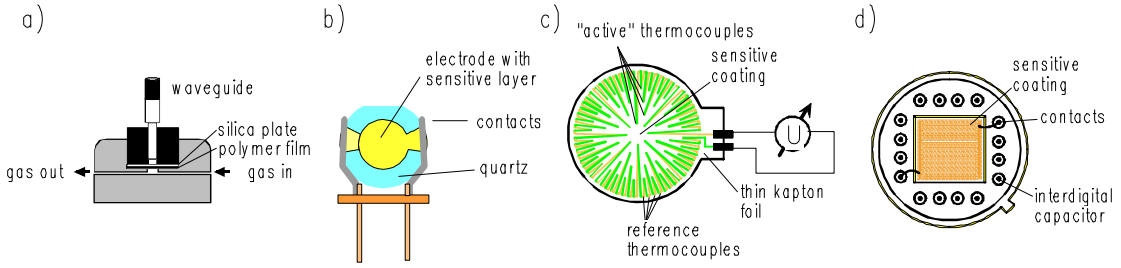



*Fig. 2: Schematic representation of the transducers used in a comparative study of polymeric coating for VOC detection. (a) Set-up for optical measurements of thickness changes Dd of a polymer film by means of reflectometric interference (RIfS) (b) 10 MHz quartz oscillator with gold electrodes to measure Dm effects by means of frequency changes (BAW, Bulk Acoustic Wave). (c) Planar thermopile wit 64 Cu/Cu/Ni thermocouples integrated in a thin Kapton foil to measure the DT effects by means of voltage measurements. (d) Interdigital capacitor with gold electrodes and 14 mm electrode separation on a glass substrate to measure DC effects [6].*

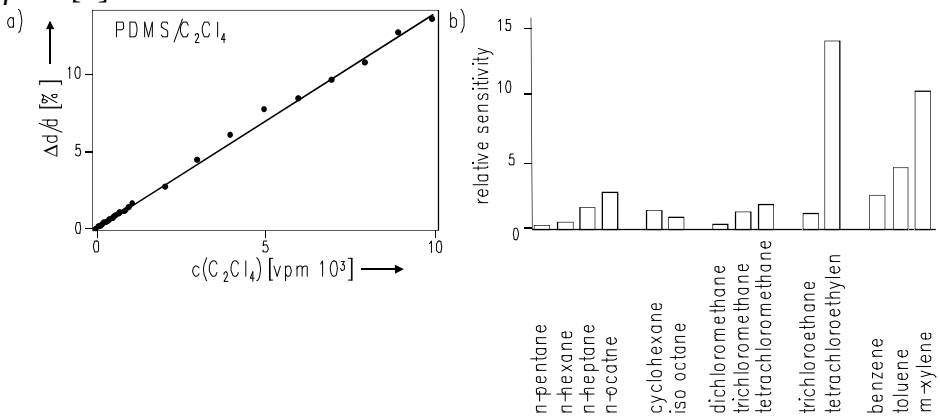



*Fig. 3: (a) Relative thickness change Dd/d versus the concentration of $C_2Cl_4$ in volumetric parts per million 10£c($C_2Cl_4$)£$10^4$vpm as deduced from optical measurements (Fig 2a). (b) relative sensitivities of the optical transducers to monitor different gases [6].*

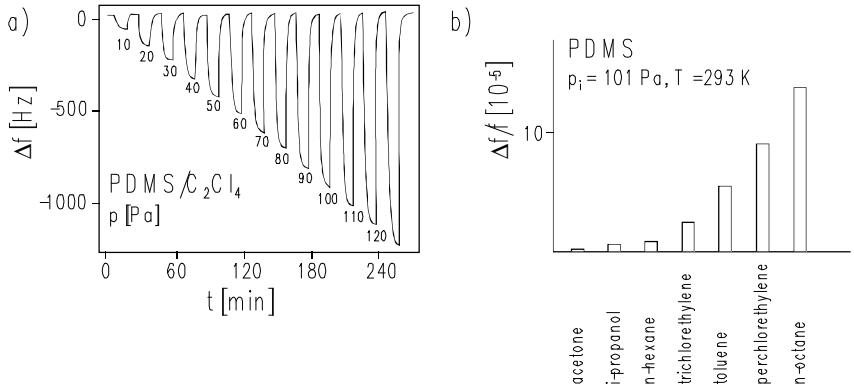



*Fig. 4: (a) Typical BAW responses Df of quartz microbalance sensors (QMB) coated with the polymer PDMS as a function of time for stepwise exposure to different partial pressures p($C_2Cl_4$) in air (numbers in the figure). (b) Relative sensitivities Df/f of the PDMS-coated quartz microbalance transducer to monitor different gases [6].*

