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Lu^{3+} poly(vinyl chloride) Membrane Electrode Based on 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene as Sensing Material

Azar Dehnavi[1], *Hassan Ali Zamani*[1,*], *Mina Shariyati*[1], *Mohammad Reza Abedi*[2], *Soraia Meghdadi*[3]

[1]Department of Applied Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran
[2]Department of Applied Chemistry, Quchan Branch, Islamic Azad University, Quchan, Iran
[3]Department of Chemistry, Isfahan University of Technology, Isfahan 84156-83111, Iran
[*]E-mail: haszamani@yahoo.com

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A Lu^{3+}-PVC membrane electrode based on 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene (HND) as a sensing ionophore has been constructed for the determination of lutetium(III) in aqueous solutions. The electrode performance exhibits a Nernstian slope of 19.6±0.2 mV decade^{-1} in the concentration range of 1.0×10^{-6} and 1.0×10^{-2} mol L^{-1} with a detection limit of 8.0×10^{-7} mol L^{-1} in the pH range of 2.3 –7.4. The response time for the proposed electrode was ~7.5 s, and its selectivity with respect to common alkali, alkaline earth, transition and heavy metal ions, including members of the lanthanide family other than Lu^{3+} was good. The proposed sensor was effectively used as an indicator electrode in the potentiometric titration of Lu^{3+} ions with EDTA. The proposed electrode sensor accuracy was studied by the monitoring of Lu^{3+} in various mixtures of different ions.

Keywords: Potentiometry, Sensor, Ion-selective electrode, PVC membrane, 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene

1. INTRODUCTION

Because the lanthanides illustrate similar chemical and physical properties, the lanthanides analysis is an extremely time consuming and complicated procedure, when several of their members are present, since separation as well as pre-concentration are required. Among lanthanides, Lutetium can seldom be found in nature. Lutetium is a rare earth metal and perhaps the most expensive of all rare elements. It is found in small amounts with all rare earth metals, and is very difficult to separate from other rare elements. Lutetium is very expensive to obtain on useful quantities and therefore it has very few commercial uses. Its use is still growing, as it is suitable for catalyzer production and glass polish. In addition, lutetium is dumped in the environment in many different places, mainly from petrol-producing industries [1-3].

There are some instrumental methods for lutetium analysis by mass spectrometry (MS), X-ray fluorescence spectrometry, inductively couple plasma mass spectrometry (ICP-MS), Isotope dilution mass spectrometry, inductively couple plasma atomic emission spectrometry (ICP-AES), etc. These methods are either time consuming, involving multiple sample manipulations, or too expensive for most analytical laboratories, as compared to the application of ion selective electrodes (ISEs). ISEs are among the most popular electrochemical devices that usually show fast and selective responses in addition to their low cost and ease of preparation and use. Literature survey revealed that only a number of lutetium(III) sensors based on different ionophores have been reported [4-10]. Lately, several studies concerning selective and sensitive PVC-membrane ion-selective electrodes were reported for some other ions [11-44]. In this study, we introduce a selective and sensitive lutetium(III) PVC membrane electrode based on 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene (HND) (Fig. 1) for potentiometric determination of a wide concentration range of Lu^{3+} ion .

