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Kinetic and conductivity study to oxidation drug ketone ,using iodoform in basic medium

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Abstract

Iodoform oxidation for drug ketone(spusforonion) at room temperature by using the conductivity study and spectrophotometric in basic medium . The conductivity study showed that molar conductance values were decreased with increasing of time at $(2 \times 10^{-4}-4 \times 10^{-4})$ concentration ,while at $(5 \times 10^{-4}-6 \times 10^{-4})$ the value of molar conductance was increased , due to the reaction in presence of basic medium and apply the Helmy's theory of oxidation solutions was applied in ethanol for the initial infinity Molaric conductivity $\Lambda_o$at 25^0 $^{\circ}$was found the liner relationship between the $\ln\Lambda$ and $C^{1/2}$for the weak electrodes . The second study was the spectrophotometric in basic medium which showed that the reaction is related to the first order for the oxidation drug concentration by the iodofrom .

الخلاصة

عند اكسدة الايودوفورم على المركب الدوائي (سبايزوفيورانون) عند درجة حرارة المختبر باستخدام دراسة التوصيلية الكهربائية وجهاز الاشعة فوق البنفسجية الوسط القاعدي. وجد ان قيم التوصيلية المولارية تتناقص مع زيادة الزمن عند التراكيز($2 \times 10^{-4}-4 \times 10^{-4}$) ، بينما عندالتراكيز ($5 \times 10^{-4}-6 \times 10^{-4}$) تزداد التوصيلية المولارية بزيادة الزمن خلال تفاعل الاكسدة في الوسط القاعدي ، وتم تطبيق نظرية حلمي للمحاليل المؤكسدة في الايثانول كمذيب قطبي لايجادΛ_oللالكتروليتات الضعيفة بشكل اكثر ملائمة للدراسة وقد وجد علاقة خطية بين $\ln\Lambda$ و جذر التركيز . اما الدراسة الثانية فهي الدراسة الحركية الطيفية في جهاز الطيفي في الوسط القاعدي لميكانيكية تفاعل الاكسدة وقد وجد ان التفاعل يتبع قانون الرتبة الاولى بالنسبة للكيتون الدوائي المؤكسد باستخدام ايودوفورم المؤكسد .

Introduction

This study was divided into two parts. The first part included the study of the chemical ketone oxidation using the electrolysis and the second part included the kinetic study of the oxidation of the ketone drug using the iodoform .Oxidation is a term that refers to the degree of oxidation for an atom of a chemical compound[1] . The conception of oxidation state the electrical charge which is gained by another atom that sharing ionic bond when connecting with others in 100%. The oxidation state is represented in true numbers, could be negative , positive signal or even zero[2] . in some cases the oxidation state could be a fraction value such as 3/8 for Iron in (Fe_3O_4) .The higher degree of oxidation are (+8) in XeO_4, ReO_4 and OsO_4 , while the lower ones are (-4) which were found in some of carbon group elements[3]. The increasing in oxidation state for an atom during the chemical reaction is called "oxidation" , the Oxidation is a key reaction for different organic synthesis. While the decreasing called "reduction"[3] . These reactions involve transformation of electrons ,the accepting of electrons is regarded a reduction while losing of electron is oxidation[4] . Chemical Kinetics of the substance is exactly how reactant set changed over into things furthermore of all physical and compound strategies which happen in midst obviously of reaction part of reaction gives clear photograph of sanctioned complex[5]. It is said that "Frame work is to Science as semantic use is to lingo". Examination of vitality is truly isolated in to two areas. IUPAC defies the oxidation state as follows[6] :

1- Oxidation state for monoatomic ion is equal to its charge .
2- Oxidation state to free element (unconnected)equal zero .
3- Oxidation state for hydrogen equal(+1) , oxygen (-2) , when they exist in most of compounds in special cases the oxidation of hydrogen is (-1) as in active metals hydrides , like LiH, and the oxidation state in peroxides is (-1) like H_2O_2.
4- The total combination of oxidation state of atoms in isoelectric molecules , must be zero while in atoms which form ions must be equal to its charge , for example , the oxidation state of sulpher in H2S, S8(primary sulpher), SO_2, SO_3 and H_2SO_4 is equal :-2, 0,+4, and +6 respectively . The oxidation state for atom would be higher , in case of increasing oxidation and would be lower in case of increasing reduction .

iodoform is an important and useful tool in the hands of chemists both for preparative and analytical purposes[7]. The utility of iodoform as an oxidant in the kinetic study may be attributed to the vital role of it in various iodoform synthesis also in various media such as basic or even in organic solvents[7]. The present investigation reports the oxidation of drug ketone by iodoform under first order reaction conditions in basic medium.

