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Determination of the Ciprofloxacin Hydrochloride Drug in Some Pharmaceuticals using Manufactured Membrane Selective Electrodes

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ABSTRACT
This research includes estimation of the Ciprofloxacin Hydrochloride drug using manufactured liquid selective membranes electrodes , where the selective electrodes of drug were prepared from the Active ingredient with Sodium tetra phenyl borate and using organic plasticizer (Di-n-butyl phthalate, DBPH) with (poly vinyl chloride, PVC) as a substrate. The results indicated that the tendency of the electrode CIP-STPB is 29.4 mV / decade at a range of the PH (2.5-4.0) and the electrode response was good for the concentrations of the pharmaceutical drug and the concentration was 10^{-1}-10^{-4} molar, correlation coefficient 0.994, detection limit 2.5 x 10^{-8} µg.ml^{-1} and the electrode life time was fourteen days, and the electrode selectivity was measured with mono- and Dual-compounds and ions, and the electrode proved successful by estimating the drug with a regenerative not less than 98%.

Keywords: ciprofloxacin hydrochloride, membrane selective electrodes CIP-STPB-DBPH

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INTRODUCTION

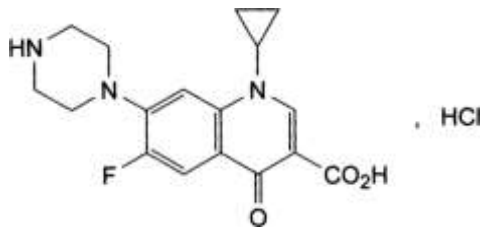
The glass electrode that responds to hydrogen ions is the beginning of the development of membrane electrodes as discovered by the scientist Cremer [(1)], and the method of selective electrodes is better than spectral methods in the analysis processes because it is fast and have a wide linear range and it is not affected by the color of the sample also it is simple, inexpensive and easy to prepare and operate [(2 3)].

In the 1960s, solid electrodes (4) with heterogeneous membranes were discovered, including the selective fluoride electrode (5).at the end of the 1960s, the liquid ion selective electrodes were used, and the calcium electrode was the first of these used electrodes (6).

The scientific name of the drug is

1-Cyclo propyl -6-floro-4-oxo-7(piperazin-1y1)-1,4-dihydroquinoline -3- carboxylic acid hydrochlorid.

The molecular formula of the drug is: $C_{17}H_{18}FN_3O_3H_2O.HCl$, its molecular weight is 385.8 and its Structural formula [(7)] is::



This drug is in the form of yellow crystals of a pale color, low in solubility in methanol and ethanol, soluble in acetone and highly soluble in water. Its melting point is 311-320 ° [(8)] and it has an absorption spectrum in distilled water λmax = 276 nm and a molar absorption coefficient of 37710 liters. Mol^{-1} cm^{-1} Ciprofloxacin hydrochloride is currently entered into the General Company for the manufacture of medicines and medical supplies - Samarra with the production of Ciprofloxacin tablets (Ciprofloxacin -500mg) in the form of a 500 mg tablet as an inflammatory treatment for arthritis, bone pain and typhoid treatment [(9,10)].

Because of the medicinal importance of the drug from a medical point of view, it was estimated by many different analytical methods such as spectral methods, liquid selective electrodes method, electrical methods and high-performance liquid chromatography technique HPLC

In this research, selective electrodes have been manufactured for this drug, and they have been used for estimation by voltage measurement.

MATERIALS AND METHODS

Instruments

1- pH meter and voltage model
JENWAY PH/mV meter 3310(Orion 91-02)/Japan
2- Calomel Electrode
Calomel Reference Electrode Fisher Scientific Company/Japan
3- Silver-Silver Chloride Electrode as internal reference electrode (Orion 90-02)
4- Hot Plate with Stirrer
JENWAY Hot Plate with Stirrer
5- Sensitive balance (with four decimal places)
Precisa 220 A Swiss made/Swiss
6- Ultrasound water bath.
Ultrasonic KARL KOLB-Germany Made/Germany

CHEMICAL MATERIALS

The used solutions

Sodium tetraphenylborate solution (10^{-1}) Molar.