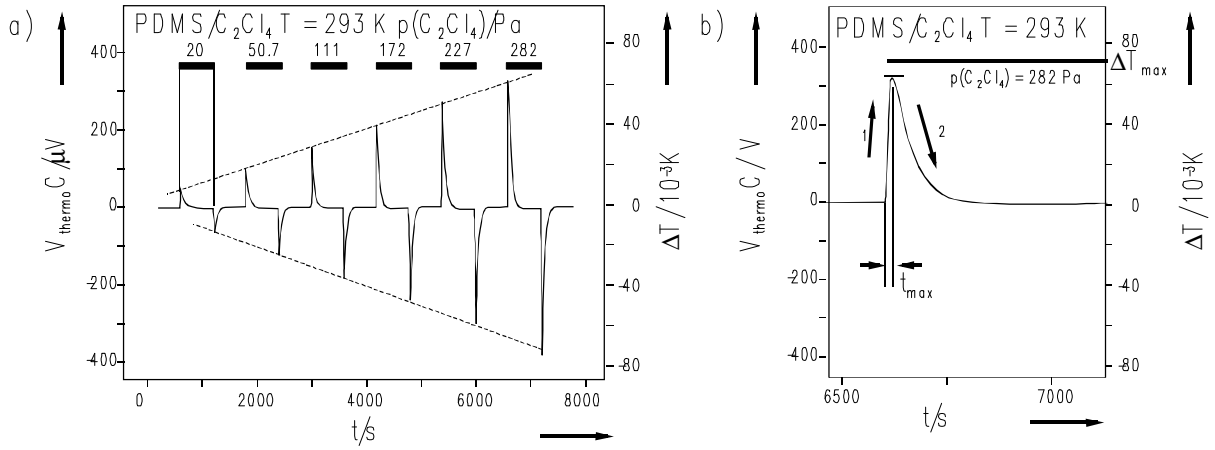



*Fig. 5: (a) Typical responses $V_{thermo}$ ~ D$T$ of the thermocouples of calorimetric thermopile sensors coated with PDMS as a function of time for stepwise exposure to different partial pressures p($C_2Cl_4$) in air. $V_{thermo}$ is the signal of three thermopiles connected in series, (b) details for one specific absorption cycle with response time $t_1$ and decay time $t_2$ characteristic for specific molecules. Values of $t_i$ could be used as features in a subsequent feature extraction [6].*

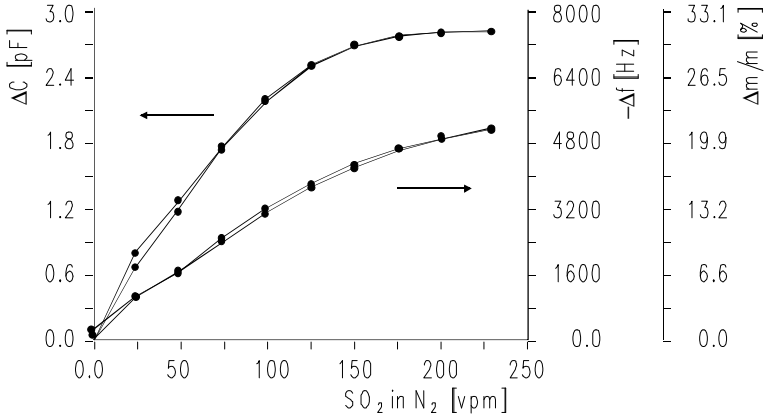



*Fig. 6: Dielectric- and mass-adsorption isotherms for $SO_2$ in $N_2$ at 30°C. D$m/m$ (~ D$f$) is the relative mass change of the sensitive coating [9].*

The use of GC column material for enantiometric separation of chiral molecules in the gas phase by means of gas sensors was shown recently by Bodenhöfer et. al. [10, 11]. Comparative mass sensitive and optical measurements are depicted in Fig. 7.

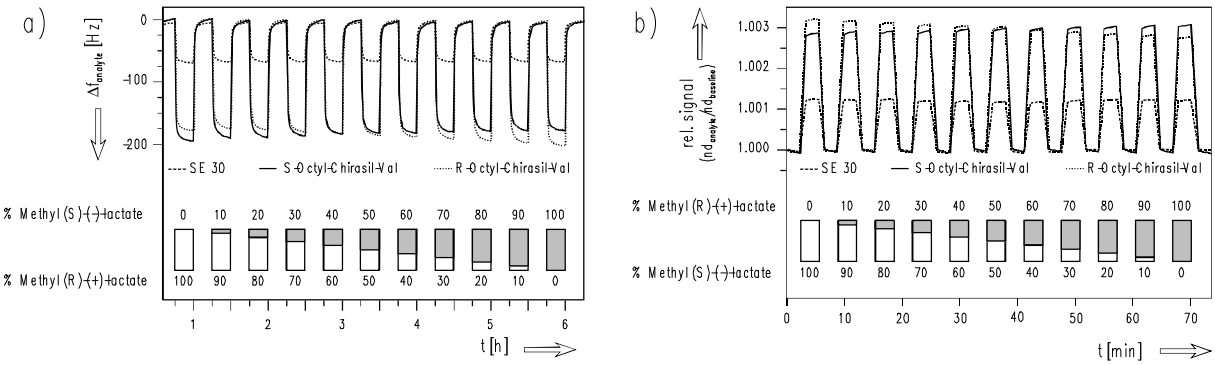



*Fig. 7: (a) BAW-signals (frequency shifts in [Hz]) of two chiral sensors ((S)-sensor, solid line, (R)-sensor, points) of similar layer thickness and an additional non-chiral SE-30-sensor (solid line, SE-30 = poly(dimethylsiloxane)) upon exposure to mixtures of different enantiomer contents (in %) of methyl lactate.*
*(b)Relative RIfS-sensor signals (normalised with regard to the layer thickness without analyte gas, as described in the text) of two chiral sensors ((S)-sensor, solid*

*line; (R)-sensor, dotted line) and an additional SE-30-sensor (dashed line) upon exposure to different enantiomeric compositions (in %) of methyl lactate [10, 11].*

Comparative mass, work function and optical absorbance changes have been measured on conducting polymers by Josowicz and Topart [12, 13]. The experimental setup is shown in Fig. 8a, comparative mass and work function changes are presented in Fig. 8b. Exposure of different polymers to methanol lead to an increase of the mass. Depending on the polymer this increase of the mass is associated with either an increase or a decrease of the work function .

