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Fabrication of PVC Enrofloxacin-Selective Electrodes for Estimating Enrofloxacin in Pure Form and as Preparation Formula

Omar Salih Hassan¹ 

Sahar S M Alabdullah^{2*} 

Amina M. Abass² 

Ahmed Ahmed² 

¹ Department of Chemistry, College of Education for Pure Science, Tikrit University, Tikrit, Iraq

² Department of Chemistry, College of Science, Al-Nahrain University, Al-Jaderia, Baghdad-Iraq.

*Corresponding author: sahar1982@gmail.com

E-mail addresses: Omar.s.hassan@st.tu.edu.iq, amina.mohsen@nahrainuniv.edu.iq, asab1977@yahoo.com

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Abstract:

This study explored the development and qualities of the response of electrochemical properties of enrofloxacin-selective electrodes using precipitation based on producing phosphotungstic, after utilizing a matrix of polyvinyl chloride (PVC) and dibutyl phthalate or dibutyl phosphate as a plasticizer. The resulting membrane sensors were an enrofloxacin-phosphotungstic electrode (sensors 1) and an ENR-DOP-PTA electrode (sensors 2). Linear responses of (ENR-DBPH-PTA) and (ENR-DOP-PTA) within the concentration ranges of 2.1×10^{-6} - 10^{-1} and 3.0×10^{-6} - 10^{-2} mol. L⁻¹, respectively, for both sensors were observed. Slopes of 51.61 ± 0.24 and 39.40 ± 0.16 mV/decade and pH ranges equal to 2.5-8.5 and 2.0-9.0 were observed for sensors 1 and 2, respectively. The coefficients of selectivity of the created sensors demonstrated phenomenal selectivity for ENR. The proposed sensors showed useful scientific properties for the assurance of ENR in drug dosage and pure form.

Keywords: Detection limit, Enrofloxacin, Electrodes, Pharmaceutical preparations, Plasticizers, Selectivity.

Introduction:

Enrofloxacin, also called ethyl ciprofloxacin, is a synthetically blended second-generation fluoroquinolone¹. The United States Food and Drug Administration (FDA) endorsed enrofloxacin as a quinolone anti-toxin for animals and oceanic items in October 1996. Enrofloxacin displays great antibacterial action against an assortment of Gram-positive (G⁺) microscopic organisms and affects mycoplasma. The benefits of enrofloxacin include a wide range of antibacterial properties, robust bactericidal effect, quick activity, and wide-ranging dissemination in the body. In addition, enrofloxacin, as a fluoroquinolone medication for animals, kills microscopic organisms by reproducing the DNA of tissue bacteria^{2,3}. Enrofloxacin can be mixed with additional antimicrobial specialist chemicals to kill pathogenic microorganisms, and there is no cross-opposition amid different anti-toxins. In previous studies on the treatment of *Legionella pneumophila*, fluoroquinolone anti-infection agents have likewise been utilized in conjunction with macrolides⁴ and

for the treatment of bacteremia brought about by Gram-negative bacilli in combination with beta-lactam antimicrobials⁵. Fig. 1 shows the chemical structure of enrofloxacin.

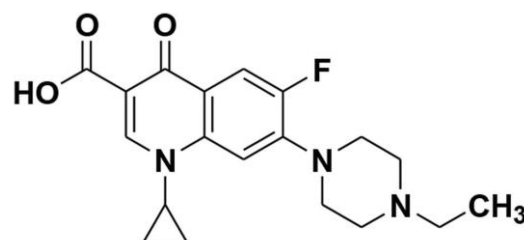


Figure 1. Chemical structure of Enrofloxacin⁶

The literature has revealed several logical strategies for quantitative assessment of enrofloxacin by spectrophotometry⁷⁻⁹, HPLC^{10,11}, fluorescence^{12,13}, and electrochemical technique¹⁴. The aim of this study is to foster an original particle-specific terminal method to determine enrofloxacin ion-selective electrodes in drug measurement. The established electrodes were effectively employed

for the potentiometric determination of enrofloxacin in pure form and in drug form, with no desire to clean up methodology primer extraction or clean up methodology. The technique has high affectability, selectivity, and exactness and can directly evaluate medication in colored and turbid solutions.

Materials and Methods:

Methods

For potential measurements, a PH-meter (HANNA) was used; for pH corrections, a pH glass electrode (HANNA) was used.