It was prepared by dissolving 3.4223 grams of the substance in water in a small baker and adding it to a 100ml volumetric flask and filling the volume with water to the mark.

Pharmaceutical (CIP) solution (10^{-1}) molar.

The stocked pharmaceutical (CIP) solution was prepared at a concentration of 10^{-1} molar by dissolving 3,858 g in a 100 mL volumetric flask and complete with water to the mark. Other standard solutions (10^{-5}-10^{-2}) molar were prepared by dilution.

Ciprofloxacin Tablet Solution (500 mg), at a concentration (10^{-1}) mol.

Ten tablets of the drug (made by Samarra Pharmaceutical Company) were ground in a mortar and the Pill weight was 0.6641. A solution of 0.1 molar was prepared by dissolving 1.2809 g of ciprosam tablet content in a volumetric flask 25 ml in a volume of deionized water, then complete the volume to Mark for the purpose of obtaining the required concentrate.

Prepare the CIP drug complex with STPB solution

The complex was prepared by adding 3 ml of the drug CIP solution with 1 ml of STPB solution at a concentration of 10^{-1} for each one with continuous stirring to create a precipitate, the precipitate was filtered and washed several times with water and left for 72 hours at the laboratory temperature 25 ° C until dry.

Manufacture of selective membrane with the presence of a DBPH plasticizer

Dissolve 0.45 g of PVC in a mixture of 10 ml of acetone and 20 ml of tetrahydrofuran (THF) and add 0.1 g of the drug complex to mixture that a above prepared with continuous stirring until dissolving and using the ultrasound, add 0.43 g of plasticizer with stirring Pending homogeneity.

All the mixture was poured into glass Petridish dish (10 cm diameter) and allowed to dry for 72 hours at laboratory temperature and evenly and then the membrane was carefully raised with Tong forceps and the membrane thickness was 0.3 mm and this is consistent with what Metzger and his group [11].

Structure of the selective membrane electrode

A section of 5 cm in length and an outer diameter of 1.5 cm was cut from the PVC tube and one end was leveled by holding it upright and moving in a circular motion on a glass plate with a few drops of THF. A circular Disk is cut from the membrane with a diameter greater than the outer diameter of the PVC tube and pasted to the end of the tube with great care. The other end of the PVC tube was connected to a glass tube containing a silver-silver chloride electrode - Ag / AgCl and attached to the potentiometer and with an insulated wire. The glass tube is filled with two-thirds of it with an internal filling solution of the pharmaceutical substance and immersed for a period of time in the pharmaceutical substance solution with the same concentration of the internal filling solution and until its saturation and the ion exchange process is completed regularly and in reverse [12].

RESULTS AND DISCUSSION

Membrane electrodes were constructed for the CIP drug, relying in its preparation on the drug complex with sodium tetraphenyl borate STPB as a sensor using the DBPH plasticizer with the presence of PVC as a substrate for these electrodes and after preparing the CIP-STPB electrode with the DBPH plasticizer the properties of each were studied after fixing the optimal conditions to get the best Nernst response.

Optimal conditions

1- Effect of the internal filling solution.

A study of the electrode was conducted when changing the internal filling solution. The best concentration was 10^{-4} molar, which gives the best Nernst response, and as shown in Figure 1. This concentration gives the best experimental value of the regression and the approximation to the theoretical value, which is (mV / decade 29.5). The results are shown in the table [1].