Second part is involve the Conductivity study , is the equivalent conductance (Λ) of electrolyte is defined as the conductivity 1 cm3 volume , contains to one gram equivalence[8]. the Equivalent conductance is represented by[8] :

$$\Lambda = L \, x \, V$$

$$\Lambda = \frac{L \cdot 1000}{C} \ldots\ldots\ldots (1)$$

Where, L = Specific conductivity

V = Volume of solution in cc. containing one gram equivalent of the electrolyte.

Two effect which are acting to prevent the ions mobility , those are :1- Asymmetric effect , 2- electrophoretric effect . Onsager equation (Kohlrausch's Law) used those effects as follows[9] :

dilution
$$\Lambda = \Lambda_o - (A + B\Lambda_o) \, C^{1/2} \ldots\ldots..(2)$$

Where Λo = molar conductance at infinite
The Kinetics provides the useful information about the mechanism and rate of chemical reaction, which helps to run a chemical reaction successfully by a way of selecting optimum condition as to get maximum yield[10]. The kinetic study also helps us to study the factors which influence the rate of reaction like temperature, pressure, substrate concentration, oxidant concentration, composition of reaction mixture and catalyst [11]. The reaction kinetics plays a very important role in the investigation of the reaction mechanism. Oxidation of organic compound carried out by oxidizing agent like iodoform[12-14].iodoform used to carry out the kinetics and mechanism of various organic compounds.The first order reaction is depended on concentration change with time[15] .

$$\ln\left(\frac{a}{a-x}\right) = k \cdot t \ldots\ldots..(3)$$

The (a) is initial concentration and (a-x) is reaction materials concentration.

The aim of this study is to show the change in kinetic of drug ketone with iodoform tracing with spectrophotometer , electro study and find rate constant , finding degree of dissociation for the reaction formed betweem drug ketone and iodoform , and finding dissociation " constant Kc" .

The mechanism Iodoform synthesis as in following diagrams[7]
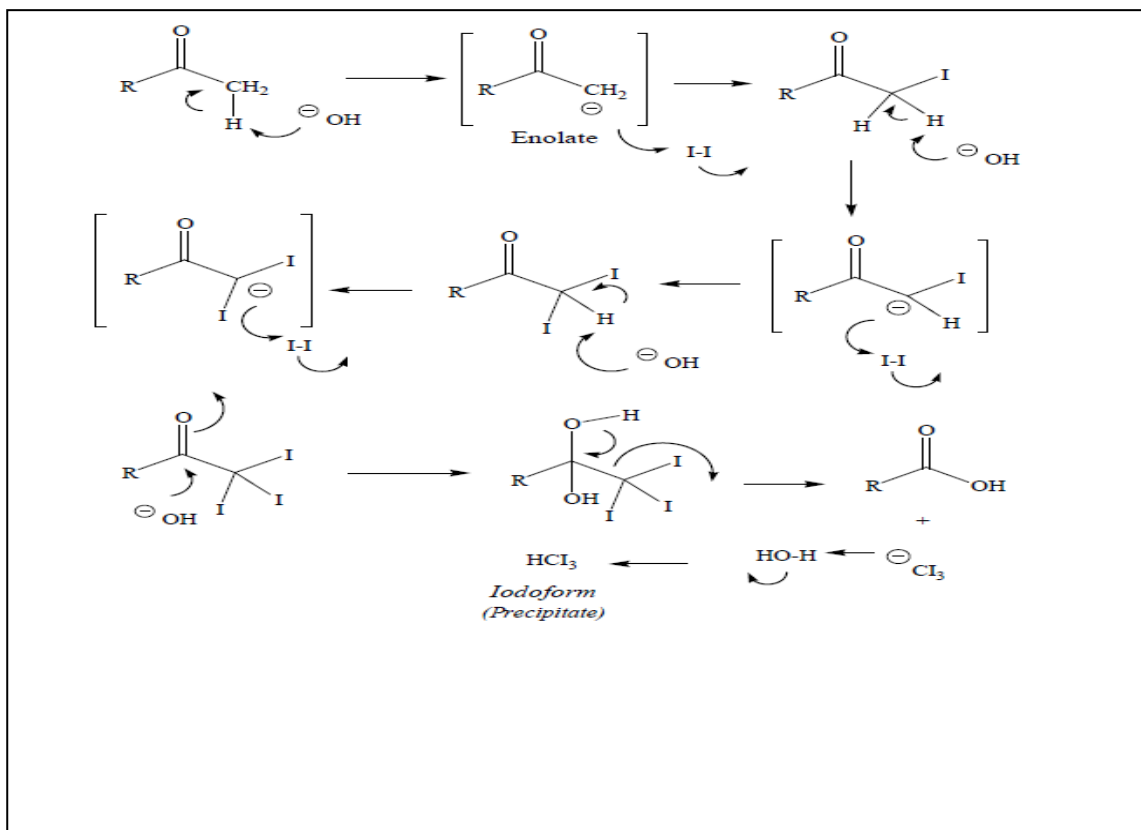
MATERIALS AND METHODS

