ISSN 1070-3632, Russian Journal of General Chemistry, 2013, Vol. 83, No. 2, pp. 260–266. © Pleiades Publishing, Ltd., 2013.
Original Russian Text © A.R. Garifzyanov, R.R. Davletshin, N.V. Davletshina, R.A. Cherkasov, 2013, published in Zhurnal Obshchei Khimii, 2013, Vol. 83, No. 2, pp. 206–212.

Synthesis, Transport, and Ionophoric Properties of α,ω-Biphosphorylated Azapodands: VI.[1] New Cesium-Selective Electrodes Based on the Phosphorylated Azapodands

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Received April 24, 2012

Abstract—New cesium-selective electrodes containing as an electrode active substance biphosphorylated azapodands: 1,8-bis(dioctylphosphorylmethylamino)-3,6-dioxaoctane (**I**), 1,10-bis(dioctylphosphorylmethylamino)-4,7-dioxadecane (**II**), and 1-(dioctylphosphorylmethylamino)-10-[(dioctylphosphorylmethyl)benzylamino]-4,7-dioxadecane (**III**) were developed, and the composition of the membranes of these electrodes was optimized. The high selectivity of electrodes based on the ionophores **I–III** toward Cs(I) ion, which can be determined on the background of the majority of the other ions of alkali and alkaline earth metals, including the close to it by size Rb(I) ion, was established.

DOI: 10.1134/S1070363213020059

The use of organophosphorus ionophores in the ion-selective electrodes (ISE) started in the earliest stages of ion-selective potentiometry [2]. The most attention in this field of analytical chemistry attracted the development of the methods for the creation of calcium-selective electrodes, since the monitoring of Ca(II) *in vivo* is required in clinical medicine. The best results were achieved using calcium salts and esters of dialkylphosphoric acids.

The ISE based on the esters of phosphorus acids turned out to be highly effective at developing the methods for the analysis of alkali metal ions, uranyl [2], and silver [3]. Phosphorus acid amides were used in the ISE for the detection of Tb(III) ions in the rock and water samples [4].

A new stage in the use of organic derivatives of phosphorus in order to develop ISEs is associated with the development of chemistry of α,ω-biphosphorylated polyethers, or podands [5]. The synthetic accessibility of these ionophores, the ability to vary widely both their structure and hydrophilic–hydrophobic balance, the high rate of ion exchange inherent in the conformationally mobile acyclic ligands, the ability of flexible polydentate molecules of a podand to take a stable conformation at the complexation, which provides a significant selective binding of metal cation – all these factors determine the continuing interest in finding new ways to use the phosphorylated podands to create high-effective ISE. Formerly we have found ionophores effective and selective towards Ca(II), Ba(II), and uranyl ions among the podands with different structures of the polyether fragment and phosphorus-containing end groups [6]. Metal complexes of phosphorylated podands formed the basis for the creation of anion-selective electrodes, in particular, for the nitrate anion [7]. Electrodes containing as ionophore the complexes of copper with phosphorylazapodands show the electrode function toward the lipophilic anions. Using a mercury-complex iodide-selective electrode the potentiometric and argentometric determination of iodide ions in different objects was carried out [8].

Oligoetheral podands synthesized by us containing terminal aminomethylphosphine oxide groups, as we have found, can be used in the ISEs suitable for the selective determination of ions of alkali and alkaline earth metals [9]. The phosphorylazapodands with aliphatic groups at the phosphoryl moiety show high

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[1] For communication V, see [1].

selectivity toward Ba(II) cation and allows its determination in the presence of ammonium ions and most of alkali metal ions. Aromatic analog of the same podand shows the greatest sensitivity to a singly charged Rb(I) cation. We found that the doubly charged ions could be determined at the minimum concentration 10^{-5} M. The limit of detection of light metal ions is worse: 10^{-3}–10^{-4} M. Thus, the use of phosphorylazapodands in the ISE provides great opportunities to create effective and innovative analytical methods.

