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Construction of a new Lu^{3+} poly(vinyl chloride) membrane sensor Based on 2,2'-[propane-1,3-diylbis(thio)]dianiline as a suitable sensing material

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This paper introduces the development of an original Lu^{3+}-PVC membrane electrode based on 2,2'-[propane-1,3-diylbis(thio)]dianiline (PDTD) as a sensing carrier. The electrode presents a Nernstian slope of 19.8±0.4 mV decade^{-1} for the in the Lu^{3+} ions across a broad working concentration range from 1.0×10^{-6} to 1.0×10^{-2} mol L^{-1} with a lower detection limit of 8.2×10^{-7} mol L^{-1} in the pH range of 2.5 –8.4. The electrode response time was rapid (~9 s), and its selectivity with respect to mono and divalent metal ions (Na^+, K^+, Ca^{2+}, Mg^{2+}, Co^{2+}, Cd^{2+}, and Pb^{2+}) and trivalent ions (Fe^{3+}, Al^{3+} and Cr^{3+}), including members of the lanthanide family (Er^{3+}, Dy^{3+}, La^{3+}, Tm^{3+}, Nd^{3+}, Ho^{3+}, Gd^{3+}, Sm^{3+}, Yb^{3+}, Pr^{3+}, Eu^{3+} and Tb^{3+}) other than Lu^{3+} was good. The making sensor has been put to use successfully as an indicator electrode in analytical applications such as the potentiometric titration and determination of Lu^{3+} ion in blend of different ions.

Keywords: PVC membrane, Potentiometry, Sensor, Ion-selective electrode

1. INTRODUCTION

Lutetium is the last element in the lanthanide series with symbol Lu and atomic number 71, and it is traditionally counted among the rare earths. Lutetium has no known biological role and has low toxicity. Lutetium metal is slightly unstable in air at standard conditions, and the aqueous solutions of most lutetium salts are colorless. Lutetium is very expensive and it has very few commercial uses. It can be used as a catalyst for cracking hydrocarbons in oil refineries and can also be used in alkylation, hydrogenation, and polymerization applications [1, 2].

A sensitive and selective method is required to gain a precise result of lutetium determination. Many techniques have been used for the determination of Lu^{3+} ion such as 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. Despite the fact that these methods are precise and sensitive, they are also expensive and time-consuming. Another method for the determination of this element is the ion-selective electrode (ISE) for Lu^{3+} potentiometric titration. The ISEs work fast, and they are easily prepared without any special or expensive equipment. There have been only a few reports of Lu^{3+} ion- selective electrodes in the literature [3-9].

Recently, a number of PVC-membrane ion-selective sensors for some other ions were reported [10-49]. In this research, the use of 2,2'-[propane-1,3-diylbis(thio)]dianiline (PDTD) (Fig. 1) as sensing ion carrier is introduced for the preparation of a highly Lu^{3+} ion-selective electrode, and its application for the measurement of the Lu^{3+} ion concentration is discussed.

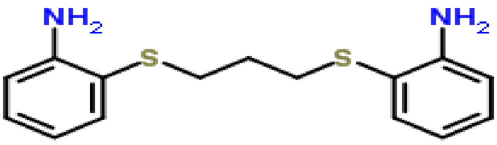

Figure 1. Chemical structure of the PDTD.

2. EXPERIMENTAL

2.1. EMF measurements

All the necessary potential measurements were performed by means of a Corning ion analyzer 250 pH/mV meter at room temperature. The electro motive force (emf) measurements of Lu^{3+} ion-selective electrode 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).

Activities were calculated according to the Debye–Huckel procedure [50].