**a)** **b)**

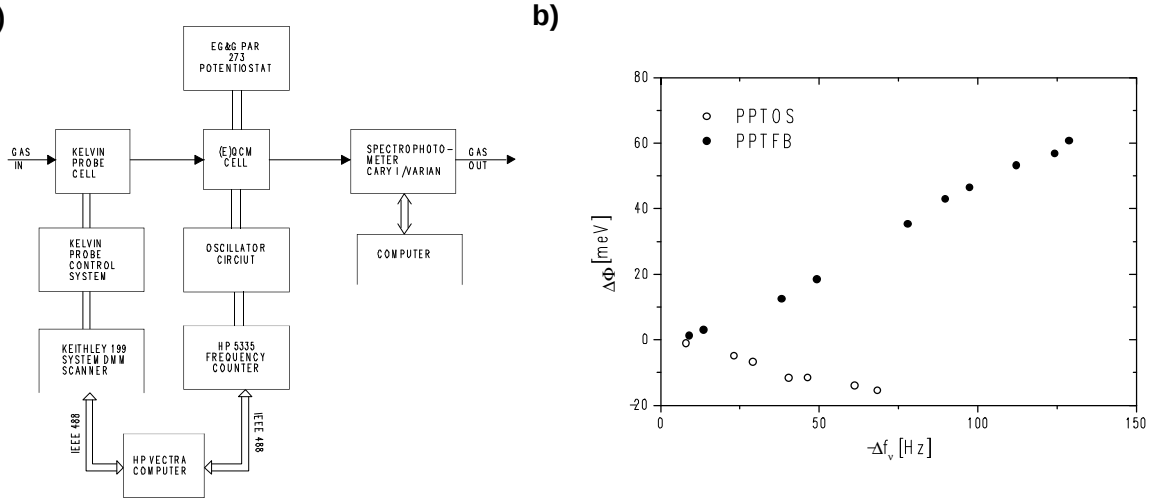



*Fig. 8: (a) Schematic diagram of the instrumentation used for mass, work function and optical measurements. (b) Correlation between equilibrium changes in work function* DF *and amount of methanol molecules sorbed in the PPTOS and PPTFB monitored by frequency changes* -D$f_n$. *Both films were 400nm thick [12, 13].*

Comparable measurements are also presented in [14].

### *(b) The same sensitive layer investigated with a single transducer, recording of different physical properties*

In this section we describe typical results obtained by measuring different physical properties of the same chemically sensitive structure.

Fig. 9a shows the experimental set-up used by Weimar et. al. [15] to measure simultaneously work function changes $\Delta\Phi$, conductance G and catalytic activity (conversion rate of CO to form $CO_2$) of a single Figaro gas sensor. Fig. 9b gives the experimental result in a three dimensional plot for a two dimensional parameter field (changes of CO and humidity concentration). As indicated by the distinct normalised signals, a determination of CO and $H_2O$ partial pressures in air may thus be performed with only one Figaro gas sensor if one enlarges the feature space.

In contrast to Fig. 9, Fig. 10 illustrates the correlation of two features by the logarithm of the relative conductance and relative work function change. The exponential dependence indicates a Schottky conduction mechanism [16].

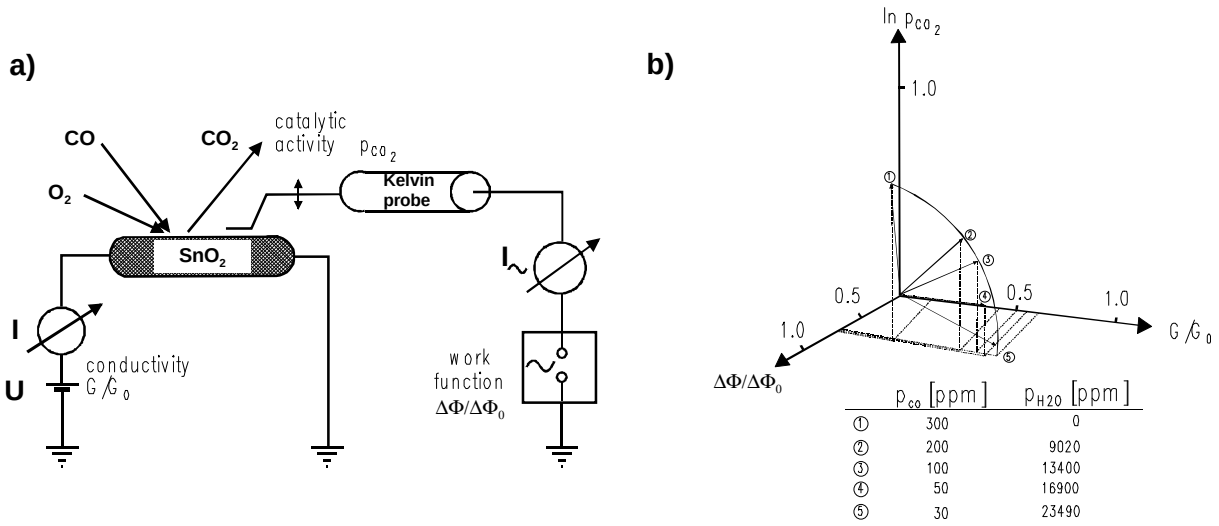



*Fig. 9: (a) Schematic set-up for measurements of conductivities, changes of work functions and catalytic activities (reaction rates) on $SnO_2$-based sensors.*
*(b) Vectorial representation of normalised signals $G/G_0$, $\Delta F/\Delta F_0$, and catalytic activities $p_{co2}$ (array III) obtained by the same sensors for different partial pressures of CO and $H_2O$. The vector $\boldsymbol{X}=((G/G_0),(\Delta F/\Delta F_0),(\ln p_{co2}))$ shown in this Figure were normalised according to $\boldsymbol{X}/\|\boldsymbol{X}\|$ [15].*