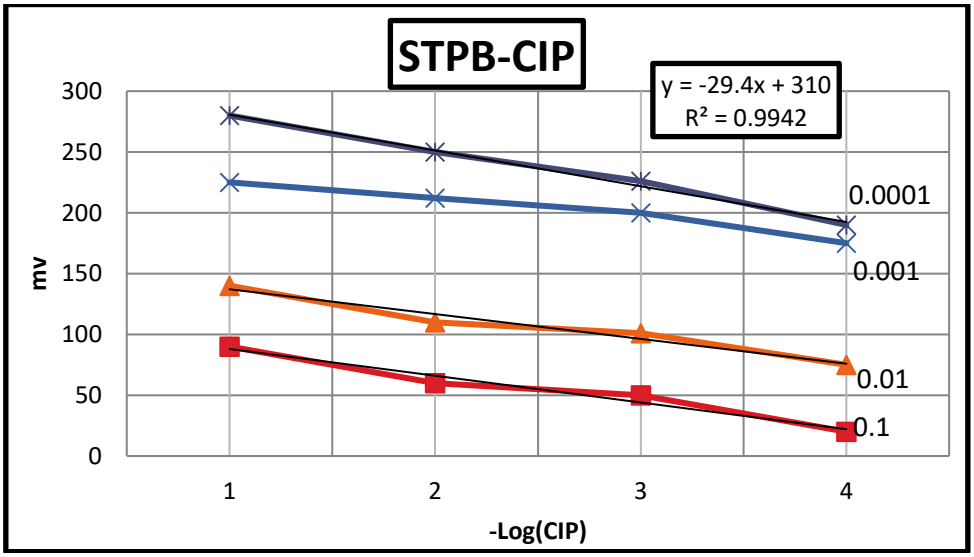


Figure 1: Effect of internal filling solution using external solutions with four different concentrations

Table 1: The best experimental value of the regression is an approximation of the value calculated in theory

Concentration Mol/L)(	10^{-1}	10^{-2}	10^{-3}	10^{-4}
The tendency)mv/decade(	22.0	20.4	16.2	29.4
correlation coefficient r^2	0.9683	0.9640	0.9661	0.9942

2- Effect of Temperature

The effect of the Celsius temperature on the response of the aforementioned electrode was studied at a concentration of 10^{-1}-10^{-3} molars five degrees for each graduation and successively. It was found that the best Celsius temperature at which the CIP-STPB electrode worked was (20-30) C° and the results are shown in Figure (2) With a noticeable increase in the values of the difference in voltage at higher temperatures, which can be attributed to the increase in the movement of the drug solution particles inside and outside the electrode, in addition to increasing the surface area of the membrane of the manufactured electrode.

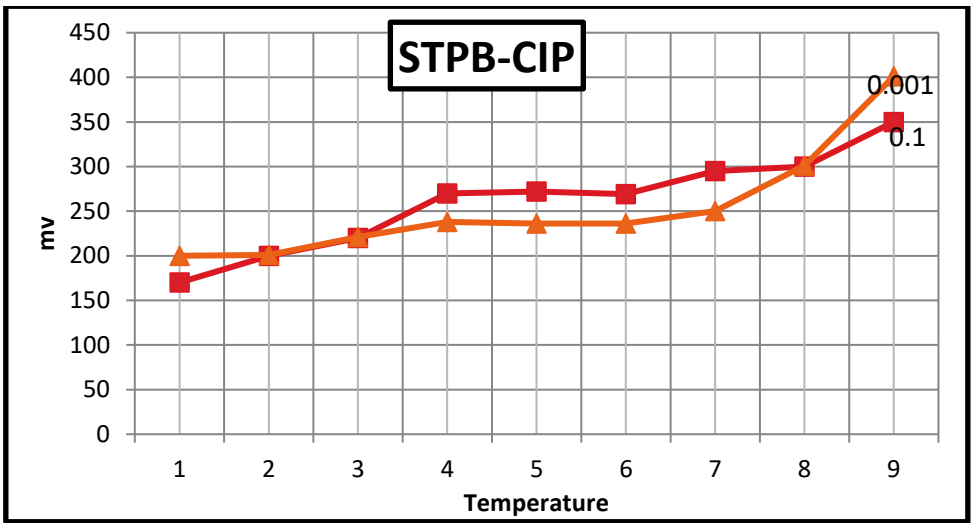


Figure 2: Effect of temperature on the electrode response using external solutions in two different concentrations

3- Effect of PH

The effect of the pH on the response of the STPB-CIP electrode was studied separately using an inner filling solution with a concentration of 10^{-4} molar to the concentrations of the outer solution of the drug (10^{-3},10^{-1}) and it was found that the best pH of the electrode CIP-STPB can be worked at a range between (2.5-4.0) and the results are shown in Figure (3). The high pH values have been neglected due to the formation of a white precipitate with the drug when using a base solution in addition to the membrane sintering while giving an irregular voltage value and possibly the cause is an alkaline error [13].