In this paper we present the results of studying the possibility of using in cesium selective electrodes the phosphorylazapodands of the general formula $Oct_2P\cdot(O)CH_2NHZNRCH_2P(O)Oct_2$ (**I–III**), where R = H, $Z = (CH_2CH_2O)_2CH_2CH_2$ (**I**); R = H, $Z = (CH_2)_3O(CH_2)_2\cdot O(CH_2)_3$ (**II**); R = CH_2Ph, and $Z = (CH_2)_3O(CH_2)_2O\cdot(CH_2)_3$ (**III**), varying the membrane phase composition through the use of the materials of different nature in the membrane and plasticizers.

To study the ionophore properties of the azapodand **I** with respect to the ions of alkali and alkaline earth elements, two ISEs were prepared, the first one containing in the membrane 5% of the electrode-active reagent **I** and sodium tetraphenylborate as a hydrophobic counterion to suppress the anionic function of the electrode. Dioctyl phthalate (60%) as the plasticizer and polyvinyl chloride (30%) were used. The internal electrode solution contained cesium nitrate at a concentration of 10^{-2} M. Another cesium selective electrode contained the same components in the ratio (%): 2.5:2.5:65:30. For each membrane the sensitivity to the Cs(I) ion was determined in a concentration range of 1×10^{-1}–1×10^{-6} M.

Both electrodes based on the ionophore **I** were tested for reproducibility and stability of response with respect to the Cs(I) ion. The first of these membranes had relatively short life time, one week, so it obviously has no prospects for the practical use. Second membrane was a more stable, and its ionophore properties were investigated in more detail.

While measuring the electrode potential in the $CsNO_3$ concentration range 10^{-1}–10^{-5} M we found that the potential drift was small, and reached a constant value in 60 s. The Nernst plot in the E–$\log c_{Cs+}$ coordinates is linear, and the electrode function Eq. (1) calculated by least squares indicates that, although its slope is less than the theoretical value for singly charged ions, however, the processes at the membrane–aqueous solution interface are reversible.

$$E = 110.4 + 49.9\log c_{Cs+}, \quad (1)$$

$$R\ 0.999,\ N\ 5,\ S\ 1.6,\ t_s\ 3.19.$$

The most important characteristic of any electrode is its selectivity that determines a possibility of its use for measuring a concentration of an individual ion in a real sample. Table 1 shows the selectivity coefficients ($-\log K_{Mz+/Cs+}$) for several alkali and alkaline earth metals calculated with the use of ion-selective electrodes based on compound **I**, according to the method of separate solutions for a series of ions of these metals, by Eq. (2)

$$K_{ij}^{pot} = (E_2 - E_1)/(2.303RT/FZ_i) + [1 - (Z_i/Z_j)]\log a_1, \quad (2)$$

where E_1 and E_2 are the potentials of investigated (i) and interfering (j) ions, respectively. The found selectivity coefficients (Table 1) were close to each other in the range 3.08–3.35, and for the Rb(I), which is isoelectronic with the cesium ion, this value is close to the upper limit (3.27), and the values obtained show that the electrode based on phosphorylazapodand **I** has a high selectivity to cesium ions.

Since the Rb(I) is closest by properties to the Cs(I) cation, we carried out determination of its permissible concentrations in the mixed solutions of these two ions. Figure 1 shows that at a constant concentration of Cs(I) 1×10^{-5} M. the presence of Rb(I) ions has no significant effect on the potentiometric response of the electrode and the membrane can be used in full working range of concentrations of rubidium ion from 1×10^{-1} to 5×10^{-4} M. When exceeding this limit, the membrane can be used in narrower concentration range 10^{-1}–10^{-4} M and 1×10^{-1}–5×10^{-3} M, in the presence of Rb(I) at a concentration of 10^{-4} and 10^{-3} M and 1×10^{-2} M, respectively. The dependence of the electrode potential on the medium acidity was obtained for 0.001 M solution of cesium nitrate in the pH range 1.0–10.0. The electrode potential remains constant in the pH range 7.0–10.0, which can be taken as the working pH range of this electrode. At pH < 6 the potenial grows, which may be due to the competing action of protons at recording the electrode potential.

