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Determination of Loperamide HCL in Pharmaceutical Preparations using Modified Ion Selective Electrode

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ABSTRACT
Another electrochemical technique was utilized to examine this medication in pharmaceutical arrangements where the anode was made utilizing the graphite segment in the batteries in the wake of cleaning it well with nitric acid and drying it with acetone. After that the mixture was readied utilizing polyvinyl chloride just as the precipitant phosphomolipidlic acid corrosive and furthermore was utilized as a substance butyl phthalate is viewed as a substance Builds the intelligibility of polymeric pasta, as great outcomes are gotten, DL1.023*10^{-8}M and connection coefficient 0.9999

Keywords: electrochemical, phosphomolipidlic acid, graphite segment
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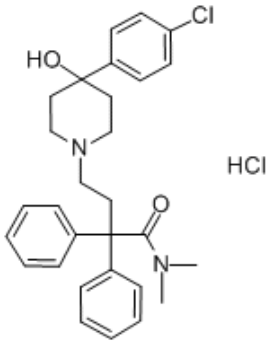
INTRODUCTION

Loperamide is a widely used antidiarrhoicopioid which is to be had without pre-scrimption(1). Initial dosage is between 4 mg and 8 mg every day (2). Subsequently, depending on man or woman response, the daily dosage can be elevated to a most of 16 mg. After ingestion the drug is extensively metabolized within the liver (3). Although N-de-methylation, oxidative N-dealkylation,and hydroxylation of the a-phenyl group have been observed(4), unique data concerning the formation and kinetics of the metabolites in people are very limited(5). One reason for this is probably the very low plasma levels, which make it difficult to determine loperamide and its metabolites quantitatively in body fluids. Because in their limits of determination maximum analytical techniques are suitable The analysis of loperamide in bulk tablets and pharmaceutical(6,7) Loperamide Hydrochloride (4-(p-chlorophenyl)-4-hydroxy-N,N-dimethyl-diphenyl-1-piperidine bytyramide monhydrochloride) (8,9) is a white powder which is slightly soluble in water, freely soluble in alcohol and in methanol. Its chemical formula is C29H34Cl2N2O2. It is a piperidine derivative [10,11] as nicely is a drug that reduces intestinal mobility and for that reason extensively used for the manipulate and symptomatic remedy of diarrhea (12). Moreover, it's been mentioned that Loperamide Hydrochloride may want to have some hobby as an ant-ihyperalgesic agent reducing pain with out causing any side impact on central fearful system (13)

STRUCTURE



EXPERIMENTAL

1- Preparing chemical solutions

0.1 molar was prepared from the pure pharmaceutical substance lepromide. As well as 0.1 molar of the precipitant phosphomolybdic acid A 0.1M solution of hydrochloric acid was prepared, as well as a 0.1M solution of sodium hydroxide and a THF solvent was used to dissolve the polymeric material used as a binder for the electrode components

2- Preparing the ion exchange complex

The ionic complex was prepared by adding the precipitating agent to the drug, where I found that the best ratio that gives the best precipitate is 1/2 ratio, which is a green precipitate of sufficient amount to make the electrode. The separation process was done by the filter paper, and the filtering process was done again for the leachate to ensure that there was no precipitate in the leachate. As 20 ml of precipitant were added to 10 ml of the drug As the spectrum below shows the complex spectrum formed

