

## **PRACTICAL APPLICATION OF INVERSE PROBLEM SOLUTION ALGORITHMS FOR WATER POLLUTION MEASUREMENTS USING ION SELECTIVE TRANSISTORS**

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**Abstract** - The paper deals with an inverse problem of ion mixture composition estimation using electronic sensors based on conventional MOS transistors. The device sensitivity to various ions is obtained by replacing the traditional transistor gate structure by special polymer membranes, which are sensitive to the ions in an electrolyte flowing over the gate. Unfortunately, the membrane selectivity is limited and ions other than the main one also influence the measurements, hence rendering the problem ill-conditioned. Thus some special methods must be employed so as to be able to obtain robust ion activity estimates from noisy sensor measurements. In this paper, for given membrane selectivity values, the ion mixture composition is estimated employing the sequential Beck's function specification algorithm. This algorithm has been implemented also in a practical data processing system as a digital filter.

### **1. INTRODUCTION**

The pollution of the natural environment is nowadays one of the most serious problems of industrialized countries. The growing importance of the environmental protection gave rise to the program of sustainable development, which sets up the standards of economical growth and technological progress with special consideration given to the environmental care. This idea is being realized in practice also by the 5<sup>th</sup> Framework Programme of the European Union supporting the SEWING project, whose results are described in this paper.

The main aim of the project is to provide a cheap and efficient system dedicated to real time water pollution monitoring. This system is based on miniature electronic sensing elements, the ion selective transistors. The base structure for all developed sensors constitutes the Ion Sensitive Field Effect Transistor (ISFET) dedicated to the detection of the hydrogen ion. Ions, other than the hydrogen ones, can be detected when the sensors are provided with special ion selective membranes. Such sensors are known as Chemically Modified Field Effect Transistors (CHEMFETs). Special measurement heads, developed within the project activities, containing sets of sensors equipped with membranes dedicated to the detection of different ions will be mounted in the so-called field posts. The processed data collected from the sensors will be transmitted to the main water quality monitoring station. Then, according to the sensor indication, appropriate actions will be taken when necessary.

The next three sections of the paper will provide basic information on the principles of the CHEMFET operation, the method of their modelling and the experimental model validation leading to the determination of model parameters and the membrane selectivity coefficient values. Subsequently, the inverse problem of ion mixture composition estimation based on noisy sensor measurements will be discussed and the possibility of improving the accuracy of obtained estimates using the function specification method will be demonstrated. Finally, the electronic system realizing the data processing in practice will be presented.

### **2. SENSOR DESCRIPTION**

The ion selective transistors are solid-state miniature potentiometric sensors based on silicon technology, which transform chemical information into an electrical signal. These sensors, CHEMFETs, can represent an attractive alternative to standard potentiometric sensors because of their relatively good analytical performance, low price, and small size. As shown in Figure 1, in CHEMFETs the classic gate of an ordinary FET is replaced by a more complex structure consisting of a reference electrode, an analysed solution, an ion selective membrane and a gate dielectric. The variable ion concentration in the solution influences the gate potential, which in turn modifies the transistor threshold voltage. In this way, the ion concentration exercises electrostatic control on the drain-source current. The structure without the membrane is capable of sensing the hydrogen ions and is used as a pH sensor.

When the dielectric is covered with an ion-selective membrane the sensor can be used for the detection of ions other than the hydrogen ones. The PolyHEMA hydrogel contains an inner electrolyte of known composition and stabilizes the sensor operation. All these additional layers introduced into the FET gate structure influence the transistor threshold voltage, and thus creating various potential drops in the gate circuit. The most important part of the sensor is the ion selective membrane, because it determines its selectivity and consequently the measurement range. The most crucial component of the membrane is the so-called ionophore (ion carrier) responsible for the transport of the main ion through the membrane. Another component is the lipophilic salt, which prevents the ions of the opposite sign from penetration to the membrane. Both the ionophore and the salt constitute only a few weight percent of the membrane and they are suspended in a polymer matrix.

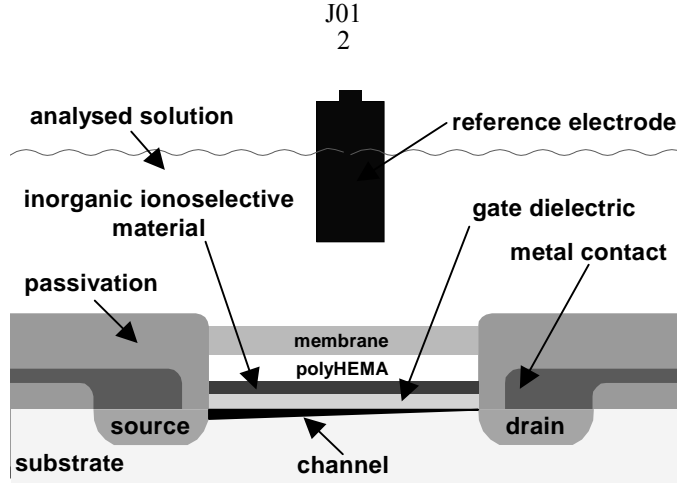


Figure 1: Cross section of CHEMFET sensor.

