

*Full Paper*

## **PVC Membrane Potentiometric Sensor Based on a Schiff Base for Determination of Ho<sup>3+</sup> ions**

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**Abstract-** Due to the importance of selective determination of the rare earth elements, construction of a novel holmium PVC membrane sensor based on 3-[(pyridine-2-ylmethylene)-amino]-2-thioxo-4-one (PMAT) as a neutral carrier, (DBP) as a plasticizer and (KTpClPB) as anionic additive is reported. The sensor has a Nernstian slope of  $21.0 \pm 0.4$  mV per decade and a linear dynamic range between  $1.0 \times 10^{-6}$  and  $1.0 \times 10^{-2}$  mol L<sup>-1</sup>. The mentioned sensor can be used in pH range of 4-7.5 and has a detection limit of  $7.0 \times 10^{-7}$  mol L<sup>-1</sup>. It has at least 4 weeks applicability life time, possesses the advantages of fast response time, and very good selectivity over a large number of cations, especially for lanthanide ions. The sensor was used as an indicator electrode in the potentiometric titration of fluoride ions of mouthwash with  $1.0 \times 10^{-3}$  mol L<sup>-1</sup> solution of Ho<sup>3+</sup> ion.

**Keywords-** Holmium, Ion-Selective Electrode (ISE), Potentiometry, Sensor, Mouthwash

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### **1. INTRODUCTION**

Lanthanide elements have gained a great attention in the last few decades owing to their unique properties and wide range of applications that utilize these elements in huge quantities. These elements and their compounds have found a variety of applications especially in metallurgy, ceramic industry, and nuclear fuel control [1-3]. Holmium is used to

create the strongest artificially generated magnetic fields and also is used in yttrium-iron-garnet (YIG) and yttrium-lanthanum-fluoride (YLF) solid-state lasers [4]. Holmium compounds have specialized uses in ceramic, glass, and phosphor lamps. Holmium nanoparticles and nanopowders provide ultra-high surface area which nanotechnology research and recent experiments demonstrate function to create new and unique properties and benefits. Oxides are available in powder and dense pellet form for such uses as optical coating and thin film applications. Holmium can absorb fission-bred neutrons. It is also used in nuclear reactors to keep the atomic chain reactions from running out of control [5].

Bioaccumulation of holmium ions in the body can be a threat to the liver. Today we know that with water animals, holmium causes damage to cell membranes, which has several negative influences on reproduction functions of the nervous system [6]. Therefore analysis of this element is very important. However, conventional methods that can be used for the analysis of this element, such as inductively coupled plasma atomic emission spectroscopy (ICP-AES) [7], neutron activation analysis (NAA) [8], are either expensive or difficult to work with. Hence the ability to analyze this element in different matrices is of great importance. Recently, a number of sensors for lanthanide group [9-12] and other elements [13-18] have been reported. Some of them have been constructed for determination of  $\text{Ho}^{3+}$  ions [19-23].

In this research a new membrane electrode based on a neutral carrier (Figure 1) for  $\text{Ho}^{3+}$  ion determination is proposed.

## 2. EXPERIMENTAL

### 2.1. Reagents

Reagent grade o-nitrophenyloctyl ether (o-NPOE), dibutyl phthalate (DBP), sodium tetraphenylborate (NaTPB), potassium tetrakis 4-chlorophenylborate (KTPCIPB) tetrahydrofuran (THF), and high relative molecular weight PVC (all from Merck) were used as received. The nitrate and chloride of all cations used (from Merck and Aldrich) were of the highest purity available and used without any further purification. The ionophore 3-[(pyridine-2-ylmethylene)-amino]-2-thioxo-4-one (PMAT) was purchased from ABI Chemicals, GmbH, Germany and used without further purification.

