ORIGINAL ARTICLE

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Estimation of Cyclic Voltammetry Data for $Srcl_2$, $Cacl_2$ and Their Interaction with Ceftriaxone Sodium Salt in KNO_3 Using Palladium Working Electrode

Esam A. Gomaa, Mohamed M. El-Defraway, Safa Q. Hussien

ABSTRACT

The cyclic voltammetry data for the redox reaction for strontium chloride and calcium chloride in 0.1M KNO_3 pollution were estimated. New palladium electrode was prepared and used as working electrode. Palladium electrode has already redox waves which used as analytical method for the estimation of calcium and strontium in solutions. Also, interaction of both strontium and calcium ions with drug ceftriaxone sodium salt was studied and the data obtained are used for the analytical evaluation for both calcium and strontium. The different kinetic and thermodynamic data was evaluated for the two kinds of ions alone and in the presence of ceftriaxone sodium salt and their data were discussed.

Keywords: Estimation voltammetry parameters –strontium chloride – calcium chloride – kinetic parameters- thermodynamic parameters.

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I. Introduction

Strontium is a mineral found in seawater, we get mainly from sea food, but also small amounts found in milk wheat bran, meat, poultry and vegetables.

Strontium is similar to calcium .It plays a role in how body makes new bone while it slows the breakdown of old bone .That means affect the strength of the bone. Some research proves that women with osteoporosis not absorb strontium as they need. We can buy different forms like strontium citrate at supermarkets and health stores.

There is no enough research known if strontium fight osteoporosis. In many countries like Australia a form of strontium known as strontium ranelate (Osseor, Protelos) is available as a prescription medication to treat and prevent osteoporosis and bone fractures. The drug containing strontium makes bones stronger and lowers getting fractures [1]. Strontium is the 15th most common element on the plant. It has several applications in medicine; radioactive strontium -89 is given to relieve bone pain. Strontium chloride is added to toothpaste to reduce pain in sensitive teeth [2]. Calcium is nutrient which all living organisms need, including humans. It is the most abundant element in the body and is vital for bone health.

Humans need calcium to build strong bones and most human body contain calcium in bones and teeth. Calcium plays a role in muscle movement and cardiovascular function [3].

Therefore, our aim of the work here is to give many data for strontium and calcium ions which help the biologist for the estimation of the two elements.

II. Experimental

Strontium chloride and calcium chloride are provided from Adwic Co.KNO_3 is provided from El gomheria Pharmaceutical Co.

Palladium electrode was prepared by cutting small pies from 99.99% BDH pure palladium sheet, jointed with copper wire and isolated by heat shrink polymer to avoid contact with the solutions. The electrochemical DY 2000 potentiostat in cell containing 30 ml of 0.1M KNO_3 at 19^{o}C. The three used electrodes are palladium working electrode, platinum wire counter electrode and Ag/AgCl/put in saturated KCl as reference electrode. The cell used has the symbol:

$Ag/AgCl_{(s)}$, $KCl_{(salt)}$/aqueous$_{KNO3\ 0.1M}$ /Pd E $_{palladium\ electrode}$

It is important to clean the working electrode with fine emery paper then washed well with bidistilled water.

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III. Results And Discussion

A. The electrochemical behavior of $SrCl_2$ alone in 0.1M KNO_3

The electrochemical behavior of $SrCl_2$ alone in 0.1M KNO_3 using, Palladium electrode PdE show three different wave two for reduction and one for oxidation. Actually, $SrCl_2$ form and $CaCl_2$ form peaks very far in the volt scale above 2.5V. But the waves we saw in Fig. 1 are the peaks for palladium. Then we study the effect of $SrCl_2$ on the redox waves for palladium in absence and presence of Ceftriaxone sodium salt drug.