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 sodium tetraphenyl borate (NaTPB), benzyl acetate (BA), dibutyl phthalate (DBP), acetophenon (AP), nitrobenzene (NB), tetrahydrofuran (THF) and high relative molecular weight PVC. 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 sensor preparation

The optimized membrane solution preparation involved the total dissolution of the following compounds: 3.0 mg of PDTD, 1.0 mg of NaTPB, 66 mg of NB and 30 mg of powdered PVC, in 3 mL of fresh THF. The resulting clear mixture was evaporated slowly up to the point that an oily concentrated mixture was obtained. A Pyrex tube (3-5 mm o.d. on top) was dipped into the mixture for about 10 s, so that a transparent membrane of about 0.3 mm thickness was formed [51-79]. The tube was pulled out from the mixture and kept at room temperature for 24 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 initial experiments, in order to check the sensitivity and selectivity of PDTD, polymeric membrane electrode (PME) containing 2,2'-[propane-1,3-diylbis(thio)]dianiline (PDTD) were applied for the detection of various cations. Due to the existence of both N and S donor atoms in the chemical structure of PDTD, it was expected to act as a sensing ionophore for special lanthanide ions in the PVC membranes. Based on the obtained results, among the metal ions used, Lu^{3+} with the most Nerstian response, can be suitably determined with the PVC membrane electrode.

3.1. Membrane composition effect

It is well understood that the nature and amount of ion carrier and additives used, nature of the plasticizer, the ratio of plasticizer/PVC, significantly influence the sensitivity and selectivity of the ion-selective electrodes [80–85]. The performance characteristics of the membranes having ingredients of different proportions are summarized in Table1. As it is seen, the increasing amounts of PDTD result in membranes that display larger slopes (electrode Nos. 5, 8 and 9). Table1 also displays that among the four various plasticizers used (BA, AP, NB, and DBP), NB with a higher polarity than BA, AP and DBP, revealed the best selectivity. The constructed membranes with a plasticizer/PVC ratio of about 2.2 were found to have the highest sensitivity. The presence of lipophilic anions in cation-selective membrane electrodes not only diminishes the ohmic resistance and enhances the potential behavior and selectivity, but also increases the sensitivity of the membrane electrodes if the extraction capability is poor [85-87]. As can be seen from Table1, addition of 1% NaTPB (membrane No. 9) will increase the sensitivity of the Lu-polymeric membrane electrode (LuPME) response to a great extent. The best performance was obtained with 30%PVC, 1%NaTPB, 3%PDTD and 66%NB.

Table 1. Optimization of the membrane ingredients.

Electrode No.	Composition of Electrode (wt.%)							Slope (mV/decade)	Dynamic linear range (M)
	PVC	NB	AP	BA	DBP	NaTPB	PDTD		
1	3	-	-	68	-	1	1	24.8±0.4	1.0×10^{-6}-1.0×10^{-2}
2	30	68	-	-	-	1	1	17.5±0.2	1.0×10^{-6}-1.0×10^{-2}
3	30	-	68	-	-	1	1	15.0±0.1	1.0×10^{-6}-1.0×10^{-2}
4	30	-	-	-	68	1	1	26.2±0.4	1.0×10^{-6}-1.0×10^{-2}
5	30	69	-	-	-	0	1	16.0±0.2	1.0×10^{-5}-1.0×10^{-2}
6	30	67	-	-	-	2	1	23.2±0.3	1.0×10^{-6}-1.0×10^{-2}
7	30	66	-	-	-	3	1	24.1±0.1	1.0×10^{-6}-1.0×10^{-2}
8	30	67	-	-	-	1	2	18.7±0.5	1.0×10^{-6}-1.0×10^{-2}
9	30	66	-	-	-	1	3	19.8±0.4	1.0×10^{-6}-1.0×10^{-2}

