

Construction of a New Selective and Sensitive Nd³⁺ ion-selective Electrode Based on 1-nitroso-2-naphtol as a Neutral Ion Carrier

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In this work, we found that 1-nitroso-2-naphtol (NN) can be used as a sensing material for the fabrication of a new highly selective neodymium(III) ion-selective membrane sensor. The best characteristics was obtained for the proposed Nd³⁺ sensor having a composition of PVC: nitrobenzene (NB): sodium tetraphenyl borate (NaTPB): NN in the ratio of 30:67:2:1 (wt%). The sensor displays a Nernstian slope of 19.7±0.4 mV decade⁻¹ for Nd³⁺ ion with detection limit of 6.5×10⁻⁷ mol L⁻¹ between pH 2.5 and 9.6. The working concentration range of the constructed sensor was 1.0×10⁻⁶ to 1.0×10⁻² mol L⁻¹ with a short response time (~8 s). The selectivity coefficients obtained using matched potential method (MPM) indicate good selectivity for neodymium ion with respect to other cations. The created sensor was successfully utilized as an indicator electrode for the potentiometric titration of neodymium ions with EDTA and the determination of neodymium contents in presence of various ions mixture.

Keywords: Potentiometry, PVC Membrane, Sensor, Ion-Selective Electrode

1. INTRODUCTION

Neodymium, with symbol Nd on the periodic table, is one of the rare-earth metal of the lanthanide series and a soft silvery-white metal with atomic number 60. Neodymium was used in equipment such as energy-saving lamps, fluorescent lamps, colour televisions and glasses. Neodymium has no known biological role. It is moderately toxic and irritating to eyes. The important use of neodymium is in high-performance electric motors and generators. It was also used in spindle magnets for computer hard drives and wind turbines, in steel manufacture, industry of electronics, and in ferrous and nonferrous alloys (used for lighter flints) [1, 2].

A review of the literature revealed that there are different instrumental methods for the determination of neodymium in different sample matrices including: ICP-MS, ICP-AES, isotope dilution

mass spectroscopy, absorption spectra of 4f electron transitions, capillary electrophoresis, gravimetric determination. Although these techniques provide an accurate measurement in trace amount of elements, they destroyed the samples. In comparison, ion selective electrodes offer advantages of fast response and low cost method, low detection limit, portability, wide concentration range, good selectivity, and simple design. They also provide an analysis method without destruction of sample. A few reports on neodymium potentiometric sensors based on different ionophores were reported in the literature survey [3-10].

Recently, a thorough literature survey has revealed that several potentiometric polyvinyl chloride (PVC)-ion selective electrodes based on various ion carriers have been reported by our group and other researchers for the determination of different cations [11-49]. In this work, we reported our study on the construction of a new polymeric Nd^{3+} -ion selective electrode based on 1-nitroso-2-naphtol (NN) as an excellent ionophore for the determination of Nd(III) ions in solution.

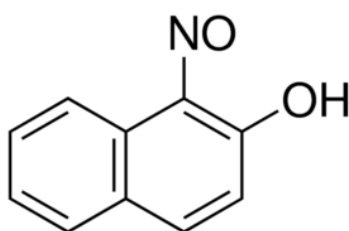


Figure 1. The NN structure.

2. EXPERIMENTAL

2.1. Reagents

For polymer membrane preparation, sodium tetraphenyl borate (NaTPB), tetrahydrofuran (THF), nitrobenzene (NB), acetophenone (AP), dibutyl phthalate (DBP), and benzyl acetate (BA) were purchased Merck Chemical (Germany) and Aldrich Co. (USA). The nitrate and chloride salts of the all used cations were also purchased from Merck and Aldrich at the highest purity available. All aqueous solutions were prepared with doubly distilled deionized water.

2.2. The emf measurements

The potential measurements were performed at room temperature with the following assembly:

Ag-AgCl | internal solution, $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ NdCl}_3$ | PVC membrane | sample solution | Hg-Hg₂Cl₂, KCl (satd.)

Corning ion analyzer 250 pH/mV meter was used for the potential measurements. The activities were evaluated according to the Debye-Huckel procedure [50].