Reagents and Chemicals

Reference enrofloxacin (ENR) was kindly donated by the State Company for the Drug Industry and Medical Appliances in Samarra (IRAQ-SDI, Samarra). ENR tablets contain 150 mg per tablet supplied by the manufacturer Pharm of India. In this work, all the utilized reagents and chemicals were of analytical grade. The water used was distilled. Sodium hydroxide and hydrochloric acid were obtained from Fluka. Polyvinyl chloride (PVC), phosphotungstic acid (PTA), dibutyl phthalate (DBPH) and dibutyl phosphate (DBP), and tetrahydrofuran (THF) were obtained from Sigma-Aldrich, USA. The ions used as interferers in the selectivity coefficient were obtained from Fluka, BDH.

Standard Solutions

Standard (Stock) Solution of ENR 1×10^{-1} M

The stock solution of ENR was prepared by adding 3.595 g of ENF to a 100 mL volumetric flask, then dissolved in distilled water plus conc. HCl dropwise, then closed tightly. Solutions of 1×10^{-6} - 1×10^{-2} M of the medication ENR were newly prepared by appropriate dilute from the stock solution of ENR utilizing distilled water.

Methods

Preparation of Ion-pair Based on PVC-membrane Sensor

By using two beakers, 50 mL of ENR solution plus the solution of phosphotungstic acid was mixed with 50 mL of water 1.0×10^{-2} M. At the same time, precipitates were filtered utilizing Whatman no. 42 paper, washed with distilled water, left at room temperature then dried and ground into a fine powder. Then, analytical measurements were made.

Construction of Sensors

First, 0.40 g of ion exchanger was individually placed in glass petri dishes 5 cm diameter. Then, it was mixed with 0.36 g of

plasticizer DBPH and DOP and 0.18 g of PVC. With 6 ml of THF, the mixture was dissolved. The Petri dishes were covered with filter paper, then left to permit dissolvable dissipation at room temperature. A thickness of 0.1 mm was obtained from the main membrane. Circles ≈ 8 mm measurement were cut using a stopper drill and glued using THF plus PVC tips that were cut into the bottom of the electrode for the glass body. A 10^{-2} M solution of watery medication ENF and 10^{-2} M of KCl were mixed in equal parts and were used as a reference inner solution. As a reference inner electrode, 1 mm-thick Ag/Ag Cl wire was inundated in the reference inner solutions. For 24 hours, the sensors were maintained at equilibration in 10^{-2} M fluid medication.

Standardization of Electrodes

The pre-arranged electrodes were drenched in relation to the twofold intersection Ag/Ag Cl reference electrode in watery medication solution ENR in a range of 1×10^{-6} to 1×10^{-1} M. During stirring, they were permitted to equilibrate with the recording of the readings of e.m.f. readings. The membrane was placed in 1×10^{-2} M ENR, then washed completely with distilled water. The e.m.f. readings were recorded as a component of concentration for medication at a particular time. Adjusted charts of the recorded response of electrode mV versus log medication concentrations were drawn up. These processed relapse equations or alignment plots were utilized for producing estimations of unclear concentrations of medication ENR.

pH Effect

The impact of pH on the data of potential recommended electrodes was concentrated on inundating the electrodes in 10^{-3} M medication solution ENR in pHs ranging from 0.5 to 14.0, utilizing 0.1 M concentrations of NaOH and HCl.

Influence of Interfering

The potential response of the considered electrodes was examined from the perspective of various associated components. The coefficient of selectivity for potentiometric, $-\log(K_{Pot} \text{ main ion, interfering})$, was utilized to assess the degree to which an unfamiliar particle would interfere with the reaction of an electrode to an essential particle. The separate solutions method was used to calculate the coefficient of selectivity. The values of potentials were estimated for 10^{-3} M of watery medication solution and afterward for 10^{-3} M fluid interference solution, independently. Then, the

potentiometric coefficients of selectivity were determined utilizing the following Eq. ¹⁵⁻¹⁸:

$$\text{Log Pot.}_{K1,2} = [(E_1 - E_2) / (2.303 RT / ZAF)] + [1 - (ZA / ZB)] \log [ENR]^{-1}$$

Where E_1 , E_2 are the values of potential for 10^{-3} M solution of (ENR) interference solution; ZB, ZA are the charges of interfering ion and drug, respectively; $2.303 RT/ZAF$ represents the slope of the examined sensor (mV/concentration decade); and ENR is the activity of medication.