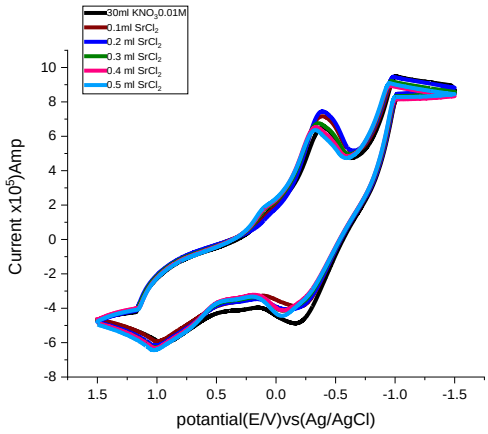

Fig. 1. Cyclic voltammograms of different $SrCl_2$ concenrations in 0.1 M KNO_3.

The reduction peaks show in Fig. 1 are due to the following reactions.

$$Pd(OH)_2 + 2e - = [PdCl4]_2 - + 2OH- \text{ at } \sim 0.3V \quad (1)$$

and

$$[PdCl2]^{2-} + 2e- == Pd^{2+} + 2Cl^- \quad (2)$$

This mechanism for the reduction processes. The oxidation mechanism is the oxidation of the second process as:

$$Pd^{2+} + 2Cl^- = [PdCl2]^{2-} + 2e- \text{ at } \sim 0.2V \quad (3)$$

We took in consideration the reversible waves the second reduction one and the oxidation peak. The redox reaction is here reversible one because ΔE is less than 0.59V.

The selecting reversible reduction and oxidation precede consuming two electrons. We noticed that all the three used waves are sharp and good defined.

Estimation of the cyclic voltammetry data:

The different equations used for the estimation of the analytical redox parameters are [4]-[10]:

$$I_P = 0.4463\ n^{3/2} F^{3/2} D^{1/2} AC / (RT)^{1/2} \nu^{12} \quad (4)$$

$$D^{1/2} = (\text{slope, } I_P \text{vs.. } \nu^{1/2}) \times (RT)^{1/2} / 0.4465 n^{3/2} F^{3/2} AC \quad (5)$$

$$\Delta E_P = E_{Pa} - E_{Pc} = 2.303RT/nF \quad (6)$$

$$\Psi = \gamma\ \alpha k_s / \sqrt{\pi nF/RT\nu Do} \quad (7)$$

$$\gamma = \sqrt{D_a/D_c} \quad (8)$$

Where ψ is the charge transfer parameters taken as one foe better approximation [11]-[16], α is the charge transfer coefficient, k_s is the standard rate constant for electron transfer, ν is the scan rate , D_a is the anodic diffusion coefficient, D_c is the cathode diffusion coefficient, F is faradays constant, T is the absolute temperature, n is the number of electrons and α =0.5 for reversible processes. A is the surface area of the working electrode.

The k_s which is the heterogeneous electron rate constant was evaluated by applying equation (9) [16]-[20].

$$k_s = 2.18\ [D\ na\ F\ \nu / RT]^{1/2} \exp[\alpha^2 nF(E_{p,c} - E_{p,a})/RT] \quad (9)$$

The electrode surface coverage was estimated for both the selected reduction and oxidation processes by using equation (10) [21]-[23].

$$\Gamma = i_P\ 4\ R\ T / n^2 F^2 A\ \nu \quad (10)$$

The quantity of electricity used for the redox reactions was estimated on applying equation (11) [24].

$$Q = n\ \Gamma\ A\ F \quad (11)$$

All the above parameters following equations (4-11) for the selected couple of peaks were calculated for $SrCl_2$ alone and tabulated in Table (1).

It was shown from Table 1 the increase in most of the estimated analytical parameters like k_s, ΓC, Q_c and Q_a parameters with the increase in $SrCl_2$ concentration favoring diffusion control reaction.