2.3. The Nd^{3+} Sensor preparation

The membranes were prepared by dissolving (1-3) mg of NN, (1-3) mg NaTPB, 30 mg PVC, (65-69) mg of the plasticizer (NB, BA, DBP and AP) in 3-5 mL of tetrahydrofuran. The resulting mixture was transferred into a glass dish of 2 cm diameter. The solvent 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 10 s, so that a transparent membrane of about 0.3 mm thickness was formed. 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 ($1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ NdCl}_3$). The electrode was finally conditioned for 24 h by soaking in a $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Nd}^{3+}$ ion solution [51-79]. A silver/silver chloride electrode was used as an internal reference electrode.

2.4. Measurement of selectivity coefficient

In this work, the values of selectivity coefficients were calculated by match potential method (MPM) [80-83]. According to MPM, the selectivity coefficient is defined as the activity ratio of the primary ion (A) and the interfering ion (B) which gives the same potential change in a reference solution. Thus, one should measure the change potential upon changing the primary ion activity, and then the interfering ion would be added to an identical reference solution until the same potential change is obtained. The matched potential method selectivity coefficient, K^{MPM} , is then given by the resulting primary ion to interfering ion activity (concentration) ratio, $K^{MPM} = \Delta a_A / a_B$.

3. RESULTS AND DISCUSSION

3.1. Potential response and the influence of membrane composition

In order to test whether NN could be used as a selective substance for neodymium ions, in the preliminary experiments, NN as suitable carrier was used in the fabrication of membrane sensors for a wide variety of cations, including alkali, alkaline earth, transition and heavy metal ions. Among different cations tested, Nd^{3+} ion illustrated a strong response to the PVC membrane based on NN. This is most probably due to the specific interaction of NN and Nd^{3+} ions, and the rapid exchange kinetics of the resulting NN- Nd^{3+} complex.

It has been known that some important ingredients of PMEs based PVC, such as the plasticizer/PVC ratio, the features of plasticizer, the amount of ion carrier, the nature of ionophore and additives used, significantly influence the selectivity and sensitivity of the PVC-based membrane electrodes [84-90]. Thus, different aspects of the constructed membrane based on NN were optimized. The resulting data are summarized in Table 1. As shown from Table 1, except NB, among three other plasticizers used, the resulting electrodes displayed no stable potential response. This is due to the high polarity of NB that facilitates the extraction of neodymium ions with high charge density from aqueous

solution to the organic membrane phase. The nature of the plasticizer influences the dielectric constant of the polymeric membranes, the mobility of the ionophore and its complex.

It is well accepted that the presence of lipophilic anions in a cation-ion selective sensor diminishes the ohmic resistance, enhances the response behavior and selectivity, and increases the sensitivity of the membrane electrodes, and in cases where the extraction capability is poor, increases the sensitivity of the membrane electrodes [90-94]. As can be seen from Table 1, addition of 2% NaTPB as a suitable additive (No. 6) will increase the slope of the potential response of the electrode from the poor value of mV decade⁻¹ (No. 5) to a Nernstian value of 19.7 mV decade⁻¹ (No. 6). Nevertheless, the best performance was obtained with a membrane composition of 30% PVC, 67% BA, 1% NN and 2% NaTPB (No. 6).

Table 1. Membrane composition optimization of the Nd³⁺ sensor.

Electrode No.	Composition of Electrode (wt.%)							Slope (mV/decade)	Dynamic linear range (M)
	PVC	NB	AP	BA	DBP	NaTPB	NN		
1	30	-	-	68	-	1	1	16.0±0.4	1.0×10 ⁻⁵ -1.0×10 ⁻²
2	30	68	-	-	-	1	1	14.1±0.5	1.0×10 ⁻⁵ -1.0×10 ⁻²
3	30	-	68	-	-	1	1	13.4±0.3	1.0×10 ⁻⁵ -1.0×10 ⁻²
4	30	-	-	-	68	1	1	13.2±0.4	1.0×10 ⁻⁵ -1.0×10 ⁻²
5	30	-	-	69	-	0	1	15.8±0.5	1.0×10 ⁻⁵ -1.0×10 ⁻²
6	30	-	-	67	-	2	1	19.7±0.4	1.0×10⁻⁶-1.0×10⁻²
7	30	-	-	66	-	3	1	22.1±0.4	1.0×10 ⁻⁶ -1.0×10 ⁻²
8	30	-	-	66	-	2	2	23.7 ±0.5	1.0×10 ⁻⁶ -1.0×10 ⁻²
9	30	-	-	65	-	2	3	24.2±0.4	1.0×10 ⁻⁶ -1.0×10 ⁻²