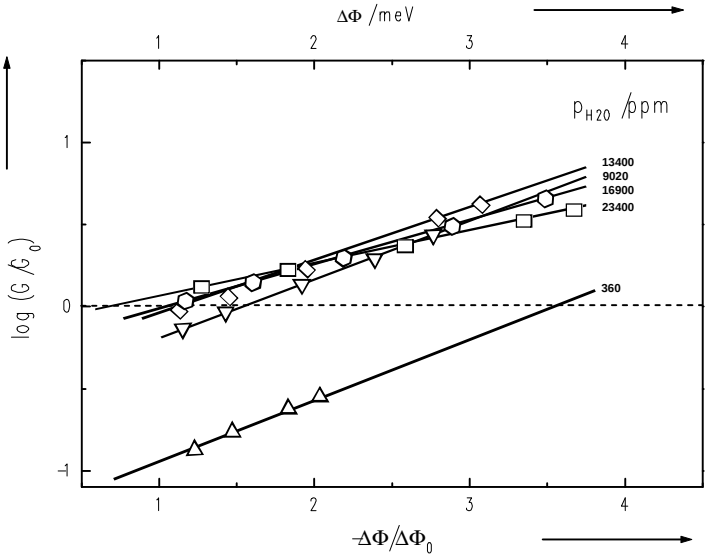



*Fig. 10: Logarithm of relative changes of conductance ($G/G_0$) as a function of relative work function changes ($\Delta F/\Delta F_0$) [16].*

The method of simultaneous measurements of work function changes and resistivities for studying the gas sensing mechanism was introduced by Mizsei and Harsányi [17] by using tin oxide thin films. Mizsei and Lantto [18] used this technique also for tin oxide thick films. Fig. 11 depicts the results obtained on palladium doped tin oxide.
For conducting polymers Kunugi et. al. [19] have performed simultaneous measurements of resistance and mass changes. The poly(pyrrole) film was deposited on a quartz crystal oscillator with an additional electrode for resistance measurements. Fig. 12a and b give the schematic side and top view of the device. In Fig. 12c the results of simultaneous measurements are shown. The increase of the mass can go with an increase or decrease of the resistance for different vapours.

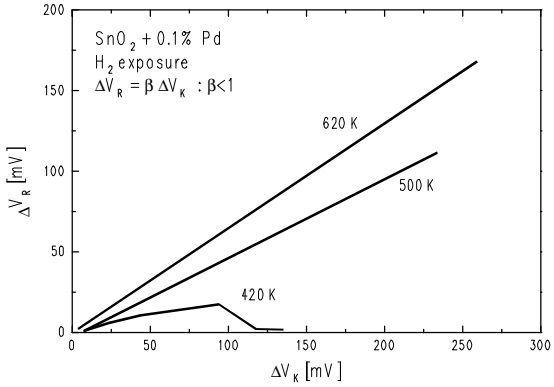



*Fig. 11: The values of* $\Delta V_R$ *as a function of* $\Delta V_K$ *at 420, 500 and 620 K of an* $SnO_2$*+ 0.1 wt.% Pd thick-film samples to* $H_2$ *exposure in laboratory air.* $\Delta V_R$ *is the estimated barrier height change from resistance measurements,* $\Delta V_K$ *is the barrier height change measured directly by Kelvin probe measurements. For details see [18].*

As shown in the examples the latter allows to discriminate e.g. between organic solvents and water vapour.

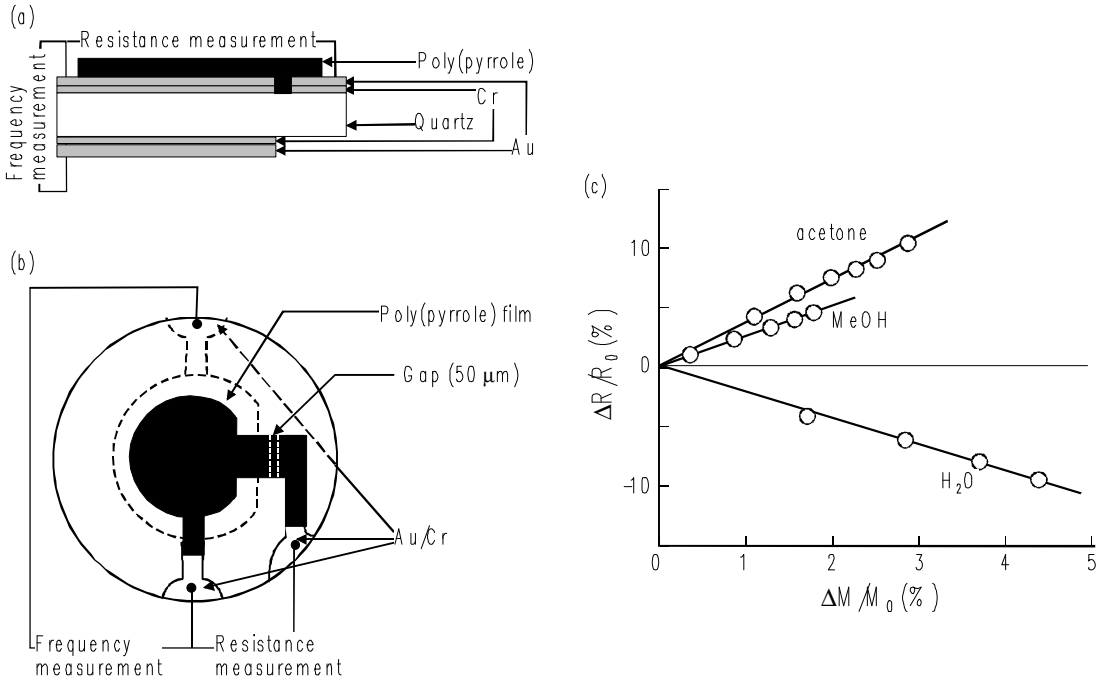
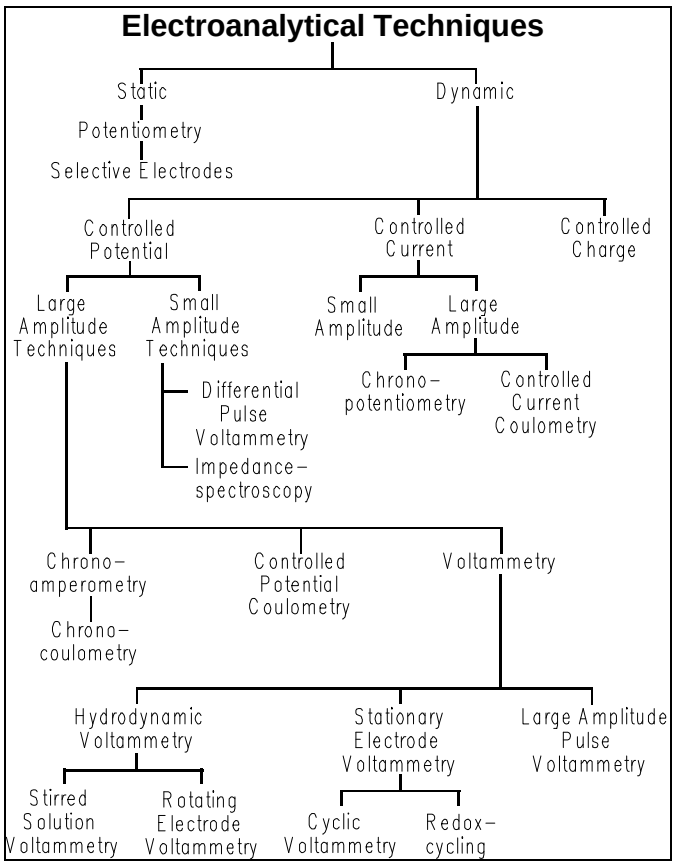



