

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

PHYSICAL CHEMISTRY DIVISION
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TERMINOLOGY AND CONVENTIONS FOR MICROELECTRONIC ION-SELECTIVE FIELD EFFECT TRANSISTOR DEVICES IN ELECTROCHEMISTRY

(IUPAC Recommendations 1994)

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Terminology and conventions for microelectronic ion-selective field effect transistor devices in electrochemistry (IUPAC Recommendations 1994)

ABSTRACT

In anticipation of the widespread use of *Ion-Selective Field Effect Transistors* (ISFETs) in electrochemistry and analytical chemistry it is appropriate to make recommendations about terminology and presentation of results. The terms gate voltage, drain current and gate bias potential are defined and experimental techniques for measurements with ISFETs are summarised. The output signal is usually a potential difference whose magnitude varies with change in logarithm of sensed ion activity or concentration in the same way (but not necessarily in sign) as the corresponding ion-selective electrode (ISE). It is proposed that the graphical representation of results should be in accordance with previously made recommendations for ISEs, that is, that the output potential difference be plotted so that variation with increase of logarithm of activity or concentration has a positive slope for cation-responsive devices and a negative slope for anion-responsive devices.

1. INTRODUCTION

Since the original pioneering work of Bergveld [1,2] on *ion-selective field effect transistors* (ISFETs), interest has grown, in spite of very real practical difficulties, in this interdisciplinary area linking solid state integrated circuit technology with analytical potentiometry using *ion-selective electrodes* (ISEs) [3,4,5,6]. Developments have been slow but steady, so, with some devices now readily available, it is appropriate to make recommendations about terminology and presentation of results, in order to avoid the confusion that can easily arise in interdisciplinary areas.

2. NOTATION AND TERMINOLOGY

Not all chemical sensitive semiconductor devices are based on the *field effect transistor* (FET) [7], *metal oxide semiconductor field effect transistor* (MOSFET), or *insulated gate field effect transistor* (IGFET). Also, not all *chemical sensitive FETs* (ChemFETs) are ion-sensitive (ISFETs), but we shall restrict attention here to ISFETs. Generally, ChemFETs are sensitive to gases and to enzyme substrates as well as to ions. Structurally, the ISFET is very similar to the IGFET, and a typical construction of an n-channel IGFET is shown in Figure 1.

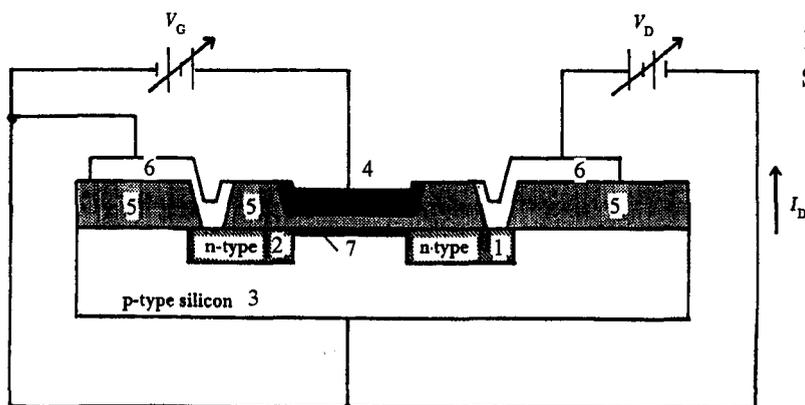


Figure 1.
Schematic diagram of an IGFET.
(1) drain,
(2) source,
(3) substrate,
(4) gate,
(5) insulator,
(6) metal contacts,
(7) conducting channel.

It consists of a p-type silicon substrate with source and drain diffusions separated by a channel which is overlain by SiO₂ as insulator and a metal gate. The polarity and magnitude of the gate voltage (V_G) applied between the substrate and the gate are chosen so that an n-type inversion layer forms in the channel between the source and drain regions. The magnitude of the drain current (I_D) will be determined by the effective electrical resistance of the surface inversion layer and the voltage difference (V_D) between the source and the drain.

V_G

I_D

V_D

The ISFET (Figure 2) differs from the IGFET in several respects: (i) the solution (analyte) is in direct contact with the gate insulator layer(s) and a reference electrode in the solution replaces the metal gate. Previously it had been thought [1,2] that the reference electrode was unnecessary, but this is not so [8]. (ii) silicon nitride, Si₃N₄, overlying the SiO₂ is used to provide a charge blocking interface and improved pH response. (iii) other membranes such as poly(vinyl chloride) containing valinomycin, which is the ion-selective electroactive material used in potassium ion-selective electrodes, can be added to confer other ion selectivities to the ISFET. (iv) the successful encapsulation of all regions of the device other than the gate region to be exposed to the analyte solutions is mandatory [9].