### 2.2. Preparation of membrane sensor

The PVC membranes were prepared according to following procedure. Precisely weighted amounts of 32 mg of powdered PVC, 58 mg of DBP and 4 mg of additive (KTPCIPB) were dissolved in 3-5 mL of THF, and 6 mg of Ionophore (PMAT) was added. After completely mixing the solution it was transferred into a glass dish of 2 cm diameter, and then its solvent was allowed to evaporate so as to gain an oily concentrated mixture. The

membrane was then formed on the tip of a Pyrex tube of 3–5 mm thickness by inserting the tube into the viscose mixture for about 10 s, so that a transparent membrane of about 0.3 mm thickness was formed. The tube was pulled out and given enough time to dry at room temperature for about 10 h, before being filled with an internal filling solution  $1.0 \times 10^{-3}$  mol  $L^{-1}$   $HoCl_3$ . As a final step, the electrode was conditioned for 24 h by soaking in a  $1.0 \times 10^{-3}$  mol  $L^{-1}$   $HoCl_3$  solution. The internal reference electrode was a silver/silver chloride electrode.

### 2.3. Electromotive force measurements

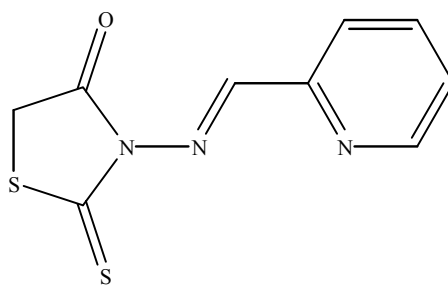
The following cell was assembled for the emf measurements:

$Ag-AgCl$  | internal solution,  $1.0 \times 10^{-3}$  mol  $L^{-1}$   $HoCl_3$  | PVC membrane | sample solution |  $Hg-Hg_2Cl_2$ ,  $KCl$  (sat'd) A Corning ion analyzer 250 pH/mV meter was used for the potential measurements at  $25.0 \pm 0.1$  °C.

## 3. RESULTS AND DISCUSSION

### 3.1. Complex formation study

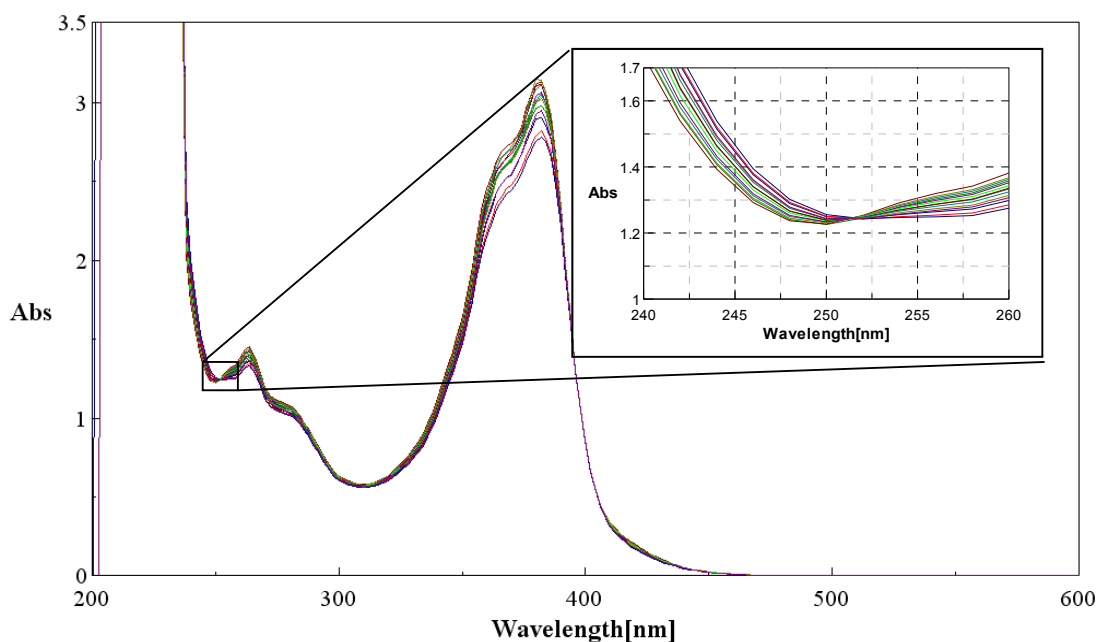
The primary spectrophotometric study for complexation, showed the tendency of the derivative PMAT (Figure 1) toward selective complexation with  $Ho^{3+}$  ions, one of the lanthanide members. Thus, this ionophore was used as a sensing material in construction of  $Ho^{3+}$  membrane sensor.