All chemicals were reagent grade or better and used without being further purified.
ketone drug :5-Acetyl Spiro [benzofuran - 2(3H),Iprime-Cyclopropan]-3-one ($C_{12}H_{18}O_3$) the concentration is (0.0001M)

sodium hydroxide(0.002M) , Iodine(0.0001M) , potassium iodide (0.0001M) , all this is solvent ethanol absolute .

the apparatus used is :Uv/vis spectrometers T90+ two beam and spectrophotometers one beam , conductivity is type Lab 720 INLAB .

procedure[12]:

1- Oxidation prepared (reagent) (0.001M) of I2 , (0.00025 gm) was dissolved in a beaker canting (5ml) of ethanol , in another canting (5ml) of ethanol (0,0001M) of KI (0.0016 gm)was also dissolved , after completion of dissolving the two beakers , they were mixed together to have new reagent with wavelength = 363nm .

2- Preparation of drug ketone

(0.001 M) of the ketone (0.0021 gm) was dissolved in (10ml) of ethanol in a beaker with wavelength=325 nm .

3- NaOH preparation

(0.002 M) of the ketone (0.0008 gm) was dissolved in (10ml) of ethanol in a beaker

4- preparation of Mixture

The solution of ketone and base were mixed together for five minutes , after that the reagent containing (KI+I2) was added in volumetric flask with 10 ml capacity , the volume was completed to 10ml by adding absolute ethanol using range of ketone concentration , wavelength for the mixture is 498nm .

Table(1) : show the volumes using for (drug ketone, oxidation , and base volume)

Ethanol volume	volume of Oxidation	Volume of NaOH	Volume of drug ketone	Volume	Concentration
7ml	1ml	1ml	1ml	1ml	2×10^{-4}
6ml	1.5ml	1.5ml	1ml	1.5ml	3×10^{-4}
5ml	2ml	2ml	1ml	2ml	4×10^{-4}
4ml	2.5ml	2.5ml	1ml	2.5ml	5×10^{-4}
3ml	3ml	3ml	1ml	3ml	6×10^{-4}
2ml	3.5ml	3.5ml	1ml	3.5ml	7×10^{-4}
1ml	4ml	4ml	1ml	4ml	8×10^{-4}