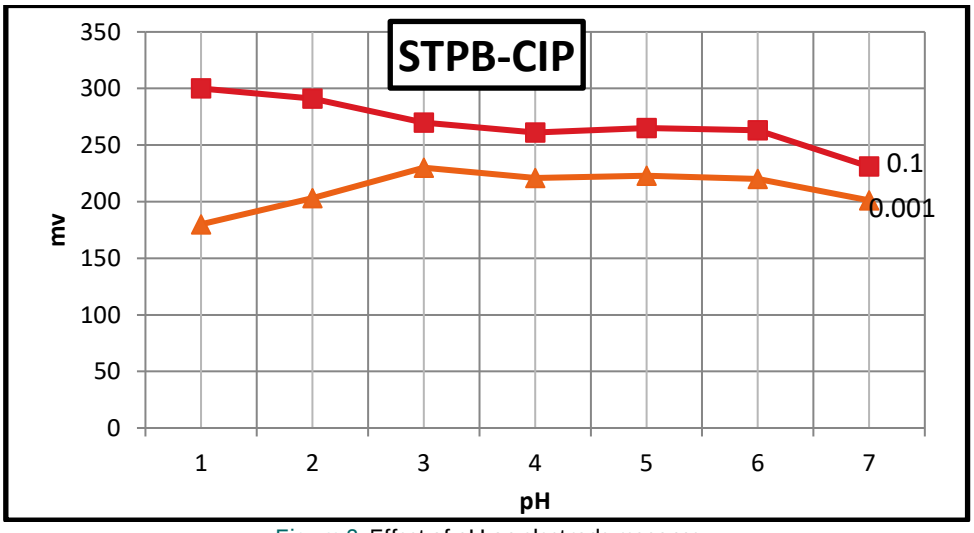


Figure 3: Effect of pH on electrode response

4- Life of Electrode

The life of the electrode was estimated by recording the potential difference using the standard drug solution at a concentration of 10^{-1} molar every two days respectively. The life of the electrode CIP-STPB was within fourteen days and after that it showed a negative deviation, as shown in Figure (4). The reason for the expiration of the electrode's life in general is due to the loss of the membrane contents (active and plasticized material) (14).

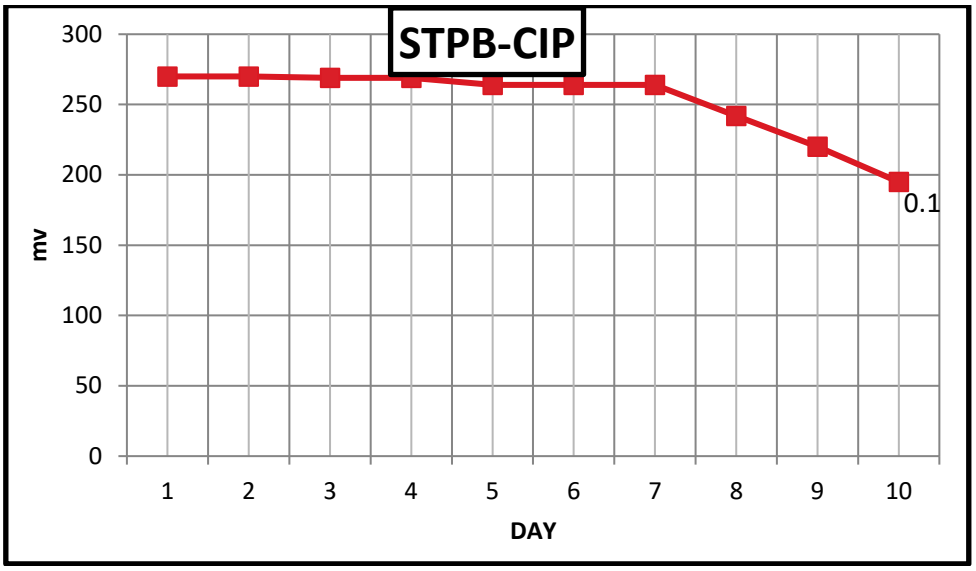


Figure 4: The effect of time on the life of the electrode

5- Standard Curve

After determining the optimal conditions, the standard curve (Figure 5) of the electrode was drawn. The detection limit for the CIP-STPB electrode was extracted and it was 2.5×10^{-8} molar as shown in the figure below.