Potentiometric Application of the ENR Electrodes

Four potentiometric methods direct, standard, multi-standard method, and potentiometric method were applied to determine ENR by taking 10 tablets, which were weighed and reduced to powder, then independently moved to two 100 mL volumetric flasks and used to set up a 10^{-1} M watery solution of ENR. Then, by reasonable dilution with distilled water, concentrations of 10^{-3} and 10^{-4} M of the medication were obtained. The e.m.f. was documented by drenching the proposed electrode

related to the twofold intersection. The concentration of medication ENR was evaluated from the curve of calibration of the matching electrode by using the reference electrode: Ag/Ag Cl.

Results and Discussion:

The current experiment showed the possibility of quantitative evaluation of ENR by utilizing ion exchanger PTA in the membrane of the electrode utilizing PVC as a matrix. It was ascertained that the ionic exchanger had little solvency and appropriate grain size. ENR was established by mixing particle affiliation complex with PTA as demonstrated by analysis of elemental investigation and the obtained Nernstian slope, as shown in Table 1. The suggested sensors were utilized for the assurance of ENR in pure and drug form.

Properties of Sensors

The electrochemical properties of ENR electrodes, briefly detailed in Table 1, were evaluated.

Table 1. Response of electrochemical attributes of the explored electrodes

Parameter	DBPH electrode	DBP electrode
Slope (mV.decade ⁻¹)	51.61±0.24	39.40±0.16
Intercept point	446.1	481.4
Range of Conc. (mole.L ⁻¹)	2.1×10 ⁻⁶ -1.0×10 ⁻¹	3.0×10 ⁻⁶ -1.0×10 ⁻²
D.L (mole.L ⁻¹)	5.7×10 ⁻⁶	9.3×10 ⁻⁶
Correlation Coefficient R ²	0.9988	0.9968
pH range	2.0-9.0	2.5-8.5
Life time (days)	44	17

It has been accounted for that matrix of PVC is a standard help and reproducible snare for association complexes of ion in the film membrane sensors. In the current work, di-n-butyl phthalate DBPH and di-n-butyl phosphate DBP were utilized in manufacturing the proposed membrane of sensors. This changed the permittivity for the organic membranes and versatility of the ion exchanger locales. In THF, the films membranes were dissolved and left to evaporate gradually at room temperature. Sensor 1 provided the greatest sensitivity, where linearity was obtained in the

range of 2.1×10^{-6} - 1.0×10^{-1} M, with a large slope of 51.61 ± 0.24 mV/decade. Typical alignment plots are displayed in Fig 2. Likewise, sensor 2 gave a non-Nernstian slope of 39.40 ± 0.16 mV/decade with a concentration range of about 3.0×10^{-6} - 1.0×10^{-2} M. Deviation from the ideal Nernstian incline was due to the response of the electrode to the cation's activity based on the medication rather than its concentration. Limit of detection for DBPH and DBP sensors was equivalent to 5.7×10^{-6} and 9.3×10^{-6} M, respectively.

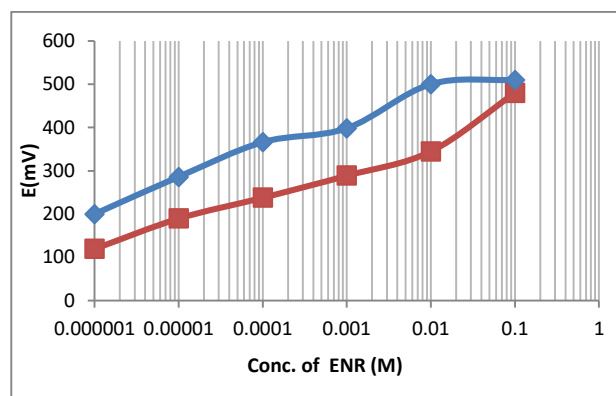


Figure 2. Calibration graph of (ENR+DBPH+PTA) and (ENR+DOP+PTA) electrodes.

