

## A Novel Potentiometric Sensor for the Determination of Iron Based on N,N'-Bis(2,4dimethoxybenzylidene)Ethylenediamine

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### Abstract

A new PVC membrane potentiometric sensor that is highly selective to Fe(III) ions was prepared by using N,N'-bis (2-hydroxybenzylidene) ethane-1,2-diamine as membrane carrier. The sensor exhibits a super-Nernstian slope of 19.5mV per decade over a  $7.3 \times 10^{-8}$  -  $1.0 \times 10^{-1}$ M concentration range. It has a response time of about 30 s and can be used for at least 8 weeks without any divergence in potentials. The proposed sensor revealed very good selectivities for Fe<sup>3+</sup> over a wide variety of other cations over a pH range 3.6–10. It was applied to the direct determination of iron in tap water samples and, as an indicator electrode, in potentiometric titrations of Fe<sup>3+</sup> ion. The electrode was also successfully applied to the speciation of iron in aqueous solutions.

**KEYWORDS:** Fe(III) ions, Ion selective electrode, PVC membrane, Potentiometry, Schiff base.

**1. Introduction:** The ion-selective electrode (ISE) dynamic response is generated by selective complexation of target ion by carrier dispersed in a poly(vinyl chloride) (PVC) matrix and useful tool for the potentiometric measurement of the activity of an ion in the presence of other ions. The ISEs based on polymeric membranes incorporated with ionophores are very useful tools for clinical, chemical and environmental analysis. Construction and application of an ion-selective electrode as a potentiometric sensor offers interesting advantages such as speed, fast response, simplicity, low cost and wide concentration range [1]. Specific metal-ligand interactions are important recognition mechanism that can be utilized in the development of potentiometric sensors [2]. Such interactions have been used in the development of cation and anion-selective electrodes based on different ionophores [3]. Until now, a large number of ionophores with the high selectivity for the specific metal ions have been developed for the potentiometric sensors for the determination of respective metal ions. A large number of PVC membrane electrodes for diverse ions such as iron were reported [4-24].

Iron is one most abundant trace mineral in the body and is the most important elements in the biological systems, playing a significant role in the oxygen transport, storage and in the electron transport [25- 26]. Iron provides a fundamental structure of haemoglobin, myoglobin, haem enzymes and many cofactors involved in enzyme activities. It is well known that an iron deficiency is the most common cause of anemia. On the other hand, too much iron can cause several health problems. High levels of iron are associated with an increased risk of cancer, heart disease and other illnesses such as haemochromatosis [27-28]. Hence, the need to iron ion determination in clinical, medicinal, environmental

and different industrial samples has created several methods to measure this analyte [29-32].

There are many methods for the detection of iron ions such as atomic absorption spectroscopy (AAS), inductively coupled plasma (ICP), etc. are employed for the determination of trace amount of iron. However, these methods are disadvantageous in terms of cost and unsuitability for routine analysis. The potentiometric methods of analysis with ion-selective electrodes are simple, cheap, convenient, fast and applicable to real samples. Hence, the development of highly sensitive and rapid methods for determination of trace amounts of iron is of considerable interest. In the present research work, I wish to introduce a highly  $\text{Fe}^{3+}$ -selective sensor based on *N,N'*-bis(2,4-dimethoxybenzylidene)ethylenediamine Schiff base as a suitable ionophore for the potentiometric determination of  $\text{Fe}^{3+}$  ions over a wide concentration range.

## 2. Experimental

### 2.1 Reagents

Reagent grade dibutyl phthalate (DBP), dioctylphthalate (DOP) sodium tetraphenyl borate (NaTPB), tetrahydrofuran (THF), AR grade ferric nitrate and high relative molecular weight PVC were purchased from E. Merck (Mumbai, MH, India) and used as received. 2,4-dimethoxybenzaldehyde and ethylenediamine purchased from Merck and Aldrich. (Mumbai, MH, India) were reagent grade materials. Chloronaphthalene (CN), Merck (Germany); *o*-nitrophenyloctyl ether (NPOE), Acros Organics (Belgium) were used and obtained. Double distilled water was used to prepare stock solution (0.1 M) of metals which was then diluted to prepare solutions of different concentrations.