RESULTS AND DISCUSSION

1- Results of conductivity experiments

The reaction kinetic was studied using conductivity meter , the following results were obtained via the tables (2-6) shown below the molar conductance of drug ketone had been calculated with oxidation factor, according to general law :

$$\Lambda = \frac{1000\,L}{C} \quad \ldots\ldots (1)$$

Conductance at in finite dilution , according to Kohlrausch's Law, were also calculated , through plotting the relationship between equivalent conductance versus the square root of concentration that shown , the equivalent conductance for the synthesized compound with oxidized factor , is weak :

$$\Lambda = \Lambda\circ - (A + B.\Lambda\circ), \sqrt{c} \ldots..(2)$$

The study stated that the conductivity for these compounds , is weak and few , according the values of dissociation reaction , which was calculated according to the following equation :

$$\propto = \frac{\Lambda}{\Lambda\circ}$$

It is stated in table (7) and the figure (6) , this is due to occupation of electronic pair for drug ketone during the addition because of increasing the number of collisions per time . for this values of conductivity decreased , indicating that the synthesized compound is a weak electrolyte.

Table (1)

state conductivity values and rate constant for the effect of oxidation factors at [iodoforme] $= 2\times10^{-4}$

Time(min)	L µS/cm	Λ s.eq^{-1}.cm^2	K(min^{-1})
0	31.0	15.5	
2	30.6	15.3	
4	30.4	15.2	
6	30.2	15.1	
8	30.0	15	
10	29.9	14.95	
12	29.8	14.9	
14	29.5	14.75	**5.12x10^{-3}**
16	27.9	13.95	
18	27.8	13.9	
20	27.7	13.85	
22	27.6	13.8	
24	27.5	13.75	
26	27.4	13.7	
28	27.3	13.56	
30	27.2	13.6	

Table (2)

state conductivity values and rate constant for the effect of oxidation factors at [iodoforme] $= 3\times10^{-4}$

Time(min)	L µS/cm	Λ s.eq^{-1}.cm^2	K(min^{-1})
0	16.25	5.41	
2	16.12	5.37	
4	15.83	5.27	
6	15.51	5.21	
8	15.63	5.17	
10	15.23	5.09	
12	15.28	5.07	
14	14.82	4.94	2.78x10^{-3}
16	14.77	4.92	
18	14.77	4.90	
20	14.52	4.84	
22	14.41	4.80	
24	14.10	4.76	
26	13.87	4.62	
28	13.84	4.61	
30	13.78	4.59	

Table (3)
state conductivity values and rate constant for the effect of oxidation factors at [iodoforme] = 4×10^{-4}

Time(min)	L µS/cm	Λ s.eq^{-1}.cm^2	K(min^{-1})
0	4.49	1.1225	
2	4.40	1.11	
4	4.43	1.1075	
6	4.42	1.105	
8	4.41	1.1025	
10	4.39	1.0925	
12	4.37	1.0925	
14	4.35	1.0875	
16	4.33	1.085	**1.2x10$^{-3.}$**
18	4.33	1.0845	
20	4.33	1.0838	
22	4.33	1.0833	
24	4.33	1.0825	
26	4.34	1.0820	
28	4.35	1.0812	
30	4.35	1.0808	

Table (4)
state conductivity values and rate constant for the effect of oxidation factors at [iodoforme] = 5×10^{-4}

Time(min)	L µS/cm	Λ s.eq^{-1}.cm^2	K(min^{-1})
0	30.4	0.608	
2	30.5	0.610	
4	30.55	0.611	
6	30.6	0.612	
8	30.64	0.6128	
10	30.7	0.614	
12	30.77	0.6154	
14	30.8	0.616	
16	30.83	0.6166	**5x10^{-5}**
18	30.9	0.618	
20	30.97	0.6194	
22	30.99	0.6198	
24	31.03	0.6206	
26	31.05	0.621	
28	31.07	0.6214	
30	31.12	0.622	

Table (5)
state conductivity values and rate constant for the effect of oxidation factors at [iodoforme] = 6×10^{-4}

Time(min)	L µS/cm		Λ s.eq^{-1}.cm^2	K(min^{-1})
0	3.74		0.62	
2	3.80		0.633	
4	3.82		0.636	
6	3.86		0.64	
8	3.91		0.65	
10	3.96		0.660	
12	3.98		0.663	
14	4.02		0.67	$2.8x10^{-4}$
16	4.06		0.676	
18		4.08	0.68	
20		4.10	0.683	
22	4.12		0.686	
24		4.14	0.69	
26		4.18	0.696	
28		4.22	0.703	
30		4.28	0.713	

