

CHEMFET SENSORS MODELING FOR WATER MONITORING SYSTEMS DESIGN

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ABSTRACT: Computer aided design of water monitoring microsystems requires efficient and accurate models of chemical sensors. Sensors considered in this paper use Field Effect Transistors (FETs) with gates replaced by a composition of an intrinsic electrolyte layer covered by a dedicated ion-selective membrane. Foundations of physical modeling of this membrane given by Van den Berg [1] have been extended in [2] to several interfering ions. Unfortunately Van den Berg (VDB) model is too complicated and in practice a simple empirical Nikolski-Eisenman (NE) model is preferred. This work is dedicated to bridge a gap between the VDB and NE models by investigation of a few intermediate models. They show how physical VDB parameters influence empirical NE parameters and provide parameters estimation from the measured calibration curves. These estimations can be good starting points for more accurate identification of the VDB model. Another result of this work is sensitivity analysis explaining how the objective function is sensitive and numerically conditioned. This analysis is useful for selection of identifiable parameters and their scaling factors providing good numerical properties of the identification task. Obtained results are useful in simulation and optimisation of water monitoring microsystems.

INTRODUCTION

A CHEMFET sensor is an important part of modern water testing instruments [6]. It is dedicated to measurements of mole activity of one kind of ions from many kinds dissolved in a solution [2,3,4,5]. A structure of this device is shown in Fig. 1. In place of gate of the traditional FET a POLYCHEMA layer is located as a reference electrolyte and then covered by an ion-selective membrane. This membrane contacts the extrinsic electrolyte subject to test.

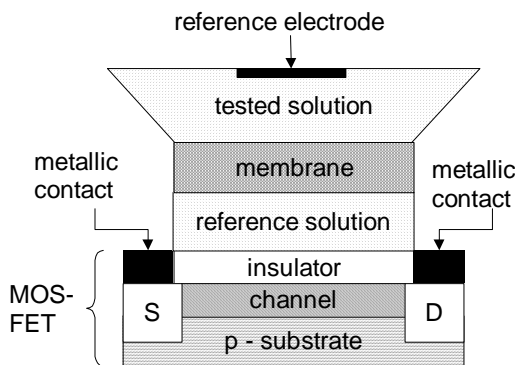


Fig.1. A schematic cross-section through a CHEMFET sensor

Let an aqueous electrolyte contain several kinds of cations C_j^+ and anions A_i^- of respective mole concentrations c_j^+ , c_i^- , electrovalences z_{C_j} , z_{A_i} and activities $a_i = \gamma_i c_i$, where activity coefficient γ_i depends on the

ionic strength of the solution $J = \frac{1}{2} \sum_i z_i^2 c_i$ according to

$\gamma_i = 10^{-Az_i^2 \sqrt{J} / (1+B\delta_i \sqrt{J}) + CJ}$ (δ_i is the effective ion diameter in Å, $A = 7.2 \cdot 10^{-6} \cdot t^2 + 6.6 \cdot 10^{-4} \cdot t + 0.488$, $B = 2 \cdot 10^{-3} \cdot t + 0.325$, $C \in [0.05, 0.15]$ are dependent on temperature t in °C).

The physical model VDB of the ion-selective membrane (Van der Berg [1]) takes into account the following effects. Activities \bar{a}_{C_j} , \bar{a}_{A_i} (equal to concentrations) of

C_j^+ and A_i^- in the membrane depend on activities of these ions in the bulk electrolyte a_{C_j} , a_{A_i} and on the voltage E_B between the membrane surface and the solution bulk according to the Boltzmann Law:

$$\bar{a}_{C_j} = a_{C_j} k_{C_j} \exp(-z_{C_j} E_B / \psi_0), \text{ for } C_j^+, \quad (1)$$

$$\bar{a}_{A_i} = a_{A_i} k_{A_i} \exp(-z_{A_i} E_B / \psi_0), \text{ for } A_i^-,$$

where k_{C_j} , k_{A_i} are division constants for cations and anions, $\psi_0 = n_i kT / q$ with Boltzmann constant k , temperature T , electron charge q and nonideality index n_i .