The electrolyte flowing over the sensor gate closes the electric circuit. The ion concentration in the solution influences the gate potential, which in turn modifies the transistor threshold voltage thus exercising electrostatic control on the drain current. Usually the ion selective transistors are operated in the constant current mode. Then, the drain current variations caused by the threshold voltage shift due to the change of the ion concentration are compensated by the adjustment of the reference electrode potential. Thus, the CHEMFET sensitivity is typically expressed as the gate voltage change per decade of ion concentration. More detailed analysis of the phenomena occurring in the device and the theory of its operation can be found in [2], [9], [16] and [17].

### 3. CHEMFET MODELLING

Most of the physical phenomena occurring in the semiconductor part of the sensor are already modelled in the well-known SPICE environment. Thus, following the idea presented in [10], for the numerical simulations, the authors adapted the existing SPICE Level 3 MOSFET model, in which the threshold voltage is chemically modified through the surface potential induced by the ions present in the analysed solution. The fact that the transistor under consideration was wide and long allowed the model simplification by neglecting most of the terms related to the short channel effects. Then, the transistor drain current can be described by the following equation, [5]:

$$I_D = \frac{C_{ox} \mu_{eff}}{2} \frac{W_{eff}}{L_{eff}} (2(V_{GS} - V_T) - V_{DS}) V_{DS} \quad (1)$$

where  $C_{ox}$  is the gate capacitance per unit area,  $I_D$  the transistor drain current,  $L_{eff}$  the effective channel length,  $V_{GS}$ ,  $V_{DS}$  the gate-source and drain-source voltages,  $V_T$  the threshold voltage,  $W_{eff}$  the effective channel width and  $\mu_{eff}$  the effective carrier mobility.

The most important parameter in equation (1), from the CHEMFET modelling point of view, is the device threshold voltage  $V_T$ , which is strongly material and temperature dependent. This is due to the fact that this voltage is a function of various mutually interdependent quantities such as the Fermi level, the intrinsic carrier concentration, the flat-band voltage, the energy band gap, the substrate dopant concentration and the intrinsic semiconductor carrier concentration. Another strongly variable parameter in equation 1, dependent both on the electrical field and temperature, is the effective carrier mobility  $\mu_{eff}$ . The exact formula to compute the mobility can be expressed as follows:

$$\mu_{eff} = \frac{\mu_0}{1 + \Theta(U_{GS} - V_T) + \frac{\mu_0 V_{DS}}{v_{max} L}} \left( \frac{T}{T_0} \right)^{-a} \quad (2)$$

where  $\Theta$  and  $a$  are the electrical field and temperature mobility modulation coefficients respectively, which have to be estimated based on measurements,  $v_{max}$  is the maximal carrier velocity in the channel and  $\mu_0$  the low field carrier mobility.

When the transistor is operating in the saturation region, the drain-source voltage  $V_{DS}$  must be substituted by the voltage  $V_{DS,sat}$ . More detailed considerations on the SPICE sensor model can be found in [15]. Compared to the standard MOS transistors, the threshold voltage of the CHEMFET has some additional components, summarised in equation (3), which are described later on in this paper. The threshold voltage computed using this equation can be substituted subsequently to some electrical simulator, so as to determine the sensor electrical characteristics, namely:

$$V_T^{CHEMFET} = E_{ref} - E_M - \Psi_s + V_T^{MOSFET} \quad (3)$$

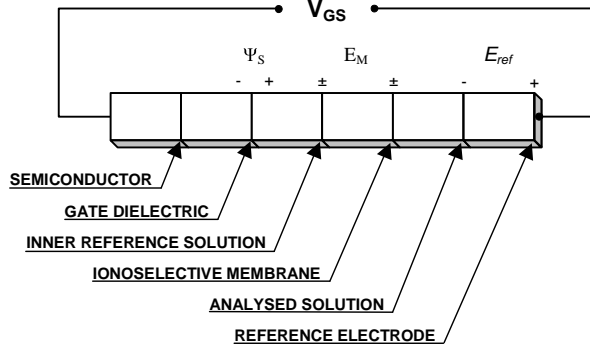


Figure 2: Electric potential in the gate circuit.

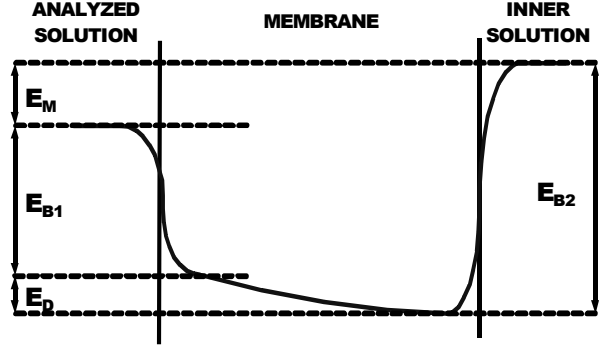


Figure 3: Distribution of membrane potentials.