3.2. Slope and working concentration range of the Nd³⁺ sensor

Fig. 2 displays the emf response of the Nd³⁺-PVC membrane at varying concentration of Nd³⁺ ions in the concentration range of 1.0×10⁻⁶ to 1.0×10⁻² mol L⁻¹. The slope of the calibration curve was 20.7±0.3 mV decade⁻¹. The lower detection limit (LOD) of the Nd³⁺ electrode was determined from the intersection of the two extrapolated segments of the calibration plot was 6.5×10⁻⁷ mol L⁻¹.

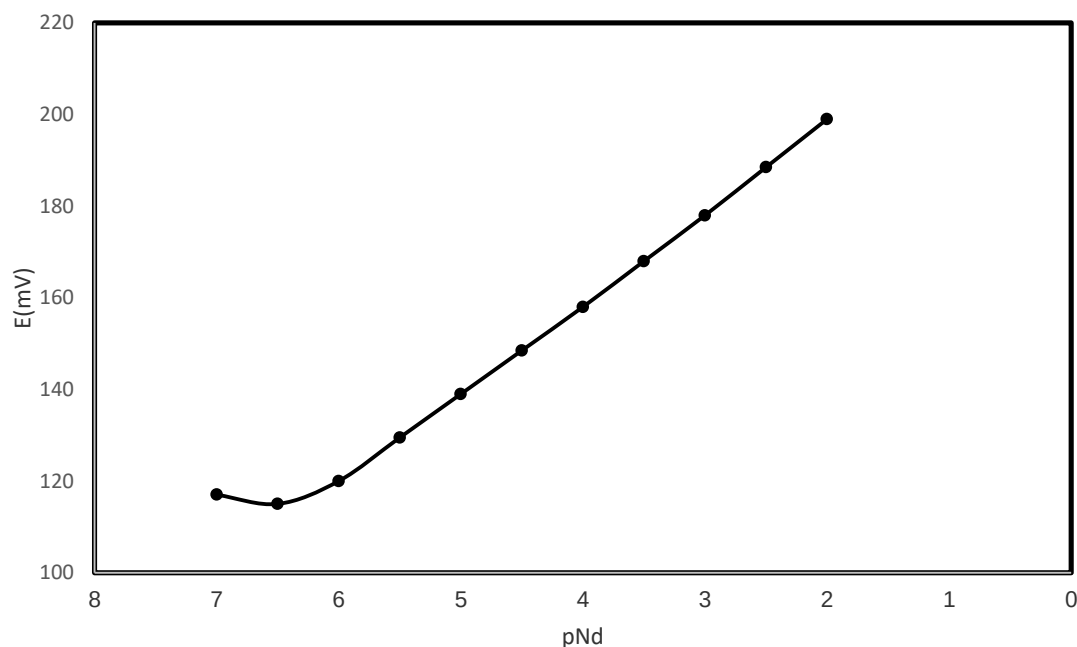


Figure 2. Calibration characteristics of the Nd^{3+} sensor based on NN (Conditions: Concentration range 1.0×10^{-6} - 1.0×10^{-2} mol L^{-1} , Slope 20.7 ± 0.3 mV decade $^{-1}$ and Detection limit 6.5×10^{-7} mol L^{-1}).