**Fig. 1.** The structure of the ionophore 3- [(pyridine-2-ylmethylene)-amino]-2-thioxo-4-one (PMAT)

Being a lipophilic ligand and also presence of nitrogen and sulfur in the structure of PMAT, suggested the possibility of complex formation with Lanthanide metal ions. In order to investigate the complex formation between  $Ho^{3+}$  ion and PMAT, in preliminary experiments, UV-VIS spectra of a solution containing a constant concentration of PMAT ( $5 \times 10^{-5}$  mol  $L^{-1}$ ) in acetonitrile were recorded between 250-500 nm by stepwise addition of 10  $\mu L$  of  $1 \times 10^{-3}$  mol  $L^{-1}$   $Ho^{3+}$  ion solution in each step. The spectra are overlaid and shown in Figure 2. As seen these spectra show an isobestic point which is a good indication of complex

formation. The same procedure was repeated for some other lanthanides such as  $\text{Nd}^{3+}$ ,  $\text{Pr}^{3+}$  and  $\text{Lu}^{3+}$  ions, but none of them showed an exact isobestic point.



**Fig. 2.** Isobestic point of PMAT with  $\text{Ho}^{3+}$  ion

### 3.2. Optimization of membrane composition

Because the degree of sensitivity and selectivity for a certain ionophore is greatly related to the membrane ingredients [24-26], the influence of membrane composition on the potential responses of the  $\text{Ho}^{3+}$  sensor was inspected. In this study, different membrane compositions were tested based on previous research papers on other sensors for similar ions [27-32] and results are shown in Table 1.

As can be seen, the membrane no. 10\* with the composition of 32% (PVC), 6% Ionophore (PMAT), 4% (KTPCIPB) and 58% (DBP) was the optimum one in the development of this sensor with a Nernstian slope ( $21.0 \pm 0.4$  mV/decade). This membrane composition was selected after many considerations. The high  $\text{Ho}^{3+}$  ion extraction into the liquid membrane was a result of the elevated ionophore tendency to form a selective complex with the  $\text{Ho}^{3+}$  ions. As can be seen from Table 1, the slope of the Ho sensor is affected by amount of ionophore PMAT in the membrane composition. Increasing of PMAT in the membrane up to 6% exhibited the best sensitivity. Table 1 reveals that the potentiometric response of the membranes was greatly improved in the presence of lipophilic anionic additive. It is well known that for the membranes based on neutral and charged carriers, the presence of lipophilic salts not only reduce the membrane resistance, but also enhance the response behaviour and selectivity, and reduce interference from sample anion [24].

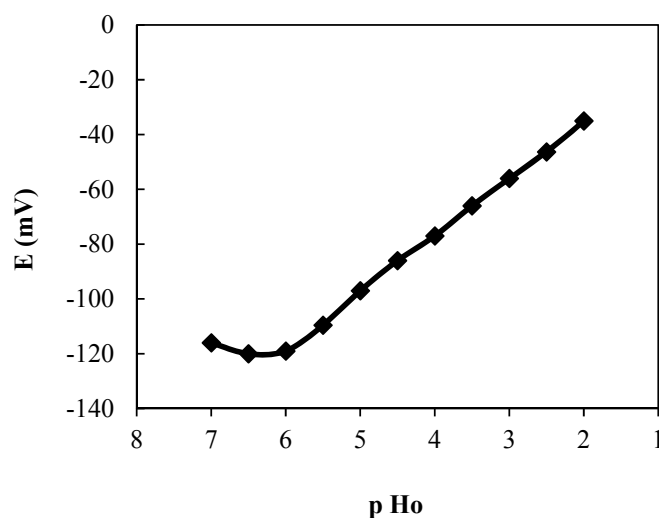
**Table 1.** The optimization of the membrane ingredients

