# Highly Selective and Sensitive Ytterbium(III) PVC Membrane Sensor Based on 1,13-bis(8-quinolyl)-1,4,7,10,13pentaoxatridecane

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In this work, we introduce a highly selective and sensitive  $Yb^{3+}$  PVC-membrane sensor. 1,13-bis(8quinolyl)-1,4,7,10,13- pentaoxatridecane (QPOD) was used as an excellent sensing chemical material in the fabrication of a  $Yb^{3+}$  poly vinyl chloride membrane sensor. This sensor illustrated very good selectivity and sensitivity towards silver ions over a wide variety of cations, including alkali, alkaline earth, transition and heavy metal ions. The sensor exhibited a Nernstian response to  $Yb^{3+}$  ions (with a slope of 20.2±0.3 mV decade<sup>-1</sup>) for a concentration range  $(1.0 \times 10^{-7} - 1.0 \times 10^{-2} \text{ mol } L^{-1})$  with a detection limit of  $7.5 \times 10^{-8}$  mol L<sup>-1</sup>. It was used as an indicator electrode in the potentiometric titration of  $Yb^{3+}$ ions with EDTA and the determination of  $Yb^{3+}$  ions in presence of metal ions mixture.

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

# **1. INTRODUCTION**

One of the lanthanides which is a soft silvery metallic element is ytterbium. It is found in the minerals gadolinite, monazite, and xenotime. It is used in metallurgical and chemical experiments. Ytterbium is a rare earth element, and it is readily attacked and dissolved by the strong mineral acids. It reacts slowly with cold water and it oxidizes slowly in air. Although ytterbium is fairly stable, it nevertheless should be stored in closed containers to protect it from air and moisture. All compounds of ytterbium should be treated as highly toxic although initial studies appear to indicate that the danger is limited. Ytterbium and other lanthanides are used for gasoline cracking catalysts, carbon arcs, and movie projectors [1].

Some techniques such as: inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma atomic emission spectrometry (ICP-AES), spectrofluorimetry, neutron activation analysis, X-ray fluorescence spectrometry, etc., have been developed for the determination

of Ytterbium; but almost all of them are time consuming because need multiple steps for sample preparation and expensive.

However, a simple method that offers great advantages such as speed and eases of preparation and procedures, relatively short response times, reasonable selectivity, wide linear dynamic ranges, and low cost, is the potentiometric detections which is based on ion-selective sensors [2-26]. Literature review shows that there are a limited number of reports on the determination of Yb(III) ions by ion selective electrochemical sensor [27-33]. Recently, several greatly selective and sensitive PVCmembrane ion-selective electrodes for various metal ions have been reported [34-52]. Nevertheless, this paper focuses on the introduction of a highly ytterbium(III)-selective sensor based on 1,13-bis(8quinolyl)-1,4,7,10,13- pentaoxatridecane (QPOD) (Fig. 1), as a novel neutral ionophore for monitoring ytterbium concentration.



Figure 1. Structure of the ligand QPOD.

## 2. EXPERIMENTAL

#### 2.1. Chemicals and reagents

Reagent-grade dibutyl phthalate (DBP), *o*-nitrophenyloctyl ether (*o*-NPOE), oleic acid (OA), acetophenon (AP), nitrobenzene (NB), dibutylsebacate (DBS), sodium tetraphenyl borate (NaTPB), 1,13-bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane (QPOD), tetrahydrofuran (THF) and high relative molecular weight PVC were purchased from the Merck and the Aldrich Chemical Companies. 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.