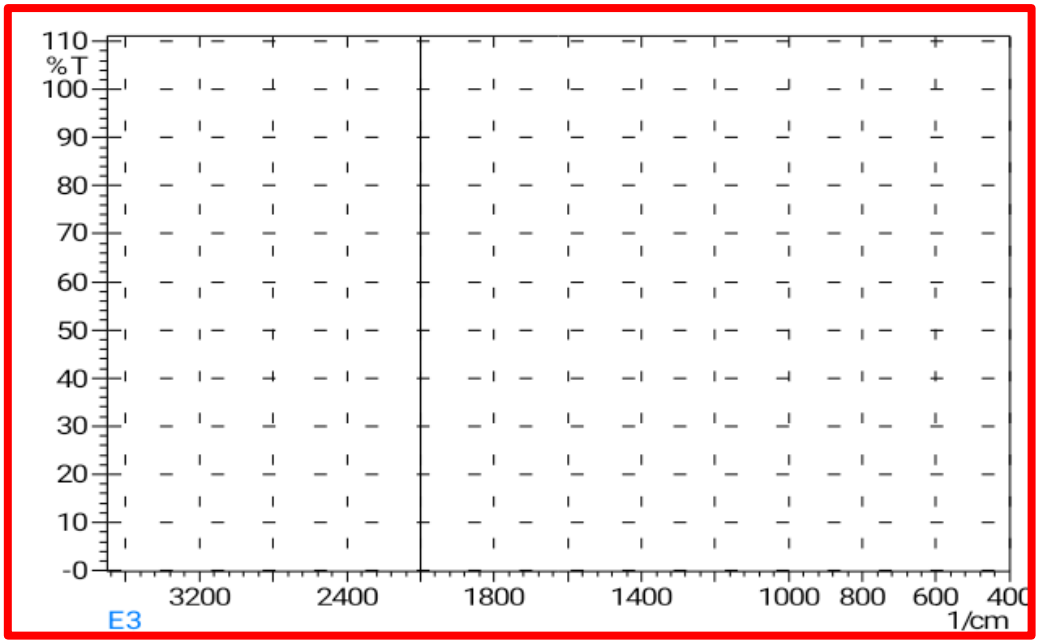


Fig 1: IR spectrum for ion exchange complex

Prepare the electrode components

This process was done by dissolving 0.5g of polyvinyl chloride in a solvent tetrahydrofuran and then adding to it 0.45 g of the complex that was prepared previously and thus left for 30 minutes and then added to the substance that gives it the flexibility which is tetra vinyl Purrette and add 5 ml of acetone to it and leave it on a motor with heating until the volume of this material decreases and it becomes 5 ml and thus we get a sticky material with a thick texture and becomes good suitable for making the membrane of the pole

3- The electrochemical sensor industry

The graphite column is taken and then perforated from the top by a hole with a depth of 3 cm and then the wire that connects to the electric cell is immersed and from the other end of the graphite column the column is immersed in the dough that was made and so it is submerged for ten consecutive times and between each time and again Leave for ten seconds, and thus we will obtain a graphite coated membrane that senses the medicinal substance. Then this electrode is immersed from the membrane side with a concentration of 0.1 molar of the medicinal substance for 6 hours to complete the ion exchange process and thus the electrode becomes ready to work after studying the factors affecting the response

4-Response time

After the manufacture of the electrode, the response time for it has been studied, and the response time is intended to be the moment when the electrode is immersed in the solution until it reaches a stable reading. Whenever the response time is few, it indicates that the electrode works well and is valid for work. The graph below shows a study of the response time of the electrode.

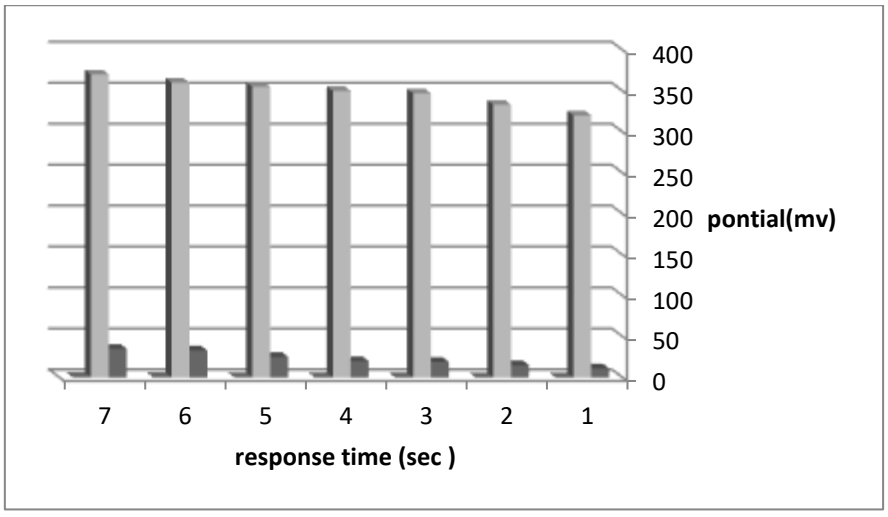


Fig 2: Response time

5-Extent of response

The extent of response to the electrode was studied by means of the voltage of a series of prepared concentrations, which is where this electrode showed a response range between the curve and the curve below, showing the response to concentrations of the processed electrode