3.2. Slope and detection limit

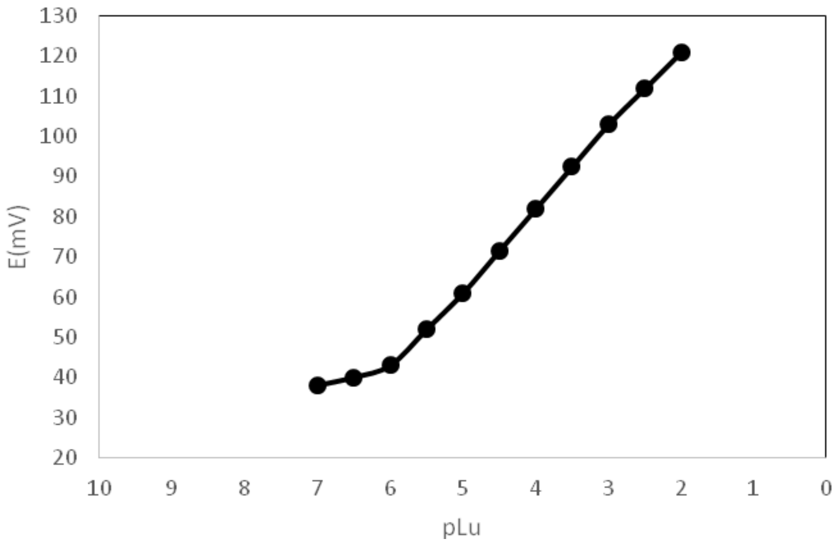

Figure 2. Calibration curves of the PDTD-based Lu^{3+} sensor (no electrode. 9).

The LuPME potential response at different concentrations of lutetium ion (Fig.2) indicates a linear working concentration range from 1.0×10^{-6} to 1.0×10^{-2} mol L^{-1}, a Nernstian slope of 19.8±0.4 $mVdecade^{-1}$ of lutetium ions concentration. The limit of detection, defined as the concentration of Lu(III) obtained when extrapolating the linear region of the calibration curve of the sensor with the optimum concentration (Fig.3) to the baseline potential, is 8.2×10^{-7} mol L^{-1}.

3.3. pH influence

The pH response profile on the response of the proposed membrane sensor to a 1.0×10^{-3} mol L^{-1} Lu(III) solution was studied over a pH range from 1.0 to 9.5 and the results are shown in Figure 3. The pH was adjusted by introducing small drops of nitric acid (0.1 mol L^{-1}) or sodium hydroxide (0.1 mol L^{-1}). As can be seen from Fig.5, the potential was found to stay fairly constant in the pH range 2.5–8.4. Beyond this range, a gradual change in potential was detected. The potential decrease at higher pH values (>8.4), is due to the formation of insoluble of $Lu(OH)_3$ while the observed increases in the potential response of the sensor. At lower pH than 2.5, the potentials increased, indicating that the membrane sensor responds to hydrogen ions.

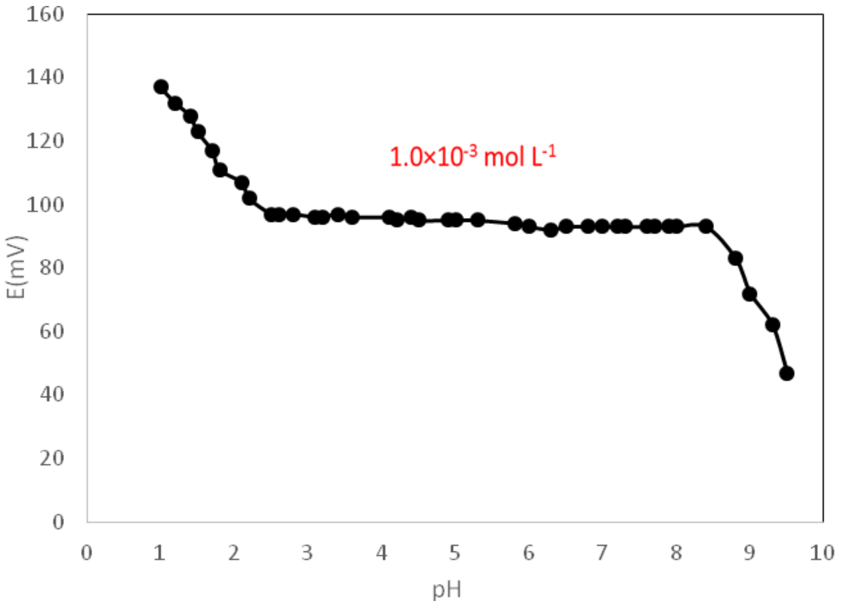

Figure 3. pH effect of the test solution (1.0×10^{-3} mol L^{-1} of Lu^{3+}) of the Lu^{3+}sensor based on PDTD (no electrode. 9).