#### 2.2. Preparation of membrane sensor

PVC-based ion-selective membranes were prepared according to a general procedure. The required ingredients were formulated by dissolving appropriate amounts of ionophore (3 mg), anionic additive OA (15 mg), plasticizer NB (52 mg), and PVC (30 mg) in 5 mL THF. The resulting mixture was transferred into a glass dish (2 cm in diameter) and the solvent was evaporated slowly until an oily concentrated mixture could be obtained. A Pyrex tube (3–5 mm in top) was dipped into the oily

mixture for about 5 s, so that a transparent film of about 0.3 mm thickness was formed. After the tube removal from this mixture, the tube was kept at room temperature for about 12 h and it was filled with the internal filling solution  $(1.0 \times 10^{-3} \text{ mol } \text{L}^{-1} \text{ YbCl}_3)$ . In the end, the electrode was conditioned by soaking in a  $1.0 \times 10^{-3} \text{ mol } \text{L}^{-1} \text{ YbCl}_3$  solution for 24 h [40–52]. A silver/silver chloride wire was used as an internal reference electrode.

#### 2.3. Electromotive force (EMF) measurements

All electromotive force was carried out with the membrane sensor using the following cell assembly:

Ag–AgCl| internal solution  $1.0 \times 10^{-3}$  mol L<sup>-1</sup> YbCl<sub>3</sub> | PVC membrane: sample| Hg–Hg<sub>2</sub>Cl<sub>2</sub>, 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 [53].

#### **3. RESULTS AND DISCISSION**

## 3.1. Potential electrode responses

In the QPOD structure, the existence of seven donating nitrogen and oxygen atoms was expected to increase both the stability and selectivity of its complexes with transition and lanthanide ions, more than other metal ions. Therefore, in the primary experiments, QPOD was used as a potentially suitable neutral carrier in the construction of the plasticized PVC membrane sensors for a number of metal ions. Among the examined metal ions, only the resulting Yb<sup>3+</sup>-selective sensor possesses a Nernstian behaviour over a very wide concentration range.

#### 3.2. Effect of membrane composition

Because the sensitivity and selectivity of a given ionophore depend significantly on the membrane ingredients, the nature of the solvent mediator and the used additive [54, 55], the membrane composition influences on the potential responses of the Yb<sup>3+</sup> sensor were monitored. The corresponding results are summarized in Table 1. The conclusion that can be derived from this Table is that the ionophore amount increase up to a value of 3% leads to the optimum sensitivity, in the presence of 15% OA and 52% of polar solvent (NB). The membrane plasticizer plays a key role in determining the selectivity, working concentration range and response time of the membrane electrode [55, 56]. From Table 1, among five different solvent mediators tested, NB is superior with respect to dibutylphthalate, *o*-nitrophenyloctyl ether, benzylacetate and acetophenone. The higher dielectric constants (DC) of NB helps the better extraction of the polar Yb<sup>3+</sup> ion, which is a cation with high charge density from the aqueous layer to the organic layer of the membrane and causes a better and faster response.

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 [56-58]. However, the membranes with the composition of 30 % PVC, 3 % QPOD, 15 % OA and 52 % NB show a Nernstian potential response.

Electrode	Composition (wt%)				Slope	Linear range
No.	PVC	C Plasticizer Additive QPOD		QPOD	(mV decade <sup>-1</sup> )	$(\text{mol } L^{-1})$
1	30	NB, 66	NaTPB, 2	2	$16.9 \pm 0.3$	$1.0 \times 10^{-6}$ -1.0 ×10 <sup>-2</sup>
2	30	DBP, 66	NaTPB, 2	2	$23.4 \pm 0.6$	$1.0 \times 10^{-5}$ -1.0 $\times 10^{-2}$
3	30	NPOE, 66	NaTPB, 2	2	$31.5 \pm 0.7$	$1.0 \times 10^{-5}$ -1.0 $\times 10^{-2}$
4	30	DBS, 66	NaTPB, 2	2	$30.5 \pm 0.4$	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$
5	30	AP, 66	NaTPB, 2	2	$14.3 \pm 0.3$	$1.0 \times 10^{-5}$ -1.0 ×10 <sup>-3</sup>
6	30	NB, 67	NaTPB, 2	1	$13.7 \pm 0.2$	$1.0 \times 10^{-5}$ -1.0 $\times 10^{-2}$
7	30	NB, 65	NaTPB, 2	3	$17.6 \pm 0.4$	$1.0 \times 10^{-6}$ -1.0 ×10 <sup>-2</sup>
8	30	NB, 64	NaTPB, 2	4	$22.4 \pm 0.5$	$1.0 \times 10^{-5}$ -1.0 $\times 10^{-2}$
9	30	NB, 63	NaTPB, 2	5	$25.6 \pm 0.8$	$1.0 \times 10^{-5}$ -1.0 $\times 10^{-2}$
10	30	NB, 62	OA, 5	3	$17.1 \pm 0.1$	$1.0 \times 10^{-6}$ -1.0 ×10 <sup>-2</sup>
11	30	NB, 57	OA, 10	3	$18.5 \pm 0.5$	$1.0 \times 10^{-6}$ -1.0 ×10 <sup>-2</sup>
12	30	NB, 52	OA, 15	3	$20.2 \pm 0.3$	$1.0 \times 10^{-7}$ -1.0 $\times 10^{-2}$

