



## FABRICATION OF ION SELECTIVE PVC MEMBRANE SENSOR FOR POTENTIOMETRIC DETERMINATION OF ESCITALOPRAM OXALATE IN ITS PURE FORM AND PHARMACEUTICAL TABLET

Ali Mohammed Atiyah<sup>1\*</sup>, Kameran Shukur Hussein<sup>1</sup>, Abdul Majeed Khorsheed Ahmed<sup>2</sup>

<sup>1</sup>Department of Chemistry, College of Science, University of Kirkuk, Kirkuk, Iraq.

<sup>2</sup>Department of Chemistry, College of Education for Pure Science, University of Kirkuk, Kirkuk, Iraq

A new PVC membrane, ion selective electrode (sensor) was constructed and used for the determination of Escitalopram oxalate (ESC) in pure form and pharmaceutical preparation; the electrode was based on the use of the ion association complex of sodium tetra phenyl borate (STPB) as an anion with ESC as a cation in polyvinyl chloride (PVC) matrix with tri-butyl phthalate (TBPH) as a plasticizer. The results of this electrode showed stability (lifetime 30 days), near a Nernstian slope of 55.9 mv/decade, over a concentration range from  $1 \times 10^{-1}$ – $1 \times 10^{-5}$  M, the electrode was found to be usable within the pH-range 3.0–6.0. This study also encompassed the measurements of the selectivity of this electrode in the existence of common cations, anions, and some drug excipients, and the selectivity factor was less than 1.0. The electrode was successfully applied to determine ESC in pharmaceutical preparation with a recovery of no less than 98.87%.

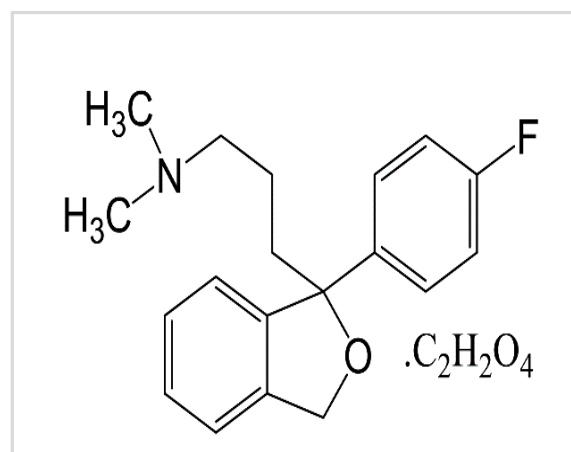
**Keywords:** Escitalopram oxalate, Potentiometric determination, PVC membrane electrode

### INTRODUCTION

Ion-selective electrodes (ISEs), essential components of the electrochemical sensor family and it is an important topic in analytical chemistry with a wide range of applications, are among the fastest-developing detection techniques<sup>1</sup>. When an ISE and a reference electrode are used together, the activity of a target ion is transformed into an electrical potential that can be spotted with a pH-meter or voltmeter<sup>2</sup>. In parse procedures, potentiometric techniques are superior to spectroscopic techniques because they are accurate, easy to prepare and use, rapid, have a large linear range, and are not impacted by the color of the sample<sup>3</sup>.

Escitalopram oxalate (ESC) is an antidepressant of the selective serotonin reuptake (SSRI) class. It is used in the treatment of major depressive disorder in adolescents and adults<sup>4</sup>. ESC is chemical, 1- [3-(Dimethyl amino) propyl]-1-(4-fluorophenyl)-13-dihydro-

5-iso benzo furan carbo nitrile oxalate<sup>5</sup>, **Fig. 1**. Its molecular formula is  $C_{20}H_{21}FN_2O.C_2H_2O_4$  with a molecular weight<sup>6</sup> of 414.40g/mol. It is a crystalline, white to off-white substance with a melting point of 152–153°C and it has a slight odor.



**Fig. 1:** The structure of ESC.

Numerous methods have been applied for the detection of ESC, such as spectrophotometry<sup>7-10</sup>, high-performance liquid chromatography (HPLC)<sup>11-17</sup>, potentiometry<sup>18</sup>, and voltammetry methods<sup>19-21</sup>. The present study, reports the development of a simple, accurate, precise, and economical potentiometric method for the quantification of ESC in pure and in tablet formulation.

## MATERIALS AND METHODS

All of the chemicals employed in the tests were of the analytical reagent grade, deionized water was utilized in each one. Escitalopram oxalate (ESC) was introduced by the state company of drug industries and medical appliances (SDI), Samara-Iraq, Losiram tablet was obtained from (Bilim Pharmaceuticals, Beyoglu, Istanbul), (STPB), (TBPH), tetrahydrofuran (THF), and polyvinyl chloride (PVC) were bought from the Fluka AG, Switzerland. A pH meter (pH Professional benchtop model BP3001) was used to test pH and potentiometric measurements, the saturated calomel electrode (SCE) served as the reference electrode. The Silver-Silver chloride model Orion 90-02 was utilized as the internal electrode, in addition to hot plate with magnetic stirrer-Germany, and ultrasonic KARL KOLB - Germany.