3.3. Influence of pH

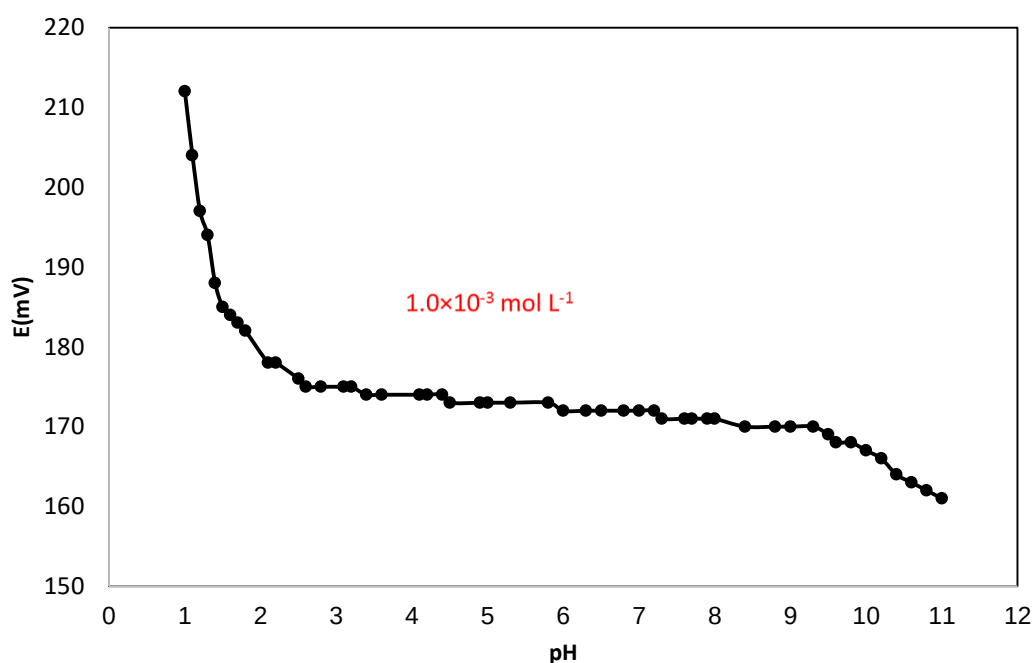


Figure 3. Effect of pH of the test solution (1.0×10^{-3} mol L^{-1}) on the potential response of the Nd^{3+} sensor.

The effect of pH on the response of membrane sensor was examined over the pH range from 2.0 up to 12.0 at a certain Nd^{3+} ion concentration (1.0×10^{-3} mol L^{-1}). The pH was adjusted by introducing small drops of hydrochloric acid (0.1 mol L^{-1}) or sodium hydroxide (0.1 mol L^{-1}) into the test solution and the results are given in Figure 3. Evidently, the potential stays constant from pH 2.5 to 9.6, beyond

which some potential drifts took place. The potential decrease at higher pH values (>8.4), could be attributed the formation of some insoluble of $\text{Lu}(\text{OH})_3$ in the solution. At lower pH values than 2.5, the potentials increased, indicating that the proposed Nd^{3+} sensor responded to protonium ions.

3.4. Dynamic response time of the created Nd^{3+} sensor based on NN

The response time, as an important factor for any sensor, was measured by changing the Nd^{3+} ion concentration in the test solution ($1.0 \times 10^{-3} \text{ mol L}^{-1}$), over a concentration range of 1.0×10^{-6} – $1.0 \times 10^{-2} \text{ mol L}^{-1}$. The potential response versus time is shown in Figure 4. As can be seen, the dynamic response time of the proposed electrode was found almost 8 s at different concentrations of the test solution.