By measuring the electrode potential in the two solutions with the concentration of Cs(I) 1×10^{-3} and 1×10^{-2} M, we found that the potentiometric response of the electrode is reversible (Fig. 2.) The detection limit of the cesium selective electrode, as determined from the intersection of the two extrapolated segments of the calibration graph, was 1.03×10^{-5} M. To establish the dynamics of the response time we recorded the

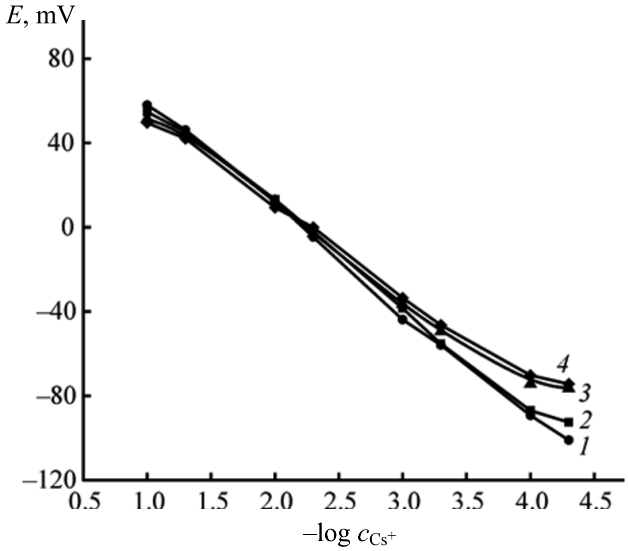

Fig. 1. Effect of the Rb(I) concentration on the electrode function of cesium selective electrode based on ionophore **I**. (*1*) 1.0×10^{-5}, (*2*) 1.0×10^{-4}, (*3*) 1.0×10^{-3}, and (*4*) 1.0×10^{-2} M.

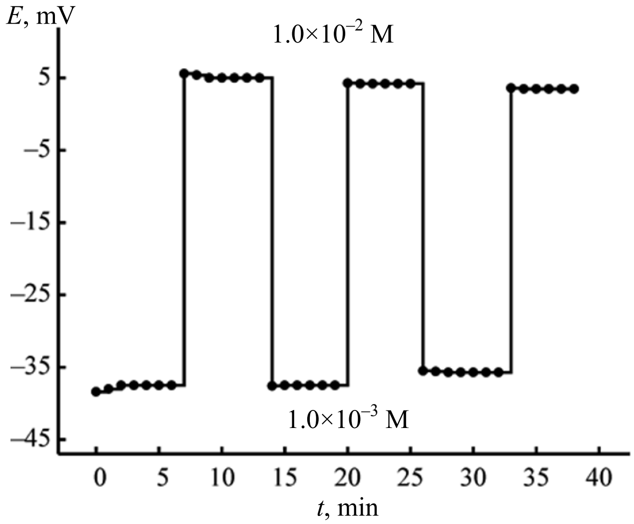

Fig. 2. Dynamics of the potentiometric response of cesium selective electrode based on ionophore **I**.

response time for sequential changes in the concentrations of cesium ions in solution in the range 10^{-1}–10^{-5} M, and found that the constant value of the potential is reached in an average of 60 s.

Thus, the electrode based on compound **I** is reversible and sensitive to the Cs(I) ions. These ions can be determined selectively in the presence of alkali and alkaline earth metals, as well as the Rb(I) ions in the concentration range of 10^{-5}–10^{-2} M, which is of particular interest. Potentiometric response of the cesium selective electrode with the ionophore **I** is stable in the pH range 7.0–10.0, the limit of detection of the Cs(I) ions is 1.03×10^{-5} M, the response time is 60 s.

As we have shown previously [6, 9], the characteristics of ion-selective electrodes noticeably depend on the compliance of the potential pseudo-cavity of the phosphorylazapodand ionophore and the metal ion to be determined. We decided therefore to trace the influence of the size of polyether bridge between the terminal aminomethylphosphoryl groups on the characteristics of cesium selective electrode. For this purpose we selected 1,10-bis(dioctylphosphorylmethylamino)-4,7-dioxadecane, the azapodand **II** with two 1,3-propylene (but not 1,2-ethylene, as in the ionophore **I**) bridges between the amine nitrogen and ether oxygen atoms.