The membrane is filled with the ionophore containing ligands L which are able to create charged complexes $C_j L$ with tested ions (say cations). Let the ionophore be strongly sensitive to the primary cation C_1^+ and weakly sensitive to interfering cations C_j^+ , $j = 2, \dots, m$. Complexation of ions is proportional to activity of these ions in the membrane \bar{a}_{C_j} and on activity of free ligands

\bar{a}_L according to $\bar{a}_{LC} = \beta_j \bar{a}_{Cj} \bar{a}_L$, $j = 1, \dots, m$, where $\beta_1 \gg \beta_2, \beta_3, \dots$ are complexation coefficients.

Balance of the ionophore with a total concentration of ligand \bar{a}_{Ltot} assumed yields $\bar{a}_L + \sum_{j=0}^m \bar{a}_{LCj} = \bar{a}_{Ltot}$.

The ionophore also contains ions Y of a lipophylic salt which associate with the complexes C_jL producing complexes C_jLY . Their activity is proportional to activity of free lipophylic ions \bar{a}_Y and to concentration of complexes C_jL : $\bar{a}_{YLCj} = K_{aj} \bar{a}_{LCj} \bar{a}_Y$, $j = 1, \dots, m$.

Balance of the ions Y , where a total concentration \bar{a}_{Ytot}

of ions Y is assumed, gives: $\bar{a}_Y + \sum_{j=0}^m \bar{a}_{YLCj} = \bar{a}_{Ytot}$.

Finally electrical neutrality of the membrane yields the balance of all charged molecules:

$$\sum_{j=1}^m (\bar{a}_{Cj} + \bar{a}_{LCj}) - \sum_{i=1}^n \bar{a}_{Ai} - \bar{a}_Y = 0 \quad (2)$$

THE IMPLICIT VDB MODEL

The above introduced physical relations yield a physical model of a border of phases between the membrane and the solution [2]. It can be written in the following form. Let:

$$\begin{aligned} e_B &= \exp(E_B / \psi_0), \quad C_0 = \sum_{j=0}^m k_{Cj} a_{Cj}, \\ C_1 &= \sum_{j=1}^m \beta_j k_{Cj} a_{Cj}, \quad C_2 = \sum_{j=1}^m K_{aj} \beta_j k_{Cj} a_{Cj}, \quad (3) \\ C_3 &= C_1 + C_2 \bar{a}_{Ltot}, \quad k_{Ai} = k_A, \quad a_{Ai} = a_A. \end{aligned}$$

The CHEMFET membrane implicit model can be written as a sum of four components:

$$M_1 + M_2 + M_3 + M_4 = 0 \quad (4a)$$

where

$$M_1 = \frac{-\bar{a}_{Ltot} C_1}{C_1 + e_B}, \quad M_2 = \frac{\bar{a}_{Ytot} (C_1 + e_B)}{e_B + C_3}, \quad (4b)$$

arise from complexation and association effects, while

$$M_3 = -\frac{C_0}{e_B}, \quad M_4 = k_A a_A \quad (4c)$$

are resulted from cations and anions in the membrane.

If we assume ion activities a_{Cj} , a_{Ai} in the bulk extrinsic electrolyte, and the primary cation C_1^+ and a corresponding anion activities \bar{a}_{C1R} , \bar{a}_{AiR} in the intrinsic electrolyte then the model (4) can be solved twice for e_B to get the membrane to extrinsic and intrinsic electrolyte potentials: E_B and E_{BR} respectively. Voltage across the membrane is now $E_M = E_B - E_{BR}$ while the diffusion effect in the membrane is neglected.

VDB MODEL APPROXIMATION

Example

Explicit approximations and sensitivities of the VDB model in Fig. 2-4,6,7 will be demonstrated for the following typical parameters. The sensor is dedicated to the primary cations K^+ , interfering cations Na^+ of concentration $c_{C2} = 10^{-1}$ [mole/l] and anions Cl^- . Model parameters are: $\delta_1 = \delta_2 = 3$, $\beta_1 = 10^9$ [l/mole], $\beta_2 = 10^5$ [l/mole], $t = 25$ [°C], $\bar{a}_{Ytot} = 5 \cdot 10^{-4}$ [mole/l], $\bar{a}_{Ltot} = 10^{-3}$ [mole/l], $K_{a1} = 5 \cdot 10^3$, $K_{a2} = 100$, $c_{1R} = 10^{-3}$ [mole/l], $k_{C1} = k_{C2} = k_A = 10^{-6}$.