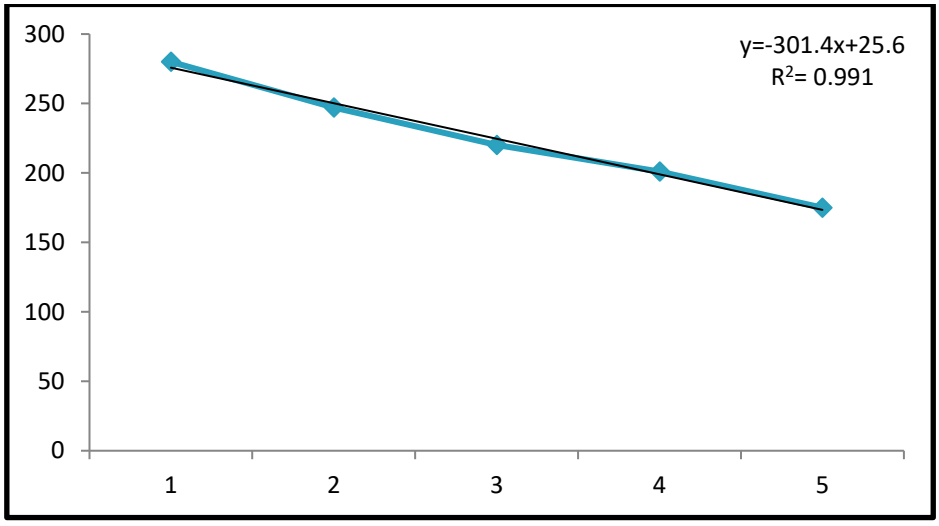


Figure 5: standard curve of electrode

6- Selectivity Measurements

The selectivity coefficient of the CIP-STPB electrode with the DBPH plasticizer was calculated using the mixed solutions method, where the electrode showed a high selectivity towards the drug without its voltage being affected by the chosen interfering ions, and this is illustrated by the selectivity coefficient values less than one [15].

b- Quantitative analysis of 500 mg sprosam tablets in a constitutional manner (16).

20 microliter of 0.05 mg / ml solution of ciprofloxacin hydrochloride were injected in the form of a preparation by taking an average weight of ten tablets dissolved in the mobile (dynamic) phase consisting of phosphoric acid 0.025 molar at PH = 3 and acetonitrile at a ratio of (6.5: 4.4) ml, respectively and at a velocity of flow. 1.5 mL / min from the mobile (dynamic) phase and column C18 type where the response (peak area) was recorded at wavelength $_{max}$ = 278 nm.

The unknown was calculated by injecting 20 microliter and at the same concentration of the standard solution, the response was recorded and the peak area was calculated and the results are shown in figures (3-5) and the concentrations were calculated by comparison as follows:

$$\% = \frac{Peak\ area^{*}\ of\ test}{Peak\ area^{*} of\ standard} \times 100$$

Application

The ciprofloxacin drug in ciprosam was estimated by applying the direct analysis method (the proposed method) and the standard (constitutional) method by measuring 20 ml for each of the prepared solutions after fixing the optimal conditions and using the CIP-STPB-DBPH electrode and from the titration curve extract the drug concentration and the results are shown in Table (2).

Table 2: results of applications by direct method of electrode

Percentage recoverability Recovery %	Standard deviation RSD%	The electrode response from the straight-line equation	*Electrode response mv	Response time (sec)	Molar concentration	Electrode type
105.57	0.08	211.22	223	43	10^{-3}	CIP-STPB-DBPH

CONCLUSIONS

The research shows the success of manufacturing selective electrodes for CIP drug with DBPH plasticizer, active substance and STPB, using PVC as the basis for these electrodes. The ability to use these electrodes to estimate the above drug shows a wide linear range of concentration, a low detection limit, and good selectivity. The drug was estimated in the preparation with a regression 105.57% .

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