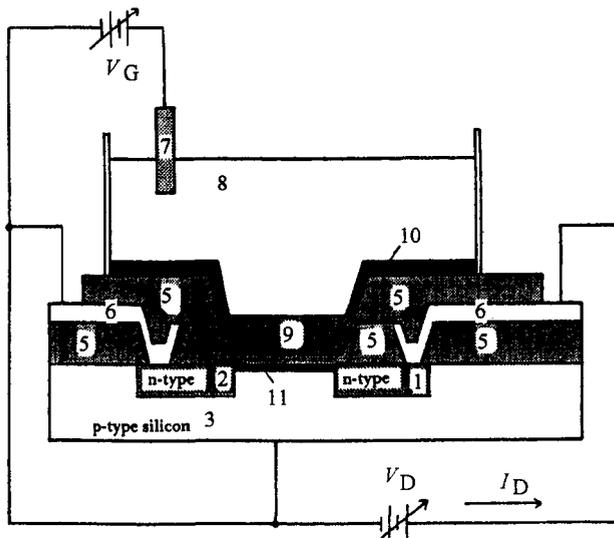


Figure 2. Schematic diagram of a composite gate, dual dielectric ISFET.

- (1) drain,
- (2) source,
- (3) substrate,
- (5) insulator,
- (6) metal contacts,
- (7) reference electrode,
- (8) solution,
- (9) electroactive membrane,
- (10) encapsulant,
- (11) conducting channel.

A diagram of the complete electrochemical system, together with the relevant electrical potentials (i.e. differences in inner potentials between the bulk phases), is shown in Figure 3. Detailed analysis of the system is available [10,3,4,6]. By analogy with the IGFET gate voltage (V_G), it is possible to define an equivalent ISFET gate voltage (V_G^*) as the electrical potential difference between the bulk phases of the semiconductor and gate material

$$V_G^* = E_I + V_{ref} + V_B$$

V_G^*

V_B

E_I

V_{ref}

where V_B is an additional series polarising voltage (gate bias potential), E_I is the interfacial membrane-solution potential difference generally given by the Nernst or Eisenman-Nikolsky equations, and V_{ref} is the reference electrode potential.

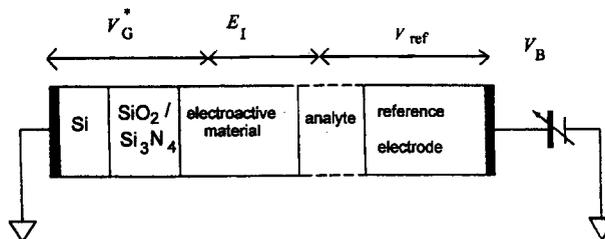


Figure 3. Schematic diagram of composite gate, dual dielectric ISFET showing potential difference contributions.

The I_D/V_G^* characteristics of ISFET devices are essentially those of the FET substructure on which it is based and depend on device design, in particular channel geometry and structure, materials and processing conditions. Several techniques of measurement can be used. If the applied gate bias potential (V_B) is fixed, then changes at the solution-electroactive material interface are reflected in changes in I_D . However, if the drain current is maintained at a constant value by means of an operational amplifier, which directly controls the applied gate bias potential using a negative feedback loop (Figure 4), then the output, V_{out} , is a potential difference which varies with change in activity of the sensed ion (a_i) in accordance with the Nernst equation. The output is therefore effectively the same as that from an ISE. Problems of representation and comparison arise, however, because V_{out} may or may not have the same sign associated with it as the ion-selective electrode depending on the inverting characteristics of the operational amplifier(s) used. For this reason, a uniform method of presentation of results from ISFETs and ISEs is recommended below.

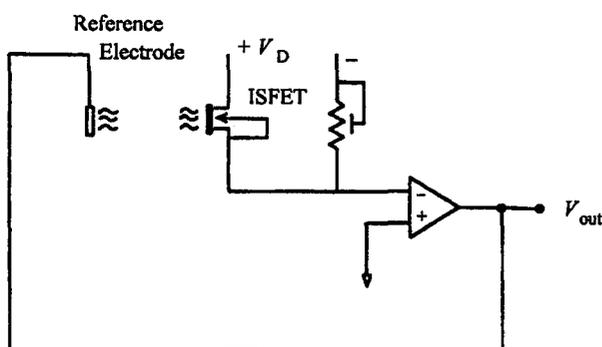


Figure 4.
Constant drain current (I_D) operating mode schematic circuit with negative feedback to the reference electrode.