*Fig. 12: (a) Schematic set-up of an BAW transducer for combined measurements of mass increase and conductivity change ( side view); (b) top view.*
*(c)Plots of the relative resistance change* $\Delta R/R_0$ *as a function of the mass change* $\Delta M/M_0$ *for the poly(pyrrole) film [19].*

An idea for the improvement of the device by using a fourth electrode and therefore using no common electrode (for circuitry reasons, offset voltages etc.) is propose by Ingleby et. al. [20].

### *(c) The same sensitive layer and transducer, same physical property investigated by different modes of operation*




As one very successful approach, suitable operation modes known for a long time in electrochemistry (Fig. 13, [21]) are currently transferred to various kinds of sensors.

*Fig. 13: Measurement techniques used in electrochemistry. An overview is given e.g. in [21].*

A typical example is extracting resistance changes for different frequencies in an a.c. measurement (impedance spectroscopy). This leads to detailed features for a subsequent pattern recognition algorithm. The technique was illustrated by Weimar and Göpel [22] for tin oxide and by Amrani et. al. for conducting polymers [23].
Fig. 14 gives an idea how to increase the sensitivity and selectivity by measurements at different frequencies. The equivalent circuit which was used for the fit is shown in the upper left corner. (FRA in this context denotes the Frequency Response Analyser, |z| is the modulus of the impedance). For CO [22] the largest signal is reported at d.c. but for $NO_2$ at around 20kHz.

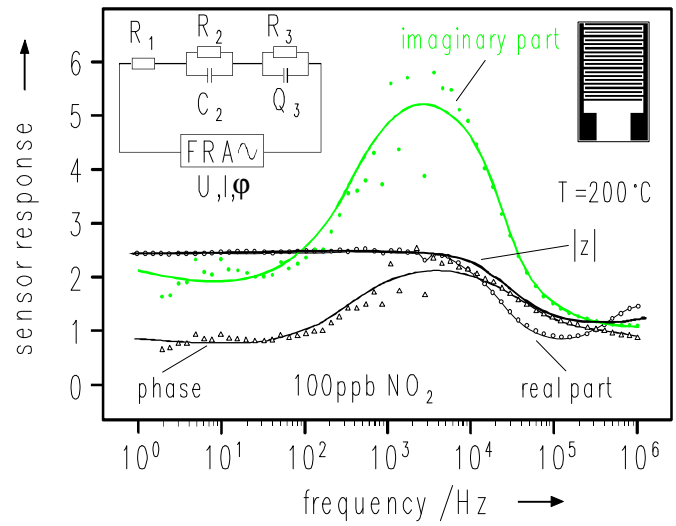



*Fig. 14: Two probe a.c. impedance which shows the sensor response plotted against the measurement frequency during the exposure of 100ppb $NO_2$. The sensor response is defined as measurement value for a selected frequency in carrier gas with $NO_2$ divided by the measurement value in carrier gas at the same frequency. The symbols represent the measured values, while the lines show a non-linear fit [22].*

Using conducting polymers and focusing at the dissipation factor (energy loss) as a function of frequency (Fig. 15) as well as other features (capacitance, conductance, resistance and reactance values) Fig. 16 gives different patterns for different parameters and different vapours.

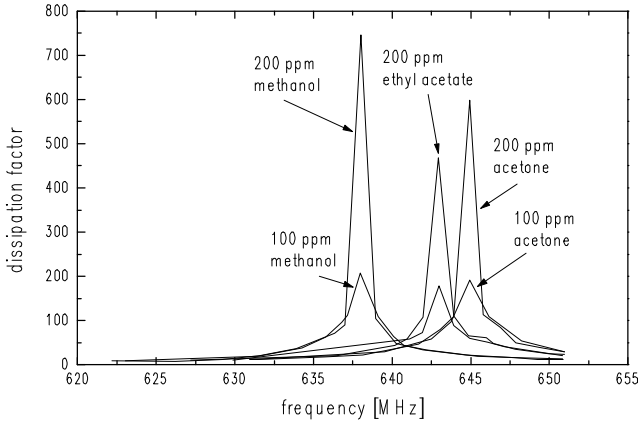



*Fig. 15: Dissipation factor vs. frequency response of poly-N-(2-pyridyl)-pyrrole to methanol, acetone, and ethyl-acetate vapour at 21°C [23].*