Table 1. Selectivity coefficients (–log $K_{Mz+/Cs+}$) of cesium selective electrode on the basis of phosphorylated azapodands **I**–**III**

Ion[a]	I	II	III
Rb^+	3.27	2.98	2.47
K^+	3.31	3.28	3.17
Na^+	3.35	3.40	3.23
Li^+	3.34	3.36	3.24
Ba^{2+}	3.08	3.27	3.19
Sr^{2+}	3.28	3.34	3.21
Ca^{2+}	3.15	3.33	3.23
Mg^{2+}	3.29	3.38	3.18

[a] Concentration of the ion 0.01 M.

To study the ionophore properties of the azapodand **II** with respect to the ions of alkali and alkaline earth elements six electrodes were produced with different composition of the membranes, which include electrode-active reagent **II**, sodium tetraphenylborate, polyvinyl chloride and a plasticizer, dioctyl phthalate or *p*-nitrophenyl octyl ether. The characteristics of the obtained ISE are shown in Table 2. In all cases, the electrode internal solution contained cesium nitrate at a concentration of 0.01 M.

Nernst dependence in the E–log c_{Cs^+} coordinates obtained for the ISE no. 1 is linear in the concentration range of potential-determining ions from 10^{-1} to 5×10^{-4} M. The slope found from the equation of the electrode function (3) was 35.4 mV, lower than the theoretical value calculated by the Nernst equation for singly charged cations.

$$E = 64.4 + 35.4\log c_{Cs^+}, \quad (3)$$
$$R\ 0.997,\ N\ 6,\ S\ 2.4,\ t_s\ 2.79.$$

Table 2. The composition of plasticized membranes based on phosphorylated azapodand **II**, sodium tetraphenylborate (Ph_4BNa), dioctyl phthalate or *p*-nitrophenyl octyl ether, and polyvinyl chloride (PVC)

Run no.	Membrane composition, %	Range of operating concentrations, M	Slope, mV	Response time, s
1	**II**:Ph_4BNa:dioctyl phthalate:PVC, 20:20:30:30	10^{-5}–10^{-1}	35.4±0.1	60
2	**II**:Ph_4BNa:*p*-$NO_2C_6H_4OOct$:PVC, 20:20:30:30	10^{-4}–10^{-1}	29.3±0.3	120
3	**II**:Ph_4BNa:*p*-$NO_2C_6H_4OOct$:PVC, 2.5:2.5:65:30	10^{-4}–10^{-1}	47.9±0.1	60
4	**I**:Ph_4BNa:*p*-$NO_2C_6H_4OOct$:PVC, 2.5: 5:63.5:30	10^{-4}–10^{-1}	46.5±0.1	140
5	**I**:Ph_4BNa:*p*-$NO_2C_6H_4OOct$:PVC, 5:5:60:30	10^{-5}–10^{-1}	44.0±0.2	120
6	**I**:Ph_4BNa:*p*-$NO_2C_6H_4OOct$:PVC, 5:7.5:57.5:30	5×10^{-5}–10^{-1}	45.2±0.3	60

For ISE no. 2 this option was even lower: 29.3 mV. So we decided to analyze the effect of membranes on the electrode response in the concentration range of 10^{-1}–10^{-6} M. For this purpose, in the composition of membranes nos. 3–6, similar to those of the membrane no. 2, was changed the ratio of ionophore **II**, sodium tetraphenylborate and *p*-nitrophenyl ester, the content of PVC in all cases remained 30%.

We found that the best characteristics has the ISE no. 3 with the membrane composition containing the above components in the ratio (%) 2.5:2.5:65:30. The equation of the electrode function (4) for ISE shows linear dependence in the Nernst potential-determining ion concentration range of 10^{-1}–10^{-4} M.

$$E = 118.4 + 47.9\log c_{\mathrm{Cs}^+}, \quad (4)$$
$$R\ 0.998,\ N\ 7,\ S\ 3.3,\ t_s\ 2.57.$$

In this case the potential drift is small, the value becomes constant in 60 s.