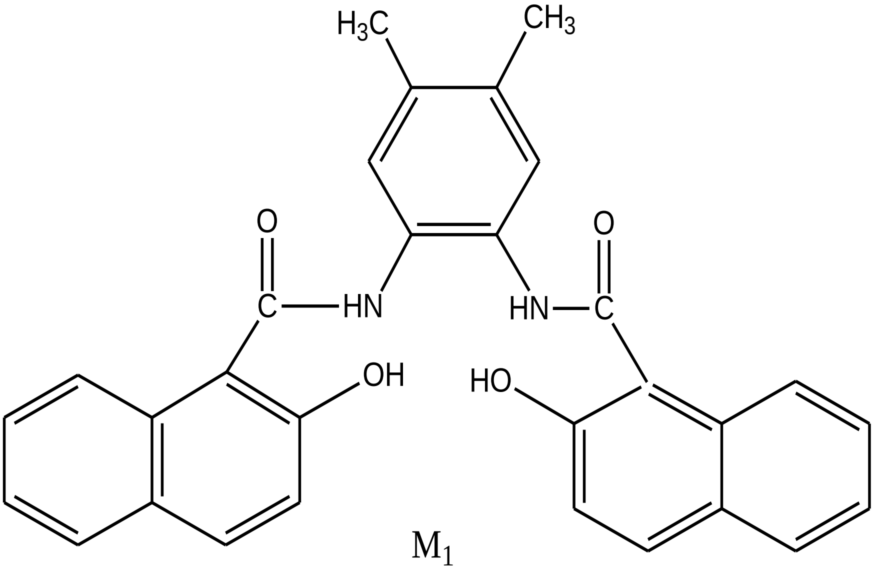

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Figure 1. Structure of the ligand HND.

2. EXPERIMENTAL

2.1. EMF measurements

The electro motive force (emf) measurements of polymeric membrane were carried out with the following cell assembly:

Ag–AgCl| internal solution 1.0×10^{-3} mol L^{-1} $LuCl_3$ | PVC membrane: sample| $Hg–Hg_2Cl_2$, KCl (satd).

A Corning ion analyser 250 pH/mV meter was used for the potential measurements at 25.0 °C. Activities were calculated according to the Debye–Huckel procedure [45].

2.2. Chemicals and reagents

The Merck Chemical and the Aldrich Co. were the suppliers for the nitrate and chloride salts of all cations and the reagent grades of dibutyl phthalate (DBP), benzyl acetate (BA), acetophenon (AP), nitrobenzene (NB), sodium tetraphenyl borate (NaTPB), tetrahydrofuran (THF) and high relative molecular weight PVC. The ligand 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene was synthesized and purified as described elsewhere [46]. The nitrate and chloride salts of all cations used (all from Merck) were of the highest purity available and used without any further purification except for vacuum drying. Doubly distilled and deionized water was used throughout.

2.3. PVC membrane electrode preparation

Membrane solutions were prepared by thoroughly dissolving 5 mg of HND, 30 mg of powdered PVC, 63 mg of NB and 2.0 mg of NaTPB in 3 mL of fresh THF. The resulting clear mixture was evaporated slowly until an oily concentrated mixture was obtained. A Pyrex tube (3-5 mm o.d. on top) was dipped into the mixture for about 5 s, so that a transparent membrane of about 0.3 mm thickness was formed [47-50]. The tube was pulled out from the mixture and kept at room temperature for 12 h. The tube was then filled with an internal solution (1.0×10^{-3} mol L^{-1} $LuCl_3$). The electrode was finally conditioned for 24 h by soaking in a 1.0×10^{-3} mol L^{-1} Lu^{3+} ion solution. A silver/silver chloride electrode was used as an internal reference electrode.

3. RESULTS AND DISCISSION

In order to evaluate the suitability of HND as an ionophore for different cations, the HND was employed as a potentially suitable neutral carrier in the construction of a number PVC membrane ion-selective sensors under identical conditions for a large number of metal ions, including alkali, alkaline earth, transition and heavy metal ions. The results revealed that except Lu^{3+} ion with the most sensitive response, in all other cases the slopes of the corresponding emf versus pM^{n+} are much lower than the expected slopes (59, 29, and 19 mV per decade for mono, di and trivalent cations, respectively).

3.1. Membrane composition effect

Since the sensitivity and selectivity for a given ionophore depend significantly on the membrane ingredients, such as the properties of the plasticizer, the plasticizer/PVC ratio, the nature and the amount of the ionophore and, especially, the nature and the amount of the used additives, the membrane composition influences were investigated on the potential responses of the Lu(III) sensor [51-55]. It should be noted that the presence of lipophilic anions in the cation-selective membrane electrodes not only diminishes the ohmic resistance, but it also enhances their response behavior and selectivity [55, 56]. The results are summarized in Table 1. Obviously, among the four solvent mediators used, NB with higher polarity than DBP, AP and BA, was used. Consequently, the use of

NB increases Lu(III) ions extraction with high charge density from the solution to the membrane. This phenomenon can be due to the dielectric constant of the plasticizer on membrane phase. As can be seen from Table 1, the slope of the Lu(III) sensor is affected by amount of ionophore HND in the membrane composition (membrane nos. 7–11). Increasing of HND in the membrane up to a value of 5%, in the presence of 2% NaTPB and 63% of polar solvent (NB), results in the best sensitivity (membrane no. 11).