3.4. Response time of the Lu^{3+}-PME

For analytical purposes, the response time of the ion selective electrode is of critical significance. The dynamic response time of the electrode was measured when the lutetium ion concentration rapidly increased from 1.0×10^{-6} to 1.0×10^{-2} mol L^{-1}. The results are shown in Figure 4.

From the results, the sensor reached equilibrium in a short period of time about 9 s in the whole concentration range.

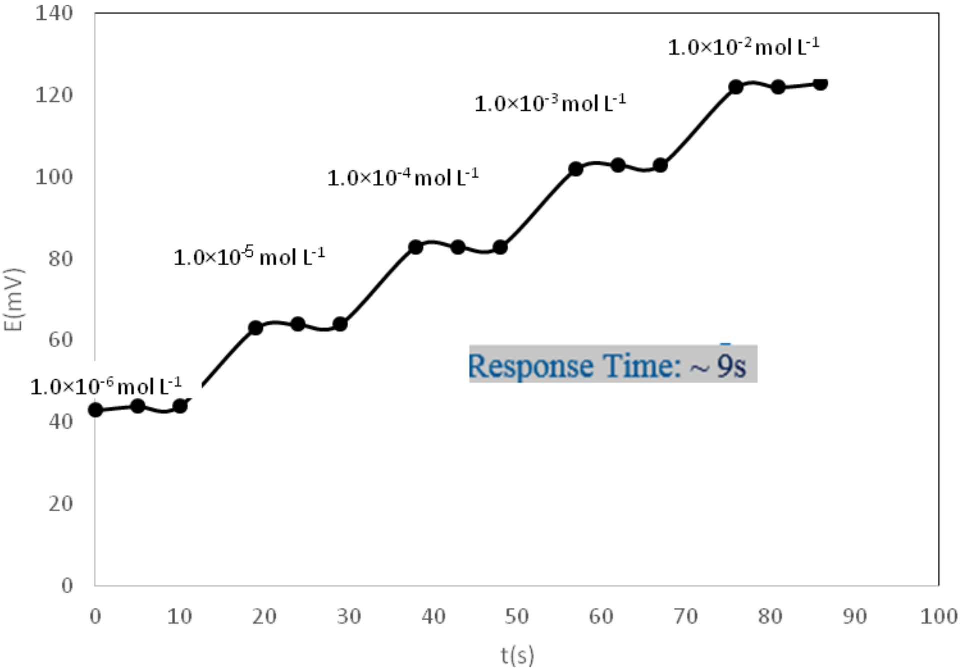

Figure 4. Dynamic response time of Lu^{3+}-PME based on PDTD (no electrode. 9).

3.5. The selectivity of Lu^{3+}-PME

Table 2. The Selectivity coefficients ($K_{Lu^{3+}}^{MPM}$) of proposed Lu^{3+}-PME.

Interfering Ion	$K_{Lu,B}^{MPM}$	**Interfering Ion**	$K_{Lu,B}^{MPM}$
Pr^{3+}	2.5×10^{-4}	Yb^{3+}	5.8×10^{-4}
La^{3+}	6.7×10^{-4}	Fe^{3+}	2.5×10^{-5}
Tm^{3+}	7.5×10^{-4}	Al^{3+}	4.0×10^{-5}
Nd^{3+}	9.0×10^{-4}	Cr^{3+}	6.0×10^{-5}
Eu^{3+}	3.2×10^{-4}	Pb^{2+}	8.0×10^{-5}
Ho^{3+}	1.5×10^{-4}	Co^{2+}	8.0×10^{-5}
Gd^{3+}	5.7×10^{-4}	Cd^{2+}	3.0×10^{-5}
Sm^{3+}	5.4×10^{-4}	Ca^{2+}	6.5×10^{-5}
Er^{3+}	8.6×10^{-4}	Mg^{2+}	5.5×10^{-5}
Tb^{3+}	3.8×10^{-4}	K^{+}	3.7×10^{-5}
Dy^{3+}	9.5×10^{-4}	Na^{+}	4.0×10^{-5}