Effect of pH

Ion-selective electrodes for quantitative estimations were designed to meet ideal trial conditions. An outline of potential pH, shown in Fig. 3, demonstrated that the responses of sensors 1 and 2 were genuinely consistent over the pH range 2.5-8.5 and 2.0-9.0 for sensors 1 and 2, respectively, and the steady working pH was 2.0-9.0. Hence, pH 5 was picked as an ideal pH for operation via the two sensors.

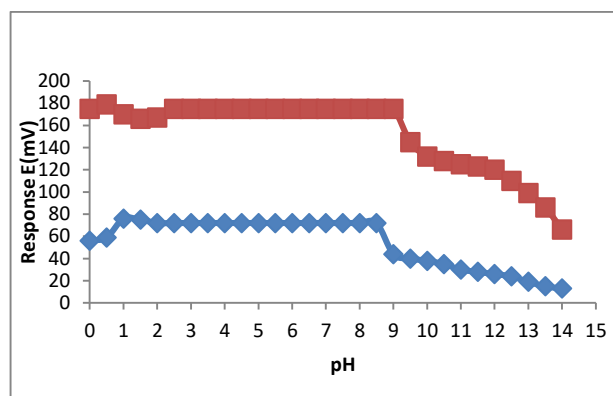


Figure 3. Values of pH for (ENR+DBPH+PTA) and (ENR+DOP+PTA) sensors at 10^{-3} M of drug.

Sensor Selectivity

Sensor 1 exhibited high values for the coefficient of selectivity, which is due to more interfering by meddling cations on the film membrane of the sensor. The upper values for the coefficient of selectivity are a result of the electrode membrane being attacked by the meddling cations. Table 2 displays the potentiometric selectivity coefficients of the sensor in the presence of some inorganic material, and the outcomes reveal that the sensor displayed great selectivity and that no critical obstruction was observed from meddling species.

Table 2. Values of selectivity coefficients of (ENR+DBPH+PTA) electrode.

Ion	Electrode (ENR+DBPH+PTA)				
	Concentration of Enrofloxacin				
	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}
Na ¹⁺	2.9241×10^{-5}	4.5337×10^{-8}	4.8238×10^{-7}	3.9270×10^{-6}	2.9241×10^{-5}
K ¹⁺	8.4029×10^{-4}	3.1728×10^{-8}	3.2285×10^{-7}	2.6283×10^{-6}	2.1397×10^{-5}
Ca ²⁺	5.6731×10^{-10}	6.7740×10^{-7}	2.7720×10^{-4}	6.4151×10^{-4}	1.4446×10^{-2}
Mg ²⁺	1.2664×10^{-9}	1.2650×10^{-6}	1.7521×10^{-3}	1.1457×10^{-3}	2.4675×10^{-2}
Fe ³⁺	5.3341×10^{-10}	8.9476×10^{-7}	4.5291×10^{-5}	1.6723×10^{-3}	6.0480×10^{-2}
Al ³⁺	3.9033×10^{-10}	6.9414×10^{-7}	3.5380×10^{-5}	1.3989×10^{-3}	5.0595×10^{-2}

Applied Potentiometric Methods for Determining ENR in Pure and Pharmaceutical Form by Using ENR Electrodes

The abovementioned sensor was useful for the evaluation ENR in pure and tablet form. The results demonstrate the suitability of the methods, as

shown in Tables 3 and 4. The results obtained by applying the potentiometric methods are presented in Figs. 4,5,6,7, which show the standard addition methods, and Fig. 8, which shows the potentiometric method by titrating the drug ENR vs. phosphotungstic acid (PT) as a titrant.

Table 3. Determination of ENR in pure drug by (ENR+DBPH+PTA) proposed electrode.

Type of Electrode	Sample	Response by potentiometric method			
		Direct	SAM	MSA	Titration
ENR+DBPH+PTA	1.0×10^{-3}	0.9446×10^{-3}	0.9577×10^{-3}	0.9652×10^{-3}	0.9894×10^{-3}
	RSD%	0.75	0.36	-	-
	Recovery %	94.46	95.77	96.52	98.94
	Error%	-5.54	-4.23	-3.48	-1.06
	1.0×10^{-4}	0.9530×10^{-4}	0.9614×10^{-4}	0.9768×10^{-4}	0.9864×10^{-4}
	RSD%	0.55	0.29	-	-
Recovery %	95.30	96.14	97.68	98.64	
Error%	-4.70	-3.86	-2.32	-1.36	

