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Effect of interferents present in the internal solution or in the conducting polymer transducer on the responses of ion-selective electrodes

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Abstract The effect of interferents present on the opposite side of the Pb^{2+} -selective membrane has been studied for both internal solution and all-solid-state sensors with a conducting polymer (CP) transducer. For interferents with moderate selectivity coefficients (sodium cations) present in the internal solution or in the CP transducer phase, super-Nernstian responses were obtained. For sensors containing strongly discriminated interferents (lithium ions), however, responses typical of conventional electrodes are observed, despite the low activity of primary ions on the opposite side of the membrane. This effect is attributed to hindered incorporation of interfering ions into the membrane, which also impairs the long term stability of the potential. Because of the relatively small absolute amounts of interferents in the transducer of all-solid-state sensors, their exchange for primary ions occurs quickly. Thus, transformation of the sensor to one with a micromolar detection limit and high potential stability is observed.

Keywords Ion-selective electrodes · All-solid-state potentiometric sensor · Selectivity · Stability

Introduction

In recent years significant progress has been achieved in the optimization of the analytical performance of ion-selective electrodes (ISEs) [1]. Both for internal-solution (IS-ISEs) [1–4] and all-solid-state conducting polymer-based sensors (ASS-CP-ISEs) [5–12] ion fluxes through ion-selective membrane (ISM) phases have been taken into

account. In good accordance with theoretical predictions [13, 14], experimental results [1–11] have shown that minimizing the sample-oriented flux of primary ions, originating on the opposite side of the ion-selective membrane leads to lower detection limits. Irrespective of the configuration of the sensor, however, primary ion transport through the ISM phase is always accompanied by a co-ion (interferent) flux in the opposite direction [1–14]. For this reason, high activity of interfering ions on the opposite side of the ion-selective membrane and constant, low, flow of primary ions, is beneficial, irrespective of whether it is an IS-ISE or an ASS-CP-ISE [1–11]. Although the general recipe is the same for both types of sensor, significantly different procedures are used to achieve improvement [3, 5–10]. For IS-ISEs an internal solution with optimized activity of both interferent and primary ions is simple to prepare. For ASS-CP-ISEs, however, appropriate conditioning is usually required to introduce the interferent, via an ISM, to the transducer phase [6, 7, 11].

The role of interfering ions in achieving balanced transport through the ion-selective membrane phase and improving analytical performance of ISEs is crucial, as theory [14] and experiment [15] show. A theoretical approach has considered different interferents of the same valence as primary ions (monovalent ions) and predicted that selectivity affects the dependence of the potential on the logarithm of ion activity (the magnitude of the super-Nernstian potential shift) [13, 14]. This affect has not yet been studied experimentally, however, either for internal solution or all-solid-state ISEs. The objective of this work was, therefore, to study experimentally the effect of interferents with different selectivity coefficients on the potentiometric characteristics and stability of recorded potentials. These results were compared for both kinds of sensor, IS-ISEs and ASS-CP-ISEs, using Pb^{2+} -selective electrodes as an example. Poly(3,4-ethylenedioxythiophene), known for its high stability [16], or poly(pyrrole), both doped with poly(4-styrenesulfonate) ions, characterized by cation binding properties [17, 18], were used as CP transducers.

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Experimental

Apparatus

A multi-channel data-acquisition setup and software from Lawson Laboratories (USA) were used in the potentiometric experiments. Systems 700 Dosino and 711 Liquino pumps (Metrohm, Switzerland) were used to achieve sequential dilution of calibration solutions. A CH-Instruments (USA) model 660A galvanostat–potentiostat and conventional three electrode cell, with platinum sheet as counter electrode and Ag/AgCl reference in 1 mol L⁻¹ KCl gel, were used for electropolymerization and other electrochemical experiments.

The double-junction Ag/AgCl reference electrode with 1 mol L⁻¹ lithium acetate in the outer sleeve (Möller Glasbläserei, Zürich, Switzerland) was used in potentiometric experiments. The recorded potential values were corrected for the liquid junction potential calculated in accordance with the Henderson approximation. The change of calculated liquid junction potential related to the range of changes of Pb(NO₃)₂ concentration did not exceed 10 mV.