Table 1. Optimization of membrane ingredients.

#### 3.3. Calibration graph



Figure 2. Calibration curve of the Yb<sup>3+</sup> sensor based on QPOD.

As it can be observed from Figure 2, the developed sensor exhibits a Nernstian response slope of  $(20.2\pm0.3 \text{ mV decade}^{-1})$  across a broad concentration range of  $1.0\times10^{-7}$ - $1.0\times10^{-2}$  mol L<sup>-1</sup>. The detection limit, defined as the Yb<sup>3+</sup> concentration obtained after the extrapolation of the linear region

of the standard plot to the baseline potential, was  $7.5 \times 10^{-8} \text{ mol } \text{L}^{-1}$ . The standard deviation for ten replicate measurements was  $\pm 0.6 \text{ mV}$ .

## 3.4. pH effect

For the pH effect observation on the sensor performance, the potentials were determined in the pH range of 1.0-12.0 (concentrated NaOH or HCl was used for the pH adjustment) at one Yb<sup>3+</sup> concentration  $(1.0 \times 10^{-3} \text{ mol } \text{L}^{-1})$ . The reported data of Figure 3 exhibit that the potential remained constant from the pH value of 3.7 up to 9.6, beyond which some drifts in the potentials were observed. The observed drift at higher pH values can be due to the formation of insoluble of Yb(OH)<sub>3</sub> or other soluble intermediate products including Yb(OH)<sup>2+</sup>, and Yb(OH)<sub>2</sub><sup>+</sup> which may not necessarily form stable complexes with the ionophore. At lower pH values, the potentials increased, indicating that the membrane sensor responded to protonium ions, as a result of the extent nitrogen atom protonation of the ionophore.



**Figure 3.** Effect of the pH of test solution  $(1.0 \times 10^{-3} \text{ mol } \text{L}^{-1} \text{ of } \text{Yb}^{3+})$  on the potential response of the Yb<sup>3+</sup> sensor based on QPOD.

#### 3.5. Dynamic response time

For analytical purposes, response time is one of the most important factors that are taken into account. In this work, the practical response time was recorded by immediate and successive changing of Yb<sup>3+</sup> concentration from  $1.0 \times 10^{-7}$  - $1.0 \times 10^{-2}$  mol L<sup>-1</sup> and the results are shown in Figure 4. As it can be seen, in the whole concentration range, the electrode reaches to its equilibrium response in a very short time (<10 s).