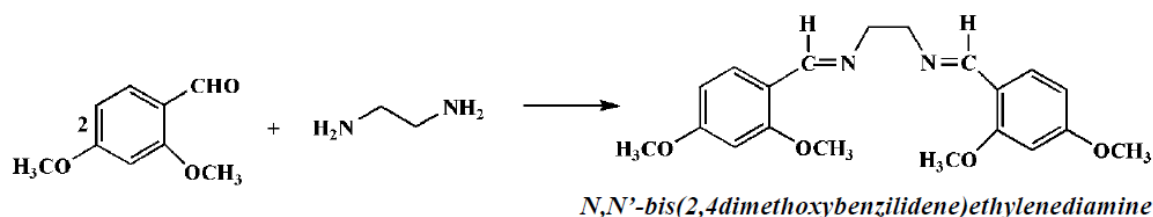
### 2.2 Apparatus

All potentiometric measurements were made at  $25 \pm 0.1^\circ\text{C}$  with a pH/mV meter [ESICO INTERNATIONAL Digital Potentiometer Model-118] using proposed sensor in conjunction with a double junction Ag/AgCl reference electrode. pH measurements were made on a digital pH meter [ESICO INTERNATIONAL Digital pH meter Model-101]. Glass electrode as a pH electrode and calomel as a reference electrode were used.

### 2.3 Synthesis of ionophores

The Schiff base *N,N'*-bis (2-hydroxybenzylidene) ethane-1,2-diamine (L) was prepared by the following reported procedure [54].

0.2 ml (3 mmol) of Ethylenediamine and 1 g (6 mmol) of 2,4-dimethoxybenzaldehyde were added to 20 ml of absolute ethanol. The reaction mixture was refluxed for 3h at  $40^\circ\text{C}$ . A powder was collected by vacuum filtration and dried overnight in vacuum. Fig.1 shows synthetic scheme of the ligand (L).



**Figure 1.** Synthetic scheme of Schiff base ligand (L).

The yield 83.0 %, m.p.159 °C. <sup>1</sup>H NMR δ<sub>H</sub> (CDCl<sub>3</sub>): 3.81 (s, 3H, OCH<sub>3</sub>), 3.83 (s, 3H, OCH<sub>3</sub>), 3.91 (s, 2H, -CH<sub>2</sub>-N=), 6.42 (s, 1H, HdAr), 6.51 (d, 1H, HbAr), 7.27 (s, 1H, HaAr), 7.88 (s, 1H, HcAr) and 8.62 (s, 1H, -CH=N-). IR (KBr, cm<sup>-1</sup>): 533, 578, 830, 1031 (ν C-O), 1124, 1165 (ν C-N), 1205, 1265 (ν C-C), 1421, 1466, 1503, 1607 (ν C=C), 1636 (ν -CH=N-), 2833 (ν C-H, Ar), 2881, 2996 (ν C-H, Me). UV-Vis. λ<sub>max</sub> (CHCl<sub>3</sub>): 275 nm, 320 nm. Anal. Calc. for L: C, 72.37; H, 4.96; N, 10.74. Found: C, 65.75; H, 6.57; N, 7.67%.

#### 2.4 Electrode preparation

The PVC based membrane was prepared by the general procedure [33]. The membrane ingredients (ionophores, anion additives, PVC and plasticizers) were dissolved in 5 mL of tetrahydrofuran(THF). After complete dissolution, the mixture was poured in the acrylic rings placed on a smooth glass plate and allow to evaporate at room temperature for 24h. Transparent membranes of about 0.1mm thickness were obtained, which were then cut to circular disc (diameter ~5 mm) and attached to one end of a Pyrex glass tube with araldite. The ratio of membrane ingredients, time of contact and concentration of equilibrating solution was optimized so that the potentials recorded were reproducible and stable. Membrane-to-membrane reproducibility was assured by following carefully the optimum condition of fabrication. The membrane that gave reproducible results and best performance was selected for detailed studies.