Reagents

Tetrahydrofuran (THF), poly(vinyl chloride) (PVC), 2-nitrophenyl octyl ether (oNPOE), sodium tetrakis[3,5-bis(trifluoromethyl)phenyl]borate (NaTFPB), and lead ionophore IV (*tert*-butylcalix[4]arene-tetrakis(*N,N*-dimethylthioacetamide)) were from Fluka (Switzerland). Doubly distilled and deionised water (resistance 18.2 MΩ cm; Milli-Q plus, Millipore, Austria) was used. All salts used were of analytical grade from POCh (Poland) except for sodium poly(4-styrenesulfonate), NaPSS, and the monomers pyrrole (Aldrich, Germany) and 3,4-ethylenedioxythiophene (Bayer, Germany).

Preparation of internal solution electrodes

The Pb-selective membrane contained (*w/w*): 0.4% lead ionophore IV, 0.15% NaTFPB, 62.3% oNPOE, and 37.15% PVC. A total of 200 mg membrane components were dissolved in 2 mL THF and the mixture was poured into a glass ring of internal diameter 25 mm. After evaporation of the solvent, discs of diameter 7 mm were cut out and placed in Philips commercial electrode bodies. Membranes of thickness 150 μm were obtained.

A series of mixtures of 0.1 mol L⁻¹ citric acid with sodium hydroxide and sodium chloride (C-Na) or 0.1 mol L⁻¹ citric acid with lithium hydroxide and lithium chloride (C-Li), of constant pH=6.4 and total concentration of sodium or lithium ions 0.3 mol L⁻¹, spiked with Pb(NO₃)₂, yielding a calculated lead activity, *a* Pb²⁺ of 1.5×10⁻⁹ mol L⁻¹ (I) or 1.5×10⁻¹¹ mol L⁻¹ (II) (both in C-Na and C-Li series of solutions), were used as the internal filling solutions of Pb²⁺-ISEs. These solutions were

denoted C-Na-I, C-Na-II, C-Li-I, or C-Li-II. Unless otherwise stated these IS-ISEs were conditioned in 10⁻³ mol L⁻¹ Pb(NO₃)₂.

Preparation of all-solid-state electrodes

For ASS-CP-ISEs poly(3,4-ethylenedioxythiophene) films were obtained by galvanostatic electrodeposition, by passage of a current of 14 μA, on glassy carbon disc electrodes of surface area 0.07 cm², previously polished with Al₂O₃, 0.3 μm. Polymerization was from aqueous solution containing 6×10⁻³ mol L⁻¹ monomer and 0.1 mol L⁻¹ sodium poly(4-styrenesulfonate), to yield PEDOT-PSS. Polymerization charge was 25 mC, yielding a polymer film thickness ca 2.5 μm [19]. The films were rinsed with deionised water and conditioned overnight in either 0.1 mol L⁻¹ LiCl or 0.1 mol L⁻¹ NaCl solution. They were then partially reduced at -0.5 V for 3 min in the same electrolyte as used for conditioning.

Poly(pyrrole) films were obtained by potentiostatic polymerization at 0.9 V from aqueous solution containing 0.2 mol L⁻¹ monomer and 0.1 mol L⁻¹ sodium poly(4-styrenesulfonate), to yield PPy-PSS. Polymerization charge was 100 mC, yielding an approximate thickness of 6 μm [20]. The films were rinsed with deionised water, then polarized under conditions of cyclic voltammetry in 0.1 mol L⁻¹ NaCl (five cycles at 20 mV s⁻¹ within the potential range from -0.8 to 0.5 V). The films were then partially reduced at -0.5 V for 3 min in 0.1 mol L⁻¹ NaCl.

When cathodic polarization was complete, electrodes coated with either polymer were immediately removed from the solution, well rinsed, left to dry in the laboratory atmosphere, and placed in the upside-down position. The top of the electrode was covered with 25 μL THF and, immediately, 30 μL THF solution of the membrane components mixture was pipetted on the top of the conducting polymer resulting in a membrane ca. 100 μm thick. After evaporation of the membrane solvent, all-solid-state sensors were obtained.

Unless otherwise stated, these sensors were conditioned overnight (before first testing) and were stored in 10⁻³ mol L⁻¹ Pb(NO₃)₂ solution between tests.