These additional electric potential drops in the gate circuit are visualised in Figure 2, [7]. Starting with the analysis from the transistor channel side, the first potential drop is the dielectric surface potential  $\Psi_s$ . Unlike in the case of ISFET, where the value of this potential serves as the measure of the hydrogen ion concentration, CHEMFET operation does not depend significantly on the properties of the gate dielectric because the dielectric contacts directly only with the polyHEMA hydrogel containing the electrolyte of known and constant composition. Thus, the surface potential, at least theoretically is constant, if good enough adhesion of the hydrogel is ensured.

From the CHEMFET operation point of view, the most important is the membrane potential  $E_M$  since it is the only one component directly dependent on the composition of the analysed electrolyte. The membrane separates two electrolytes containing the ion to be detected, however the concentrations of this ion in each electrolyte are different. The first electrolyte is the analysed solution, the other one is the reference solution of known main ion concentration contained in the polyHEMA hydrogel. The membrane potential can be expressed as the sum of the membrane diffusion potential  $E_D$  and the difference of the potential drops induced at the phase boundaries  $E_B$ , see equation (4). This difference results from the inequality of the ion concentrations in the measured and the inner reference solution as shown in Figure 3. The membrane potential is usually modelled by an empirical formula presented in the next section, namely:

$$E_M = E_{B1} - E_{B2} + E_D \quad (4)$$

The last component is the reference electrode potential  $E_{ref}$ . This potential results from the Fermi level difference between the solid metal electrode (silver) and liquid solution containing a salt of the electrode metal (silver chloride). The reference electrode potential does not depend on the electrical current or the chemical composition of the solution and is assumed to be the sum of the electrode material dependent potential  $E_0$  and some absolute temperature dependent component, as it is shown in the following equation, [14]:

$$E_{ref} = E_0 + E_T (T - 300) \quad (5)$$

#### 4. MEMBRANE SELECTIVITY

The most important problem encountered during measurements of the ion concentration with CHEMFETs is related to the limited membrane selectivity. As a result, the membrane potential varies not only with the concentration of the main ion to be detected, but also it is dependent on the concentration of some other ions, called interfering ions. The most commonly employed model of the phenomena occurring in the sensor membrane is based on the semi-empirical Nikolski-Eisenman equation, derived from the Nernst equation, [8]. The main advantage of this approach is that it is time efficient, quite accurate and it can be easily implemented in any simulation environment. Moreover, the model parameters, such as the selectivity coefficients, can be found analytically without any difficulties. According to this model, the membrane potential changes  $\Delta V_T$  in presence of various ions in the analysed solution can be expressed by the following equation:

$$\Delta V_T = \frac{RT}{z_i F} \ln \left[ a_i + \sum_{j \neq i} k_{ij}(T) a_j^{\frac{z_i}{z_j}} \right] \quad (6)$$

where F is the Faraday constant, R the gas constant, T the absolute temperature, a the ion activity, i, j the main and interfering ion indices and z the ion electrovalence.

The issue, which has to be commented on, is the difference between the ion concentration and its activity. These two quantities are related through the so-called ionic strength of the solution. However, except for very strong solutions, they are equal to each other and thus these terms will be used interchangeably although all the simulations the ion activity is always taken into account.

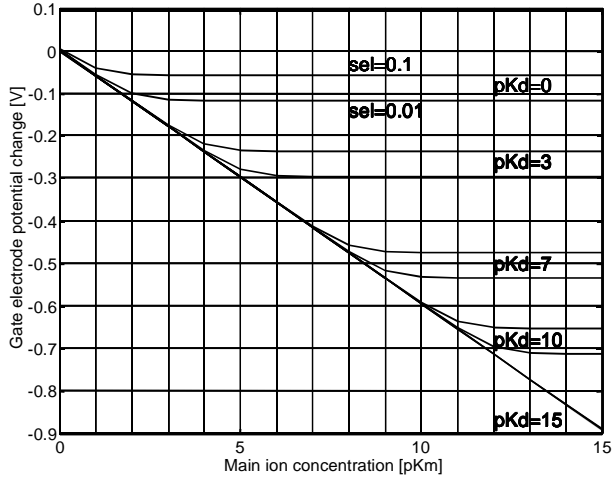


Figure 4: Gate potential change vs. ion activity.