#### 2.5 Conditioning of membranes and Potential Measurements

The membranes were equilibrated for 3 days in 1.0M Fe(NO<sub>3</sub>)<sub>3</sub> solutions. The potentials have been measured by varying the concentration of test solution in the range 1.0×10<sup>-8</sup>M to 1.0×10<sup>-1</sup> M. The standard Fe(NO<sub>3</sub>)<sub>3</sub> solutions have obtained by gradual dilution of 0.1M Fe(NO<sub>3</sub>)<sub>3</sub> solution. The electrode potential (EMF) measurements were performed at 25°C by using the following electrochemical system

**Ag/AgCl| KC (satd.)|0.1M Fe(NO<sub>3</sub>)<sub>3</sub> ||PVC membrane || test solution| Ag/ AgCl| KCl (std.)**

A 10<sup>-1</sup>M Fe(NO<sub>3</sub>)<sub>3</sub> was taken as an inner reference solution. The performances of electrodes were accessed by measuring the EMFs of the test solutions from high (1×10<sup>-1</sup> M) to low (1×10<sup>-8</sup> M) concentrations.

The Activity coefficients of  $\text{Fe}^{3+}$  ions were calculated according to the following modified Debye-Huckel procedure, using following equation

$$\log \gamma = -0.511 z^2 [\mu^{1/2} / (1 + 1.5 \mu^{1/2}) - 0.2 \mu]$$

Where  $\gamma$  the activity coefficient,  $\mu$  is the ionic strength and  $z$  is the charge on ion.

## 2.6 Methodology

### 2.6.1. pH Measurement:

The pH dependence of the electrodes was determined by measuring the potential response of  $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{ Fe}^{3+}$  ion solution as a function of pH in the range of 1.0 to 13.0. The pH was adjusted by 0.1M solutions of HCl and NaOH.

### 2.6.2. Selectivity Determination:

In this study, IUPAC recommended fixed interference method (FIM) were used. In this method, Selectivity Coefficients was determined by a fixed concentration of interfering ion ( $a_B = 1.0 \times 10^{-2} \text{ mol L}^{-1}$ ) was added to the primary  $\text{Fe}^{3+}$  ion solutions ranging from  $1.0 \times 10^{-8}$  to  $1 \times 10^{-1} \text{ mol L}^{-1}$  and potentials were measured. The potential values obtained were plotted versus the activity of the primary ion. Potentiometric selectivity coefficients were determined graphically using the expression [34].

$$K_{\text{Fe},B}^{\text{Pot}} = \frac{a_{\text{Fe}^{3+}}}{(a_B)^{\frac{z_{\text{Fe}}}{z_{\text{Fe}}z_B}}}$$

## 3. Results and Discussion

### 3.1 Potential response study and optimization of membrane composition

The principle of the working of PVC-based membranes is selective recognition by the incorporated carrier via complex formation with the analyte ion. It is reported that the N,N'-bis(2,4dimethoxybenzilidene)ethylenediamine ligand form strong complexes with iron(III) [54], thus heterogeneous membranes of N,N'-bis(2,4dimethoxybenzilidene)ethylenediamine and PVC matrix in different ratios were fabricated and equilibrated in  $0.5 \text{ M Fe}^{3+}$  solution for 3 days to obtain stable potentials. The potentiometric response characteristics of the membrane sensors were carried out with a fixed concentration of  $1.0 \times 10^{-2} \text{ M Fe}^{3+}$  as internal solution and varying the  $\text{Fe}^{3+}$  concentration in test solutions in the range of  $1.0 \times 10^{-8}$  to  $1.0 \times 10^{-1} \text{ M}$ . In this work the potential responses of most sensitive common cations are shown in Fig.1 can be seen in most cases except for the iron ion-selective membrane electrode the slope of the corresponding potential is much lower than 59.5 and 29.5 mV per decade for the mono and divalent anions respectively. The influences of the membrane composition, the nature and amount of plasticizers and amount of NaTPB as lipophilic additives on the potential response of the proposed Fe(III) sensor with different metal ions were therefore investigated and the results are summarized in Table 1, Fig1 and Fig2. It was observed