TABLE 1: Effect of different concentrations of $SrCl_2$ by using palladium electrode at 292.15 K and scan rate 0.1V.S^{-1}

[M] x10^3 mol.L^{-1}	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-) Ip,a x10^5 Amp	Ip,c x10^5 Amp	$I_{p,a}/I_{p,c}$	E° Volt	Da x10^5 $cm^2.s^{-1}$	Dc x10^5 $cm^2.s^{-1}$	αnac	Ksc x10^2	Γ c x10^9 mol.cm^{-2}	(+) Qc x10^5 C	Γ a x10^9 mol.cm^{-2}	(-) Qa x10^5 C
3.32	-0.2272	-0.3807	0.1535	-1.13	1.32	-0.8606	-0.3039	0.0159	2.15	0.7127	2.49	4.3602	1.32	-3.7528	-1.14
6.62	-0.2317	-0.3746	0.1429	-1.12	1.36	-0.8190	-0.3031	0.0039	5.82	0.6168	1.08	4.5179	1.37	-3.7003	-1.12
1.32	-0.1223	-0.2838	0.1615	-7.40	1.44	-0.5136	-0.2031	0.0004	1.65	1.0302	8.96	4.7732	1.45	-2.4515	-7.43
1.64	-0.0905	-0.2965	0.2060	-7.40	1.78	-0.4156	-0.1935	0.0002	1.62	0.7684	1.20	5.8995	1.79	-2.4524	-7.43
1.64	-0.0905	-0.2965	0.2060	-7.40	1.78	-0.4156	-0.1935	0.0002	1.62	0.7684	1.20 1.82	5.8995	1.79	-2.4524	-7.43

B. Effect of different scan rates on the cyclic voltammograms of $SrCl_2$

The effect of different scan rates 0.1,0.05 and 0.02 VSec-1 was studied on $SrCl_2$ in 0.1M KNO_3.

The data given in Fig. 2 for the relation between peaks currents and square root of scan rate is given in Fig. 2 showing approximate straight line indicating the diffusion process.

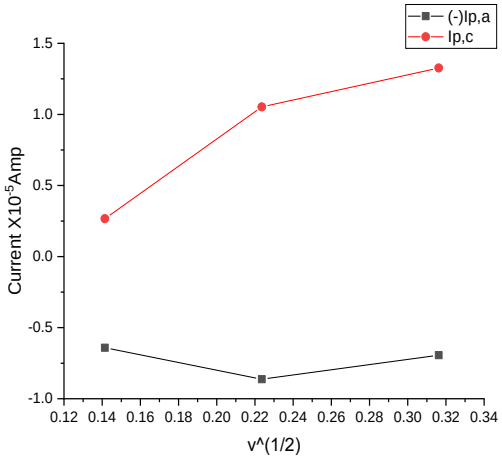

Fig. 2. The relation between peak current square root of the scan rates.

C. Effect of Ceftriaxone sodium salt drug on the electrochemical behavior of $SrCl_2$

Effect of Ceftriaxone sodium salt drug on the electrochemical behavior of $SrCl_2$ in 0.1M KNO_3 using palladium electrode at 19 °C was experimentally studied (see Fig. 3).

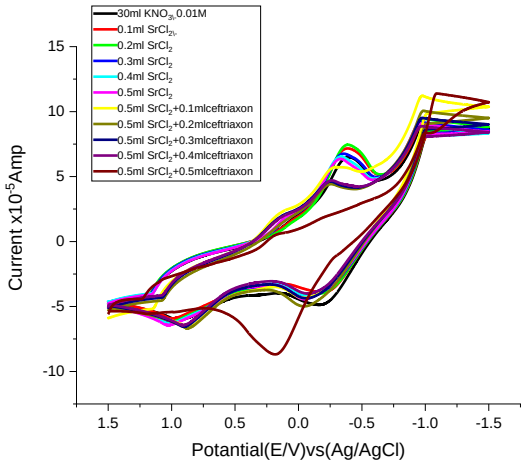

Fig 3. Cyclic voltammogram of 0.5 ml $SrCl_2$ 0.01M in Ceftriaxone (Pd electrode).

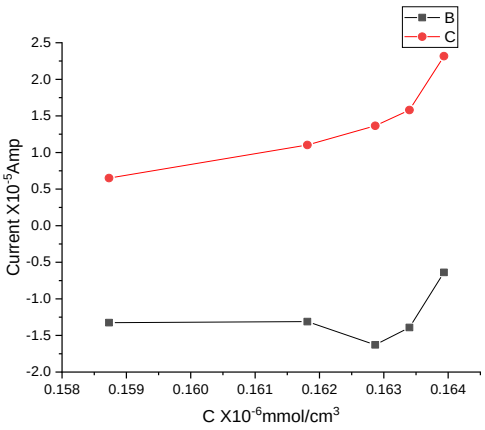

Fig. 4. Effect of 0.01M in Ceftriaxone on 0.5 ml $SrCl_2$ in 30 ml KNO_3 0.01M (Pd electrode).