**Figure 4.** Dynamic response time of the Yb<sup>3+</sup> sensor for step changes in the Yb<sup>3+</sup> concentration.

### 3.6. The sensor selectivity

$M^{n+}$	$\mathrm{K}^{\mathrm{MPM}}_{\mathrm{Yb}^{3+},\mathrm{B}}$	$M^{n+}$	$\mathrm{K}^{\mathrm{MPM}}_{\mathrm{Yb}^{3+},\mathrm{B}}$
Eu <sup>3+</sup>	$1.1 \times 10^{-4}$	Tm <sup>3+</sup>	$7.0  imes 10^{-5}$
Ce <sup>3+</sup>	$3.5 \times 10^{-5}$	Cr <sup>3+</sup>	$1.1 \times 10^{-4}$
Tb <sup>3+</sup>	$1.8  imes 10^{-4}$	Fe <sup>3+</sup>	$6.3 \times 10^{-5}$
Sm <sup>3+</sup>	$1.3 \times 10^{-4}$	Ni <sup>2+</sup>	$1.2 \times 10^{-4}$
Lu <sup>3+</sup>	$1.3 \times 10^{-4}$	Co <sup>2+</sup>	$1.1 \times 10^{-4}$
Nd <sup>3+</sup>	$2.2 \times 10^{-4}$	$\mathrm{Cd}^{2+}$	$1.3 \times 10^{-5}$
Er <sup>3+</sup>	$1.6 \times 10^{-5}$	Pb <sup>2+</sup>	$1.2 \times 10^{-3}$
Dy <sup>3+</sup>	$1.2 \times 10^{-4}$	Ca <sup>2+</sup>	$1.4 \times 10^{-4}$
La <sup>3+</sup>	$4.5 \times 10^{-5}$	$Mg^{2+}$	$5.7 \times 10^{-4}$
Gd <sup>3+</sup>	$1.1 \times 10^{-4}$	$Na^+$	$2.5 \times 10^{-5}$
Pr <sup>3+</sup>	$1.1 \times 10^{-4}$	$\mathbf{K}^+$	$1.0 \times 10^{-3}$

**Table 2.** Selectivity coefficients of the Yb<sup>3+</sup> electrode.

The potentiometric selectivity coefficient, which reflects the relative response of the membrane sensor for the primary ion over other ions that are present in solution, is perhaps the most important characteristic of an ion-selective electrode. In this work, the potential response of the recommended  $Yb^{3+}$  ion-selective sensor was investigated with the aid of the matched potential method (MPM) [59, 60]. In accordance with the MPM, the selectivity coefficient is defined as the activity ratio of the primary ion (A) and the interfering ion (B), which gives some potential change in a reference solution. The reported values of Table 2 present that the selectivity coefficients are in the order of  $1.0 \times 10^{-3}$  or

smaller for all the tested ions, indicating they would not significantly disturb the function of this Yb<sup>3+</sup> sensor.

In Table 3, selectivity, the dynamic linear range, the pH range, the detection limit and response time of the developed electrode are compared with the best data of the previously reported  $Yb^{3+}$  selective membrane electrode [27-29]. Evidently, the suggested  $Yb^{3+}$  sensor in terms of selectivity coefficients, dynamic linear range, detection limit and response time is superior to the former  $Yb^{3+}$  ionselective electrode.

**Table 3.** Comparison of selectivity coefficients, detection limit, linearity range, response time and pH range of the proposed Yb<sup>3+</sup> sensor and the formerly reported Yb<sup>3+</sup> sensor.

Parameters	Ref. 27	Ref. 28	Ref. 29	This work
Detection limit (mol $L^{-1}$ )	7.0×10 <sup>-7</sup>	5.0×10 <sup>-7</sup>	4.2×10 <sup>-7</sup>	7.5×10 <sup>-8</sup>
Linear range (mol $L^{-1}$ )	$1.0 \times 10^{-6} - 1.0 \times 10^{-2}$	$1.0 \times 10^{-6}$ - $1.0 \times 10^{-2}$	$1.0 \times 10^{-6} - 1.0 \times 10^{-1}$	$1.0 \times 10^{-7} - 1.0 \times 10^{-2}$
Response time (s)	~15	8-10	<10	<10
Interfering ion (B)	Cu	Dy	Pr, Dy, Fe, K, Ni	-
$K_{sel}$ More than $5.0 \times 10^{-3}$				
pH range	3.0-8.0	3.5-9.0	3.2-8.3	3.7-9.6

#### 3.7. Analytical application



Figure 5. Potential titration curve of 25.0 mL from a  $1.0 \times 10^{-4}$  mol L<sup>-1</sup> Yb<sup>3+</sup> solution with  $1.0 \times 10^{-2}$  mol L<sup>-1</sup> of EDTA.