that membrane which contained only the ligand (I) and anion excluder NaTPB in PVC matrix (sensor no. 1) in the ratio 5:75:2(I:PVC:NaTPB) (w/w, mg) exhibited a working concentration range of  $1.0 \times 10^{-5}$  to  $1.0 \times 10^{-1}$  M of  $Fe^{3+}$ . The sensor has a super Nernstian slope of 30.5 mV/decade of activity. It is well documented that plasticizers used also influence selectivity and sensitivity of ISEs. Hence, various membranes doped with different plasticizers, viz., DOP, DBP, CN and NPOE have been prepared and studied their response characteristics to see the effect of these plasticizers. The optimum composition with response characteristics of these membranes are listed in Table 1 and showed in Fig. 1. As evident from Fig. 1 and Table 1, the addition of plasticizers showed a marked improvement in the working concentration range of the sensor except DOP. The working concentration range is widened to  $5.0 \times 10^{-6}$  to  $1.0 \times 10^{-1}$ ,  $7.3 \times 10^{-8}$  to  $1.0 \times 10^{-1}$ ,  $1.9 \times 10^{-1}$  to  $1.0 \times 10^{-1}$  M with the addition of CN, NPOE and DBP, respectively, while there is no change in concentration range by adding DBP. The limit of detection for sensor no. 4 is calculated from the intersection of the two extrapolated segment of the calibration curve (Fig. 3) and is found to be  $7.5 \times 10^{-8}$  M. Since the best results were obtained for NPOE and DBP being slightly better for NPOE, since they have greater polarity, since it has a slightly higher lipophilicity and lower polarity compared with DBP and 2-NPOE.

Based on the above-mentioned response characteristics of all the examined electrodes, electrode no. 4 was chosen for all subsequent studies.

Table 1.

Composition and response characteristics of Schiff base ligand (L) based membrane electrodes selective to  $Fe^{3+}$

Sensor No.	Composition of the membrane (w/w)						Working concentration range (M)	Slope (mV/decade of activity)	Response time (s)
	(I)	PVC	CN	DOP	NPOE	DBP			
1.	5	75	---	-	-	2	$1.0 \times 10^{-5}$ - $1.0 \times 10^{-1}$	30.5	55
2.	5	75	100	-	-	-	$5.0 \times 10^{-6}$ - $1.0 \times 10^{-1}$	2	13.2
3.	5	75	-	100	-	-	$1.0 \times 10^{-6}$ - $1.0 \times 10^{-1}$	2	25.6
4.	5	75	-	-	100	-	$27.3 \times 10^{-8}$ - $1.0 \times 10^{-1}$	-	19.5
5.	5	75	-	-	-	100	$1.9 \times 10^{-5}$ - $1.0 \times 10^{-1}$	2	32.0

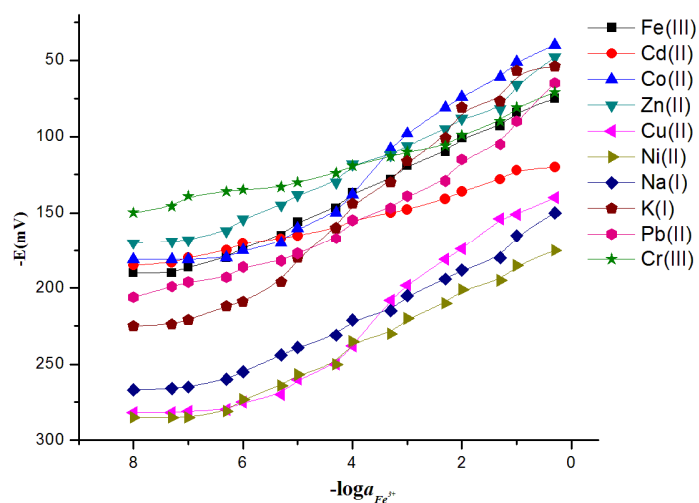


Fig.1. The potential response of anion-selective electrodes based on ligand (L)