### Standard solution of Escitalopram oxalate

A solution of  $1 \times 10^{-1}$  M was prepared by

dissolving 4.14 g of escitalopram oxalate and making the solution up to 100 ml with deionized water. The working solutions of concentrations from ( $1 \times 10^{-1}$ - $1 \times 10^{-5}$  M) were prepared by appropriate dilution.

### Standard solution of STPB

A solution of  $1 \times 10^{-1}$  M was prepared by dissolving 3.42 g of STPB and making the solution up to 100 ml with deionized water.

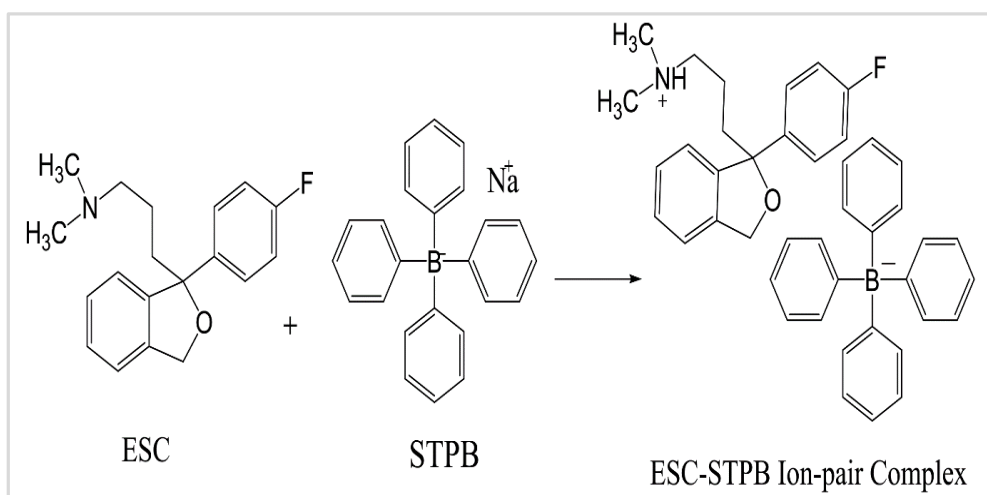
### Stock solutions of interferences

Solutions of NaCl, KCl, NaNO<sub>2</sub>, MnSO<sub>4</sub>, Sucrose, and Starch, with a concentration of  $1 \times 10^{-1}$  M, and further solutions prepared by mitigation from the stock solutions.

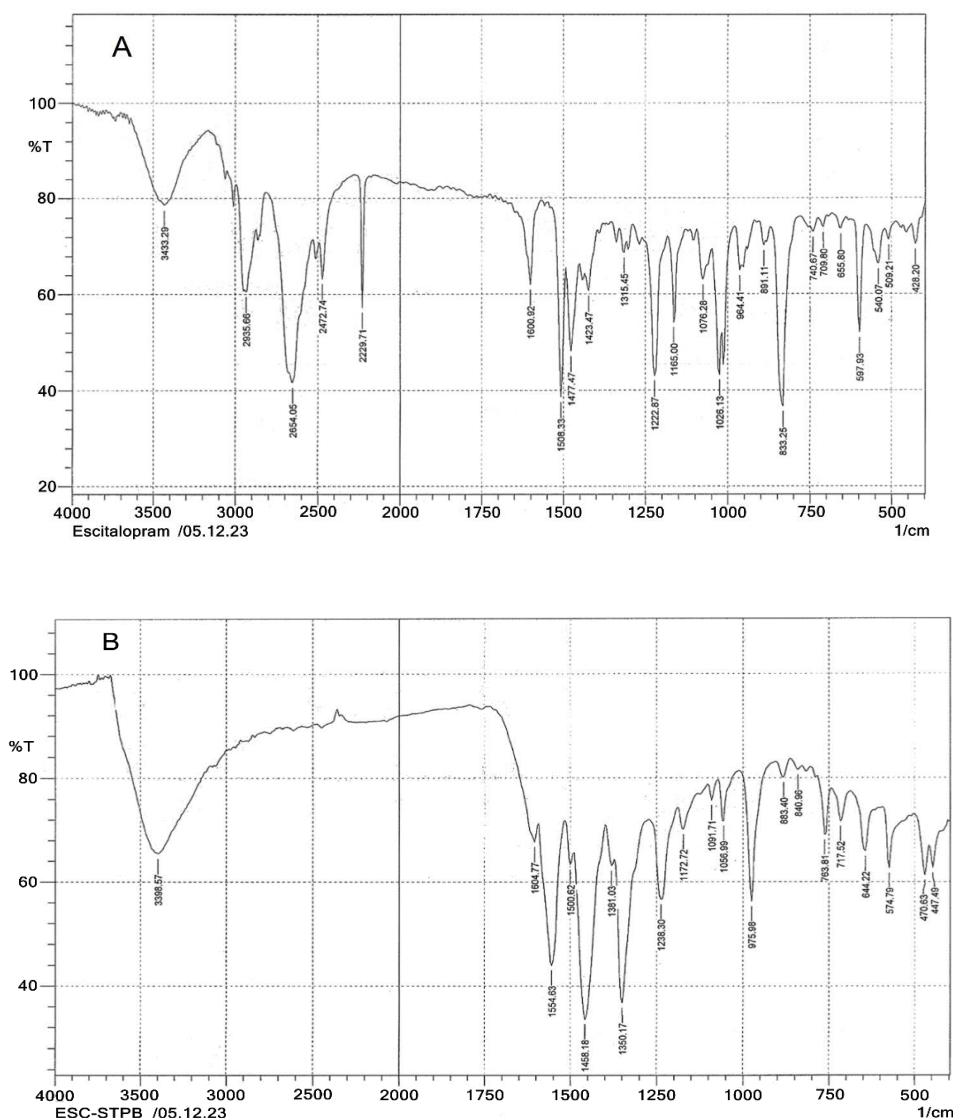
### Preparation of Ion-Pair for ESC-STPB