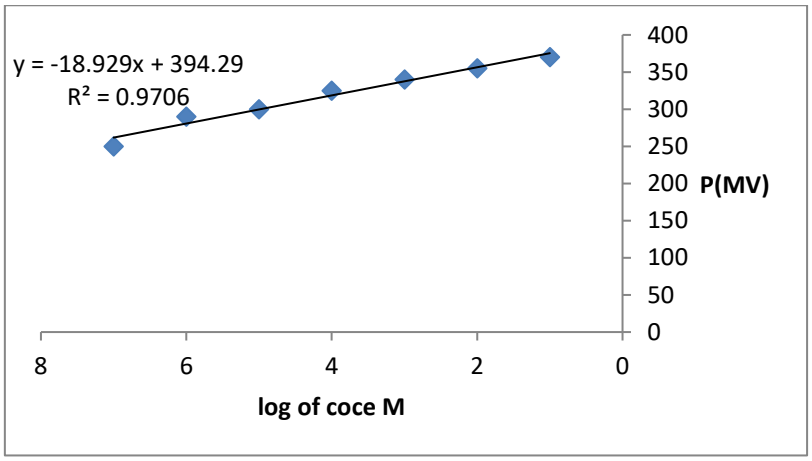


Fig 3: Extent of response

6-Study the effect of the acidity

The effect of the acidic function of the electrode was studied at a concentration of 0.001M, where the acidic and basic function was studied by adding at the beginning of the acid and then adding the base and found that the best possible range with which the electrode responds is from (2-7) and the reason for this is the acidity increase You will affect the chemical composition of the complex that gives the process of ion exchange, but the basal increase also will affect the ionic complex, and thus this response becomes disorganized with a high range of acidity and basicity As shown in the diagram below.

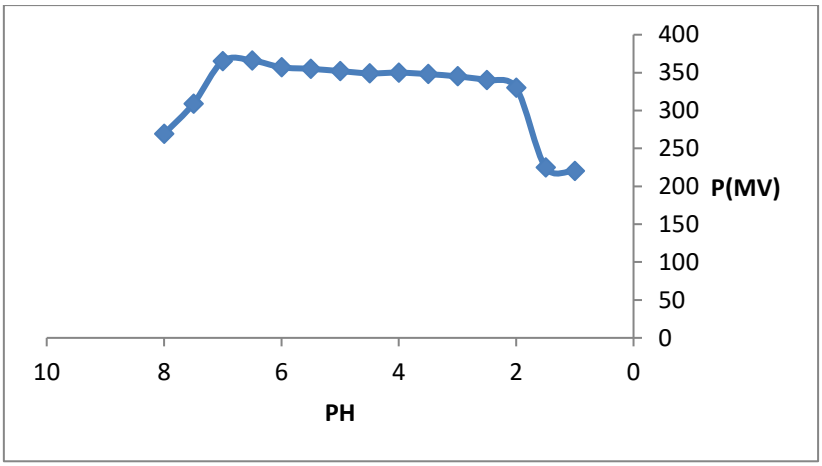


Fig 4: effect of the acidity

7-The effect of temperature on the electrode

The effects of temperature on the electrode were studied by taking 50 ml of the drug solution and placing it in the refrigerator until the surface of the solution was frozen and using the thermometer placed with the inside of the solution where the study was done from 5 ° C to 45 ° C where it was found that the electrode has a good response between temperature 15 - 40 ° C, because at high temperatures this leads to the clarity of the membrane components due to the spacing of the polymeric chains of the membrane and thus the clarity of the membrane components. As for the lower temperatures, there is a difficulty in obtaining the ion exchange process due to the freezing of the solution and this negatively affects the results that can be obtained From Electrode.