The constant drain current method has disadvantages if the circuit is broken when the analyte solution is changed. This can cause the feedback potential to assume large positive values, and lead, when it is reestablished, to voltage transients which polarise the reference electrode, and/or the electroactive gate, causing spurious offset potentials and even encapsulation breakdown. To get round this, alternative circuits have been proposed [see 3,6], for example, one in which the drain current is inverted, undergoes current/voltage conversion and the resultant output is used to drive a further circuit incorporating a matching IGFET, so that the IGFET drain current mirrors that of the ISFET. This requires several operational amplifiers, voltage reference sources and a means of gain calibration and gain adjustment to compensate for slight ISFET/IGFET differences. Again, the sign of the resulting output signal depends on the number of amplifier inversions. In all cases, the measurement is, effectively, change in V_B with change in $\lg a_i$ which is equal to the modulus of the change in E_I with change in $\lg a_i$.

3. RELATION OF PERFORMANCE CHARACTERISTICS TO ION-SELECTIVE ELECTRODES

IUPAC Analytical Nomenclature Commission made recommendations for terms and symbols for ion-selective electrodes and a summary of these has appeared and is under revision[11]. For uniformity in the representation of ISE calibration curves, it is recommended that the potential difference (emf) of an ion-selective electrode assembly (ISE combined with an identified reference electrode) be plotted on the ordinate (vertical axis) with the more positive potentials at the top of the graph and that $-\lg$ [activity of species measured] or $-\lg$ [concentration of species measured] be plotted on the abscissa (horizontal axis) with increasing activity (concentration) to the right. Essentially this leads to a cation-responsive ISE having a calibration curve with a positive slope and an anion-responsive ISE a calibration curve with a negative slope. It is recommended that calibration curves for ISFETs should be in accordance with this convention for the slopes, so the way of plotting on the ordinate of experimental values of output potential difference is chosen appropriately, and that the modulus of the values of experimental output derived from ISFETs is regarded as equivalent to potential differences from ISEs as used in the Nernst or Eisenman-Nikolsky equations [11].

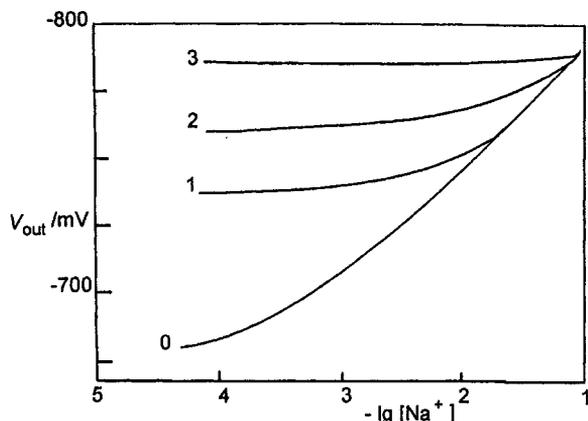


Figure 5. Calibration curve (0) and selectivity curves
 1: $10^{-3} \text{ mol dm}^{-3} \text{ K}^{+}$
 2: $10^{-2} \text{ mol dm}^{-3} \text{ K}^{+}$
 3: $10^{-1} \text{ mol dm}^{-3} \text{ K}^{+}$
 for potassium ion interference on a monensin-based sodium ion responsive ISFET.

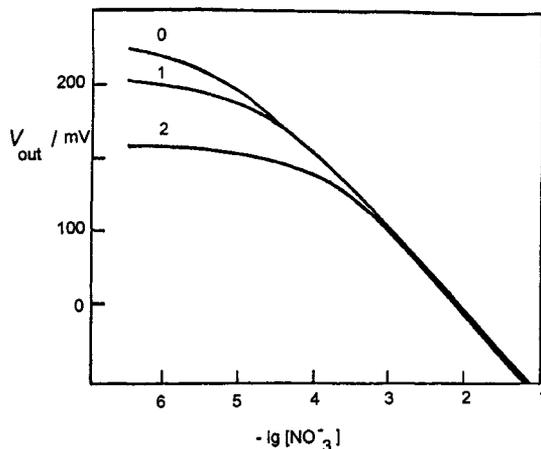


Figure 6. Calibration curve (0) and selectivity curves for the interference on a nitrate ISFET
 1: $10^{-3} \text{ mol dm}^{-3} \text{ chloride}$
 2: $10^{-3} \text{ mol dm}^{-3} \text{ nitrite}$

Other symbols and techniques relating to the performance of ISFETs in the determination of ions in solution should be in accordance with the recommendations for ISEs [11]. Recommended procedures for calibration of ISFETs are those recommended for ISEs [12]. For instance, the selectivity coefficient (k_{ij}) should be determined for preference by the mixed solution method. Attention is drawn to the continuous dilution method [12,13,14] for determining the calibration curve and selectivity coefficient. The use of this method for ISFETs is illustrated in Figure 5, where the results for a sodium-ion responsive ISFET, based on monensin, (obtained by the constant drain current method), and for a nitrate-ion responsive ISFET, based on tetradodecylammonium nitrate in Figure 6, obtained using the Optran, operational transducer [15], have been plotted in accordance with the above proposed convention for slopes.

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