Different concentrations of the ligand Ceftriaxone were studied by adding to the $SrCl_2$ solution in 0.1M KNO_3 supporting electrolyte and the data are given in Table 2 and Fig. 3. Fig. 4 shows the relation between i_P for the reduction and oxidation peaks versus $SrCl_2$ concentration which gave and approximate straight lines. The medium 0.1 M KNO_3 was illustrated in Fig. 3 with specific Pd Peaks, but on adding Cefriaxone to $SrCl_2$ specific strontium peaks were observed which here used foor the evaluation of solvation parameters. Also, more negative shift in reduction peak potential and positive shift in oxidation peak potentials on adding Ceftriaxone to $SrCl_2$ solution.

Decrease in all the limiting currents for the redox waves was observed and shift towards negative potentials for the reduction processes and more positive potential for the oxidation peak is given indicating complexion behavior.

All the estimated kinetic and cyclic voltammetry data estimated for the effect of the drug on $SrCl_2$ redox peaks show decrease in all values indicating complex reaction happened. The complexation stability constant (β) for $SrCl_2$-Ceftriaxone sodium salt complexes in 0.1M KNO_3 was calculated by the application of equation (12) [25]-[30].

$$(E_p)_c-(E_P)_M=2.303\ RT/nF\ \log\ \beta_C+2.303RT/nF\ \log\ C \qquad (12)$$

where B_c is the stability constant,$(E_P)_M$ is the peak potential for$SrCl^2$ in absence of the drug, $(E_P)_C$ is the peak potential for the complex, C is the ligand concentration (drug) and other symbols are explained before.

The Gibbs free energy of interaction between $SrCl_2$ and the drug were calculated using equation (13) [22]-[30.]

$$\Delta G= -2.303RT\ \log\ \beta_C \qquad (13)$$

TABLE 2: STABILITY CONSTANT AND GIBBS FREE ENERGIES FOR ($SrCl_2$+CEFTRIAXONE) COMPLEX

[M] x10^3 mol. L^{-1}	[L] x 10^3 mol.L^{-1}	(E_p,1/2)M	(E_p,1/2)C	ΔE v	J (L/J)	Log βj	ΔG (KJ/mol)
3.32	3.27	0.1547	0.2109	0.0561	0.9836	8.3353	-46.4670
6.62	6.51	0.1828	0.2129	0.0301	0.9837	7.5901	-42.3129
9.90	9.74	0.1974	0.1580	-0.0393	0.9837	6.2168	-34.6570
1.64	3.23	0.2006	0.1777	-0.0229	1.9677	12.3764	-68.9951

From the data given in Table 2 we conclude that the interaction between $SrCl_2$ and ceftriaxone sodium salt antibiotic forming strong complex.

From the data given in Table 2 we conclude that the interaction between $SrCl_2$ and ceftriaxone sodium salt antibiotic forming strong complex.

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D. Electrochemical behavior of $CaCl_2$ alone in 0.1M KNO_3 at 19 °C

The same medium was used for evaluating the analytical parameters for $CaCl_2$ alone in 0.1M KNO_3. Same effect as in the case of $SrCl_2$ was used and the evaluated cyclic voltammograms and analytical data are discussed.

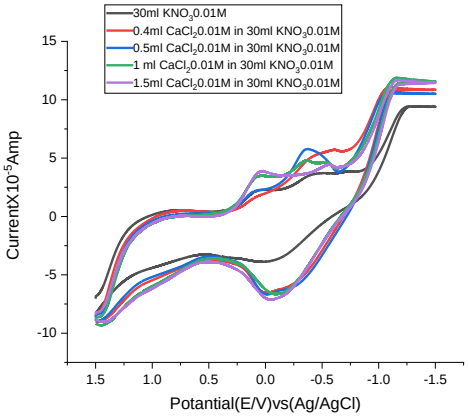

Fig. 5. Cyclic voltammogram of $CaCl_2$ 0.01M in 30 ml KNO_3 0.01 M (Pd electrode).