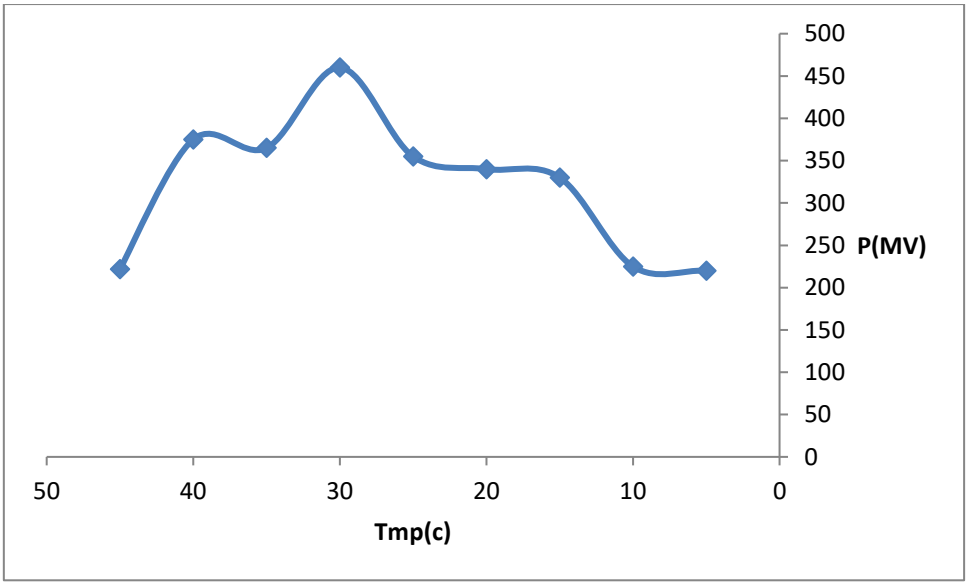


Fig 5: effect of temperature on the electrode

8-Electrode life

The age of the electrode was studied by measuring the Nernist response one time for every three days where it was found that the electrode lifetime reaches 66 days and then after that the response begins with a deviation indicating the beginning of the end of the electrode lifespan due to the clarity of the membrane components of the sensor over time and thus the response decreases as indicated in The illustration is below

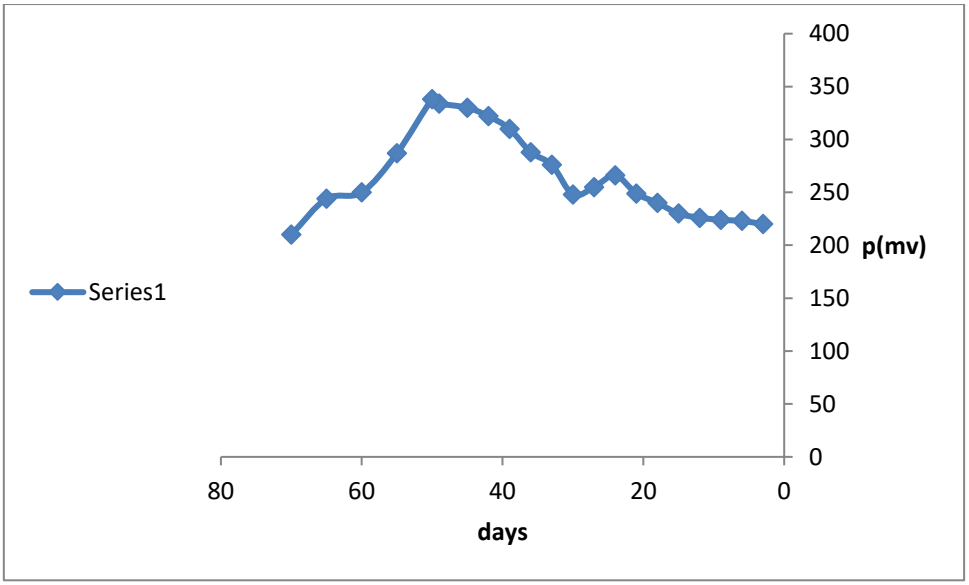


Fig 6: Electrode life

9-Calibration curve

The calibration curve was studied for concentrations from 10^{-1}M to 10^{-7}M where the concentration algorithm was drawn against the voltage and had a good correlation coefficient and good results were obtained as shown in the table below