The case of lipophylic salt consideration

It can be shown that in the very wide range of primary cations C_1^+ concentration (e.g. from 10^{-8} to 10^0) components $|M_1|$ and $|M_2|$ remain on the almost constant level α due to dominating effects of complexation and association. Moreover in a small and medium concentration range (Nikolski range) negligible are M_3 and M_4 . Hence simplified equations $\alpha = M_1 = -M_2$ give a very good approximation of VDB in the Nikolski range:

$$E_B = \psi_0 \ln(e_B) \quad (5a)$$

where

$$e_B = C_1 \left[\frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}} \left(1 + \sqrt{1 + 4\bar{a}_{Ytot} \frac{C_2}{C_1}} \right) - 1 \right] \quad (5b)$$

and C_1, C_2 include the dependence on a_{C1} . Moreover

$$\alpha = \frac{2\bar{a}_{Ytot}}{1 + \sqrt{1 + 4\bar{a}_{Ytot} \frac{C_2}{C_1}}}. \quad (5c)$$

The model (5ab) we will call super NE (SNE). Unfortunately in general it does not reduce to the Nikolski-Eisenman (NE) model [3] since the term in brackets in (5b) is not constant but depends on the primary ion activity a_{C1} through the ratio C_2/C_1 .

We find out that the SNE model is exactly equivalent to the NE model only if all association constants are equal: $K_{a1} = K_{a2} = \dots = K_a$. i.e.:

$$e_B = C_1 \left[\frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}} \left(1 + \sqrt{1 + 4\bar{a}_{Ytot} K_a} \right) - 1 \right]. \quad (6)$$

Otherwise SNE can only be approximated by the less accurate though still applicable in practice NE formula. This NE approximation of (5ab) takes the form:

$$E_B = E_{Boffset} + \psi_0 \ln(a_{C1} + \sum_{i=2}^m K_i a_{Ci}), \quad (7a)$$

where the offset voltage and selectivity coefficient can be expressed as:

$$E_{Boffset} = \psi_0 \ln \left\{ \beta_1 k_{C1} \left[\frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}} (1 + \sqrt{1 + 4\bar{a}_{Ytot} K_{a1}}) - 1 \right] \right\}, \quad (7b)$$

$$K_i = \frac{\beta_i k_{Ci} \left[\frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}} (1 + \sqrt{1 + 4\bar{a}_{Ytot} K_{am}}) - 1 \right]}{\beta_1 k_{C1} \left[\frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}} (1 + \sqrt{1 + 4\bar{a}_{Ytot} K_{a1}}) - 1 \right]}, \quad (7c)$$

while K_{am} depends on interfering cations:

$$K_{am} = \frac{\sum_{i=2}^m K_{ai} \beta_i k_{Ci} a_{Ci}}{\sum_{i=2}^m \beta_i k_{Ci} a_{Ci}} \quad (7d)$$

and the slope of (7a) is $2.3 \psi_0$ (about 57mV per decade at $t=25^\circ\text{C}$).

In the ideal Nikolski case $K_{a1} = K_{a2} = \dots = K_a$ where the formula (6) holds the selectivity (7c) simplifies to $K_i = \beta_i k_{Ci} / \beta_1 k_{C1}$. In the general case it depends on ligands, lipophilic salt total concentrations and association constants.

Quality of SNE (5ab) and NE (7abc) models is demonstrated in Fig. 2. More accurate SNE very well explains the small and medium concentration range with accuracy better than 0.3% of the range. Accuracy of NE approximation (7abc) in this region is worse, error is about 1.5% and increases when the ratio C_2/C_1 , i.e. the contrast between association constants, increases.