The ISE selectivity coefficients based on ionophore **II** determined by the procedure described above are close to each other for most of the metal ions of groups I and II and are in the range 3.28–3.40 (Table 1). The exception is Rb(I) ion: in this case, the value of log $K_{\mathrm{Rb(I)/Cs(I)}}$ 2.98 is much lower. It can be noted that the electrode based on phosphorylazapodand **II** has a fairly high selectivity for cesium ions, but it is clear that the close in ionic radius Rb(I) ion can have the greatest interfering effect at high concentrations. To determine the allowed values of concentrations at which this ion can exert a strong influence on the response of the cesium selective electrode we carried out a joint determination of these ions. As seen in Fig. 3, at concentrations of 10^{-4}–10^{-5} M the Rb(I) ion does not interfere in the determination of Cs (I). If this limit is exceeded, the ISE can be used to analyze the ions Cs(I) in the concentration range of 10^{-1}–10^{-3} M. at the content of Rb(I) ions in the solution in the range 1×10^{-2}–1×10^{-3} M.

As to the effect of pH of solution containing 0.001 M of $CsNO_3$ on the electrode response, the potential of the electrode no. 3 in the range of pH 1.0–7.0, as we established, changed relatively little. For this electrode we examined the reversibility of the ISE and the dynamics of the potentiometric response. The first of these characteristics we identified by sequential measurement of the potential in solutions of two concentrations of $CsNO_3$: 1×10^{-3} and 1×10^{-2} M, and we found that under these conditions the potentiometric response of the electrode was reversible, the detection limit of the cesium selective electrode with ionophore **II** was equal to 1.06×10^{-4} M. In order to establish the dynamics of the response, we determined the time of its sequential change at the Cs(I) concentrations in the range 10^{-1}–10^{-4} M. It turns out that the constant value of the potential is reached in 60 s on the average.

Thus, we have found the optimum composition of the membrane composition of the ion selective electrode based based on 1,10-bis(dioctylphosphorylmethylamino)-4,7-dioxadecane **II**, which is sensitive toward the Cs(I) ions and reversible, with good selectivity for cesium to measure it against the background of most of the ions of alkali and alkaline earth metals, in particular, in the presence of Rb(I) in the concentration range 10^{-2}–10^{-5} M. We found the pH range in which potentiometric response of the cesium selective electrode is optimal. The detection limit of the electrode is 1.06×10^{-4} M, the response time is 60 s.

Along with the structural factors of substrate and the ionophore, as well as compositional characteristics of the ISE membrane, an important role in the selection of a pair of metal–ligand obviously belongs to the electronic characteristics of potential coordination sites of the ionophore that contribute to its coordination ability. With regard to the ionophores discussed in this paper both the electron-donating

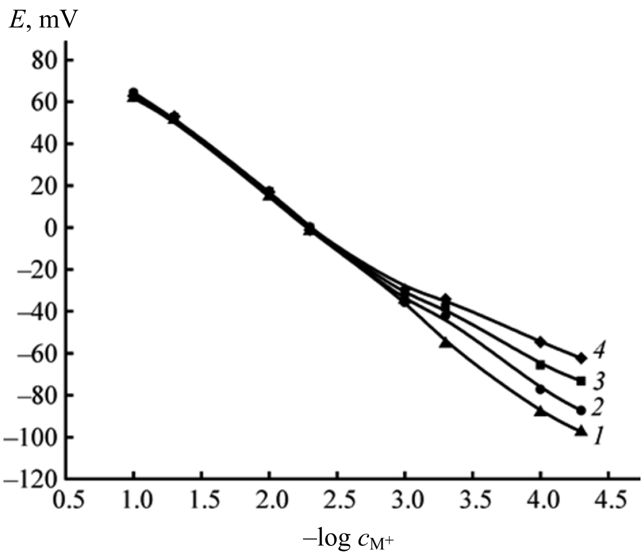

Fig. 3. The influence of the rubidium concentration on the electrode function of cesium selective electrode based on ionophore **II**. (*1*) 1.0×10^{-5}, (*2*) 1.0×10^{-4}, (*3*) 1.0×10^{-3}, and (*4*) 1.0×10^{-2} M.

ability of the terminal oxygen atoms of phosphoryl fragments and the basicity (nucleophilicity, hardness–softness) of the nitrogen atoms should be considered. We tried to evaluate in some approximation the role of the last of these factors, using as ionophore in the cesium selective electrode the structural analog of podand **II**, *N*-benzyl derivative **III**, in which the acceptor benzyl group, obviously, reduces the electron density on one of the terminal nitrogen atoms.