Results and discussion

Internal solution electrodes

To emphasise the effect of interferent present in the internal filling solution of the lead-selective electrode, citrate complex buffers solutions were prepared with either Li⁺ or Na⁺ cations. Primary ions of two different activities were used for each interfering cation. The interfering ions chosen are characterized by significantly different affinity for the Pb²⁺-selective membrane (log *K*_{Pb,Na}^{pot}=-5.57±0.02, log *K*_{Pb,Li}^{pot}=-10.2±0.64 [3]). Figure 1 shows *E* vs. log *a* Pb²⁺ dependencies obtained for IS-ISEs. All lines recorded for Pb²⁺ activity corresponding to concentrations

greater than 10^{-5} mol L⁻¹ were characterized by Nernstian slopes (within experimental error). For lower Pb²⁺ activities in sample solution a pronounced effect of internal solution composition was observed.

The E vs. $\log a$ Pb²⁺ dependancies recorded for electrodes with C-Na-I or C-Na-II internal filling solutions were, as expected [2, 3], characterized by an abrupt potential decrease for concentrations (activities) below 10^{-6} mol L⁻¹, with the potential change being greater for lower Pb²⁺ activity in the internal solution. The occurrence of this super-Nernstian region is indicative of effective transport of Pb²⁺ ions from the sample (leading to depletion of the sample boundary layer) to the internal solution. This flux is compensated by outward flow of sodium ions [1–4].

The abrupt potential decrease corresponds to almost zero concentration of Pb²⁺ ions in solution close to the membrane surface, $c(0,t)=0$. The relationship between the sample bulk, c^0 , surface concentration of Pb²⁺ ions, $c(0,t)$, and Pb²⁺ flux to the membrane, J , is [11, 21, 22]:

$$c(0,t) = c^0 - 2J \left(\frac{t}{D\pi} \right)^{1/2} \quad (1)$$

Assuming then that $c(0,t)=0$ for $c^0=10^{-6}$ mol L⁻¹, corresponding to the abrupt potential change, taking $D=6 \times 10^{-6}$ cm² s⁻¹ for Pb²⁺ ions in solution [23] and a typical experiment time of $t=300$ s, the calculated Pb²⁺

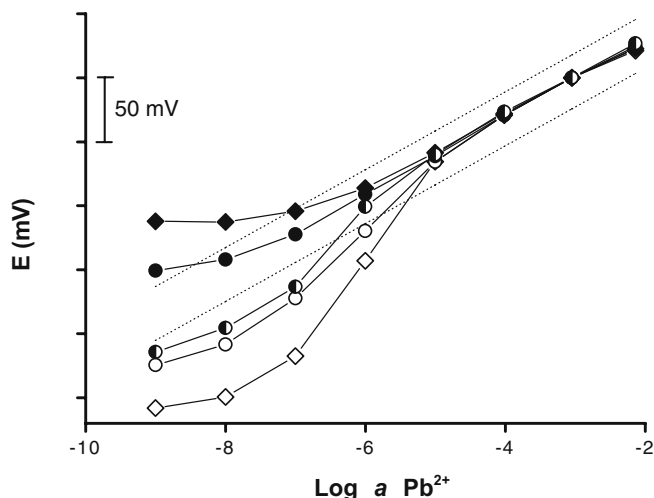


Fig. 1 Open circuit potentiometric responses of lead-selective IS-ISEs of different internal solutions: (open circles) C-Na-I (0.1 mol L⁻¹ citric acid with sodium hydroxide and sodium chloride [Na⁺]=0.3 mol L⁻¹, pH=6.4, calculated a Pb²⁺ activity 1.5×10^{-9} mol L⁻¹); (open diamonds) C-Na-II (0.1 mol L⁻¹ citric acid with sodium hydroxide and sodium chloride [Na⁺]=0.3 mol L⁻¹, pH=6.4, calculated a Pb²⁺ activity 1.5×10^{-11} mol L⁻¹); (filled diamonds) C-Li-I (0.1 mol L⁻¹ citric acid with lithium hydroxide and lithium chloride [Li⁺]=0.3 mol L⁻¹, pH=6.4, calculated a Pb²⁺ activity 1.5×10^{-9} mol L⁻¹); (filled circles) C-Li-II (0.1 mol L⁻¹ citric acid with lithium hydroxide and lithium chloride [Li⁺]=0.3 mol L⁻¹, pH=6.4, calculated a Pb²⁺ activity 1.5×10^{-11} mol L⁻¹); (half-filled circles) The same as C-Li-II but spiked with NaCl to yield [Na⁺]=0.01 mol L⁻¹. For easy comparison all curves were shifted to give equal potential at $\log a$ Pb²⁺=-3. The dotted lines represent $\pm 1n 2$ deviation from linear Nernstian responses

flux, J , is approximately 10^{-13} mol cm⁻² s⁻¹. This value is only approximate, because of the time-dependent surface concentration predicted by Eq. (1).