Membrane no.	Composition (%)				Slope (mV/decade)
	PVC	Plasticizer	Ionophore	Additive	
1	32	DBP,61	5	2(NaTPB)	12.6 ±0.1
2	32	DBP,60	5	3(NaTPB)	13.4±0.4
3	32	DBP,59	6	3(NaTPB)	16.1±0.3
4	32	DBP,58	6	4(NaTPB)	17.0±0.2
5	32	NPOE,59	6	3(NaTPB)	12.1±0.3
6	32	NPOE,58	6	4(NaTPB)	12.9±0.4
7	32	DBP,57	5	2(KTpCIPB)	13.0±0.6
8	32	DBP,61	6	2(KTpCIPB)	15.1±0.5
9	32	DBP,59	6	3(KTpCIPB)	17.6±0.4
10*	32	DBP,58	6	4(KTpCIPB)	21.0±0.4

\* The optimum composition

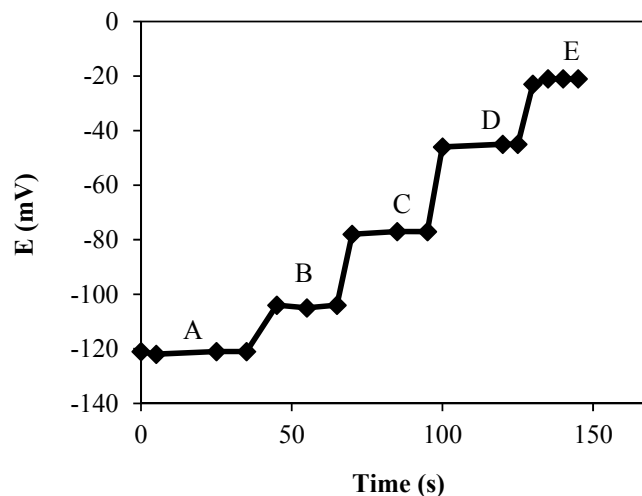
### 3.3. Calibration curve and statistical data

The critical response characteristics of the  $\text{Ho}^{3+}$  sensor were assessed according to IUPAC recommendations. The potential response of the membrane at varying concentration of  $\text{Ho}^{3+}$  ions (Figure 3) indicates a linear range from  $1.0 \times 10^{-6}$  to  $1.0 \times 10^{-2}$  mol  $\text{L}^{-1}$ . The slope of the calibration curve was  $21.0 \pm 0.4$  mV/decade of  $\text{Ho}^{3+}$  ion concentration. The lower detection limit of the sensor, determined from the intersection of the two extrapolated segments of the calibration curve, was  $7.0 \times 10^{-7}$  mol  $\text{L}^{-1}$ . The standard deviation after replicate measurements is  $\pm 0.4$  mV.



**Fig. 3.** The calibration curve of the  $\text{Ho}^{3+}$  membrane sensor

Dynamic response time is another important factor for the evaluation of any ion-selective electrode. The practical response time of the sensor was investigated by changing the  $\text{Ho}^{3+}$  ion concentration in solution, over a concentration range from  $1.0 \times 10^{-6}$  to  $1.0 \times 10^{-2} \text{ mol L}^{-1}$ , and the results are shown in Figure 4. As seen in whole concentration range the electrode reaches its equilibrium response in a very short time ( $<15$  s). This is most probably due to the fast exchange kinetics of complexation–decomplexation of  $\text{Ho}^{3+}$  ions with the PMAT at the test solution–membrane interface.



**Fig. 4.** Dynamic response time of the electrode for step changes in the concentration of  $\text{Ho}^{3+}$  solution: (A)  $1 \times 10^{-6} \text{ mol L}^{-1}$ , (B)  $1 \times 10^{-5} \text{ mol L}^{-1}$ , (C)  $1 \times 10^{-4} \text{ mol L}^{-1}$ , (D)  $1 \times 10^{-3} \text{ mol L}^{-1}$ , and (E)  $1 \times 10^{-2} \text{ mol L}^{-1}$