Potentiometric titrations were performed by using the proposed electrode as an indicator electrode for the titration of 25 mL of  $1.0 \times 10^{-4}$  mol L<sup>-1</sup> Yb<sup>3+</sup> ions against  $1.0 \times 10^{-2}$  mol L<sup>-1</sup> EDTA. Titration curves is shown in Figure 5. As can be seen from Figure 5, the sensor can monitor the amount

of ytterbium ions. This electrode was also successfully applied to the determination of ytterbium in presence of metal ions mixture and the results are given in Table 4. As can be seen, the accuracy of ytterbium determination in metal ions mixture is almost quantitative.

$_{\rm Vb}^{3+}  ({\rm mol}  {\rm L}^{-1})$	Added cations (mol $L^{-1}$ )	Found <sup>a</sup> (mol L <sup>-1</sup> )	Recovery(%)
1.0×10 <sup>-6</sup>	Gd & Pr, 1.0×10 <sup>-4</sup>	$(1.03\pm0.04)\times10^{-6}$	103
1.0×10 <sup>-6</sup>	Eu & Er, 1.0×10 <sup>-4</sup>	$(0.99\pm0.06)\times10^{-6}$	99
1.0×10 <sup>-6</sup>	La & Ce, 1.0×10 <sup>-4</sup>	$(1.02\pm0.03)\times10^{-6}$	102
1.0×10 <sup>-6</sup>	Dy & Tm, 1.0×10 <sup>-4</sup>	$(1.01\pm0.05)\times10^{-6}$	101
1.0×10 <sup>-6</sup>	Lu & Nd, 1.0×10 <sup>-4</sup>	$(1.01\pm0.04)\times10^{-6}$	101
1.0×10 <sup>-6</sup>	Na & Ca, 1.0×10 <sup>-4</sup>	$(0.99\pm0.05)\times10^{-6}$	99
1.0×10 <sup>-6</sup>	Pb & Ni, 1.0×10 <sup>-4</sup>	$(0.98\pm0.03)\times10^{-6}$	98
1.0×10 <sup>-6</sup>	Cr & Fe, $1.0 \times 10^{-4}$	$(1.01\pm0.04)\times10^{-6}$	101
1.0×10 <sup>-6</sup>	K & Mg, 1.0×10 <sup>-4</sup>	$(0.98\pm0.03)\times10^{-6}$	98
1.0×10 <sup>-6</sup>	Co & Cd, 1.0×10 <sup>-4</sup>	$(1.02\pm0.06)\times10^{-6}$	102
$1.0 \times 10^{-6}$	Pb & Ca & K, 1.0×10 <sup>-4</sup>	$(1.01\pm0.05)\times10^{-6}$	101
1.0×10 <sup>-6</sup>	Fe & Na & Ca, 1.0×10 <sup>-4</sup>	$(0.99\pm0.04)\times10^{-6}$	99

**Table 4.** Determination of  $Yb^{3+}$  ion in presence of metal ions mixture.

#### **4. CONCLUSION**

The ytterbium PVC membrane sensor based on the 1,13-bis(8-quinolyl)-1,4,7,10,13pentaoxatridecane (QPOD) ligand with the composition 3% ionophore, 30% PVC, 15% OA and 52% NB exhibited the best performance characteristics with a Nernstian behavior over the concentration range of  $1.0 \times 10^{-7}$ - $1.0 \times 10^{-2}$  mol L<sup>-1</sup> Yb<sup>3+</sup>, a detection limit of  $7.5 \times 10^{-8}$  mol L<sup>-1</sup> and a fast response time of 10 s. The sensor works well in a pH range of 3.7-9.6 and it can be effectively employed for the Yb<sup>3+</sup> estimation in presence of metal ions mixture.

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