Significantly different results—absence of the super-Nernstian region—were obtained for electrodes containing Li⁺ ions in the internal solution phase. The responses, Fig. 1, were linear with the slope close to Nernstian within the activity range 10^{-2} to 10^{-6} mol L⁻¹, detection limit $10^{-6.9}$ mol L⁻¹, and the activity range 10^{-2} to 10^{-7} mol L⁻¹, detection limit $10^{-7.9}$ mol L⁻¹, for C-Li-I and C-Li-II, respectively. Despite the constant and low activity of Pb²⁺ ions in the internal solution and the Pb²⁺ complexing agent present, exchange of Li⁺ ions on the opposite side of the membrane and concomitant lithium cation transport through the ISM phase is significantly hindered. Thus, release of Pb²⁺ ions from the sensor (most probably the membrane phase) into the sample solution sets the observed detection limit as close to that of “traditional” ion-selective electrodes. This conclusion was supported by results from calibration performed for sample solutions of buffered (because of the presence of citrate complexes) Pb²⁺ activity (results not shown). In this instance a linear, Nernstian, dependence within the activity range 2×10^{-8} to 2×10^{-11} mol L⁻¹ was obtained.

Interestingly, when the internal solution was spiked with a relatively small amount of Na⁺ ions (Na⁺ to Li⁺ ratio equal to 0.03), the super-Nernstian behavior returned, Fig. 1. Thus, the effect of relatively small amounts of other ions in the internal solution is important in practical sensor construction. It seems justified to expect that even small changes of internal solution composition can, occasionally, significantly affect responses.

All-solid-state ion-selective electrodes

The effect of choice of interfering ions is also important when preparing all-solid-state ISEs with conducting polymer transducers. In this instance, however, introduction of Li⁺ ions during conditioning of the Pb²⁺ sensor would be virtually impossible. The method of ensuring a high loading of cations was based on experience in the field of CP—reduction of a cation-exchange polymer requires uptake of cations [24, 25]. PEDOT-PSS was chosen because of its low selectivity for monovalent cations [26]; both Li⁺ and Na⁺ ions can be easily introduced to the film.

Figure 2 shows responses obtained for ASS-CP-ISEs with PEDOT-PSS contact prepared as described above. For both types of electrode and for activities corresponding to the concentration range 10^{-2} to 10^{-5} mol L⁻¹, linear responses with slopes close to Nernstian (within experimental error) were obtained. For lower activities different response patterns were observed. The sensor with the transducer phase pretreated in Na⁺ solution was characterized by a super-Nernstian effect within the activity range 10^{-6} to 10^{-8} mol L⁻¹. This points to effective transport of Pb²⁺ from the solution to the transducer phase compensated by Na⁺ flux in the opposite direction,

similarly to electrodes containing sodium ions in the internal solution. In contrast, for the electrode with the transducer phase pretreated with Li^+ solution the super-Nernstian effect was not observed and a detection limit close to $10^{-7.5} \text{ mol L}^{-1}$ was obtained.

This experiment proves that the effect of interferences present on the opposite side of the ISM on responses of ASS-CP-ISEs and IS-ISEs is essentially the same, except that the super-Nernstian effect is less apparent and lasts less time for ASS-CP-ISEs. This difference results from the absolute amount of ions present on the opposite side of the ISM. The maximum amount of mobile cations in the CP layer, as in this experiment, is approximately 10^{-8} mol [11] whereas in the internal solution electrode it is approximately 10^{-4} mol . Assuming the same Pb^{2+} flux for internal solution and all-solid-state ISEs, calculated using Eq. (1) (approximately $10^{-13} \text{ mol cm}^{-2} \text{ s}^{-1}$), the amount of Pb^{2+} ions transferred to the sensor in course of overnight conditioning is approximately 10^{-9} mol . This amount is comparable with that present in the CP transducer, but much lower than that in the internal solution phase.