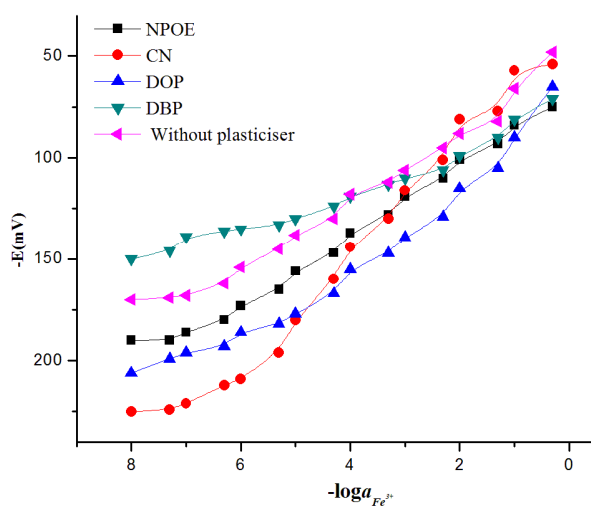


Fig.2. Potential responses of Fe<sup>3+</sup> membrane sensor based on ligand(L) with different plasticizer.

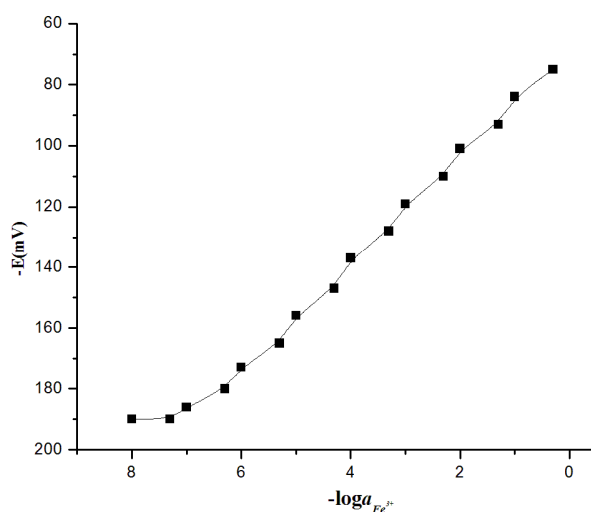


Fig.3. Calibration plot for  $Fe^{3+}$  ion selective sensor for sensor No. 4 based on ligand(L).

### 3.2 Effect of pH change

The effect of pH on the potential response of the membrane electrode was tested in the pH range 1.0 – 13.0 for  $1.0 \times 10^{-3} \text{ molL}^{-1} Fe^{3+}$  ion solution and the effect is shown in **Figure 4**. The operational range was investigated by varying the pH of the test solution with HCl/NaOH. It is seen from **Figure 4** that the potentials remains constant in the pH range 3.6 -10.0 for sensor No. 4 based on [L]. The change in potential below pH 3 is apparently due to interference of hydrogen ion and above pH 10.0 due to strong hydrolysis of Ferric ions.