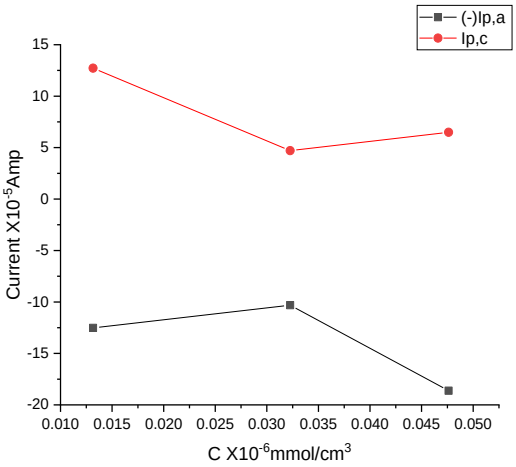

Fig. 6. Effect of Ceftriaxone sodium salt on $CaCl_2$

All the data given in Table 3 are greater than that on using $SrCl_2$ indicating more ionization and more diffusion for the calcium salt.

TABLE 3: EFFECT OF DIFFERENT CONCENTRATIONS OF $CaCl_2$ AT 295.85K AND SCAN RATE 0.1V.S^{-1} (PALLADIUM ELECTRODE)

[M] x10^3 mol.L^{-1}	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-) Ip,a x10^4 Amp	Ip,c x10^4 Amp	Ip,a/Ip,c	E° Volt	Da x10^4 $cm^2.s^{-1}$	Dc x10^4 $cm^2.s^{-1}$	αnac	ksc	Γ c x10^8 mol.cm^{-2}	(+) Qc x10^5 C	Γ a x10^8 mol.cm^{-2}	(-) Qa x10^5 C
1.32	-0.2986	-0.3665	0.0678	-1.25	1.27	-0.9836	-0.3326	0.0012	1.28	0.6100	2.40	4.2153	1.28	-4.1464	-1.26
1.64	-0.2346	-0.3199	0.0853	-1.47	2.11	-0.6988	-0.2773	0.0011	2.27	0.7415	4.18	6.9902	2.12	-4.8853	-1.48
3.23	-0.2662	-0.3393	0.0731	-1.03	4.70	-2.1927	-0.3028	0.0001	2.92	1.0805	5.06	1.5581	4.72	-3.4166	-1.04
4.76	-0.2497	-0.5339	0.2841	-1.86	6.49	-2.8694	-0.3918	0.0002	2.55	0.7827	3.30	2.1499	6.51	-6.1692	-1.87

E. Different drug

Ceftriaxone concentrations were added to $CaCl_2$ solution we observe the formation of complex between $CaCl_2$ and the drug.

The stability constant and Gibbs free energy of calculation were estimated and with smaller complexation values than that for $SrCl_2$. This means that the electrostatic complexation between the drug and $SrCl_2$ is greater than that between $CaCl_2$ and the drug. All the data given in Tables 4 are greater than that on using $SrCl_2$ indicating more ionization and more diffusion for the calcium salt whereas the data in Table 5 is smaller than $SrCl_2$ data.

TABLE 4: EFFECT OF DIFFERENT CONCENTRATIONS OF $CaCl_2$ AT 295.85K AND SCAN RATE 0.1V.S^{-1} (PALLADIUM ELECTRODE)