Table 1. Optimization of the membrane ingredients.

Membrane no.	Composition(%)							Slope (mVdecade^{-1})	Liner range (mol L^{-1})
	PVC	NB	AP	BA	DBP	HND	NaTPB		
1	30	66	-	-	-	2	2	17.2±0.4	1.0×10^{-6}-1.0×10^{-2}
2	30	-	66	-	-	2	2	24.8±0.3	1.0×10^{-6}-1.0×10^{-2}
3	30	-	-	66	-	2	2	16.0±0.4	1.0×10^{-6}-1.0×10^{-2}
4	30	-	-	-	66	2	2	26.0±0.2	1.0×10^{-6}-1.0×10^{-2}
5	30	-	-	-	65	3	2	27.4±0.4	1.0×10^{-6}-1.0×10^{-2}
6	30	67	-	-	-	2	1	26.7±0.4	1.0×10^{-6}-1.0×10^{-2}
7	30	65	-	-	-	2	3	15.3±0.1	1.0×10^{-6}-1.0×10^{-2}
8	30	67	-	-	-	1	2	16.1±0.1	1.0×10^{-6}-1.0×10^{-2}
9	30	65	-	-	-	3	2	17.9±0.1	1.0×10^{-6}-1.0×10^{-2}
10	30	64	-	-	-	4	2	18.5±0.2	1.0×10^{-6}-1.0×10^{-2}
11	30	63	-	-	-	5	2	19.6±0.2	1.0×10^{-6}-1.0×10^{-2}

3.2. Slope and detection limit

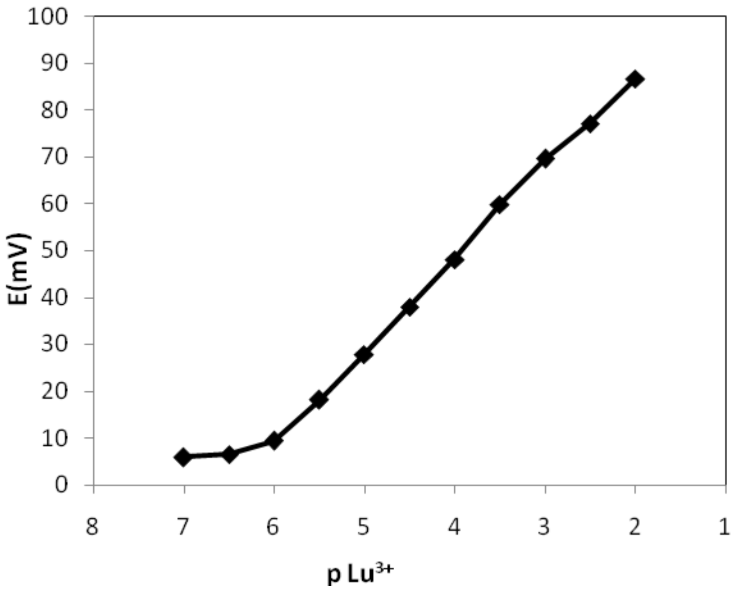

Figure 2. Calibration curves of the HND-based Lu^{3+} sensor.

The emf *vs*. pLu^{3+} plot (Fig. 2) for optimal membrane ingredients indicates that it has a Nernstian behavior over a broad concentration range of Lu^{3+} ions. The slope and the linear range of the resulting calibration graph were 19.6±0.2 mV per decade and 1.0×10^{-6}-1.0×10^{-2} mol L^{-1}, respectively. The limit of detection, defined as the Lu^{3+} ion concentration obtained when the linear region of the calibration graph was extrapolated to the base line potential, was 8.0×10^{-7} mol L^{-1}. The standard deviation for ten replicate measurements was ±0.5 mV.

3.3. pH influence

The pH dependence of the membrane sensor was tested from the pH value of 1 up to 11 in a 1.0×10^{-3} mol L^{-1} Lu(III) solution. The associated resulting data are illustrated in Figure 3, where it can be seen that the potential remains fairly constant in the pH range of 2.3-7.4. Beyond this range, a gradual change in the potential was detected. The observed potential drift at higher pH values (>7.4) could be caused by the formation of some Lu(III) hydroxyl complexes in the solution. The increase in the potential response at lower pH values of <2.3, is most probably due to the responds of the sensor to the hydronium ions (the protonation of the nitrogen atoms of ion carrier in acidic media).