Mixing 50 ml of ESC  $1 \times 10^{-1}$  M solution with 50 ml of STPB  $1 \times 10^{-1}$  M solution with panning. The product was liquidated, laundered with water, and dehydrated at 25 °C for three days in an evacuated desiccator. The product is comprised of a stable 1:1 water insoluble ion association complex and a proper grain-size precipitate. (Scheme 1) shows the likely structure of ESC and STPB.

The FT-IR spectra of pure ESC (Fig. 2-A) offered peaks at 3062 and 3090  $\text{cm}^{-1}$  for C-H stretching (aromatic) vibration, 2935.66  $\text{cm}^{-1}$  for C-H stretching (aliphatic) vibration, 2229.71  $\text{cm}^{-1}$  for  $\text{-C}\equiv\text{N}$ , 1043-1272  $\text{cm}^{-1}$  for  $(\text{CH}_3)_2\text{-N-CH}_2\text{-}$ , and 550-850  $\text{cm}^{-1}$  for C-F stretching. The data shown in (Fig. 2-B) are similar to those expected from the reported study showing the functional groups for ESC with an ion-pair.



**Scheme. 1:** The process by which the ESC-STPB ion-pair complex forms.



**Fig. 2:** FT-IR spectrum of (A) pure ESC and (B) ion pair ESC-STPB.

### Construction of liquid membranes for ESC with the plasticizer and PVC

0.040 g of ESC-STPB was blended with 0.360 g of TBPH and 0.17 g of PVC. Next, while stirring, 7.0 ml of tetrahydrofuran was added till a viscous solution formed, as shown in **Fig. 3**.

This sticky mixture was spilled into a glass casting ring of 30 mm in height and 35mm in diameter. It is composed of two segments; one of them is the glass cylinder, and the other is the glass plate. The two pieces were splice together using PVC-THF mixture, and the top side of the cylinder was sheeted with a pad of filter paper. The blend was left for 48 hours to let evaporation of the solvent, after which the glass cylinder was

removed from it. The membrane thickness 0.13 mm.

### Assembling the ion-selective electrode (sensor fabrication)

A 2-3 cm PVC tube was taken, and its end was flattened by rotating it vertically on a glass plate while a few drops of THF were added to ensure there was no gap or opening between the membrane disc and the tube, then clipped a circular disk from the major membrane adequate to cover the external diameter of the PVC tube and paste it very strictly on the opening of the modified tube, making sure that there is no opening or void between the membrane disc and

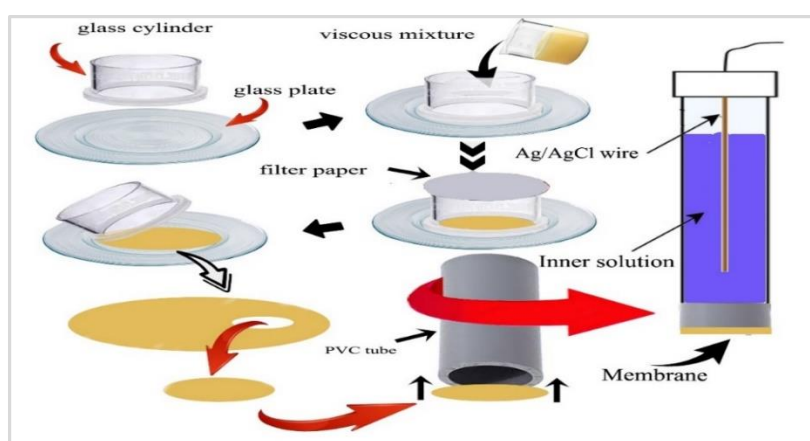
the tube. The other end of the PVC tube is pinned to the internal electrode, as seen in **Fig. 4**.