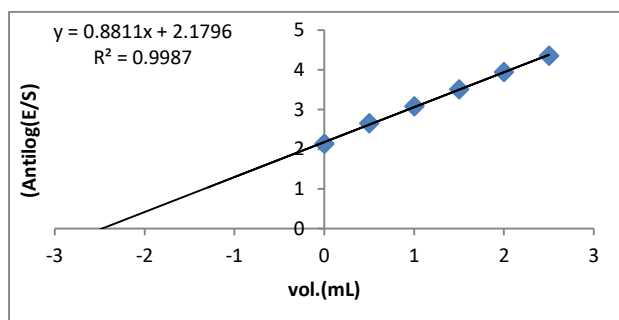


Figure 4. Potentiometric method (standard addition method) for electrode (ENR+DBPH+PTA) at concentration 10^{-3} M of ENR drug.

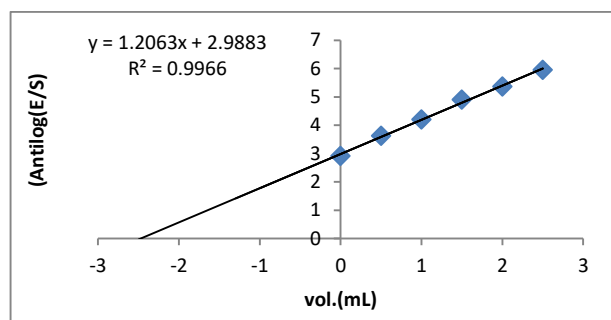


Figure 5. Potentiometric method (standard addition method) for electrode (ENR+DBPH+PTA) at concentration 10^{-4} M of ENR drug.

Table 4. Evaluation of ENR in tablet using suggested sensor.

Type of Electrode	Sample	Response by potentiometric method			
		Direct	SAM	MSA	Titration
ENR+DBPH+PTA	1.0×10^{-3}	0.9572×10^{-3}	0.9835×10^{-3}	0.9753×10^{-3}	0.9803×10^{-3}
	RSD%	0.30	0.49	-	-
	Rec%	93.72	98.35	97.53	98.03
	Error%	-4.28	-1.65	-2.47	-1.97
	1.0×10^{-4}	0.9659×10^{-4}	0.9677×10^{-4}	0.9688×10^{-4}	0.9842×10^{-4}
	RSD%	0.27	0.23	-	-
Rec%	96.59	96.77	96.88	98.42	
Error%	3.41	-3.23	-3.12	1.58	

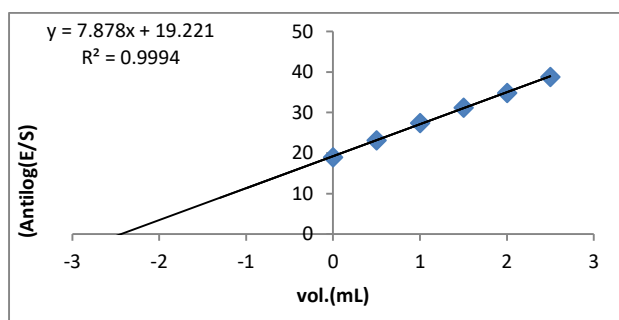


Figure 6. Potentiometric method (standard addition method) for electrode (ENR+DBPH+PTA) at concentration 10^{-3} M of ENR tablets.

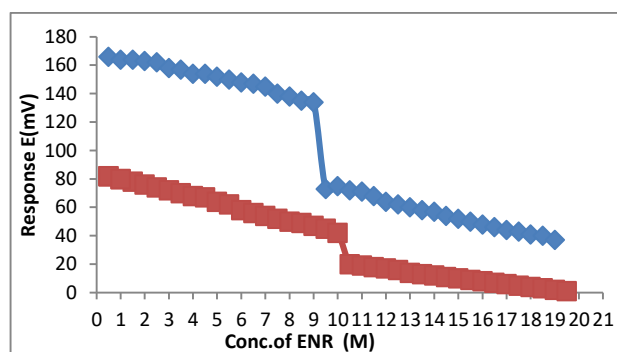


Figure 8. Potentiometric method (titration method) for electrode (ENR+DBPH+PTA) at concentration 10^{-3} M of ENR drug and PTA.