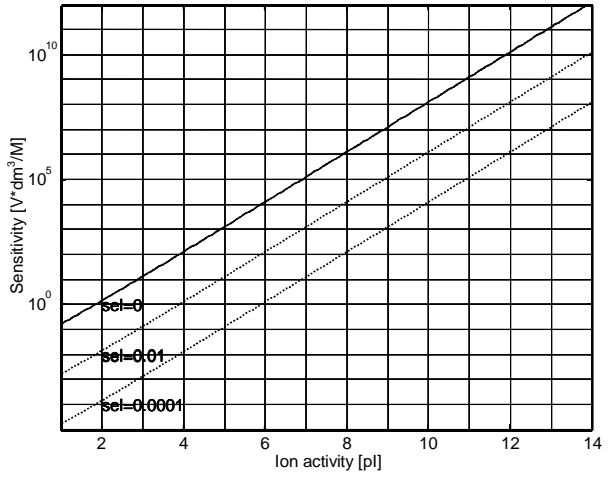


Figure 5: Derivative of gate potential vs. ion activity.

The curves resulting from equation (6) are shown in Figure 4 for two different membrane selectivity values and five different interfering ion activities. Additionally, the derivative of the threshold voltage change with respect to the ion activity was visualised in Figure 5. As can be seen, due to the limited selectivity, the ideal curve flattens out for certain activities of the main ion and consequently the sensor becomes insensitive to the main ion at its low activities. The serious impact, which this phenomenon has on the results of the ion mixture composition estimation, will be discussed in the following sections. The exact location of the bending point on the curve, and consequently the usable measurement range, are determined both by the membrane selectivity and the activity of the interfering ion.

The selectivity coefficient  $k$  is the relative selectivity to an interfering ion  $j$  compared to the main ion  $i$ . For example, if the value of the coefficient is equal to 0.1, the presence of the interfering ions in the concentration is ten times higher than the concentration of the main ion causes exactly the same change of the sensor threshold voltage. The values of the selectivity coefficients are usually given on a logarithmic scale. Similarly, the ion activities are given in the units of  $pK$ , which are equal to the negative decimal logarithm of the concentration. Then, it is much easier to analyze the influence of interfering ions on the sensor operation. Namely, for the interfering ion concentration of  $10^{-3} \text{ M/dm}^3$  and the selectivity of 0.01, the curve will be bent at the main ion activity of  $10^{-5} \text{ M/dm}^3$  ( $-3 - 2 = -5$ ) as demonstrated in Figure 4.

The parameter values of the CHEMFET model were determined based on the measurements and simulations of real sensor structures. The measured CHEMFETs were manufactured at the Institute of Electron Technology in Warsaw, Poland. The ion selective membranes were deposited at the Warsaw University of Technology where the measurements were performed using the specially designed measurement stand described in [4] and [11]. The stand allows fully automated measurements of transistor characteristics with variable ion concentrations and at different temperatures. The main goal of the measurements was to capture electrical characteristics of the devices so as to provide data necessary for sensor model parameter extraction and determination of membrane selectivity coefficients. The logarithms of the experimentally determined membrane selectivity coefficients are presented in Table 1. These values will be used in subsequent numerical experiments consisting in the estimation of ion concentration. More information on the measurements and parameter extraction can be found in [6].

Table 1: Logarithms of selectivity coefficients.

Interfering ion:		$\text{K}^+$	$\text{Na}^+$	$\text{NH}_4^+$
Main ion:	$\text{K}^+$	0.000	-2.564	-1.688
	$\text{Na}^+$	-1.479	0.000	-1.662
	$\text{NH}_4^+$	-0.483	-1.799	0.000

## 5. ION CONCENTRATION ESTIMATION

The experiments presented in this section were carried out in order to investigate the possibility of estimating the composition of ion mixture in the presence of measurement errors. For the experiment, it was assumed that there were three types of sensors monitoring the activity of potassium, sodium and ammonium cations. The hypothetical variations of ion activities in time assumed in the experiment are represented by black lines in Figure 6. Then, knowing the activity of each cation and the membrane selectivity values, the sensor responses were computed. Next, some noise was introduced into the sensor responses so as to investigate the immunity of estimates to the measurement errors. The standard deviation of the noise added to the original values equalled 10 mV. Both the exact (black lines) and the corrupted sensor responses noise (grey lines) are presented in the Figure 7.