This electrode was filled by ESC as an internal solution and tied with the SCE as a reference electrode. Before beginning any

measurements, this fabricated electrode was conditioned by immersing it for 10 hours in the same concentration of the internal solution.



**Fig. 3:** Preparation of ion-selective PVC membrane sensor for ESC.



**Fig. 4:** Assembling of ESC-STPB-TBPH sensor fabrication.

**Response time of electrode**

The time it takes the sensor to attain a steady potential within ( $\pm 1$  mv) of the eventual equilibrium value is known as the response time. It was found to be shorter for concentrations ( $1 \times 10^{-1}$  M, and  $1 \times 10^{-2}$ ), while higher for concentrations ( $1 \times 10^{-3}$ ,  $1 \times 10^{-4}$ , and  $1 \times 10^{-5}$  M); this may explain why the dilute solution reaches the equilibrium on the surface of the membrane slower than the concentrated solution. It is known that several factors impact the response time, such as viscosity, temperature, and the speed of panning the measured solution. (**Table 1**) displays the electrode's response timings.

**Table 1:** Response time of electrode.

	Concentration (M)	Response time (second)
<b>ESC-STPB-TBPH electrode</b>	$1 \times 10^{-1}$	10
	$1 \times 10^{-2}$	14
	$1 \times 10^{-3}$	26
	$1 \times 10^{-4}$	34
	$1 \times 10^{-5}$	47

## RESULTS AND DISCUSSION

### Results

Ion-selective PVC membrane was prepared by the reaction of ESC with STPB, using TBPH as a plasticizer. (Fig. 5) shows a typical arrangement for measurement utilizing an ion-selective electrode.

### Concentration of internal filling solution

We implemented this study by changing the internal filling solution concentration in the range of ( $1 \times 10^{-1}$ - $1 \times 10^{-5}$  M). The measured potentials shown in (Fig. 6) indicate that the concentration of  $1 \times 10^{-3}$  is the best because it

produced the best Nernst response; thus, it was adopted as the suitable concentration.

### Impact of pH

Three diverse external solutions of the drug ( $1 \times 10^{-2}$ ,  $1 \times 10^{-3}$ , and  $1 \times 10^{-4}$ ) were prepared and modified to the desired pH values with dilute HCl or NaOH solutions. The electrode-appropriate pH range was 3.0 to 6.0 (Fig. 7). At pH under the value of 3.0, the electrode response heightened as the analyte acidity heightened; the membrane may remove the hydrogen ions, leading to rowdy responses. Because of the configuration of the non-protonated secondary amino group, the phased downward in potential remarked at a pH value above 6.0, which gives rise to a decrease in its concentration.