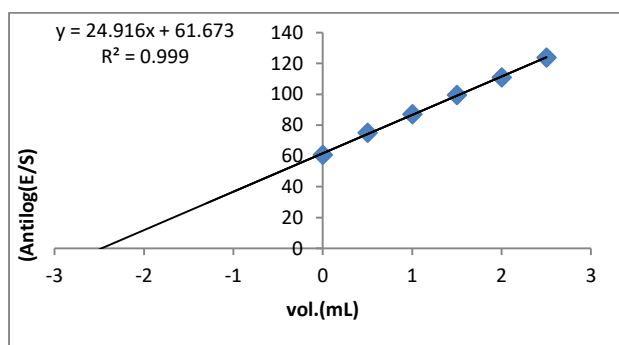


Figure 7. Potentiometric method (standard addition method) for electrode (ENR+DBPH+PTA) at concentration 10^{-4} M of ENR tablets.

The fabricated electrode was fully validated and the results showed huge success for developed electrode in quantification of ENR in pure solutions and pharmaceutical dosage units. The ability of proposed electrode to quantify the ENR concentration in the bulk dosage form was tested by measuring the concentrations of ENR in pharmaceutical formulations. The potential readings for the two concentration of drug ENR of preparation samples provided great results as obtained on the Table. 4, which ensure that the validated electrode has high selectivity.

Conclusion:

For the quantitative assurance of ENR in an unadulterated structure and in drug form, the

proposed electrodes are adequately straightforward and specific. The sensors have the advantages of rapid response, varied scope concentration and pH concentration, and direct assurance of the medication. They can thus be utilized for standard evaluation of medication in laboratories.

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Authors' Declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in Tikrit University.

Authors' Contributions Statement:

O.S.H.: investigated and wrote the original draft. A.M.A.: did the drafting. S.S.M.A.: did the data analysis and reviewed the manuscript; A.A.: reviewed the manuscript and supported with resources.

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إنروفلوكساسين الانتقائية لتقدير إنروفلوكساسين في الشكل النقي وبالصيغة PVC تصنيع أقطاب التحضيرية

أمينة محسن عباس²

سحر سمير محمد العبد الله²
أحمد عبد الرزاق أحمد²

عمر صالح حسن¹

¹قسم الكيمياء، كلية التربية للعلوم الصرفة، جامعة تكريت، تكريت، العراق.
²قسم الكيمياء، كلية العلوم، جامعة النهرين، الجادرية، بغداد، العراق.

الخلاصة:

أوجدت هذه الدراسة تطوراً لخصائص الاستجابة الكهروكيميائية للأقطاب الكهروكيميائية الانتقائية للإنروفلوكساسين باستخدام الراسب والمعتمد على إنتاج الفوسفوتونجستيك، بعد استخدام مزيج من البولي فاينيل هيدروكلورايد (PVC) وداي بيوتيل فتاليت أوداي بيوتيل فوسفيت كمواضع ملدنة. كانت أغشية المتحسسات الناتجة عبارة عن قطب كهربائي إنروفلوكساسين-فوسفوتونجستيك (متحسس 1-ENR-DBPH-PTA) و (متحسس 2-ENR-DOP-PTA). الاستجابات الخطية لأقطاب (ENR-DBPH-PTA) و (ENR-DOP-PTA) كانت ضمن المديات التركيبية 2.1×10^{-6} - 10^{-1} و 3.0×10^{-6} - 10^{-2} مول لتر⁻¹ على التوالي، والتي تم ملاحظتها لكلا المتحسسين. الانحدارات كانت 0.24 ± 51.61 و 0.16 ± 39.40 ملي فولت/حقبة. ومديات الدالة الحامضية مساوية إلى 2.5-8.5 و 2.0-9.0 التي لوحظت للمتحسسات 1 و 2، على التوالي. أظهرت أجهزة الاستشعار التي تم إنشاؤها معاملات انتقائية هائلة لعقار ENR. كما أظهرت أجهزة الاستشعار المقترحة خصائص علمية مفيدة لتقدير ENR في جرعة الدواء والشكل النقي.

الكلمات المفتاحية: حد الكشف، أقطاب إنروفلوكساسين، المستحضرات الصيدلانية، الملدنات، الانتقائية.