Stability of the open circuit potential

The choice of interferent ions present on the opposite side of the ISM phase also affects the stability of the internal solution sensors. The procedure used to test stability was to follow potential vs. time dependencies in solutions of primary ions, then in solution of interfering cations, and again in solution of primary ions. Theoretical considerations usually link the potential changes observed under these conditions with changes of composition of the thin electrolyte layer on the opposite side of the ISM of internal solution free sensors [6, 9, 10, 27, 28]. The objective of our

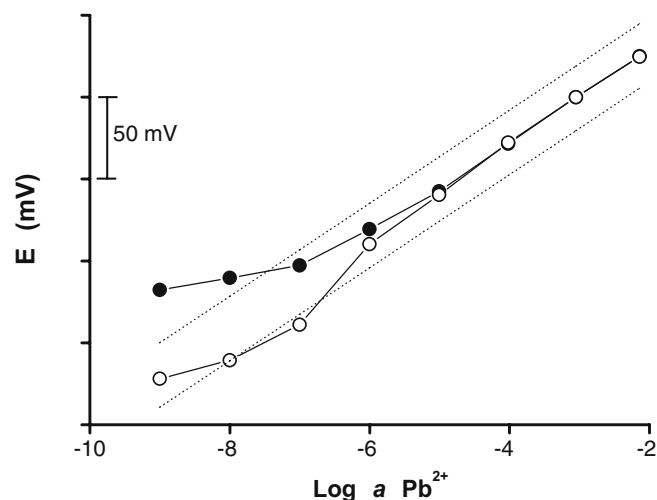


Fig. 2 Open circuit potentiometric responses of lead-selective, poly(3,4-ethylenedioxythiophene) ASS-CP-ISEs with differently prepared CP transducer phases: (open circles) sensor with CP layer pretreated in $0.1 \text{ mol L}^{-1} \text{ NaCl}$, (filled circles) sensor with CP layer pretreated in $0.1 \text{ mol L}^{-1} \text{ LiCl}$. For easy comparison all curves were shifted to give equal potential at $\log a \text{ Pb}^{2+} = -3$. The dotted lines represent ± 2 deviation from linear Nernstian responses

experiment was, however, to prove that similar changes can result from ion-exchange via ISM, irrespective of the type of the sensor, thus both IS-ISEs and ASS-CP-ISEs were studied.

Measurements were made for electrodes with a high ratio of interferent to primary ion content on the opposite side of the membrane. For IS-ISEs with C-Na-II internal solution a potential decrease was observed after placing in Pb^{2+} solution; this was attributed to progressing saturation of the ISM with primary ions (Fig. 3). This effect is consistent with the predictions of the Nernst equation. After immersion in NaCl (interferent) solution, slightly decreasing potentials were measured throughout 6 h (mean change -1.8 mV h^{-1}).

For IS-ISE containing C-Li-II internal solution a potential increase in the Pb(II) solution was observed. On transfer to NaCl solution, initially a fast potential decrease and then an increase of measured values was observed. On going back to the primary ions solution ($\text{Pb(NO}_3)_2$), the potential change of IS-ISE with C-Na-II was equal to -1 mV h^{-1} ; significantly faster changes were observed shortly after switching from NaCl to $\text{Pb(NO}_3)_2$ solution. For the C-Li-II sensor larger potential changes were recorded (-5 mV h^{-1}), indicative of lower stability of the recorded potentials.

Although similar analysis of stability can be performed for ASS-CP-ISEs, a full analogy with IS-ISEs cannot be drawn owing to rapid saturation of the CP transducer with primary ions, as reported in the previous section. The stability test was conducted for a sensor with poly(pyrrole) doped with PSS^- anions as a solid contact. In the course of conditioning or measurement in Pb^{2+} ions solution, deprotonation of the polymer, coupled with incorporation of primary ions (in this case Pb^{2+}) into the CP phase occurs [5, 11], $\log K_{\text{Pb,H}}^{\text{pot}} = -5.6$ [3].

After conditioning in $10^{-3} \text{ mol L}^{-1} \text{ Pb(NO}_3)_2$ solution, the stability test was performed, analogously as for IS-ISEs (Fig. 3). After placing in Pb^{2+} solution the recorded