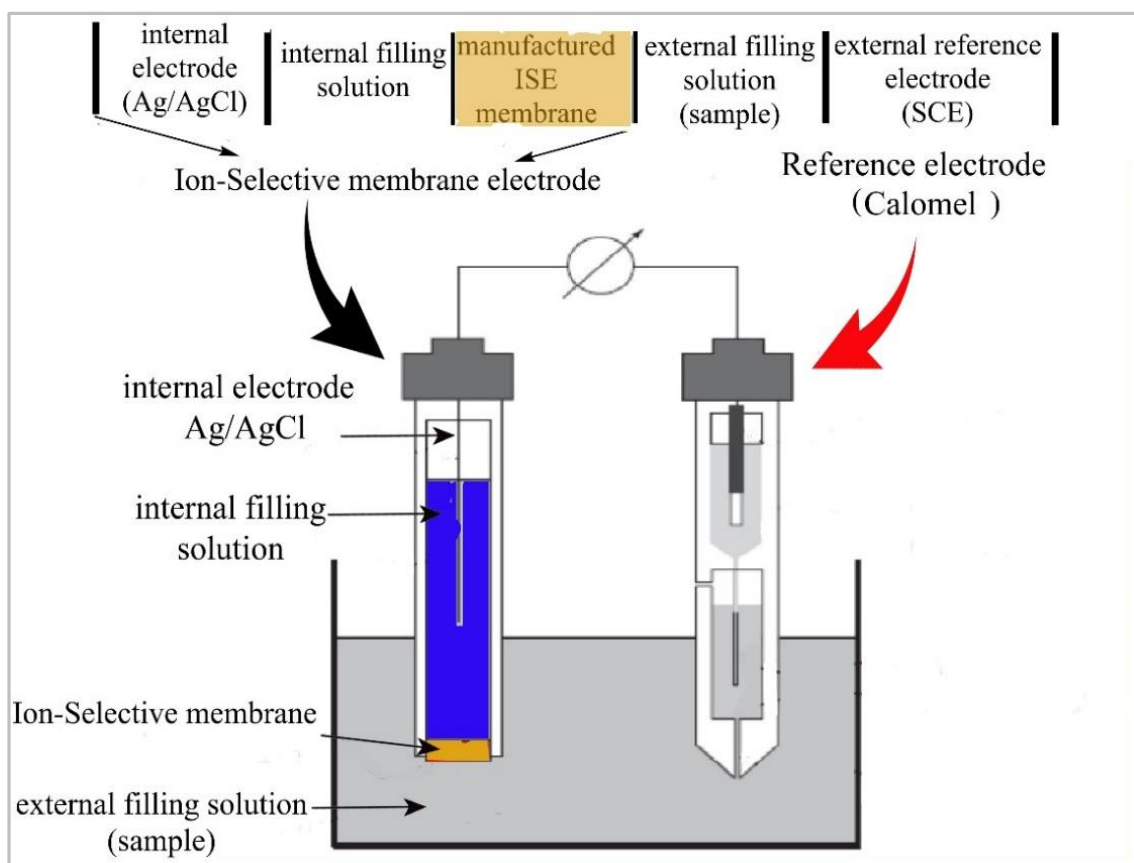
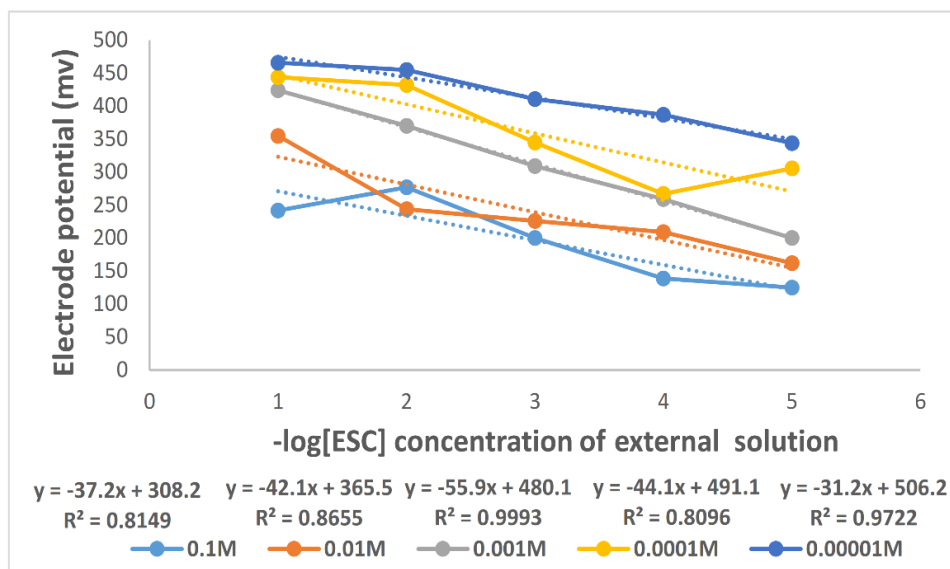
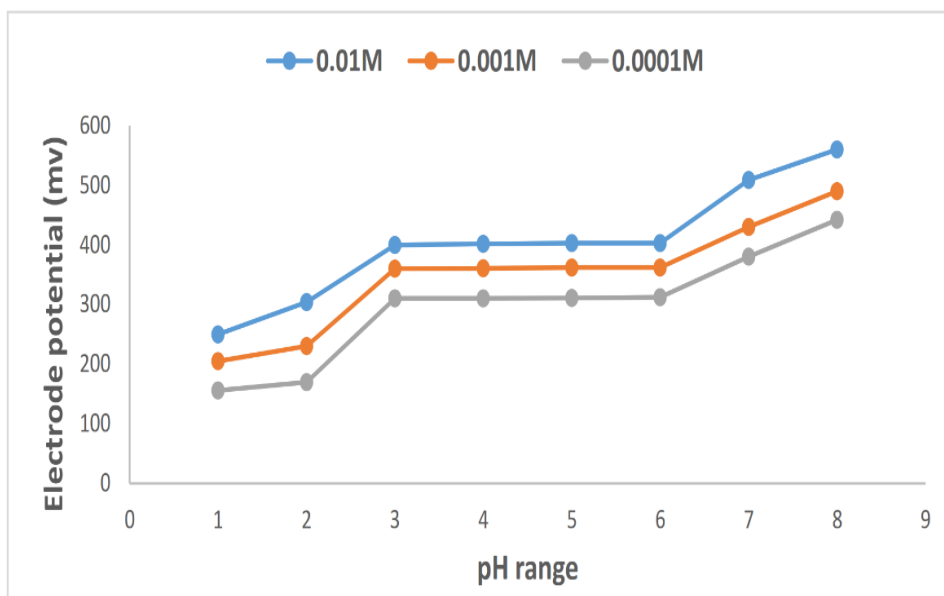


Fig. 5: Ion-selective membrane electrode in an electro-analytical cell.