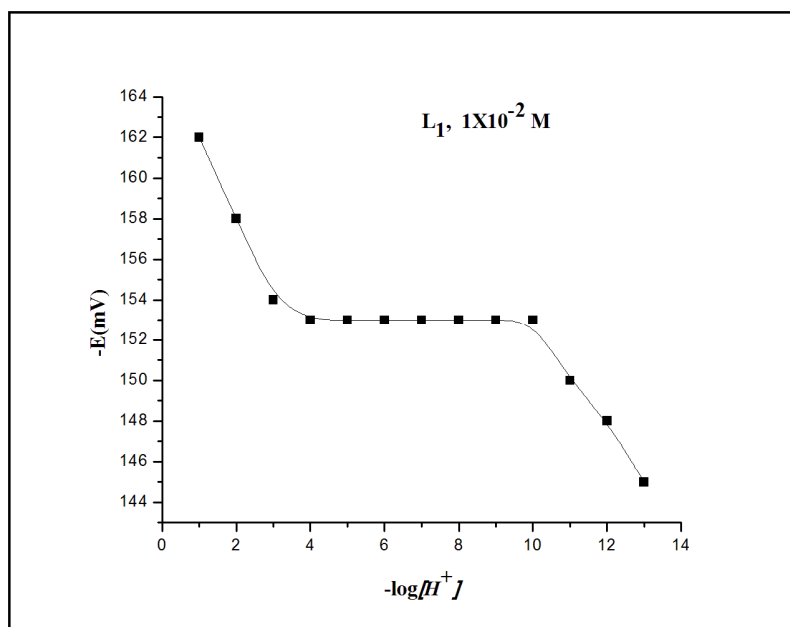


Figure 4. Effect of pH of test solution on the potential response of the  $Fe^{3+}$  ion-selective electrode.

### 3.3 Potentiometric Selectivity

Selectivity is the most important characteristics of an ISE, which defines the nature of the device and the range to which device may be successfully employed. It is determined in the terms of potentiometric selectivity coefficient ( $K_{A,B}^{Pot}$ ), which has been evaluated by the fixed interference method (FIM), using the modified form of the Nicolsky equation as suggested by Viteri and Diamond is given below.

$$K_{A,B}^{Pot} = \frac{a_A}{a_B}$$

Here  $a_A$  is the activity of the primary ion and  $a_B$  is the activity of interfering ions ( $1 \times 10^{-2}$  M)

The selectivity coefficients so calculated for the sensors based on two ionophores are summarized in Table 2. It is clear from Table 2 that the proposed sensor is selective for  $Fe^{3+}$  over many mono-, di- and trivalent cations.

Table 2.

Interfering Ions		$L_1$
$Na^+$	4.2	
$K^+$		4.0
$Co^{2+}$ 3.8		
$Mn^{2+}$ 2.5		
$Ni^{2+}$ 3.3		
$Cd^{2+}$ 3.0		
$Pb^{2+}$ 3.5		
$Cu^{2+}$ 4.7		
$Zn^{2+}$	3.4	
$Ca^{2+}$ 3.1		
$Mg^{2+}$	3.1	
$Al^{3+}$ 4.2		

### 3.4 Effect of non-aqueous solvent

The real samples may contain non-aqueous content, so the performance of the sensor was also investigated in partially non-aqueous media as 5%, 10%, 15%, and 20% (v/v) using non-aqueous content in methanol–water, ethanol–water and acetonitrile–water mixtures. The results are summarised in Table 3. It was found that the sensor did not show any appreciable change in working concentration range and slope in mixtures up to 15% (v/v)



non-aqueous content. However, above 25% non-aqueous content, the potentials showed drift with time both in working concentration range and slope which may be probably due to leaching of the ionophore at higher organic content.