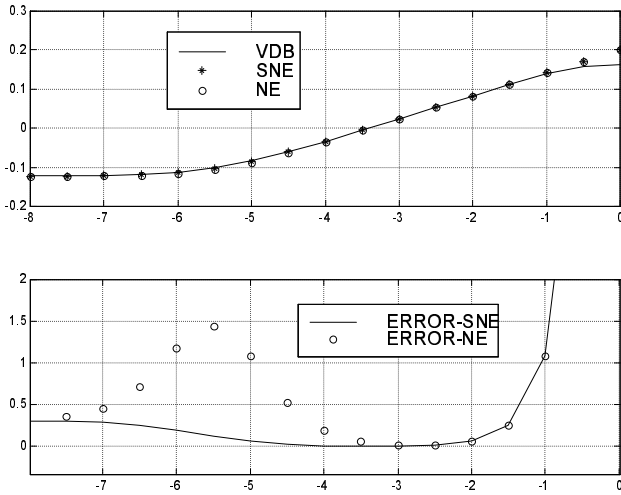


Fig. 2. E_B in volts with respect to logarithm of primary ions concentration for VDB, SNE, NE models (upper) and relative error of SNE and NE in % (lower)

The high concentration knee

High concentration operation of the model (4) can be approximated assuming that M_2 remains on the level α (5c), M_3 is negligible and concentration of ions influence components M_1 and M_4 according to the equation $M_1 + \alpha + M_4 = 0$. This gives the characteristic $E_B(a_{C1})$ of the form (7a) though with the high concentration knee. We define the knee activity as the activity where

$\psi_0 \log(2)$ drop of E_B occurs. The model including the knee (called SNE+knee) takes the form:

$$e_B = \frac{C_1 \left(\frac{\bar{a}_{Ltot}}{\alpha} - 1 \right)}{\frac{1}{2} \left(1 + \eta + \sqrt{(1-\eta)^2 + 4 \frac{\eta \bar{a}_{Ltot}}{\alpha}} \right)}, \eta = \frac{C_1 k_A a_A}{\alpha} \quad (8)$$

where in the nominator there is the model (5b), while the denominator produce the knee.

Activity of the primary ion corresponding to the knee can be estimated from:

$$a_{C1knee} = \frac{2\delta}{\sqrt{\omega^2 + 4\delta + \omega}} \quad (9a)$$

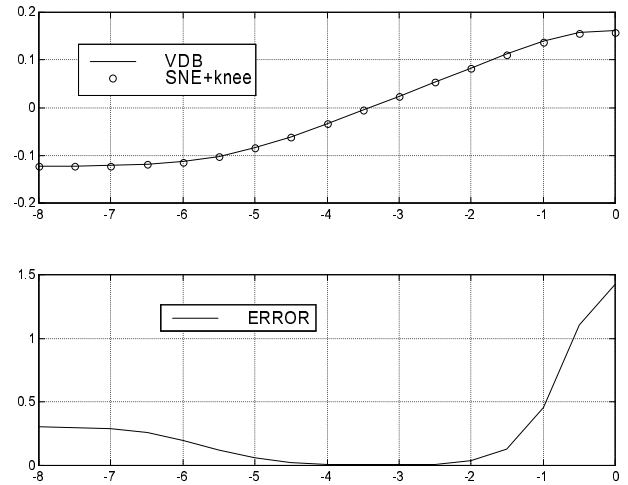
where

$$K_i = \frac{\beta_i k_{Ci}}{\beta_1 k_{C1}}, \quad \omega = \sum_{i=2}^m (K_i + 1) a_{Ci} \quad (9b)$$

$$\delta = \frac{2\bar{a}_{Ytot} / (k_{C1} \beta_1)}{k_A (1 + \frac{\bar{a}_{Ltot}}{\alpha})} - \left(\sum_{i=2}^m a_{Ci} \right) \left(\sum_{i=2}^m K_i a_{Ci} \right). \quad (9c)$$

Quality of the model (8) is shown in Fig. 3. Its inaccuracy is less than 1.5 per cent.