[M] x10^3 mol. L^{-1}	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-) Ip,a x10^4 Amp	Ip,c x10^4 Amp	Ip,a/Ip,c	E° Volt	Da x10^4 $cm^2.s^{-1}$	Dc x10^4 $cm^2.s^{-1}$	αnac	ksc	Γ c x10^8 mol.cm^{-2}	(+) Qc x10^5 C	Γ a x10^8 mol.cm^{-2}	(-) Qa x10^5 C
1.32	-0.2986	-0.3665	0.0678	-1.25	1.27	-0.9836	-0.3326	0.0012	1.28	0.6100	2.40	4.2153	1.28	-4.1464	-1.26
1.64	-0.2346	-0.3199	0.0853	-1.47	2.11	-0.6988	-0.2773	0.0011	2.27	0.7415	4.18	6.9902	2.12	-4.8853	-1.48
3.23	-0.2662	-0.3393	0.0731	-1.03	4.70	-2.1927	-0.3028	0.0001	2.92	1.0805	5.06	1.5581	4.72	-3.4166	-1.04
4.76	-0.2497	-0.5339	0.2841	-1.86	6.49	-2.8694	-0.3918	0.0002	2.55	0.7827	3.30	2.1499	6.51	-6.1692	-1.87

TABLE 5: STABILITY CONSTANT FOR ($CaCl_2$-LIGAND) COMPLEX

[L] x10^3 mol.L^{-1}	(Ep,1/2) M Volt	(Ep,1/2) C Volt	ΔE Volt	J (L/M)	Log βj	ΔG kJ mol^{-1}
3.27	-0.3918	-0.3076	0.0841	0.2	2.354	-13.124
6.51	-0.3918	-0.2694	0.1224	0.4	3.792	-21.144
9.74	-0.3918	-0.2546	0.1372	0.6	4.781	-26.655
1.29	-0.3918	-0.2657	0.1261	0.8	5.292	-29.506
1.61	-0.3918	-0.2562	0.1356	1	6.139	-34.227

F. Life Cyclic voltammograms

Here are some cyclic voltammograms like from the used instrument which explain the peaks for both calcium and strontium ions.

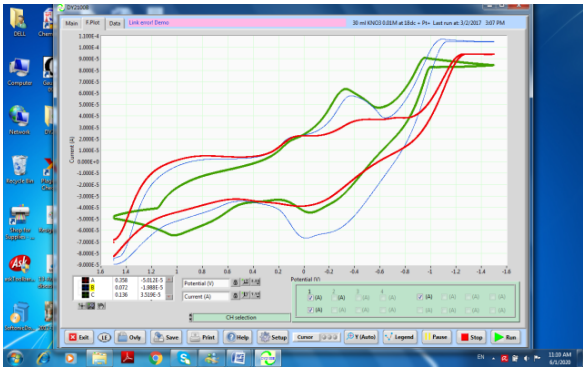

Fig. 7. Life cyclic voltammograms of medium 0.1 M KNO_3, red, plus 0.5 ml $CaCl_2$ blue, plus 0.5 ml $SrCl_2$ green at 18 °C using Pd Electrode.

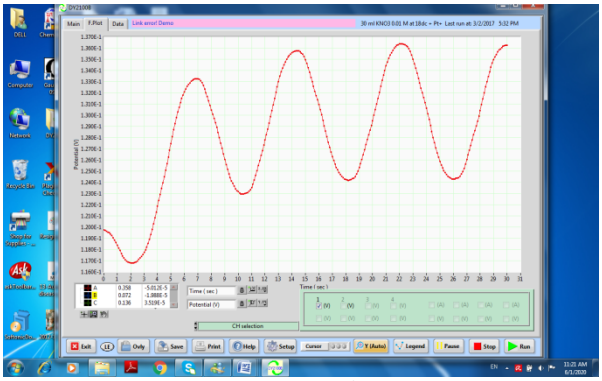

Fig. 8. Open circuit voltammetry of the medium 0.1 M KNO_3 in Pd electrode.

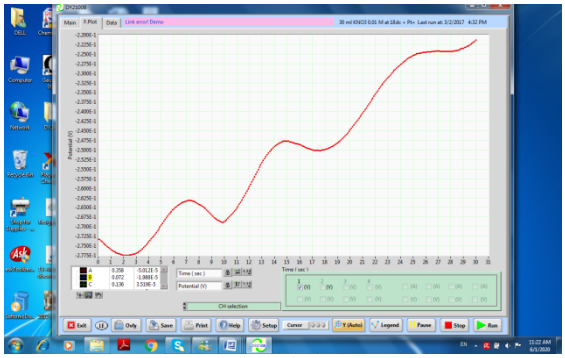

Fig. 9. Open circuit voltammetry life for 0.2 ml ceftriaxone with 0.5 ml $CaCl_2$ (0.1 M).