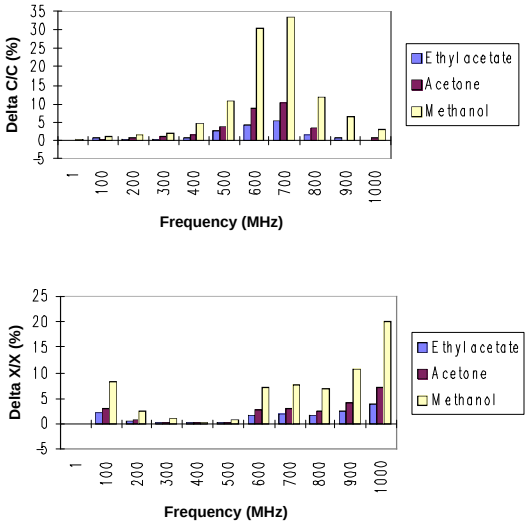



*Fig. 16: Relative change of the capacitive, conductive, resistive and reactive part of the a.m. sensor to 100ppm concentration of methanol, acetone and ethyl acetate [23].*

Another often varied parameter is the operation temperature of e.g. metal oxide gas sensors or pellistors. Sears et. al. [24] proposed to monitor conductance / time curves for different gases under conditions of thermal cycling.

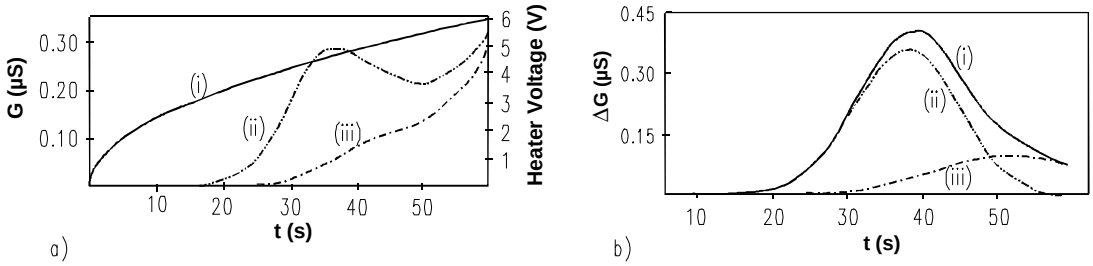



*Fig. 17: (a) Typical conductance G vs. time data for the TGS 813 sensor collected during the rising part of the thermal cycle. (i) heater voltage, (ii) 50ppm of propane, (iii) pure air. (b) Sensor response $\Delta G=G-G_0$ (µS) for 50ppm of propane and its decomposition into two Gaussian functions. (i) experimental $\Delta G$, (ii) and (iii) component Gaussian functions [25].*

Fig. 17a [25] shows the typical conductance vs. time data for a thermal cycle of the heater. Part b shows the signal shape analyis by a Gaussian deconvolution. This type of approach permits a quantitative analysis of two component mixtures if the concentration of one component is known.

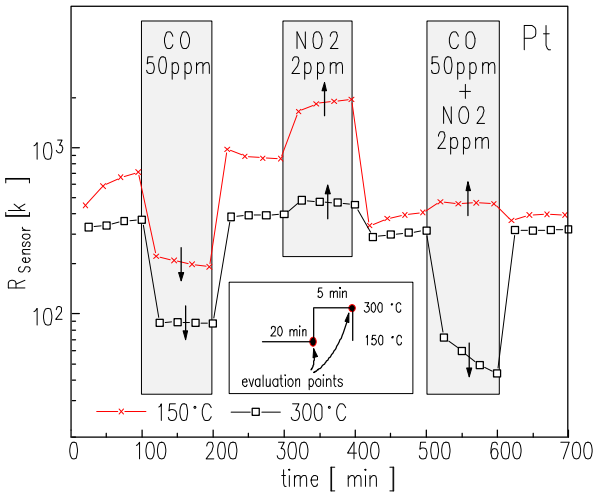



*Fig. 18: Plot of the resistance at the evaluation points, i.e. the end of the low and the end of the high temperature period. The temperature of the sensor was pulsed between 150°C and 300°C [26].*

A successful identification of CO and $NO_2$ in the ambient atmosphere by means of pulsed temperature operated single tin oxide sensors was presented by Heilig et.al. [26]. The operation mode as well as the feature extraction procedure is shown in Fig. 18.

Kunt et.al. [27] described an optimisation of micro hot plate chemical gas sensor temperature program for the discrimination of ethanol and methanol. Fig. 19a gives different signatures of different vapours when a standard temperature profile was applied, whereas Fig. 19b shows the ethanol and methanol identification by using the optimised temperature programme.