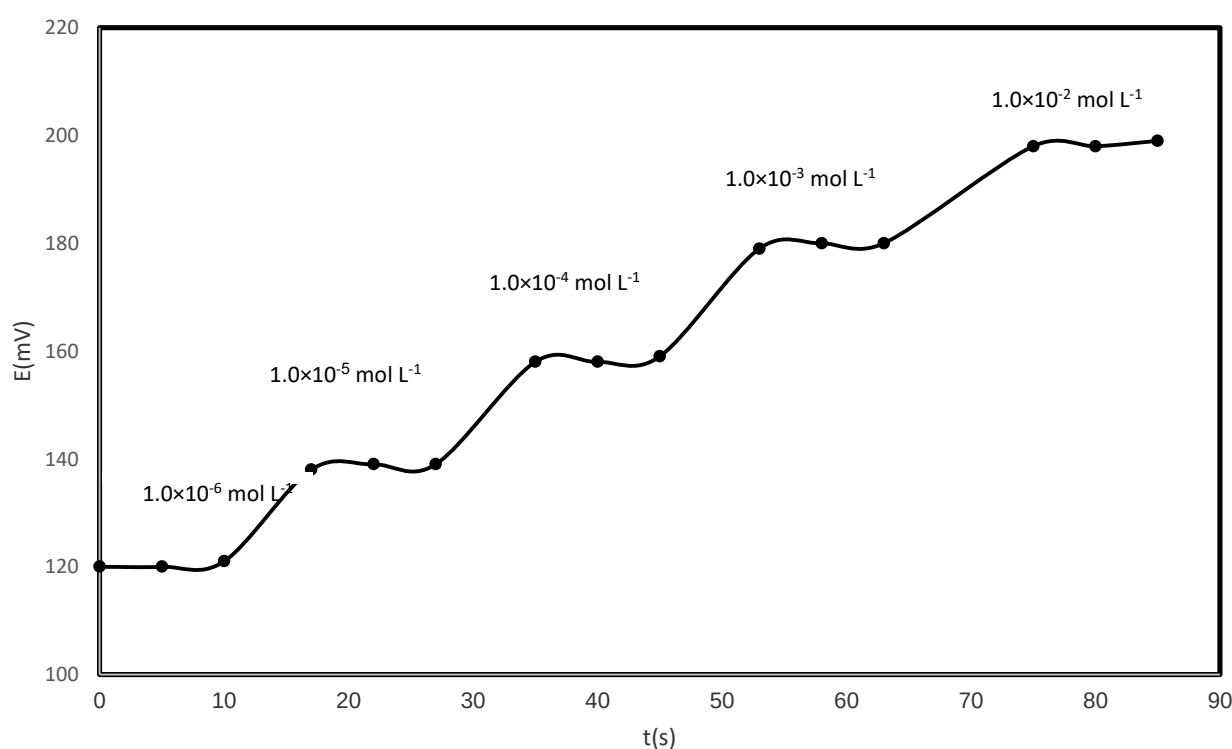


Figure 4. Dynamic response time of the Nd^{3+} sensor based on NN {for A) $1.0 \times 10^{-6} \text{ mol L}^{-1}$, B) $1.0 \times 10^{-5} \text{ mol L}^{-1}$, C) $1.0 \times 10^{-4} \text{ mol L}^{-1}$, D) $1.0 \times 10^{-3} \text{ mol L}^{-1}$, E) $1.0 \times 10^{-2} \text{ mol L}^{-1}$ } in solution of Nd^{3+} ions.

3.5. Selectivity coefficients values of the proposed Nd^{3+} sensor based on NN

The selectivity behavior of the proposed sensor was determined through the matched potential method (MPM) and the calculated values of selectivity coefficients are listed in Table 2. As it is evident from Table 2, the developed sensor is selective towards neodymium ions in the presence of various interfering ions, indicating negligible interference on the potential response of the created sensor.

Table 2. Selectivity coefficients of various interfering ions determined by matched potential method.

Interfering Ion	$K_{Nd,B}^{MPM}$	Interfering Ion	$K_{Nd,B}^{MPM}$
Pr ³⁺	5.2×10^{-4}	Yb ³⁺	3.7×10^{-4}
La ³⁺	7.5×10^{-4}	Cr ³⁺	5.0×10^{-5}
Tm ³⁺	3.5×10^{-4}	Al ³⁺	1.0×10^{-5}
Lu ³⁺	5.5×10^{-4}	Ni ²⁺	7.3×10^{-5}
Eu ³⁺	5.0×10^{-4}	Mg ²⁺	2.5×10^{-5}
Ho ³⁺	2.8×10^{-4}	Co ²⁺	3.0×10^{-5}
Gd ³⁺	9.0×10^{-4}	Cd ²⁺	4.5×10^{-5}
Sm ³⁺	8.5×10^{-4}	Ca ²⁺	6.7×10^{-5}
Er ³⁺	4.6×10^{-4}	Pb ²⁺	3.0×10^{-5}
Tb ³⁺	4.8×10^{-4}	K ⁺	5.5×10^{-5}
Dy ³⁺	5.0×10^{-4}	Na ⁺	9.0×10^{-5}

Table 3. Comparison of the proposed Nd³⁺ electrode with the previous Nd³⁺ electrodes.