To study the ionophore properties of this reagent, we prepared two electrodes with different content of the electrode-active reagent **III**, and examined the response in the concentration range of $CsNO_3$ solution 10^{-1}–10^{-6} M with each membrane.

At the percentage ratio ionophore **III**, sodium tetraphenylborate, *p*-nitrophenyl octyl ether, and polyvinyl chloride 5:5:60:30 the slope was satisfactory (43.8), but the response time was quite large (120 s). The best results were achieved at a ratio of components (%) 2.5:2.5:65:30: at this composition the ISE membrane is characterized by the slope of 45.9 and its electronic response is described by Eq. (5). Function of the cesium selective electrode is shown in Fig. 4.

$$E = 95.937 + 45.9\log c_{Cs^+}, \quad (5)$$
$$R\ 0.999,\ N\ 8,\ S\ 2.8,\ t_s = 2.57.$$

The response time was 75 s. Although the slope is below the theoretical one, however, it indicates the reversibility of the processes occurring at membrane–aqueous solution interface.

We calculated the coefficients of the selectivity of the electrode on the basis of cesium selective azapodand **III** relative to the cations of alkali and alkaline earth metals. These values, as in previous cases, were close to each other (Table 1). From the relatively narrow range of selectivity factors Cs(I) with respect to the metals of groups I and II, 3.17–3.24, also falls out the Rb(I) ion: selectivity for it is low (2.47).

It may be noted that this ISE also has a fairly high selectivity to Cs(I) ion, but as for the ISE with the ionophore **II**, the Rb(I) ion may have a hindering effect on the potentiometric response of the electrode at high concentrations. To determine the allowed values of concentrations at which this ion may have a significant impact, we also performed joint determination of these ions in a solution. As follows from the dependences in Figure 5, ion Rb(I) in solution at concentrations of 10^{-4}–10^{-5} M did not affect significantly the potential of the cesium selective electrode, but compared with the previous case there is a slight narrowing of the working range of concentrations to 5×10^{4} M. Above this limit the membrane can be used to determine the cesium ion in the concentration range of 10^{-1}–10^{-3} M at the content in solution of Rb(I) ions 10^{-2}–10^{-3} M. The detection limit of the cesium selective electrode with ionophore **III** is 1.04×10^{-5} M.

Study of the pH effect in the range of 1.0–12.0 on the electrode response of this electrode in 0.001 M solution of cesium nitrate showed the smallest change in the electrode potential (from –58.2 to –76.2 mV). Noticeable fluctuations in the acid medium at pH 1–6 are probably due to the competing action of protons on the electrode response. The response time of ISEs with ionophore **III** was registered by us at a sequental change in the Cs(I) ions concentration in the range of 10^{-1}–10^{-5} M, a constant value of potential was reached within 75 s on the average.

All the produced plasticized electrodes showed no appreciable long term change of potential (over months). Regular testing evidenced the stability of the potentiometric response.

Thus, the created new cesium selective electrodes that include in the membrane the phosphorylazapodands differing by the length of the polyether bridge and terminal lipophilic aminomethylphosphoryl groups are similar by the properties and exhibit a fairly high selectivity to the ion Cs(I), allowing its determination

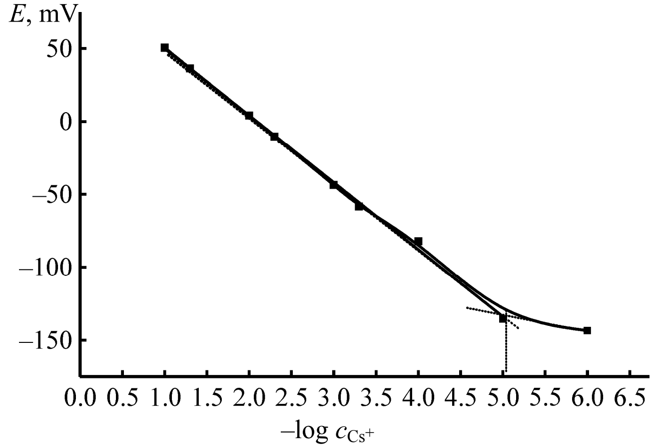

Fig. 4. The electrode function of cesium selective electrode based on ionophore **III**.