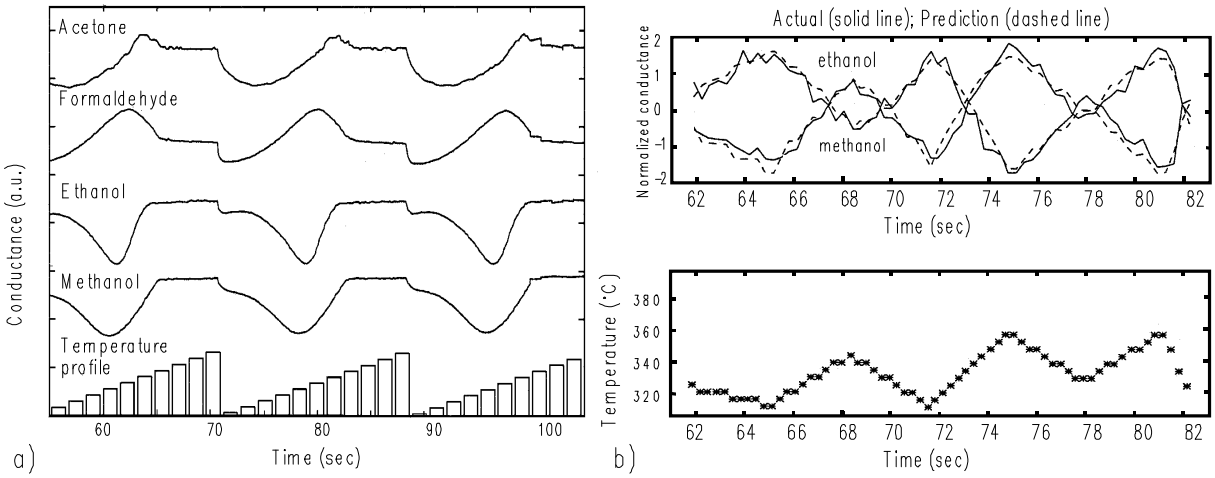



*Fig. 19: (a) Different gas responses subject to a linear temperature profile applied to micro hot plates. (b) Optimized responses for ethanol and methanol [27].*

Feature extraction by Fast Fourrier Transform analysis (FFT) of the resistance/conductance of sinusoidal temperature modulated tin oxide sensors was performed by Nakata et. al. [28] and Heilig et. al. [29].

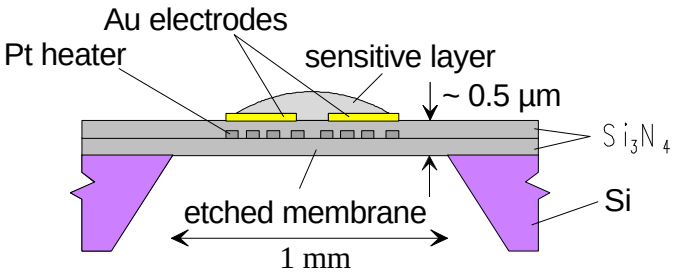



*Fig. 20: Etched microstructure used by Heilig et.al. for sinusoidal temperature modulation [29].*

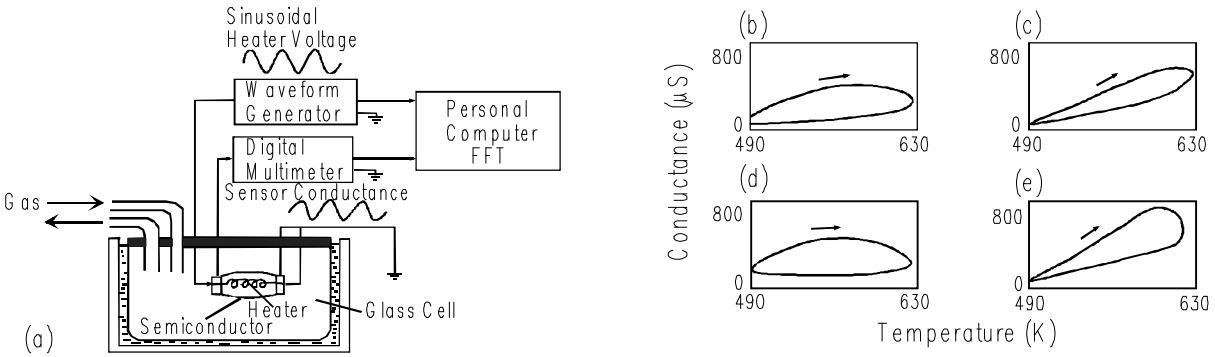



*Fig. 21: (a) Experimental apparatus for detecting the dynamic response of a gas sensor (Figaro TGS 813). Sensor conductance G versus sensor temperature T curve for 1000ppm aromatic vapors: (b) benzene, (c) chlorobenzene, (d) toluene, and (e) nitrobenzene [28].*

Fig. 21a shows the experimental set-up used by Nakata et. al. Fig. 21b to e depicts the different signatures corresponding to different hydrocarbons. The authors conclude that it is possible to quantitatively discriminate between gaseous mixtures using the higher harmonics of the sensor response.

Heilig et.al. [29] analysed hybride tin oxide sensors (micro-machined structures with thick film sensitive layers Fig. 20) using also a sinusoidal temperature modulation combined with FFT feature extraction. Fig. 22a shows the typical fingerprint for CO and $NO_2$. Fig. 22b presents a polar plot using the normalised amplitudes of the base frequency as well as the higher harmonics.

As shown it's possible to discriminate between the pure gases and the mixtures. Using an Artificial Neural Net (ANN) the quantitative analysis was successfully performed.