Parameter	Ref. 4	Ref. 5	Ref. 6	Ref. 7	Ref. 8	Ref. 9	This work
LR ^a (mol L ⁻¹)	1.0×10^{-5} - 1.0×10^{-2}	1.0×10^{-6} - 1.0×10^{-2}	1.0×10^{-6} - 1.0×10^{-1}	1.0×10^{-6} - 1.0×10^{-2}	1.0×10^{-6} - 1.0×10^{-2}	5.0×10^{-7} - 1.0×10^{-2}	1.0×10^{-6} - 1.0×10^{-2}
DL ^b (mol L ⁻¹)	2.0×10^{-6}	7.0×10^{-6}	8.0×10^{-7}	7.9×10^{-7}	6.2×10^{-7}	1.0×10^{-7}	6.5×10^{-7}
Response time (s)	<10	<15	<15	<5	<10	10	~8
pH range	3.5-8.5	4.0-8.0	3.0-7.0	4.0-6.5	3.7-8.3	4.0-8.0	2.5-9.6
Slope (mV decade ⁻¹)	19.6±0.3	19.6±0.3	19.4±0.3	20.1±0.2	19.7±0.4	19.8±0.3	19.7±0.4
Log K _{sel} >-2	La, Sm, Gd	La, Gd, Sm, Cu, Yb	La, Gd, Sm, Pr	Ce, Yb, Gd, Sr, Cu, La, Ag	La, Dy	Hg, Ni	-

^aLR: Linear Range;^bDL: Detection Limit

Table 3 compares the response characteristics of the recommended neodymium sensor such as: the linearity range, detection limit, pH range, response time, slope and selectivity coefficients are compared with those of the best Nd³⁺ PVC-membrane sensors reported previously in the literature by other researchers [4-9]. From the data given in Table 3 it is evident that the newly developed sensor is

superior to the formerly reported Nd^{3+} sensors in terms of selectivity, slope, pH range, dynamic response time, detection limit and dynamic concentration range.

3.6. Analytical applications

The constructed Nd^{3+} sensor acts well under laboratory conditions and can be successfully used in the potentiometric titrations of neodymium ion solution ($1.0 \times 10^{-4} \text{ mol L}^{-1}$) with a standard EDTA solution ($1.0 \times 10^{-2} \text{ mol L}^{-1}$) and the corresponding titration curve is shown in Figure 5. As seen, the sensor assembly can be used to determine the amount of Nd^{3+} ion concentration in solution by potentiometric titration curve providing a sharp end point.