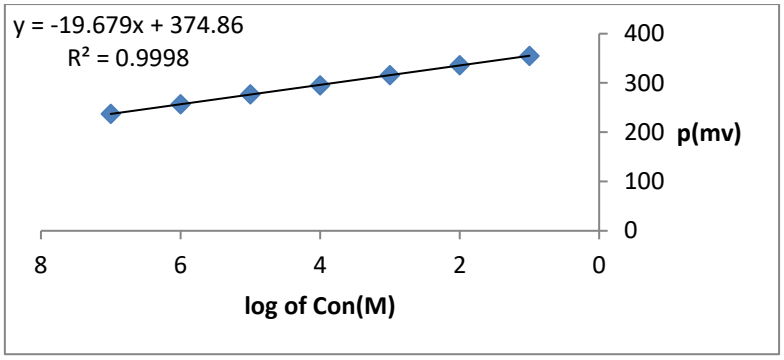


Fig 7: Calibration curve

Table 1: Statistical results of the electrode

Con(M)	The response(MV)	The response from the straight-line equation (MV)
0.1	356	355.181
0.01	335	335.502

10-Detection limit

The detection limit was calculated by the lowest response to the lowest concentration that the electrode could sense, according to the following table

Table 2: Detection limit

The lowest concentration the electrode can sense	Response rate	Standard deviation	Detection limit
10^{-7}M	236(MV)	0.4564	$1.023*10^{-8}$

Con(M)	Response of electrode	Response Straight-line equation	Relative standard deviation	Recovery
10-3	316	315.8	0.4322	99%

11-Accuracy and precision

Accuracy and compatibility were studied by taking six numbers for each response given by the electrode and for concentrations within the calibration curve

12- Application

After conducting a process of studying all the variables that affect the electrode, a study was done to estimate the pharmaceutical preparations in the drug in the commercial product of 2 mg, where ten grains were weighed and grinded all of these pills and placed in a baker and took the average weight of a grain and thus attended by a concentration and was measured by the additions method Standard and calculate the concentration of unknown substance The figure below shows the standard additions method

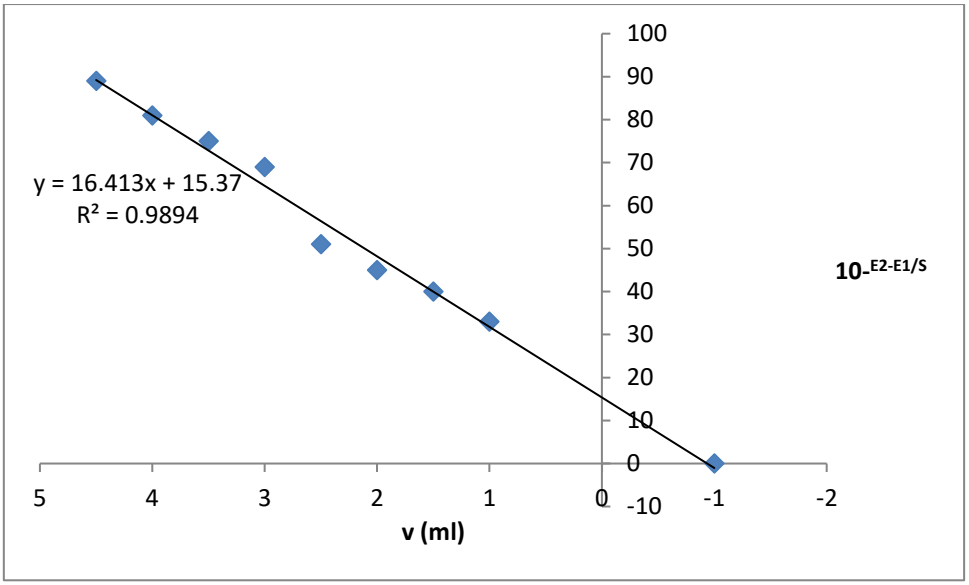


Fig 8: Standard additions method

We note that good and satisfactory results were obtained by completing the analysis process and thus can be clarified according to the following table

Table 4: Result of Standard additions method

CON(M)	Con Obtained from the electrode	REC
0.001	0.00104	102.5

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