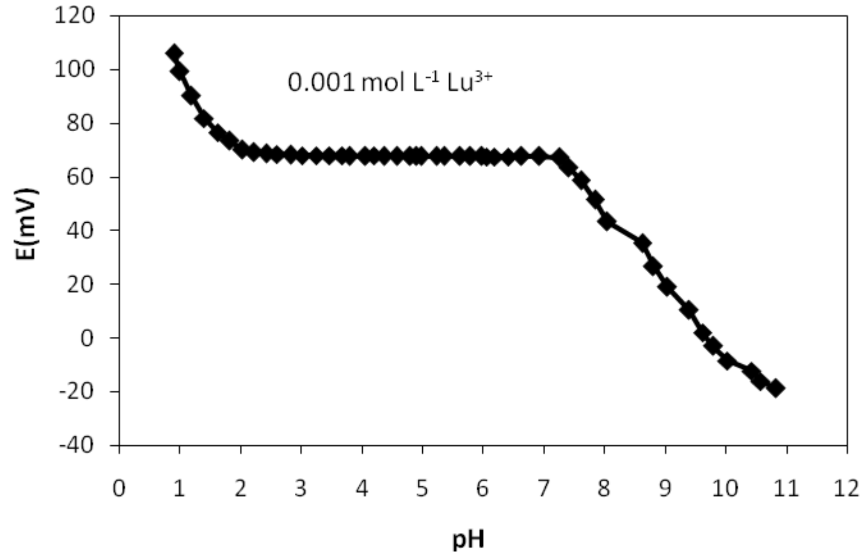

Figure 3. pH effect of the test solution (1.0×10^{-3} mol L^{-1} of Lu^{3+}) of the Lu^{3+}sensor based on HND.

3.4. Response time of the Lu^{3+} sensor based on HND

For analytical purposes, response time is one of the most important factors that are taken into account. The dynamic response time of the membrane was measured at various concentrations (from 1.0×10^{-7} to 1.0×10^{-2} mol L^{-1}) of the test solutions with different lutetium concentrations and results are shown in Figure 4. As it can be seen, the response time of the sensor is about 7.5 s in the whole concentration range.

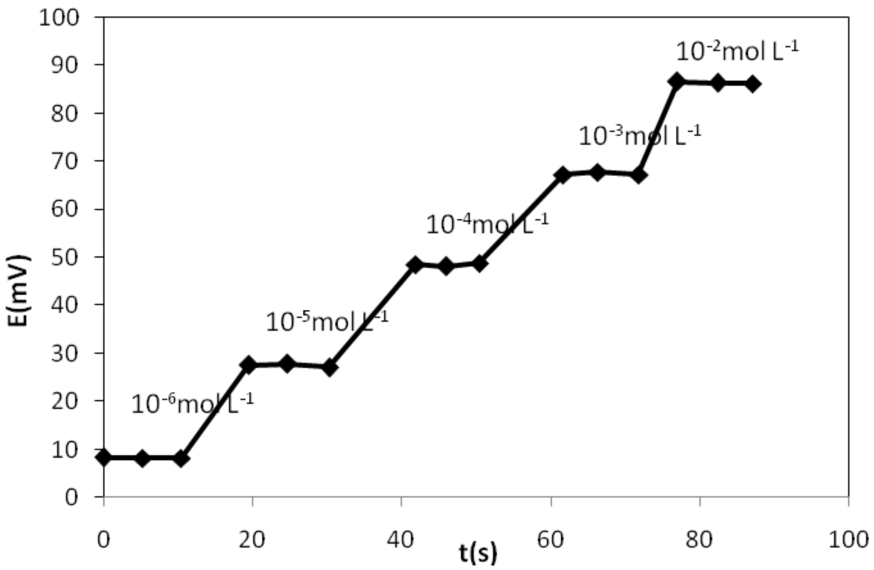

Figure 4. Dynamic response time of Lu^{3+} sensor based on HND.

3.5. The Lu(III) electrode selectivity

Table 2. Selectivity coefficients ($K^{MPM}_{Lu^{3+}}$) of proposed Lu^{3+} sensor.