Examining Figure 7, it is clearly visible that the sensors respond strongly to high activities of interfering ions. This is particularly noticeable for the sodium sensor, which at low sodium concentration follows the change in the ammonium concentration. The next step of the experiment was to compute the ion activity estimates from the noisy sensor responses. For the considered univalent ions, the earlier presented Nikolski-Eisenman equation describing the changes of threshold voltage can be transformed into the matrix form given in equation (7), where  $K$  is the sensitivity coefficient matrix containing ones on the diagonal and the adequate sensitivity coefficients outside the diagonal and  $A$  is the vector of unknown ion activities. The right hand side of this equation is the pre-processed vector of the sensor threshold voltage changes  $\Delta V$ . Thus, in order to determine the activities of each particular ion based on the measured potential changes, it is sufficient to invert the sensitivity coefficient matrix  $K$  and consequently compute the unknown ion activity matrix  $A$ . The activity estimates obtained through the simple inversion of the sensitivity matrix for the noisy data are represented by grey lines in Figure 6.

$$K A = 10^{\frac{\Delta V F}{2.3RT}} \quad (7)$$

The ion concentration values estimated from the exact data are equal to the arbitrary ones originally assumed for the simulation. This proves that in the case of error free input data it is possible to obtain accurate ion activity estimates. On the contrary, the results produced for the erroneous input data are inaccurate and the estimation error increases significantly for low ion concentration values. This is due to the fact that, because of the limited membrane selectivity, when the concentration of the main ion is low, sensors become sensitive to the interfering ions. As a result, the sensors follow rather the changes in the concentrations of the disturbing ions instead of the one of the main ion.

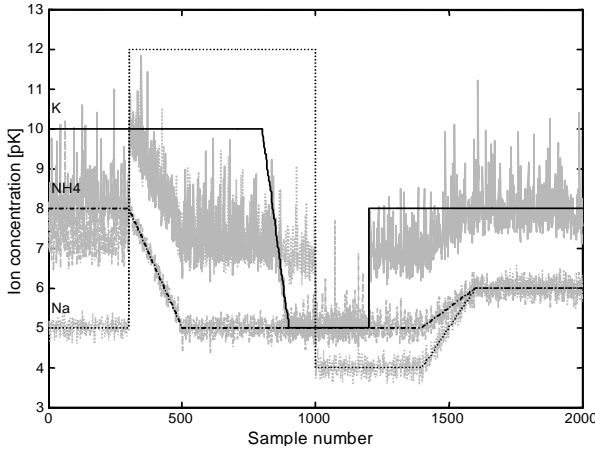


Fig. 6: Original and estimated ion concentrations.

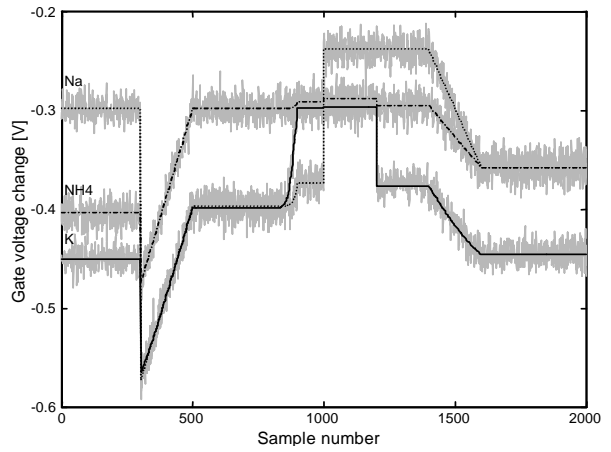


Fig. 7: Exact and noisy gate potential responses.

## 6. APPLICATION OF FUNCTION SPECIFICATION METHOD

The accuracy of the obtained estimates can be improved to some extent by introducing into the system redundant information by placing additional sensors or averaging a certain number of consecutive measurements. Then, instead of the simple inversion of the square selectivity matrix  $K$ , the pseudo inverse matrix of the rectangular selectivity matrix has to be computed. The general formula to determine the pseudo inverse sensitivity matrix  $K^{-I}$ , derived from the least mean squares method, can be expressed by the following equation [3]:

$$K^{-I} = (K^T * K)^{-1} * K^T \quad (8)$$

The exact form of the sensitivity matrix depends on the choice of the particular estimation algorithm. There exist a large variety of such algorithms, but they can be divided into two main groups; the adaptive algorithms and the specialised algorithms for solving inverse problems. For the ion concentration estimation considered here, the function specification algorithm, belonging to the latter group of algorithms, has been chosen.

The function specification algorithm is based on the assumption that the variation with time of the unknown quantity to be estimated has some functional form. In the simplest and the most commonly used version of the algorithm, it is assumed that several consecutive measurements are equal, hence the functional form is constant. In the sequential estimation, the unknown values of the ion concentration are determined successively with each new arriving data knowing the current sensor indication and some past measurements. The estimation accuracy can be further improved by introducing additional ion sensors. The final formula to estimate the activities is expressed by equation 9. For details of the function specification method refer to [1] and the methodology to construct the necessary matrices in the considered case is presented in [12], namely:

$$\hat{A}_k = ((KI)^T KI)^{-1} * (KI)^T * \Delta V \quad (9)$$

Before the practical implementation, the sequential function specification algorithm was tested in numerical experiments. Different numbers of sensors (up to 5 of each kind) combined with a variable number of averaged samples (up to 20) were explored in the simulations so as to find the best possible configuration for improving the quality of the previously obtained unfiltered activity estimates. As demonstrated in [13], the best estimation results were obtained when both additional sensors and multiple values were used. However, both the number of sensors and the future values should not be too large because the improvement in accuracy is insignificant in comparison to the increase of computational complexity.