Estimated knee concentration calculated from (9a) for the example involved is $c_{C1knee} = 10^{-0.4}$ [mole/l].



ions concentration for VDB and SNE+knee models (upper) and relative error of SNE+knee in % (lower)

The case of no lipophilic salt

The VDB model can be also considered in the case when the membrane is made of ionophore with no lipophilic salt, i.e. $\bar{a}_{Ytot} = 0$. In this case in (4) $M_2 = 0$ and M_3 is negligible, and so $M_1 + M_4 = 0$. This gives a very good approximation:

$$e_B = \frac{C_1 \bar{a}_{Ltot}}{k_A a_A} \quad (10)$$

$$\frac{C_1}{2} + \sqrt{\frac{C_1^2}{4} + \frac{C_1 \bar{a}_{Ltot}}{k_A a_A}}$$

which can be further approximated in the NE form:

$$E_B = E_{Bofset} + \frac{\psi_0}{2} \ln(a_{C1} + \sum_{i=2}^m K_i a_{Ci}) \quad (11a)$$

where K_i is given by (9b) while:

$$E_{Bofset} = \frac{\psi_0}{2} \ln \left(\frac{\bar{a}_{Ltot}}{\beta_1 k_{C1} k_A \sum_{i=2}^m a_{Ci}} \right) \quad (11b)$$

Selectivity of the model (11) depends on the ratio of division and complexation constants. Accuracy of the model (10) with respect to the VDB model (better then 0.05% in the full range) is shown in Fig. 4.

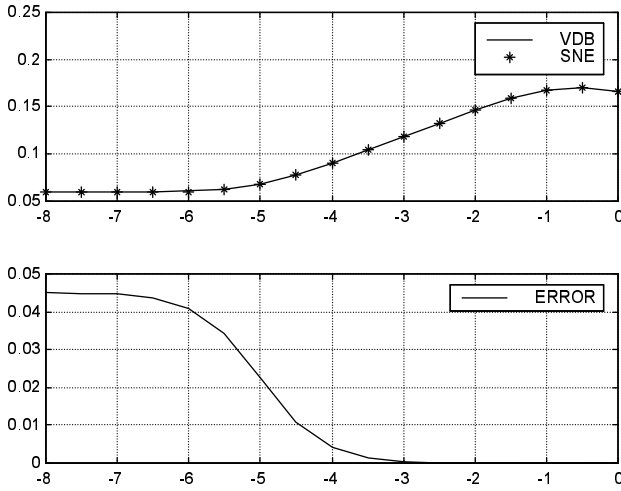


Fig.4. E_B in volts with respect to logarithm of primary ions concentration in the no lipophylic salt case for: VDB (solid), eq. (10) (asterisks) (upper) and relative error of model (10) (lower)

MODEL IDENTIFICABILITY

Parameters selection and sensitivity analysis

Efficiency and accuracy of the model identification is its the most important feature. Identification of the implicit VDB model is difficult since only the total offset voltage of the sensor: $E_{GS} = E_{FET} + E_{BR} - E_B$ is available from measurements (see Fig.5) and its semirelative sensitivities with respect to technological parameters not only are dependent on concentration but some of them also are extremely small in comparison with others. Hence the identification task of technological parameters:

- n_i - nonideality index,
- k_{Cj} - division constants for cations,

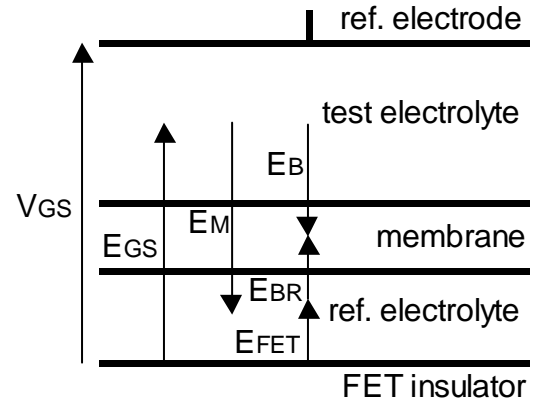


Fig. 5. Voltages arrangement in the CHEMFET

- β_j - complexation constants for cations,
- K_{aj} - association constants for cations, (12)
- \bar{a}_{Ltot} - total concentration of ligands,
- \bar{a}_{Ytot} - total concentration of a lipophilic salt,
- k_{Ai} - division constants for anions,
- a_{C1R} - primary cations activity in the reference solution,

E_{FET} - FET offset voltage,

is, in general, ill-conditioned. Thus we transform this primary set to the secondary set of parameters:

- $K_j = \frac{\beta_j k_{Cj}}{\beta_1 k_{C1}}$ - intrinsic selectivity,
- $E_{offs} = E_{FET} + \psi_0 \ln a_{C1R}$ - intrinsic offset voltage,
- n_i - nonideality index, (13)
- K_{aj} - association constants for cations,
- \bar{a}_{Ltot} - total concentration of ligands,
- \bar{a}_{Ytot} - total concentration of a lipophilic salt,
- $k_{Ai} k_{C1} \beta_1$ - normalized division constants for anions,
- $k_{Ci} / k_{C1} \beta_1$ - normalized division constants for cations,
- $1 / \beta_j$ - inverts of complexation constants

obtainable from (4) after some algebra.

Sensitivites of the voltage E_{GS} to both primary and secondary parameters may be calculated more accurately from the implicit VDB model and approximately from the explicit model (5a), (8). Discrepancy between exact and approximate derivatives varies from a few to a few tens per cent. Analytically determined semirelative sensitivities with respect to secondary parameters (13) applicable for scalling the identification least squares task have been plotted in Fig. 6.

Presented sensitivities show that in the identification task essential are the most influencing parameters: E_{offs} (sensitivity: $\sim 10^{-1}$), K_j , \bar{a}_{Ltot} , \bar{a}_{Ytot} , K_{a1} (sensitivity: $\sim 10^{-2}$). Weaker identifiable is K_{a2} (sensitivity $\sim 10^{-8}$) influencing only the small concentration region and anion parameter $k_{Ai} k_{C1} \beta_1$ (sensitivity $\sim 10^{-2}$) influencing in the high concentration knee region, which is not observable in up to date high quality sensors. Further pa-

parameters $k_{C_i}/k_{C_1}\beta_1$ and $1/\beta_j$ are negligible since they influence only the negligible component M_3 of the general model (4).

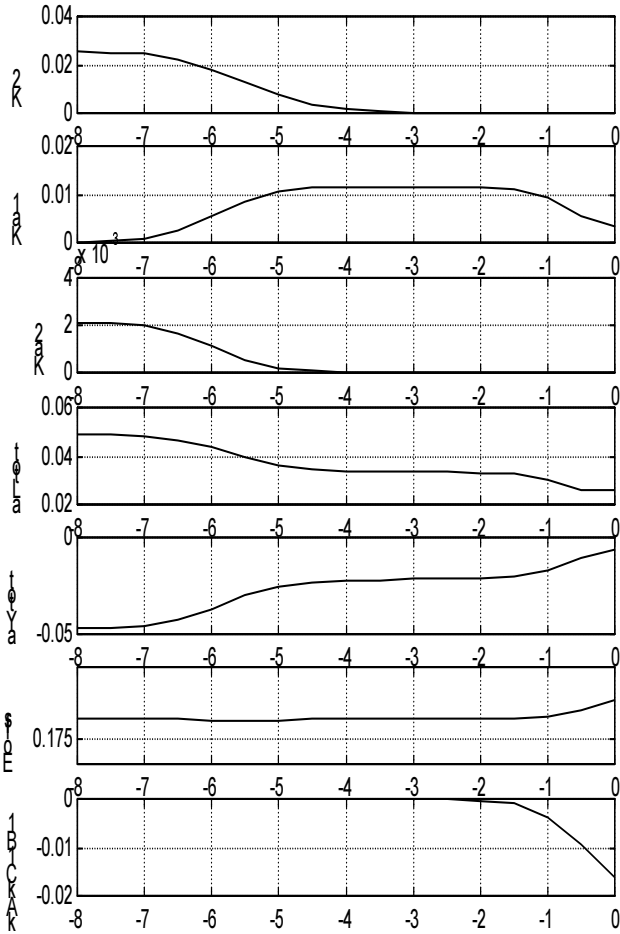


Fig. 6. Sensitivities of E_{GS} voltage to parameters: $K_2, K_{a1}, K_{a2}, \bar{a}_{Ltot}, \bar{a}_{Ytot}, E_{offs}, k_A k_{C1} \beta_1$