Interfering Ion (B)	$K^{MPM}_{Lu^{3+},B}$	Interfering Ion (B)	$K^{MPM}_{Lu^{3+},B}$
Pr^{+3}	1.0×10^{-3}	Yb^{+3}	5.6×10^{-4}
La^{+3}	7.6×10^{-4}	Mg^{+2}	1.0×10^{-3}
Tm^{+3}	9.0×10^{-4}	Pb^{+2}	2.1×10^{-3}
Nd^{+3}	8.5×10^{-4}	Na^{+}	2.0×10^{-4}
Eu^{+3}	4.0×10^{-3}	K^{+}	8.8×10^{-4}
Ho^{+3}	1.0×10^{-3}	Co^{+2}	2.6×10^{-3}
Gd^{+3}	2.4×10^{-3}	Cd^{+2}	2.2×10^{-3}
Sm^{+3}	3.2×10^{-3}	Ca^{+2}	2.4×10^{-3}
Er^{+3}	5.0×10^{-4}	Fe^{+3}	3.2×10^{-4}
Tb^{+3}	8.7×10^{-4}	Cr^{+3}	7.5×10^{-4}
Dy^{+3}	2.0×10^{-3}	Ni^{+2}	3.1×10^{-3}

Selectivity coefficients, which reflects the relative response of the membrane sensor towards the primary ion over the other ions present in the solution, is perhaps the most important characteristic of an ion-selective electrode. In this research, the potential responses of the recommended Lu^{3+} membrane sensor to a wide variety of cations were investigated by matched potential method (MPM) [57-59]. The resulting selectivity coefficient values are summarized in Table 2. In line with the information of this Table, the selectivity coefficients for the monovalent tested cations (Na^+, K^+) are smaller than 8.8×10^{-4}. Additionally, the selectivity coefficients for the divalent tried cations (Ca^{2+}, Mg^{2+}, Ni^{2+}, Co^{2+}, Cd^{2+}, Pb^{2+}) are also small in the range of $1.0\times10^{-3} – 3.1\times10^{-3}$. In the case of trivalent cations (Pr^{3+}, La^{3+}, Tm^{3+}, Nd^{3+}, Eu^{3+}, Ho^{3+}, Gd^{3+}, Sm^{3+}, Er^{3+}, Tb^{3+}, Dy^{3+}, Yb^{3+}, Fe^{3+} and Cr^{3+}), the selectivity coefficients are relatively small (from 3.2×10^{-4} to 4.0×10^{-3}). Eventually, it can be stated that because of the good selectivity coefficients values, the disturbance produced by these cations in the function of the developed membrane sensor is negligible.

3.6. Analytical application

The lutetium membrane sensor was used as an indicator electrode in the successful titration of a lutetium ion solution (1.0×10^{-4} mol L^{-1}) with EDTA (1.0×10^{-2} mol L^{-1}). The resulting titration curves are depicted in Figure 5. As seen from Fig. 5, the amount of lutetium can be determined with the proposed sensor.