**Table 3. Effect of partially non-aqueous medium on the working of Fe<sup>3+</sup> sensor based on L.**

Non-aqueous of a <sub>Fe<sup>3+</sup></sub> Content (% , v/v)	Working Concentration range (mol L <sup>-1</sup> )	Slope (mV decade <sup>-1</sup> )
0	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.5
<b>Methanol</b>		
5	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.2
10	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.3
15	1x10 <sup>-1</sup> -7.7x10 <sup>-8</sup>	19.5
20	1x10 <sup>-1</sup> -5.8x10 <sup>-6</sup>	24.5
<b>Ethanol</b>		
5	1x10 <sup>-1</sup> -7.9x10 <sup>-8</sup>	19.6
10	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.5
15	1x10 <sup>-1</sup> -7.7x10 <sup>-8</sup>	19.4
20	1x10 <sup>-1</sup> -5.9x10 <sup>-6</sup>	31.5
<b>Acetonitrile</b>		
5	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.3
10	1x10 <sup>-1</sup> -7.8x10 <sup>-8</sup>	19.5
15	1x10 <sup>-1</sup> -7.7x10 <sup>-8</sup>	19.5
20	1x10 <sup>-1</sup> -5.9x10 <sup>-6</sup>	26.5

#### 4. Analytical applications

##### 4.1 Potentiometric Titration

The utility of sensor has been further assessed by as an indicator electrode in the potentiometric titration of Fe<sup>3+</sup> solution by titrating 25 ml of 1.0 × 10<sup>-2</sup> M Fe(NO<sub>3</sub>)<sub>3</sub> against 1.0 × 10<sup>-2</sup> M EDTA solution. The pH of the solution was maintained at 4.0 throughout the titration with dil. HCl and NaOH. The titration plot obtained (Figure ) at

pH 4.0 is of standard sigmoid shape. Thus, the sensor can be used to determine  $\text{Fe}^{3+}$  ion accurately under laboratory condition.

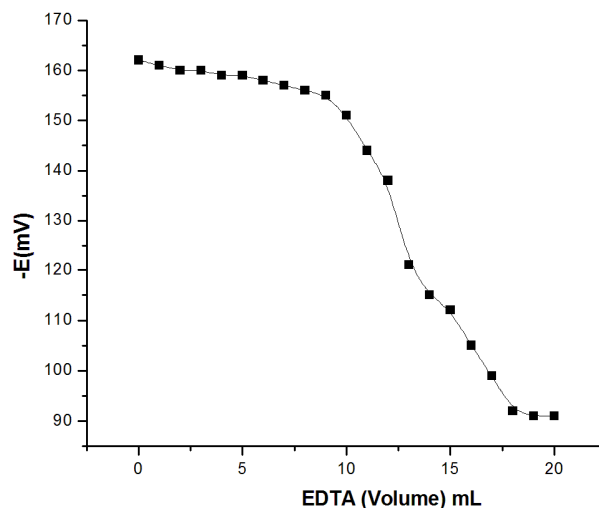


Fig.4. Potentiometric titration curve of  $10^{-2}$  M Fe (III) with EDTA solution ( $10^{-2}$  M) using the proposed electrode 4 at pH 4.

**Table 4.**Determination of Fe(III) ions in water samples

Sample	Proposed sensor (mg/L) <sup>a</sup>	AAS(mg/L)
Mineral water 1	1.6± 0.2	1.8±0.4
Mineral water 2	1.8±0.2	1.5±0.3
Waste water 1	4.5±0.3	4.1±0.2
Waste water 2	4.7±0.4	4.5 ± 0.3
River water 1	1.7±0.3	1.5±0.2
River water 2	2.4±0.2	2.3± 0.4

<sup>a</sup> The results are based on triplicates measurements

It is clear that the resulting data of the fabricated device were in satisfactory agreement with those of AAS.

### Conclusion

This work revealed that a potentiometric PVC-based membrane sensor based on ligand (L) functioned as an excellent  $\text{Fe}^{3+}$ -selective membrane sensor with a Nernstian behavior (19.5 mV/decade) in a concentration range  $7.3 \times 10^{-8}$ -  $1.0 \times 10^{-1}$  M and a fast response time of 30 s in the entire concentration range and could be used for the determination of this ion in the presence of the considerable concentrations of common interfering ions. The detection limit of the electrode is  $7.5 \times 10^{-8}$  M. Applicable pH range, lower detection limit, and potentiometric selectivity coefficients of the proposed sensor make it as a suitable device for the determinations of this ion.

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