**Fig. 6:** Effect of internal filling solution for ESC-STPB-TBPH electrode.



**Fig. 7:** Impact of pH on the responses of ESC-STPB-TBPH electrode.

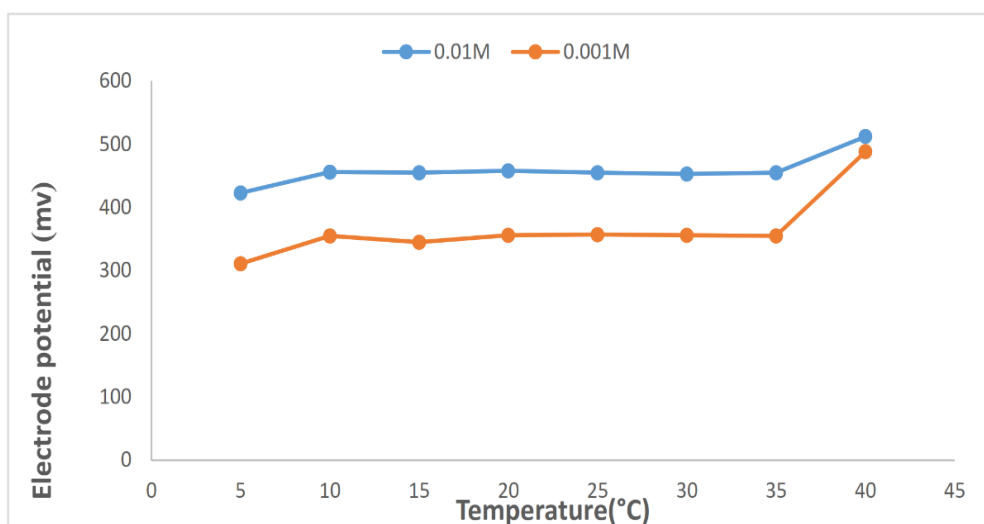
**Impact of temperature**

The findings showed that a rise in potential values was observed at temperatures above 35°C, which caused the membrane to spoil. This phenomenon may be related to both an increase in the surface district of the manufactured electrode membrane and an increase in the mobility of ESC drug solution molecules within the membrane. The results indicate that the ideal working temperature is between (15-35°C), as shown in **Fig. 8**.

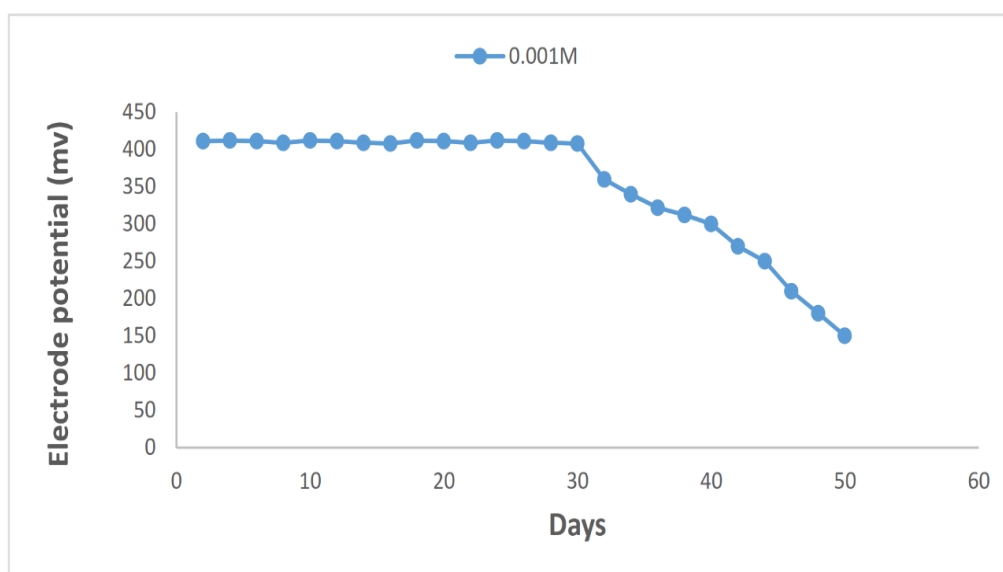
**Lifetime of electrode**

This study demonstrated that the electrode (ESC-STPB-TBPH) has a lifetime stability of about 30 days and afterward showed a negative deflection; the reason is the missing contents of the membrane (active material and plasticizer) from the polymeric layer. (**Fig. 9**) presents the results.





**Fig. 8:** Impact of temperature on the ESC-STPB-TBPH electrode's responsiveness.



**Fig. 9:** Lifetime of ESC-STPB-TBPH electrode.

### Selectivity

The selectivity factor ( $K^{pot}_{A,B}$ ) for the ESC-STPB-TBPH was calculated by the separate solution method using Nickolsky–Eisenman equation (**Equation 1**)<sup>22</sup>

$$\log K^{pot}_{A,B} = \frac{(E_B - E_A)/S + (1 - Z_A/Z_B) \log a_A}{S} \quad (1)$$

Where  $E_A$ ,  $E_B$ = potentials for the primary and interfering ions,  $Z_A$ ,  $Z_B$ = charge numbers for the primary and interfering ions,  $a_A$ = activity for the primary ions, and  $S$ = is the slope of the electrode.