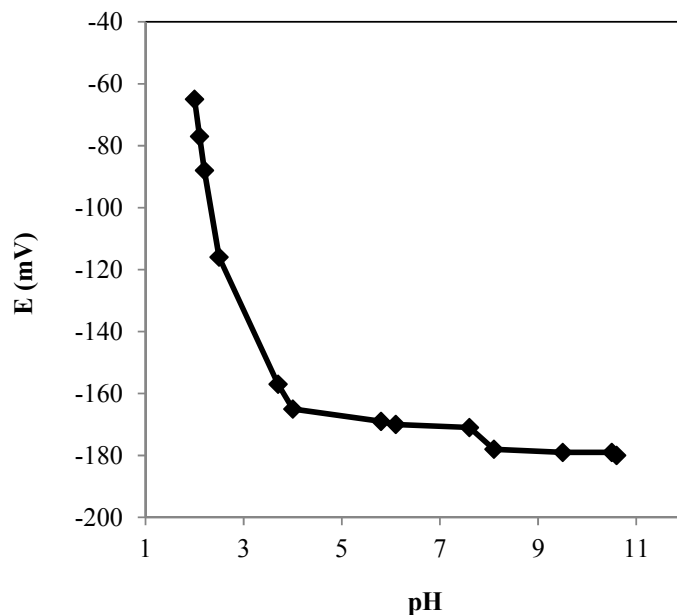
### 3.4. PH effect

Influence of pH on the response of the membrane sensor was studied for a  $1 \times 10^{-4} \text{ mol L}^{-1}$   $\text{Ho}^{3+}$  solution over a pH range of 2 – 11, and the results are shown in Figure 5.

In the pH range of 4–7.5 the potential did not change with changing pH, indicating the applicability of this sensor in this pH range. Beyond this range, a gradual change in potential was detected. The observed slightly decreased potential at higher pH values could be due to the formation of insoluble  $\text{Ho}(\text{OH})_3$  in solution. At lower pH, the potentials increased, indicating that the membrane sensor responds to hydrogen ions.

### 3.5. Stability and life-time

The stability and lifetime of the  $\text{Ho}^{3+}$  sensor, were studied by testing three electrodes over a period of 6 weeks, during which, the electrodes were in daily use over extended period of time (1 h per day). After 4 weeks, a slight decrease in the slope (from 21.0 to 18.1 mV/decade) was observed.



**Fig. 5.** Effect of the pH of test solutions  $1 \times 10^{-4}$  mol L<sup>-1</sup> on the potential response of the Ho<sup>3+</sup> ion-selective electrode

### 3.6. Selectivity of the sensor

Selectivity, which describes an ion-selective electrode's specificity toward the target ion in the presence of interfering ions, is the most important characteristic of these devices. The potentiometric selectivity coefficients of the Ho<sup>3+</sup> sensor were evaluated by MPM method. The resulting values of the log  $K_{MPM}$  are given in Table 2. As can be seen, for the metal ions and trivalent lanthanide ions tested, there are negligible interferences in the performance of the electrode.

**Table 2.** The selectivity coefficients of various interfering cations for the membrane

Interfering ion	log $K_{MPM}$	Interfering ion	log $K_{MPM}$
Na <sup>+</sup>	-1.175	Fe <sup>3+</sup>	-2.325
Ca <sup>2+</sup>	-2.241	Cu <sup>2+</sup>	-1.891
Mg <sup>2+</sup>	-3.250	Cd <sup>2+</sup>	-3.121
Pb <sup>2+</sup>	-1.109	Pr <sup>3+</sup>	-2.655
Co <sup>2+</sup>	-2.474	Ce <sup>3+</sup>	-2.282
Zn <sup>2+</sup>	-2.657	Er <sup>3+</sup>	-1.967
Lu <sup>3+</sup>	-1.353	Yb <sup>3+</sup>	-3.015
Nd <sup>3+</sup>	-2.109	Gd <sup>3+</sup>	-2.214

### 3.7. Analytical applicability

The sensor was successfully applied to direct monitoring of  $\text{Ho}^{3+}$  ions in binary mixtures and the results are summarized in Table 3. As it is obvious, the recovery of  $\text{Ho}^{3+}$  ions is very good (99.1–103.5%). The  $\text{Ho}^{3+}$  membrane sensor has been successfully used as an indicator electrode in the potentiometric titration of  $\text{Ho}^{3+}$  ion solution (20.0 mL,  $1.0 \times 10^{-4}$  mol  $\text{L}^{-1}$ ) with a standard EDTA solution and the results are illustrated in Figure 6. As can be seen, the amount of  $\text{Ho}^{3+}$  ions can be determined with the membrane sensor.