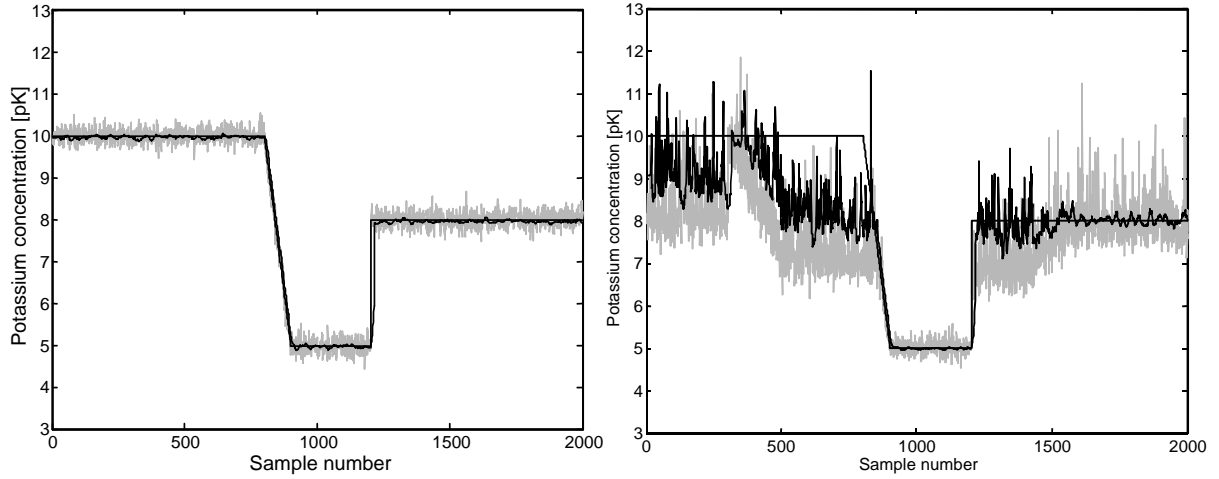


Figure 8: Potassium concentration estimates.

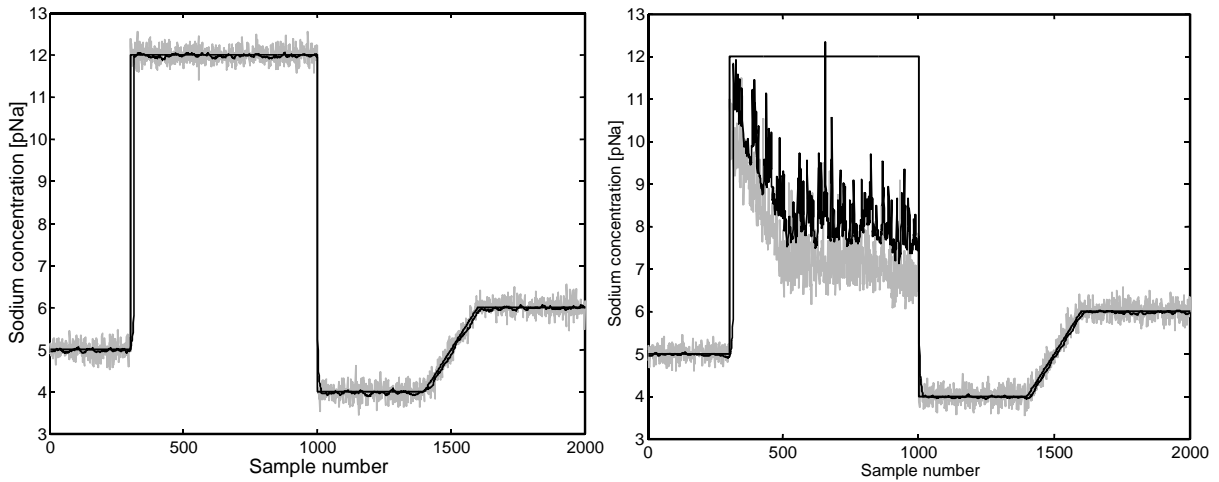


Figure 9: Sodium concentration estimates.