The values of the selectivity factor were extremely small, which implies that these

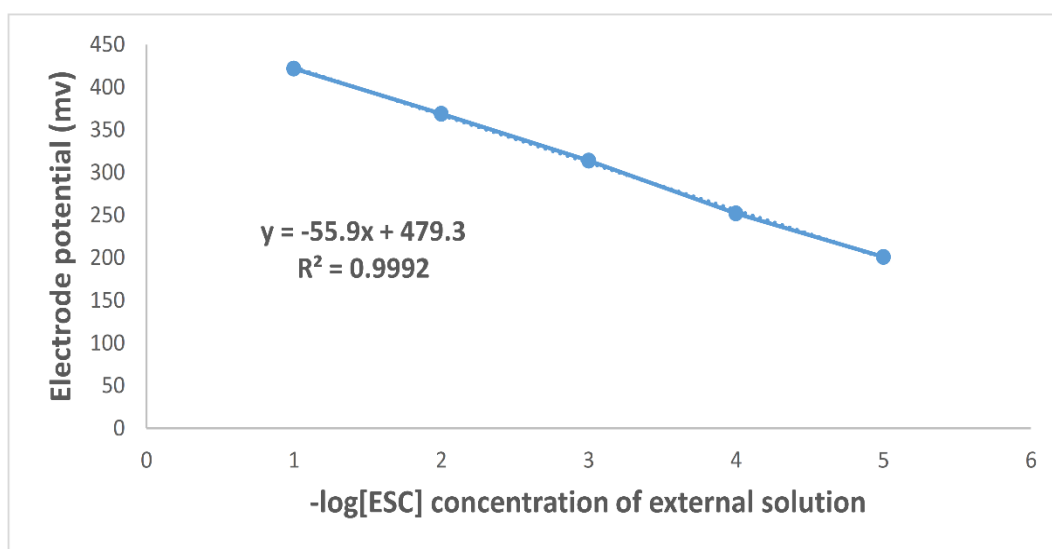
substances do not interfere with the response of the ESC electrode. The values of the selectivity factor for the electrode are outlined in **Table 2**.

### Calibration plot

After obtaining the optimal conditions of the method, the results shown in (**Fig. 10**) indicate that the plot of potentials (mv) versus -logarithm [ESC] gave a linear gradient relationship. The linear range and slope were ( $10^{-1}$ - $10^{-5}$ M), and 55.9 mv/decade, respectively. The slope is near the theoretical value (59.16 mv/decade) calculated from the Nernst equation for the uni-charged ion.

**Table 2:** Selectivity coefficient values according to separate method.

Interference ion (0.1 M)	Log $K_{A,B}$	$K_{A,B}^{pot}$
$Na^{+1}$	- 3.792	$1.6 \times 10^{-4}$
$K^{+1}$	- 3.935	$1.15 \times 10^{-4}$
$Mn^{+2}$	- 6.384	$4.1 \times 10^{-7}$
$SO_4^{-2}$	- 5.410	$3.8 \times 10^{-6}$
$Cl^{-1}$	- 4.360	$4.3 \times 10^{-5}$
$NO_2^{-1}$	- 4.007	$9.8 \times 10^{-5}$
Starch	- 4.400	$3.9 \times 10^{-5}$
Sucrose	- 4.770	$1.6 \times 10^{-5}$

**Fig. 10:** The calibration plot of ESC using ESC-STPB-TBPH electrode.**Detection limit**

The detection limit (LOD) was calculated by measuring the potential for the lowest concentration  $1 \times 10^{-5}$  M (six determinations) using the equation (Equation 2)

$$LOD = \frac{3.3 \times S \times C}{\bar{X}} \quad (2)$$

Where S= The standard deviation for the lowest concentration, C= lowest concentration,  $\bar{X}$ = Average of six determinations of potential for the lowest concentration. The results are shown in Table 3.

**Accuracy and precision**

The accuracy and precision of the method were verified by measuring the relative error (RE%), recovery, and relative standard deviation (RSD%) for five diverse concentrations ( $1 \times 10^{-1}$  -  $1 \times 10^{-5}$  M), by measuring the potentials for six determinations for each

concentration, the (RSD%) and recovery% indicates that the method is highly sensitive and exact. (Table 4) presents the findings.

**Analytical application**

The analytical applicability of the proposed ISE has been applied to determine the ESC drug in tablet formulation. Investigations were made using the standard calibration curve method. The outcomes shown in Table 5 demonstrate that the sensor is adaptable for the direct estimation of ESC in tablets, with a recovery of at least 98.87% and a relative standard deviation (RSD%) between (0.308 - 0.191).

**Comparison of the method**

The comparison between some of the analytical variables for the present method and the reported method. The results are shown in Table 6.