The selectivity coefficients determination for an ion selective electrode, as one the most important characteristics, is defined the relative response of the sensor towards the primary ion over the other ions present in the solution. In this work, the potentiometric selectivity coefficient of the Lu^{3+}-PME based on PDTD to the monovalent cations (Na^+, K^+), the divalent cations (Ca^{2+}, Mg^{2+}, Co^{2+}, Cd^{2+}, Pb^{2+}) and the trivalent cations (Fe^{3+}, Al^{3+}, Cr^{3+}, Er^{3+}, Dy^{3+}, La^{3+}, Tm^{3+}, Nd^{3+}, Ho^{3+}, Gd^{3+}, Sm^{3+}, Yb^{3+}, Pr^{3+}, Eu^{3+}, Tb^{3+}) were investigated by matched potential method (MPM) [88-91]. The selectivity coefficient values for different metal ions are summarized in Table 2.

The data revealed that in the case of trivalent cations (Fe^{3+}, Al^{3+}, Cr^{3+}, Er^{3+}, Dy^{3+}, La^{3+}, Tm^{3+}, Nd^{3+}, Ho^{3+}, Gd^{3+}, Sm^{3+}, Yb^{3+}, Pr^{3+}, Eu^{3+}, Tb^{3+}), the selectivity coefficients were in the range 9.5×10^{-4} to 2.5×10^{-5}. The obtained selectivity coefficients for mono- and di-valent cations (Na^+, K^+, Ca^{2+}, Mg^{2+}, Co^{2+}, Cd^{2+}, Pb^{2+}) were smaller than 8.0×10^{-5}, which means that any of tested ions did not affect on the response of the proposed Lu^{3+}-PME.

3.6. Analytical application

The newly created Lu^{3+} ion-selective electrode was successfully used as an indicator electrode in the titration of a lutetium ion solution (1.0×10^{-4} mol L^{-1}) with EDTA (1.0×10^{-2} mol L^{-1}) the titration curve is given in Figure 5. As shown in the figure, the end point of titration and the concentration of Lu^{3+} ions in solution can be determined using the developed Lu^{3+}-PME.

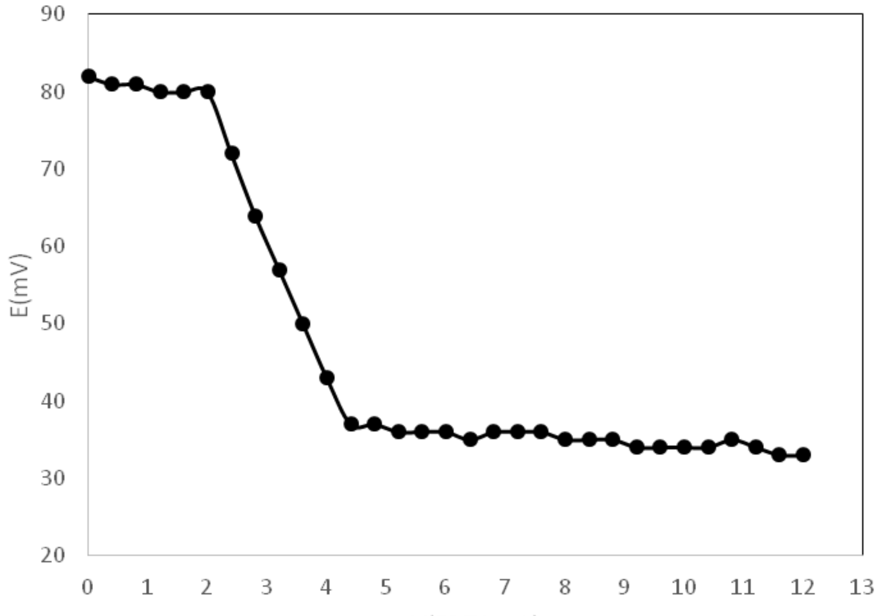

Figure 5. Potential titration curve of 25.0 mL from a 1.0×10^{-4} mol L^{-1} Lu^{3+} solution with 1.0×10^{-2} mol L^{-1} of EDTA.