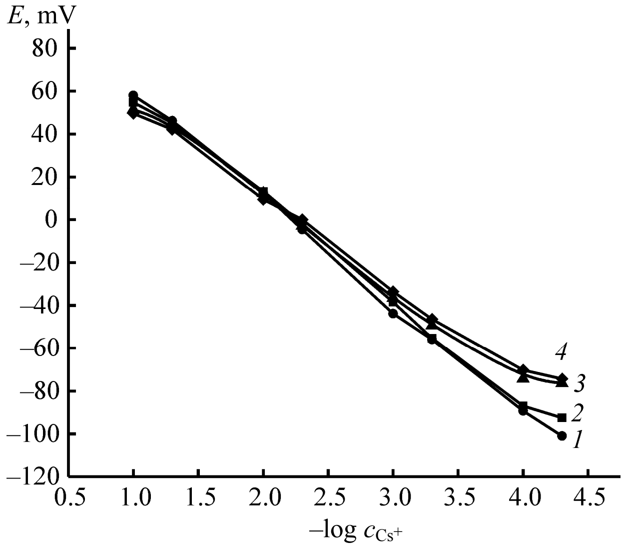

Fig. 5. The influence of the concentration of rubidium on the electrode function of cesium selective electrode based on ionophore **III**. (*1*) 1.0×10^{-5}, (*2*) 1.0×10^{-4}, (*3*) 1.0×10^{-3}, and (*4*) 1.0×10^{-2} M.

in the presence of a large number of ions of alkali and alkaline earth metals, in particular, the Rb(I) ion with the close atomic radius. Showing a reversible response to cesium ions, the new plastic ISE have good performance: stability of the electrode potential for a long time, satisfactory reproducibility of the analytical results, a very high selectivity toward the target substrate. Given these characteristics of the ISE, as well as synthetic accessibility of the used phosphorylazopodand ionophores, quite optimistic prospects for their practical use are expectable.

EXPERIMENTAL

Methods of synthesis of the studied phosphorylazapodands **I–III** were described in [1, 9, 10].

The solutions of the studied ions were prepared from the corresponding salts of pure, chemically pure, and analytical grade. If necessary the recrystallization of a salt from saturated aqueous solutions was carried out. We used the dioctyl phthalate of analytical grade, nitrophenyl octyl ether was prepared by the method [11].

Potentiometric measurements were performed in plastic cell filled with solutions of the investigated salts where the silver chloride electrode EVL-IM1 and the examined electrode were immersed. The latter was a tip of polyethylene tube, the lower end of which contained plasticized membrane with a diameter of 1–2 mm. The cell was filled with an aqueous solution of the ion to be determined with a constant concentration. Before the first use, the electrode was kept in a solution of studied salt for 24 h or, if necessary, for 48 h. The potential difference between the ISE and the reference electrode was measured using a digital pH-meter–millivoltmeter I-130.

Membranes solutions were prepared by mixing a the necessary amounts of electrod-active substance and plasticizer and together with 0.30 g of a solution of polyvinyl chloride in 5 ml of THF it was poured onto polished glass plate on which was placed a circular cylinder of 30–35 mm diameter and 30 mm ofheight to prevent spreading of the solution. The mixture was kept at room temperature till complete evaporation of the solvent, the thickness of the membranes obtained ranged from 0.1 to 0.8 mm. A disk with a diameter 5 mm was excised from the membrane, glued to the end of polyvinyl chloride tube with a solution of polyvinyl chloride in tetrahydrofuran to give, thus, the body of the electrode. The electrode was filled with internal solution and supplied with an internal reference electrode. Potentiometric measurements with membrane ion-selective electrodes were carried out in the system with transfer:

$$\text{Ag}\,\big|\,\text{AgCl, KCl}\,\brokenvert\,1\times10^{-2}\text{–}1\cdot10^{-5}\ \text{M}\,\big|\,\text{membrane}\,\big|\,1\times10^{-2}\ \text{M}\,\brokenvert\,\text{AgCl}\,\big|\,\text{Ag}$$

The internal electrode solution contained the standard solution of the investigated salts.

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research (grant 10-03-00580).

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