Rough identification of the model

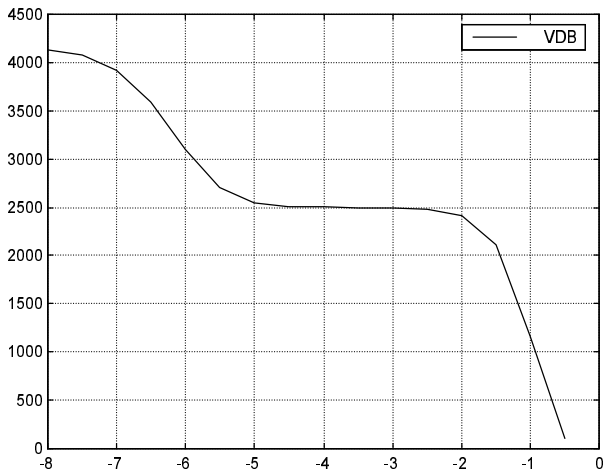


Fig. 7. The plot of dE_B/dc_{C1}

Let us consider the CHEMFET model in the primary ions concentration domain $E_{GS} = E_{offs} - \psi_0 \ln(e_{GS})$,

$$e_{GS} = c_{C1} + \sum_{j=2}^m K_j c_{Cj}, E_{offs} = E_{FET} + \psi_0 \ln c_{1R}.$$

Identification procedure consists of the following stages:

1. *Nonideality index* is identifiable from the slope of $E_{GS}(c_{C1})$ in the medium concentration linear region.

We select $E^1 = E_{GS}(c_{C1}^1)$, $E^2 = E_{GS}(c_{C1}^2)$ in this region, estimate $\psi_0 = (E^2 - E^1) / \ln(c_{C1}^1 / c_{C1}^2)$ and calculate $n_i = kT/q\psi_0$.

2. *The offset* can be estimated from a point selected in the linear region $E^1 = E_{GS}(c_{C1}^1)$. Then we obtain:

$$E_{offs} = E^1 + \psi_0 \ln c_{C1}^1 \text{ and } E_{FET} = E_{offs} - \psi_0 \ln c_{1R}.$$

3. *Association constants*. If not all K_{aj} are the same they influence the calibration curve. This can be seen on $d e_{GS} / d c_{C1} = d \exp[(-E_{GS} + E_{offs}) / \psi_0] / d c_{C1}$ plot. In Fig. 7 shown is this derivative approximated by difference quotients. In the small concentration region where the sensor calibration curve is flat we have two plateaux (left plateau - level 4200 and right plateau - level 2500) dependent on association constants. They enable estimation of K_{a1}, K_{a2} .

In the case of one interfering cation a ratio of plateau levels (of $d e_{GS} / d c_{C1}$): $r = e'_{GS \text{ left}} / e'_{GS \text{ right}}$ can be expressed by the formulae:

$$r = \frac{\left[\kappa_2 + \frac{\lambda(\kappa_1^2 - \kappa_2^2) / 2}{(\kappa_2 + 1) / \lambda - 1} \right]}{\kappa_1}, \quad (14)$$

where $\kappa_1 = \lambda(1 + \sqrt{1 + 4\bar{a}_{Ytot}K_{a1}}) - 1$, $\lambda = \frac{\bar{a}_{Ltot}}{2\bar{a}_{Ytot}}$,

$$\kappa_2 = \lambda(1 + \sqrt{1 + 4\bar{a}_{Ytot}K_{a2}}) - 1.$$

If r is estimated from a slope of the measured characteristic and K_{a2} is assumed then κ_1 and hence K_{a1} can be calculated from (14).

4. *Interfering cations contribution* $w = \sum_{j=2, m} K_j c_{Cj}$ can

be estimated from two points chosen on the low concentration knee where the curvature is highest: $E^1 = E_{GS}(c_{C1}^1)$, $E^2 = E_{GS}(c_{C1}^2)$. We calculate $w = (c_{C1}^1 - \varepsilon c_{C1}^2) / (\varepsilon - 1)$ where $\varepsilon = \exp((E^2 - E^1) / \psi_0)$. If a solution contains one type of interfering cations C_j^+ , then a selectivity constant can be estimated from $K_j = w / c_{Cj}$. Due to (7c) the intrinsic selectivity mentioned in (13) can be calculated from $K_j \kappa_1 / \kappa_2$.