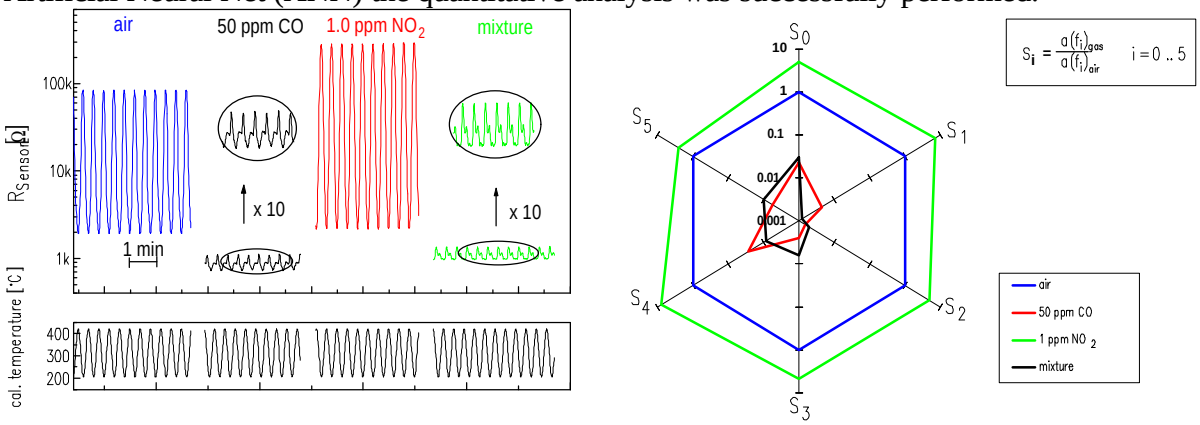



*Fig. 22: (a) Time-dependent changes of the resistance R of a micromachined $SnO_2$ sensor in synthetic air (50% r.h.) and during additional exposure to 50 ppm CO, 1.0 ppm $NO_2$ and a mixture of 50 ppm CO and 1.0 ppm $NO_2$. The nanocrystalline $SnO_2$ material was obtained by calcinating $Sn(OH)_4$ at 450°C followed by Pd surface doping [29] (b) Polar plot of the different gases by plotting the normalised amplitude $S_0...S_5$ of the base frequency as well as the amplitudes of the higher harmonics after FFT ($a(f_i)_{gas}$ ,i=0...5) [29].*

An interesting concept for the temperature modulation for miniaturised pellistors is given by Aigner et. al. [30]. Using a sinusoidal modulation at the input (heater) yields different contributions at the output (spectral components, Fig. 23).

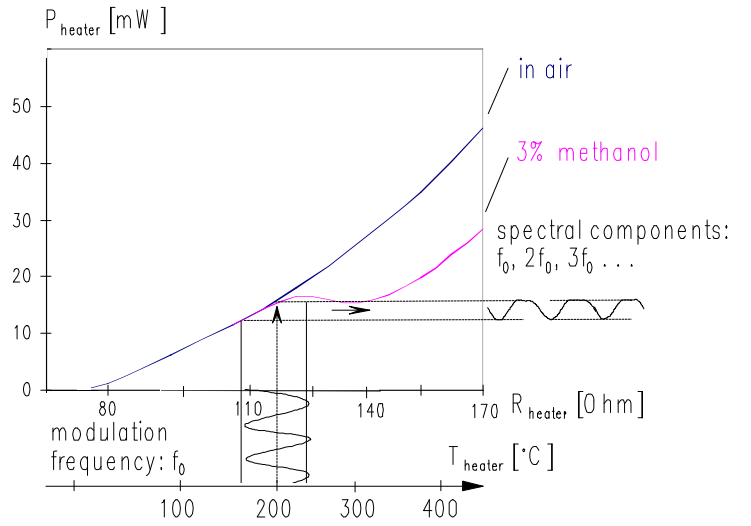



*Fig. 23: Principle of sinusoidal temperature modulation. $P_{heater}$ denotes the heating power, $R_{heater}$ the resistance of the heater structure.*

A variation of contact geometries e.g. in a transmission line measurements (TLM) is offering additional parameters for metal oxide based sensors. In principle a separation between contact and layer contribution is possible. First experiments are reported in [31].

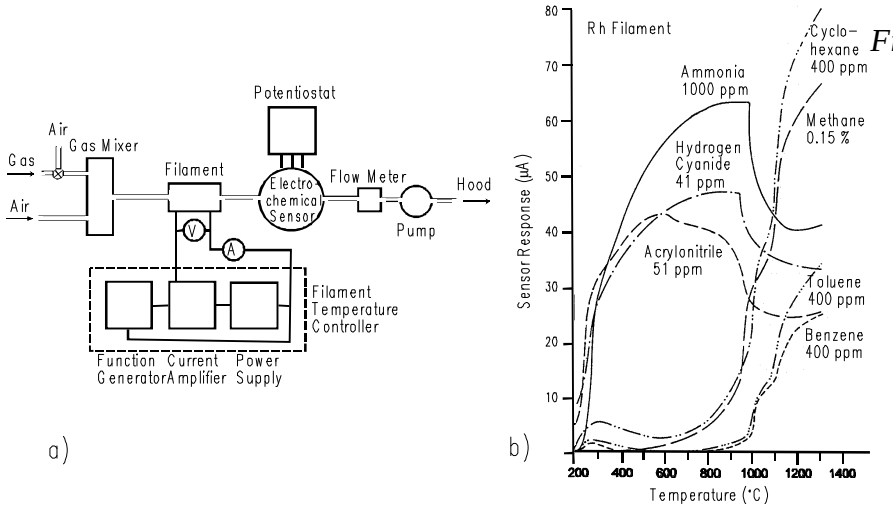



*Fig. 24: (a) Schematic of the experimental system. (b) Response of the electrochemical sensor at various temperatures of the rhodium filament.*

A classical approach of modulating an outer parameter (which does not fit exactly into our classification scheme of this article, since here the sample is varied) is the variation the temperature of a preceding catalyst .

Otagawa and Stetter [32] have used a Rh-filament and an electrochemical cell to perform these experiments. Fig. 24a shows the experimental set-up, Fig. 24b give the sensor response at different temperatures. In consequence this outer parameter variation leads to a variation of the gas composition at the surface of the sensor. A comparable set-up was used by Schweizer et.al. [33] for the study of fish freshness. Further results are given in [34, 35].

## 4 Conclusion and Outlook

As shown, multiparameter sensor systems increase the number of features per sensor element. This allows to include a higher dimensional features space for a subsequent pattern recognition or multicomponent analysis in a cheap way. As shown by the selected case studies selectivities and sensitivities can be increased and even quantitative analysis of gas mixtures can be performed. These techniques as well as a combination of those might be implemented in commercial sensor systems such as electronic noses and expert systems. A useful tool which can be used as workbench for all of these approaches has been optimised recently. This tool is

a modular sensor system in which all components shown very schematically in Fig. 1 can be exchanged and optimised easily ("MOSES" [36]).

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