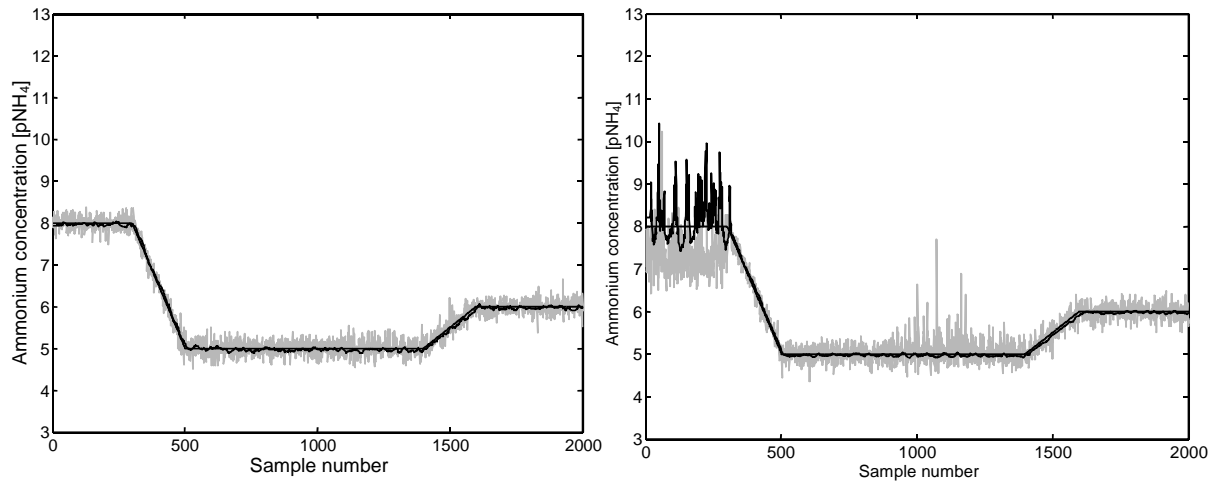


Figure 10: Ammonium concentration estimates.

Initially the operation of the algorithm was investigated without the presence of interfering ions. Then, the influence of these ions was taken into consideration. The estimates obtained in this way are presented by black lines in Figures 8-10. The left column shows the results without the ion interference and the right column with the inference. For comparison purposes, the unfiltered estimates are also shown in these figures by grey lines. As can be seen, the application of the function specification algorithm improved the accuracy of the estimates. However, the general conclusion is that the errors remain unacceptably high in all the cases. Examining the figures it can be concluded that, due to the limited membrane selectivity, the obtained estimation results are far from the actual values only for low concentrations of the main ion. Consequently, the practical estimation range is limited to ion concentrations above  $10^{-8}$  M/dm<sup>3</sup>, unless some extremely selective membranes are developed.

## 7. PRACTICAL SYSTEM REALISATION

The particular problem described here is linear because all the considered ions are univalent. Then, the function specification algorithm can be implemented in the form of a digital filter. This approach is computationally efficient and seems to be particularly well suited for real time water pollution monitoring purposes. The filter coefficients can be determined as the appropriate sensitivity coefficients as described in [1]. The most important advantage of the digital filter approach is that some of the non-linearities occurring in the real cases, such as the temperature dependence of CHEMFET parameters or selectivity coefficients, can be accommodated during the iterative updates of filter coefficients.

The developed data processing system, shown in Figure 11, consists of three parts designed at the various partner institutions participating in the SEWING project. The analogue interface, conceived at the VTT company in Finland, provides adequate bias and collects measurement data from six CHEMFET sensors, manufactured in Poland, and a commercial temperature sensor. Then, the measured analog ion activity samples are digitalized, multiplexed and sent through a serial interface to the TMS320 Digital Signal Processor board where they are stored in memory for further processing. The DSP board, programmed at Universitat Politecnica de Catalunya in Spain and Technical University of Lodz in Poland, runs concurrently several processes implementing different types of operations: The SetUp program calibrates periodically sensors and controls their bias circuits on the analog board. The Measure procedure reads the measured data out of the sensors at specified time instants. The main part of the software code processes the data so that to produce the ion concentration estimates. Except for the function specification algorithm, the program also implements the blind source separation method.

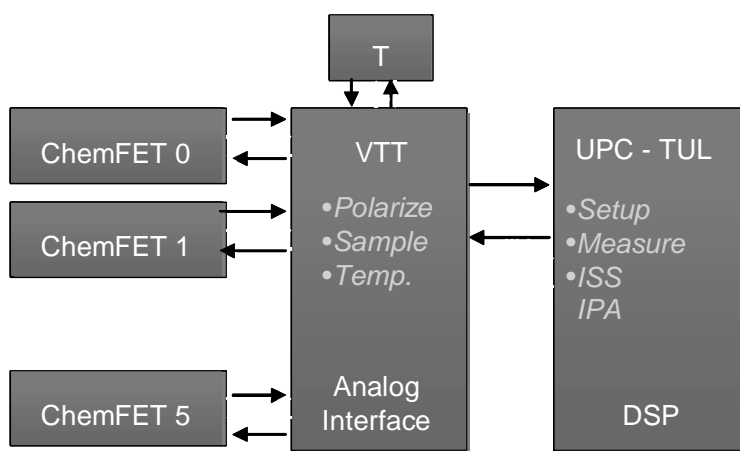


Figure 11: Data processing system for ion concentration estimation.