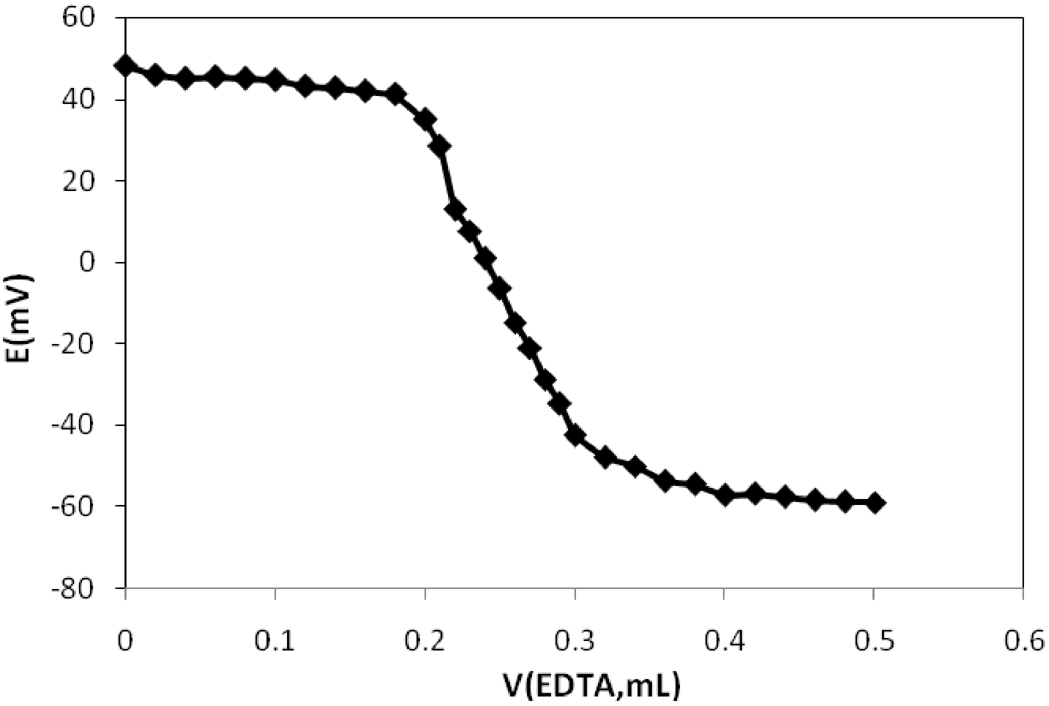

Figure 5. Potential titration curve of 25.0 mL from a 1.0×10^{-4} mol L^{-1} Lu^{3+} solution with 1.0×10^{-2} mol L^{-1} of EDTA.

To test its practical applicability of the proposed sensor, it was additionally applied to the determination of the Lu^{3+} ions in mixtures of two and three different ions. The resulting data of Table 3 indicate that the accuracy of the Lu^{3+} ions detection in different solutions of different metal ions is almost quantitative.

Table 3. Determination of Lu^{3+} ion in presence of different metal ions mixtures.

Lu^{3+} (mol L^{-1})	Added cations (mol L^{-1})	Found[a] (mol L^{-1})	Recovery (%)
1×10^{-6}	(0.001) $Gd(NO_3)_3$ & (0.001) $Pr(NO_3)_3$	1.04×10^{-6}	104
1×10^{-6}	(0.001) $Eu(NO_3)_3$ & (0.001) $Er(NO_3)_3$	1.02×10^{-6}	102
1×10^{-6}	(0.001) $La(NO_3)_3$ & (0.001) $Ho(NO_3)_3$	1.02×10^{-6}	102
1×10^{-6}	(0.001) $Dy(NO_3)_3$ & (0.001) $Yb(NO_3)_3$	1.01×10^{-6}	101
1×10^{-6}	(0.001) $Tb(NO_3)_3$ & (0.001) $Nd(NO_3)_3$	1.04×10^{-6}	104
1×10^{-6}	(0.001) $Na(NO_3)$ & (0.001) $Ca(NO_3)_2$	1.03×10^{-6}	103
1×10^{-6}	(0.001) $Pb(NO_3)_2$ & (0.001) $Ni(NO_3)_2$	1.01×10^{-6}	101
1×10^{-6}	(0.001) $Cr(NO_3)_3$ & (0.001) $Fe(NO_3)_3$	1.01×10^{-6}	101
1×10^{-6}	(0.001) $K(NO_3)$ &(0.001) $Mg(NO_3)_2$	1.01×10^{-6}	101
1×10^{-6}	(0.001) $Pb(NO_3)_2$ & (0.001) $Ca(NO_3)_2$ & (0.001) $K(NO_3)$	1.01×10^{-6}	101
1×10^{-6}	(0.001) $Fe(NO_3)_3$ & (0.001) $Na(NO_3)$ & (0.001) $Ca(NO_3)_2$	1.02×10^{-6}	102

a.Results are based on three measurements

4. CONCLUSION

Using of 1,2-bis(2-hydroxynaphtamido)-4,5-dimethylbenzene (HND) in poly vinyl chloride membrane sensor as a selective ionophore with NB as plasticizer shows the best response characteristics with Nernstian behavior over the concentration range 1.0×10^{-6}-1.0×10^{-2} mol L^{-1} Lu^{3+} and a fast response time of ~7.5 s. Applicable pH range, lower detection limit, and potentiometric selectivity coefficients of the proposed sensor make it a superior device both compared to previously reported Lu^{3+} ion selective sensor and also to other methods used for the determinations of this ion.

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