Example

Certain CHEMFET sensor's V_{GS} voltage has been measured under $V_{DS}=0.5V$ at threshold I_D current $1mA$ in a solution with primary K^+ and interfering Na^+ ions of concentration $0.1 [mole/l]$. Taken measures are collected in Table 1.

Table 1. A CHEMFET measures

$\lg c_{C1}$	$V_{GS} [V]$	$\lg c_{C1}$	$V_{GS} [V]$
-6	-0.450359	-3.5	-0.537747
-5	-0.470402	-3	-0.562922
-4.5	-0.489739	-2	-0.618414
-4	-0.517489	-1	-0.670697

Secondary parameters $n_i=1.0988$, $E_{offs}=-0.7262[V]$, $K_2=5.6434e-5$ have been identified from the voltages in Table 1. $\Delta e_{GS} / \Delta c_{C1}$ estimated from Table 1 gave (very roughly) a left plateau ~ 1.2 and right plateau ~ 0.95 (see Fig. 8). From $K_{a2}=100$ obtained is $K_{a1}=3250$. We get $E_{fet}=-0.5645[V]$ and $\beta_2=1.4108e5$ while $\beta_1=1e9$ and $k_{C1}=k_{C2}=1e-6$ have been assumed. The identified model is plotted in Fig.9 together with measured points. Maximum missfitting is about 4% of the range.

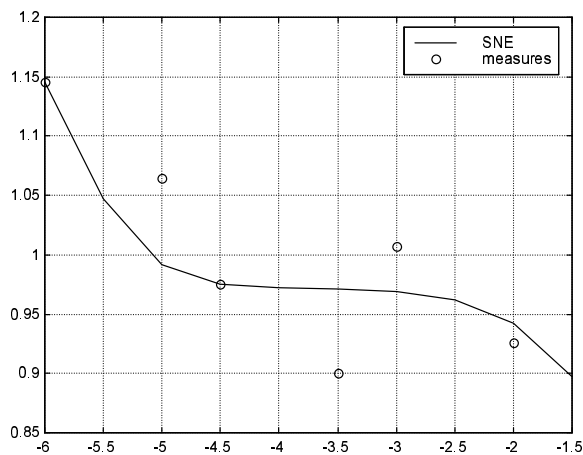


Fig 8 Measured and identified $\Delta e_{GS} / \Delta c_{C1}$

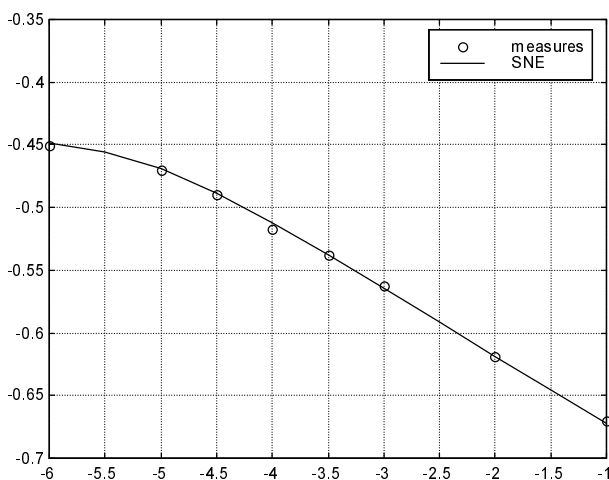


Fig. 9 E_{GS} in volts calculated from the identified model (5ab) (solid line) and measured (circles)

CONCLUSIONS

The VDB model is an implicit model with technological parameters which are hard for identification. In this paper its simplification to a few explicit models of different accuracy levels have been introduced. They bridge a gap between the VDB and NE model. Accuracy of the introduced models varies from 0.3 to a few per cent and is better than accuracy of identification (due to little accuracy of chemical measurements). So in practice these models (in conjunction with a model of the electrical part of the sensor) are quite sufficient for CAD of chemical MEMS.

Moreover a useful method for identification of a few basic parameters of the proposed models has been introduced. Other parameters less influencing the model should be assumed arbitrary. This rough identification is useful as a very good starting point for final identification based on the least p-th method.

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