The digital filter implementing the function specification algorithm has been validated using the artificial test data generated by a special circuit emulating an array of CHEMFET sensors. Knowing the membrane selectivity coefficients for each CHEMFET, for some arbitrary ion concentration values the emulation circuit modelling the Nikolski-Eisemann equation produced the corresponding sensor responses. This circuit was based on several logarithmic amplifiers (ordinary operational amplifiers with diodes in their feedback loops). The experiments were intended to demonstrate the ability of the algorithm to separate the mixed signals when the sensor array is exposed to a noisy environment. The assumed input ion activity values for each cation are shown in Figure 12 whereas the resulting estimates obtained using the digital filter running on the DSP are presented in Figure 13 (the figure shows sequentially the three output channels). As can be seen in the figures the algorithm operation is correct. The digital filter was able to separate the signals mixed by the emulating circuit and provide accurate ion concentration estimates. The system designed within the project is intended to be planted in the measurement heads at the field posts monitoring the water quality so as to test it in real conditions. The first measurement results are expected to be available in spring 2005.

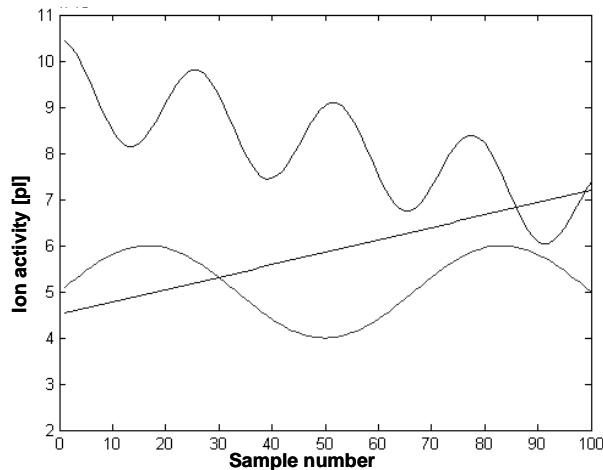


Figure 12: Input algorithm test data.

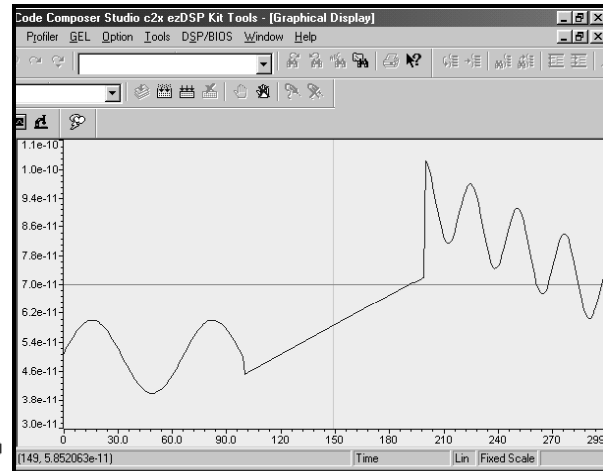


Figure 13: Output ion concentration estimates.

## 8. CONCLUSIONS

This paper presented an inverse problem consisting in the estimation of ion mixture composition estimation using CHEMFET sensors. The particular problem discussed here is especially difficult to solve due to imperfect selectivity of sensor membranes, which limits significantly the practical measurement range. The presented numerical experiments confirmed the fact that such sensors are vulnerable to the presence of interfering ions. For the simulations, a simple but efficient model of CHEMFET sensor was developed. The proposed model combines the standard SPICE MOSFET model and an electrochemical model of the membrane potential, which influences the threshold voltage of the transistor. As demonstrated, the proposed model is suitable for the simulations of the device operating in a wide range of temperatures and ion concentrations.

The function specification method might be a relatively efficient tool for improving the quality of estimates. Employing this algorithm, both the mean value and the standard deviation of the estimation error can be limited by averaging subsequent ion concentration measurements or by introducing additional sensors. Both techniques can be applied simultaneously. The averaging of measured values increases the delay between the occurrence of the ion concentration change and its detection. On the other hand, the introduction of additional sensors increases the computational effort and consequently the estimation time. Therefore, for the real time estimation of ion concentration, the function specification algorithm combining both averaging techniques to reduce the measurement error influence is suggested. The presented digital filter practical realization renders possible the design of an ion concentration monitoring unit, containing a digital filter implementing the function specification algorithm and some additional logic circuitry, which can be integrated within a single IC.

Although the application of the inverse problem algorithm can improve slightly the situation, but certainly it is not a perfect solution. Definitely, the principal limitation of the estimation accuracy constitutes the poor membrane selectivity. Because the estimates obtained for high ion activities are quite accurate, CHEMFETs can be used efficiently only for the detection of contaminant alarm levels. Decisively, in order to improve further the estimate quality the research should be focused on the development of some more selective membranes.

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