It was also applied to the determination of  $\text{F}^-$  ions in three different pharmaceutical samples, and the results of triplicate measurements are summarized in Table 4. For this approach a diluted solution of pharmaceutical sample containing  $\text{F}^-$  was titrated by a standard  $\text{Ho}^{3+}$  solution using the proposed sensor as an indicator electrode. Before the equivalent point we have  $\text{HoF}_3$  in the solution and the potential is almost constant, but after the equivalent point addition of more  $\text{Ho}^{3+}$  ions will increase the potential (Figure 7). As seen, there is a satisfactory agreement between the declared fluoride content and the determined values in Table 4.

**Table 3.** Direct monitoring of  $\text{Ho}^{3+}$  ions in binary mixtures

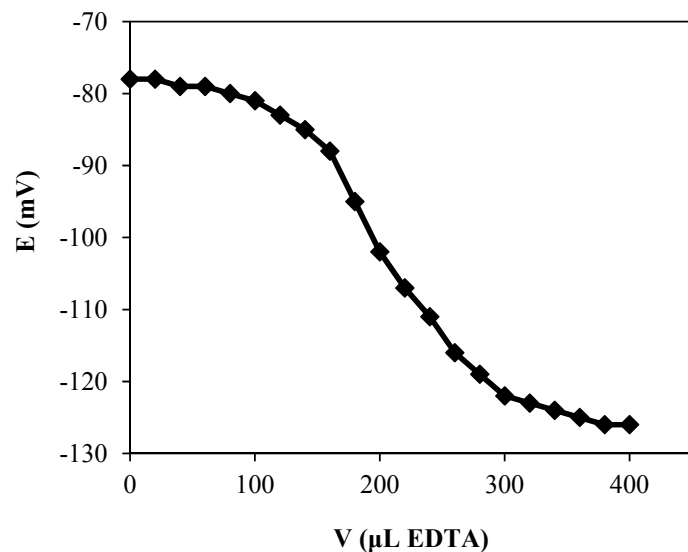
$\text{Ho}^{3+}$ (mol/L)	added cation (mol/L)	Recovery (%)
$1.0 \times 10^{-5}$	$\text{Na}^+$ ( $1.0 \times 10^{-4}$ )	103.4
$1.0 \times 10^{-5}$	$\text{Mg}^{+2}$ ( $1.0 \times 10^{-3}$ )	101.0
$1.0 \times 10^{-5}$	$\text{Ca}^{2+}$ ( $1.0 \times 10^{-3}$ )	99.1
$1.0 \times 10^{-5}$	$\text{Co}^{2+}$ ( $1.0 \times 10^{-3}$ )	102.6
$1.0 \times 10^{-5}$	$\text{Pb}^{+2}$ ( $5.0 \times 10^{-5}$ )	103.5
$1.0 \times 10^{-5}$	$\text{Zn}^{+2}$ ( $1.0 \times 10^{-3}$ )	103.2
$1.0 \times 10^{-5}$	$\text{Nd}^{+3}$ ( $1.0 \times 10^{-4}$ )	101.8
$1.0 \times 10^{-5}$	$\text{Pr}^{+3}$ ( $5.0 \times 10^{-4}$ )	99.2
$1.0 \times 10^{-5}$	$\text{Lu}^{+3}$ ( $5.0 \times 10^{-5}$ )	102.0
$1.0 \times 10^{-5}$	$\text{Gd}^{+3}$ ( $5.0 \times 10^{-4}$ )	102.2