The proposed Lu^{3+} ion-selective electrode was successfully employed to the monitoring of the Lu^{3+} ions in presence of various cations. The resulting data (after triplicate measurements) are given in

Table 4. It should be noted that the recovery of lutetium ions in all mixtures is acceptable (94.0%-104.0%).

Table 3. Determination of Lu^{3+} ion in presence of various ions mixture.

Lu^{+3} (mol L^{-1})	Added cations[a] (mol L^{-1})	Found[a] (mol L^{-1})	Recovery (%)
1.0×10^{-6}	La + Tm	0.96×10^{-6}	96
1.0×10^{-6}	Eu + Er	0.95×10^{-6}	95
1.0×10^{-6}	La + Ho	0.94×10^{-6}	94
1.0×10^{-6}	Sm + Yb	1.04×10^{-6}	104
1.0×10^{-6}	Tb + Nd	1.02×10^{-6}	102
1.0×10^{-6}	Na + Cu	0.94×10^{-6}	94
1.0×10^{-6}	Pb + Fe	0.95×10^{-6}	95
1.0×10^{-6}	Co + Cr	1.03×10^{-6}	103
1.0×10^{-6}	K + Mg	1.04×10^{-6}	104
1.0×10^{-6}	Fe + Ca + K	1.03×10^{-6}	103
1.0×10^{-6}	Al + Na + Co	1.04×10^{-6}	104

[a] Each metal ion concentration was 0.001 mol L^{-1}, all metals were introduced as nitrates.

[b] Based on three measurements.

The created Lu^{3+} sensor was also applied for the fluoride ion determination in the mouthwash solutions (three preparation samples with the same fluoride contents). After triplicate measurements, the data are given in Table 4. The resuls in the Table 4, indicate that the determination of fluoride ion in the mouthwash samples with the proposed sensor is almost quantitative.

Table 4. Determination of fluoride ions in mouth wash solutions.

Sample	Labeled (mgL^{-1})	Found ISE[a] (mgL^{-1})
Sodium fluoride mouth wash solution (Aquafresh, Brentford, U.K.)	1350	(1375 ± 21)[b]
Sodium fluoride mouth wash solution (Eurodont, DuroDont GmbH)	1450	(1482 ± 24)

[a] Suggested Lu^{3+} sensor.

[b] Results are based on three measurements.

4. CONCLUSION

A new PVC Lu^{3+}-ion selective electrode incorporating 2,2'-[propane-1,3-diylbis(thio)]dianiline (PDTD) as a sensing ionophore was successfully used to determine Lu^{3+} cations over a broad working concentration range of 1.0×10^{-6}-1.0×10^{-2} mol L^{-1} with a Nernstian behavior of 19.8±0.4 mV decade^{-1} and a lower detection limit of 8.2×10^{-7} mol L^{-1} in the pH range of 2.5 –8.4. The selectivity towards the lutetium ions was not influenced by the presence of the mono-, di- and trivalent cations used (Na^+, K^+, Ca^{2+}, Mg^{2+}, Co^{2+}, Cd^{2+}, Pb^{2+}, Fe^{3+}, Al^{3+}, Cr^{3+}, Er^{3+}, Dy^{3+}, La^{3+}, Tm^{3+}, Nd^{3+}, Ho^{3+}, Gd^{3+}, Sm^{3+}, Yb^{3+}, Pr^{3+}, Eu^{3+}, Tb^{3+}), since the interference of these substances was low. The fabricated Lu^{3+} electrode can be successfully applied to the monitoring of the lutetium ions in presence of the cations mixture.

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