On comparing the open circuit voltammetry of the two Fig. 8, 9 we observe that the number of cycles decrease on using $CaCl_2$+Ceftiaxone in period 30 second than that of the medium 0.1 M KNO_3 alone indicting the decrease in diffusion processes due to complexation.

Linear sweep voltammetry: The specific reduction wave for calcium ion is appeared at -0.6 V and became clearer on adding the drug ceftriaxone sodium salt antibiotic.

Fig. 10. Life linear sweep voltammetry of medium, re, plus 0.1 ml $CaCl_2$ (0.1M) , blue, plus 0.3 ml drug.

G. Aqueous free energies of the proton in $SrCl_2$ and $CaCl_2$ solutions by adding Ceftriaxone antibiotic

Estimation of real solvation free energy of the proton from our cyclic voltammetry data is available by using Truhlar cycle [30]-[32].

We can estimate from this cycle the solution free energy of the proton. Also, the $\Delta G°_{atm}$ (atomization free energy) and the ion solvation free energy, $\Delta G°_{ion}$ together can be detected as interaction free energies ,$\Delta G°_{rxn}$ and their values can be estimated [33].

When we study the cycle given above, we can estimate the ESHE the standard hydrogen electrode potential under our conditions applying equation (14) and the $\Delta G°_{ion}$ under the complex reaction between $SrCl_2$, $CaCl_2$ and Ceftriaxone sodium salt.

$$ESHE = - \Delta G/F \quad (14)$$

$$-\Delta G°_{rxn} = \Delta G°_{ion} + \Delta G°_{atm} + \Delta G°_{S}(H^+) = \Delta f G°(H^+) + \Delta G°_{S}(H^+) \quad (15)$$

$$\Delta G°_{ion} + \Delta G°_{atm} = \Delta_f G°(H^+) \quad (16)$$

The coresponding $\Delta_f G°(H^+)$ based on Fermi-Dirack statistics is 1095 k.J/mole [34].

The data given in Table 6 show $SrCl_2$ solutions gave bigger, $\Delta G°_{rxn}$, $\Delta G°_{S}(H^+)$ results indicating that it is more active in solvation than $CaCl_2$ solutions. The increase in negativity for $\Delta G°_{S}(H^+)$ in both solutions of $SrCl_2$+ Ceftriaxone and $CaCl_2$+Ceftriaxone indicate the complexation reaction and replacing proton by ligand molecules.

TABLE 6: SOLVATION FREE ENERGIES AND PROTON FREE ENERGIES FOR BOTH $SrCL_2$ AND $CACL_2$ SOLUTIONS IN PRESENCE OF CEFTRIAXONE SODIUM SALT

[Ceftriaxone] x10 ^{3}	$SrCl_2$ $\Delta G°_{rxn}$ K.J/Mol	$SrCl_2$ $\Delta G°_{S}(H^+)$ K.J/Mol	$CaCl_2$ ΔG_{rxn} K.J/Mol	$CaCl_2$ $\Delta G°_{S}(H^+)$ K.J/Mol
3.27	-46.4670	-1141.467	-13.124	-1108.124
6.51	-42.3129	-1137.313	-21.144	-1116.144
9.74	-34.6370	-1129. 957	-26.655	-1121.655
1.29	-68.9951	-11.63.985	-29.506	-1124.506

IV. CONCLUSIONS

We explained the redox behavior for $SrCl_2$ and $CaCl_2$ from cyclic voltammetry in 0.1 M KNO_3 at 19 °C.

The estimation of different cyclic voltammetry parameters was carried out for $SrCl_2$, $CaCl_2$ and their interaction with ceftriaxone sodium salt antibiotic. The stability constant, Gibbs free energies of complexion were obtained, and their values were discussed.

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