**Table 4.** Determination of fluoride ions in different mouthwash samples and statistical analysis of the results

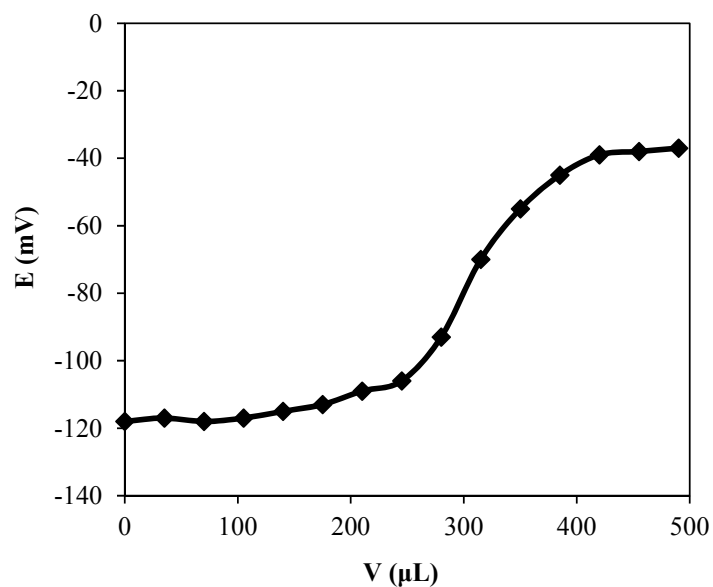
Sample	% Fluoride (labeled)	% Fluoride (found) <sup>a</sup>	%RSD	t-test calculated <sup>b</sup>
Sodium fluoride mouthwash solution (Shahredaru co., Tehran, Iran)	0.200	$0.190 \pm 0.005$	2.6	3.46
Sodium fluoride mouthwash solution (Irshashafa co., Tehran, Iran)	0.050	$0.048 \pm 0.001$	2.1	3.46

a. based on three replicates    b. The critical value is:  $t_{0.01,2}=6.965$





**Fig. 6.** Potentiometric titration curve of  $20.0 \text{ mL}$  of  $1.0 \times 10^{-4} \text{ mol L}^{-1} \text{ Ho}^{3+}$  solution with  $1.0 \times 10^{-2} \text{ mol L}^{-1}$  EDTA with the ISE prepared as indicator electrode



**Fig. 7.** Potentiometric titration curve of  $20 \text{ mL}$  of a diluted mouthwash solution with a  $(1.0 \times 10^{-3} \text{ mol L}^{-1}) \text{ Ho}^{3+}$  solution

Table 5 shows a comparison between this electrode and previously reported membrane sensors for holmium ions. As can be seen the characteristics of the proposed sensor are comparable with those of other  $\text{Ho}^{3+}$  ion selective electrodes.

**Table 5.** Comparison of the characteristics of the proposed sensor with those of the previously reported Ho<sup>3+</sup> membrane electrodes

Properties	Ref. [19]	Ref. [20]	Ref. [21]	Ref. [22]	Ref. [23]	This work
Detection limit (M)	$7.0 \times 10^{-6}$	$6.3 \times 10^{-7}$	$5.8 \times 10^{-7}$	$4.6 \times 10^{-7}$	$5.0 \times 10^{-7}$	$7.0 \times 10^{-7}$
Linear range (M)	$1 \times 10^{-5}$ - $1 \times 10^{-2}$	$1 \times 10^{-6}$ - $1 \times 10^{-2}$	$1 \times 10^{-6}$ - $1 \times 10^{-2}$	$1 \times 10^{-6}$ - $1 \times 10^{-2}$	$1 \times 10^{-6}$ - $1 \times 10^{-2}$	$1 \times 10^{-6}$ - $1 \times 10^{-2}$
Response time (s)	15	~10	<10	~5	<10	<15
Slope (mV/decade)	19.70±0.2	19.97±0.3	19.8±0.3	19.5±0.3	20.1±0.2	21.0±0.4
pH range	4.0-9.5	4.5-9.0	2.5-9.8	3.2-9.8	3.5-9.4	4.0-7.5

#### 4. CONCLUSION

The PVC-membrane Ho<sup>3+</sup> ion selective electrode based on PMAT exhibited response characteristics including Nernstian behavior with slope of 21.0±0.4 mV per decade, broad linear range of  $1.0 \times 10^{-6}$ - $1.0 \times 10^{-2}$  mol L<sup>-1</sup>, low detection limit of  $7.0 \times 10^{-7}$  mol L<sup>-1</sup>, fast response time and selectivity. The mentioned sensor can be used in pH range of 4-7.5 and has at least 4 weeks applicability life time. Also, the resulting values of the log K<sub>MPM</sub> shows that for the metal ions and trivalent lanthanide ions tested, there are negligible interferences in the performance of the sensor. The proposed electrode was used as an indicator electrode in the potentiometric titration of Ho<sup>3+</sup> ions with EDTA. It was successfully applied to determination of fluoride ions in mouthwash preparations.

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