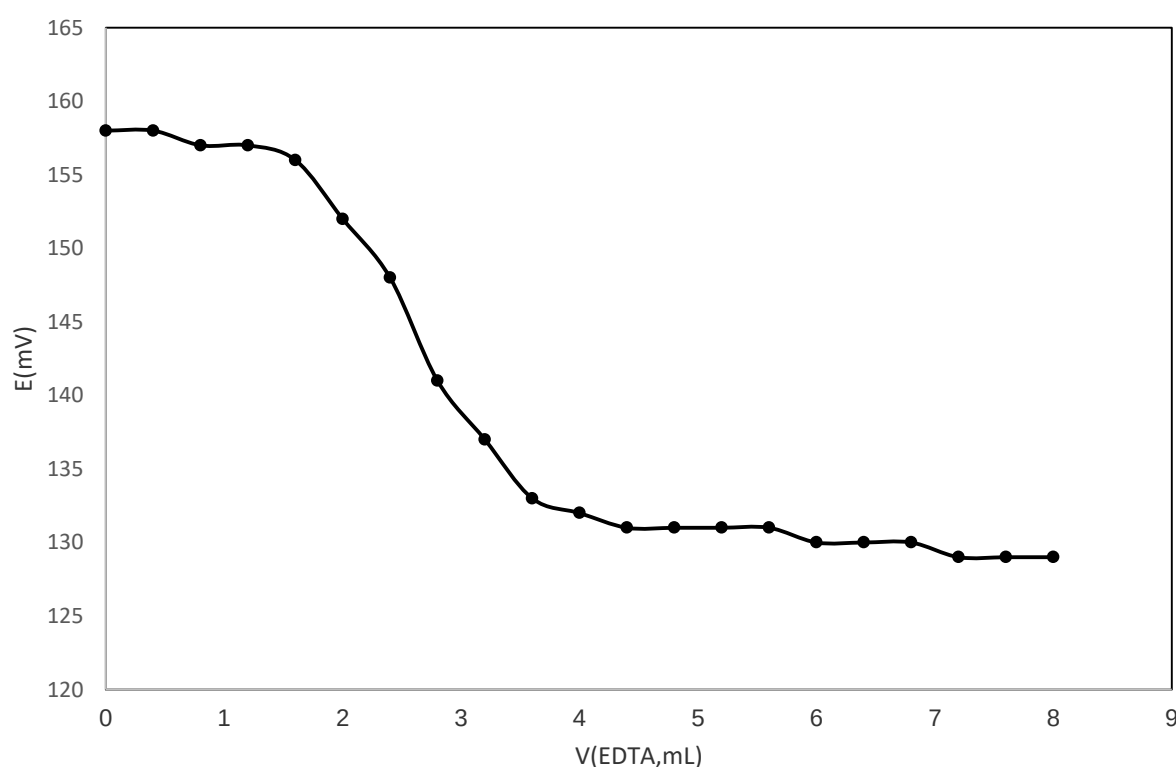


Figure 5. Potentiometric titration curve of 20.0 mL from a $1.0 \times 10^{-4} \text{ mol L}^{-1}$ Nd^{3+} solution with $1.0 \times 10^{-2} \text{ mol L}^{-1}$ of EDTA.

The recovery test was successfully used for the monitoring of Nd^{3+} ions concentration in presence of different ions mixture and the resulting values are given in Table 4. As it is obvious, the recovery of Nd^{3+} ions in different metal cations mixture is almost quantitative.

Table 4. Recovery test of Nd^{3+} ions added to the different cations mixture.

Nd^{3+} (mol L^{-1})	Added cations ^a (mol L^{-1})	Observed content ^b (mol L^{-1})	Recovery (%)
1.0×10^{-6}	La + Tm	1.05×10^{-6}	105
1.0×10^{-6}	Eu + Er	1.04×10^{-6}	104

1.0×10^{-6}	Gd + Pr	1.07×10^{-6}	107
1.0×10^{-6}	Sm + Yb	0.97×10^{-6}	97
1.0×10^{-6}	Na + Cu	10.5×10^{-6}	105
1.0×10^{-6}	Pb + Ni	0.94×10^{-6}	94
1.0×10^{-6}	Co + Cr	1.05×10^{-6}	105
1.0×10^{-6}	K + Mg	0.93×10^{-6}	93
1.0×10^{-6}	Al + Na + Zn	1.06×10^{-6}	106
1.0×10^{-5}	Fe + Ca + K	0.98×10^{-6}	98

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

^bBased on three measurements.

4. CONCLUSION

The newly fabricated PVC-membrane sensor based on 1-nitroso-2-naphtol (NN) as ionophore, NaTPB as lipophilic cationic salt and BA as plasticizer was successfully used for the determination of Nd^{3+} ions concentration in solution. The most important properties of the best performing sensors are: slope: $19.7 \pm 0.4 \text{ mV decade}^{-1}$; linear range: $1.0 \times 10^{-6} - 1.0 \times 10^{-2} \text{ mol L}^{-1}$; detection limit: $6.5 \times 10^{-7} \text{ mol L}^{-1}$; response time: $\sim 8 \text{ s}$; pH range: 2.5-9.6. The Nd^{3+} -membrane sensor based on 1-nitroso-2-naphtol (NN) displays good selectivity for Nd^{3+} ion towards the mono-, di- and trivalent cations used. The recommended sensor was successfully applied to the neodymium determination in presence of different ions mixture.

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