**Table 3:** Detection limit.

Lowest concentration (C)	Standard deviation (S)*	$\bar{X}^*$ (mv)	LOD (M)
$1 \times 10^{-5}$	2.91	205	$4.7 \times 10^{-7}$

\* Six determinations

**Table 4:** Results of accuracy and precision.

Drug Concentration M	Response (mv)	Response calculated (mv)	RE %	*RSD%	Recovery%
$10^{-1}$	422	420.6410	-0.322	0.6931	99.67
$10^{-2}$	368	367.1667	-0.226	0.3183	99.77
$10^{-3}$	310	308.6667	-0.430	0.5290	99.57
$10^{-4}$	252	254.6631	1.056	0.8482	101.05
$10^{-5}$	203	203.8333	-0.410	0.8450	99.59

\* Average of six determinations.

**Table 5:** Determination of ESC in its tablet's pharmaceutical preparations using ESC-STPB-TBPH electrode.

Concentration taken (M)	Concentration found (M)	Measured Potential (mv)*	Calculated** Potential (mv)	RSD%	RE%	Recovery %
$1 \times 10^{-2}$	$1.06 \times 10^{-2}$	366.67	368.3	0.191	-0.442	99.56
$1 \times 10^{-3}$	$9.92 \times 10^{-4}$	311.17	309.6	0.210	0.507	100.50
$1 \times 10^{-4}$	$1.13 \times 10^{-4}$	249.13	252	0.306	-1.138	98.87
$1 \times 10^{-5}$	$1.1 \times 10^{-5}$	205.33	206.7	0.308	-0.662	99.34

\*Average of six determinations. \*\*The calculated potential means the value of potential calculated from the calibration curve or the potential corresponding to the selected concentration in the calibration curve.

**Table 6:** The comparison study between the proposed and the reported method <sup>18</sup>.

Parameter	Present method	Reported method		
	PVC membrane	PVC membrane	Coated wire membrane	Coated graphite rod membrane
Active ingredient	STPB	Phosphomolybdic acid	Ammonium reineckate	Phosphotungstic acid
Plasticizer	Tri-butyl phthalate	Diocetyl phthalate	Diocetyl phthalate	Diocetyl phthalate
Slope, mv/decade	55.9	58.5	57.8	57.97
Linear range, M	$1 \times 10^{-1}$ - $1 \times 10^{-5}$	$1 \times 10^{-2}$ - $1 \times 10^{-6}$	$1 \times 10^{-2}$ - $1 \times 10^{-6}$	$1 \times 10^{-2}$ - $1 \times 10^{-7}$
Life time, day	30	40	45	40
Working pH	3-6	3-7	3-7	3-7
Working Temperature, °C	15-35	None	None	None
Detection limit, M	$4.7 \times 10^{-7}$	$5 \times 10^{-7}$	$4.8 \times 10^{-7}$	$5 \times 10^{-8}$
Correlation coefficient	0.9992	0.9999	0.9999	0.9999

## Conclusion

The suggested technique used sodium tetra phenyl borate (STPB) as an active ingredient with tri-butyl phthalate (TBPH) and PVC to produce an ion-selective electrode for measuring escitalopram oxalate. This electrode demonstrated a reasonable recovery rate and low detection limit with time stability of up to four weeks. The electrode also showed an adequate working concentration range, excellent selectivity, and quick reaction times.

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## نشرة العلوم الصيدلانية جامعة أسيوط



### تصنيع مستشعر غشاء كلوريد متعدد الفاينيل لتقدير عقار اكسالات الإسيتالوبرام جهدياً في شكله النقي وفي مستحضره الصيدلاني

علي محمد عطيه<sup>1\*</sup> - كامران شكر حسين<sup>1</sup> - عبد المجيد خورشيد احمد<sup>2</sup>

<sup>1</sup> قسم الكيمياء، كلية العلوم، جامعة كركوك، العراق

<sup>2</sup> قسم الكيمياء، كلية التربية للعلوم الصرفة، جامعة كركوك، العراق

تم بناء غشاء PVC جديد، مستشعر (قطب انتقائي للأيونات ISE) لتقدير عقار الإسيتالوبرام في شكله النقي وفي المستحضرات الصيدلانية، بالاعتماد على معقد الترابط الأيوني المتكون بين أنيونات رباعي فينيل بورات الـ صوديوم (STPB) وكاتيونات الإسيتالوبرام (ESC) باستخدام فتالات ثلاثي البيوتل (TBPH) كملدن ومتعدد كلوريد الفاينيل (PVC) ركيزة لها، بينت النتائج ثباتية القطب (العمر 30 يوم) واستجابة قريبة من القيمة النيرنستية (الميل 55,9 mV/decade) وبمدى خطي للتركيز تراوح (x10<sup>-1</sup>-1x10<sup>-5</sup> M1)، وجد أن القطب الكهربائي قابل للاستخدام ضمن نطاق الرقم الهيدروجيني (3,0 - 6,0). تضمنت الدراسة أيضاً قياس انتقائية هذا القطب بوجود مجموعة من الكاتيونات والانيونات الشائعة وبعض السواغات الدوائية ووجد أن معامل الانتقائية لجميع الأصناف المدروسة هو أقل من 1. تم تطبيق القطب بنجاح لتقدير الإسيتالوبرام في المستحضرات الصيدلانية مع استردادية لا تقل عن 98,87%.