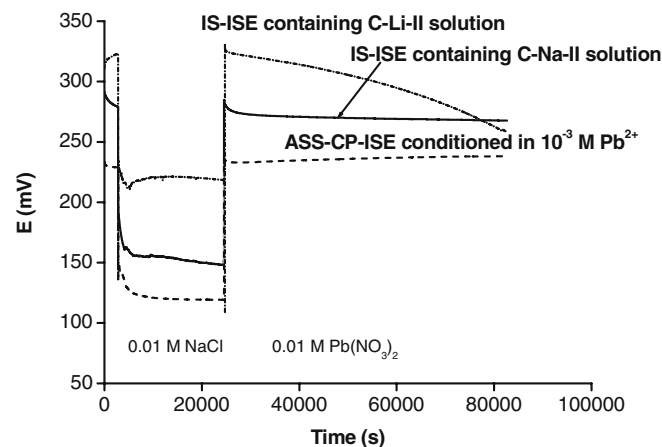


Fig. 3 Potential vs. time traces of lead-selective IS-ISEs, with internal solution containing C-Li-II or C-Na-II, and of poly(pyrrole)-based ASS-CP-ISEs containing sodium ions on the opposite side of the ISM, recorded in $0.01 \text{ mol L}^{-1} \text{ Pb(NO}_3)_2$, then in $0.01 \text{ mol L}^{-1} \text{ NaCl}$, and again in $0.01 \text{ mol L}^{-1} \text{ Pb(NO}_3)_2$

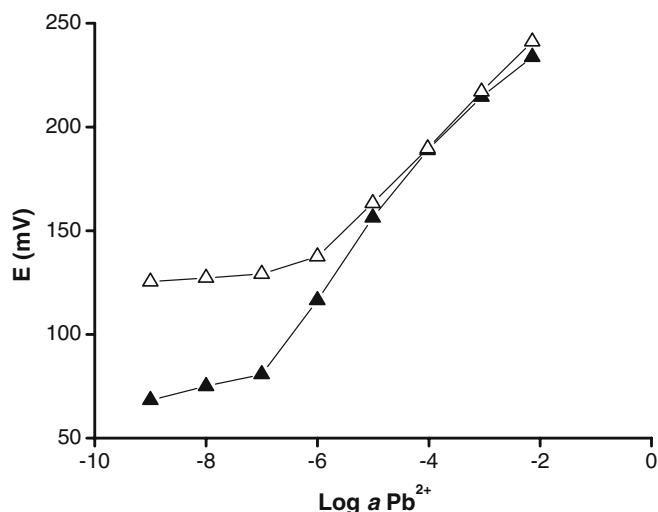


Fig. 4 Open circuit potentiometric responses of lead-selective poly(pyrrole)-based ASS-CP-ISEs conditioned in 10^{-3} mol L⁻¹ Pb(NO₃)₂ recorded before (*filled symbols*) and after (*open symbols*) potential stability test

potential remained stable, pointing to sufficient saturation of the PVC-based membrane. On transfer to NaCl solution a small potential decrease was observed and then the potential stabilized. After immersion again in the primary ions solution (Pb(NO₃)₂), the potential was stable, with an increase close to 0.3 mV h⁻¹. Thus, the electrode potentials are more stable than for IS-ISEs. The potential stability in this test is also indicative of the absence of a water layer between the CP and the membrane.

The stability of the potential for the ASS-CP-ISE is also attributed to saturation of the ISM phase with Pb²⁺ ions and accumulation of sufficient amount Pb²⁺ ions in the CP. Thus, reversible charge transfer at the ISM-CP interface is ensured [5, 6, 11]. The increase of the amount of Pb²⁺ in the sensor phases is confirmed by potentiometric characteristics recorded before and after the stability test (Fig. 4). The former calibration line is characterized by a super-Nernstian effect, which is absent after completion of the stability test. Originally, the super-Nernstian effect results from the CP oriented flux of Pb²⁺ ions, replacing H⁺ ions. After the stability test (long contact with solution of Pb²⁺ ions) the CP phase composition has been enriched in Pb²⁺ ions, thus the super-Nernstian effect disappears. A characteristic typical of a classical ISE with micromolar detection limit and high potential stability is obtained.

Conclusions

The choice of ions on the opposite side of the ion-selective membrane determines detection limits and stability for both internal solution and all-solid-state sensors. The presence of ions strongly discriminated within the sensor results in potential drifts and higher detection limit, despite the presence of primary ion binding agent. Taking into account potential stability, IS-ISEs loaded with interferents of moderate selectivity are recommended for long-term measurements in dilute solutions whereas ASS-CP-ISEs

are suitable for shorter contact times or, without time limits, at higher concentrations. The difference between the stability of IS-ISE vs. ASS-CP-ISE is attributed to the small amount of mobile interferent in the CP, which leads to a significant decrease of the interferent to Pb²⁺ ratio. This effect results in unstable characteristics in dilute solutions and ultimately to behaviour typical of classical ISEs with a micromolar detection limit.

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