The data obtained also showed that the electron mobility between these two molecules , is responsible for that increase the table (1-4) show that molar conductance values , decrease with increasing of time at ($2x10^{-4}$-$4x10^{-4}$) concentration , this refers to the conductivity of reaction , having a few conductivity , so the polarization of carbonyl group would also be decreased effecting by the inductive effect of the halogen . The attraction will stay keeping on randomly, the attraction and electrostatic forces would be having importance greatly and widely . The presence of ionic atmosphere that combine the ions round the drug ketone and the opposite ions , the importance of ionic atmosphere , decreasing in case of increasing an infinite dilution of the solution until completely disappear , and this atmosphere effects on ions mobility by increasing the concentration and due to presence unsymmetrical and electrophoretic effect which slow the mobility of ions , so it is noticed that molar conductance , decreased by increasing of time . while at ($5x10^{-4}$-$6x10^{-4}$) the value of molar conductance would be increasing , due to the reaction in presence of basic medium , effecting to the concentration of ketone , deading to increasing of the basicity of ketone , so the conductivity would be increasing the concentration on formation of carboxylate ion as, stated in the previous mechanism , that is shown in the following figures .
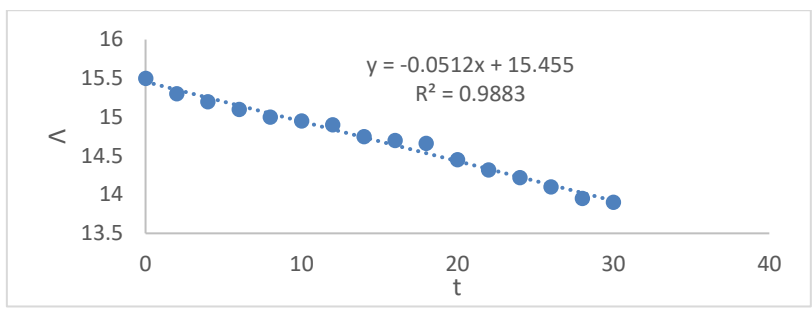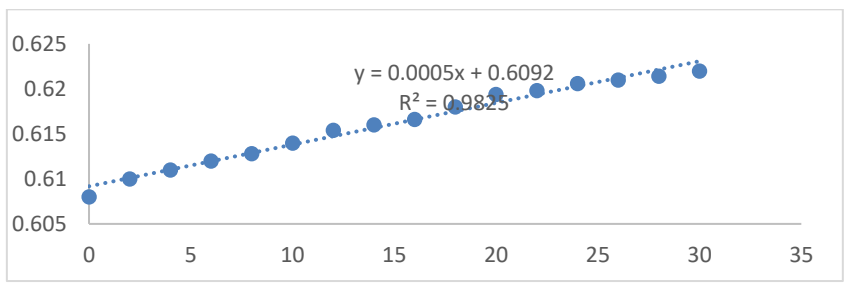
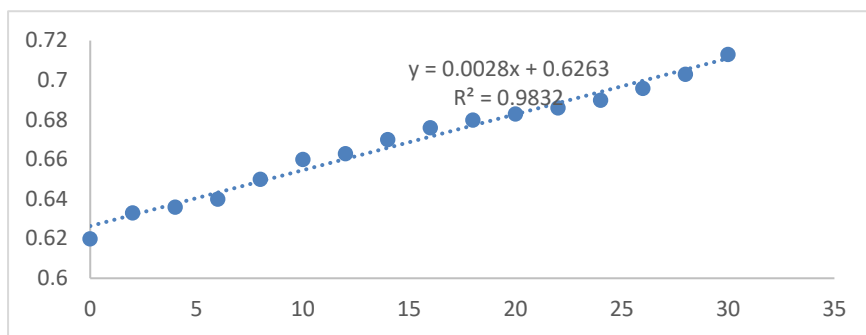**Fig(1) : effect of iodoform oxidation factor at (2x10⁻⁴) molar con.**
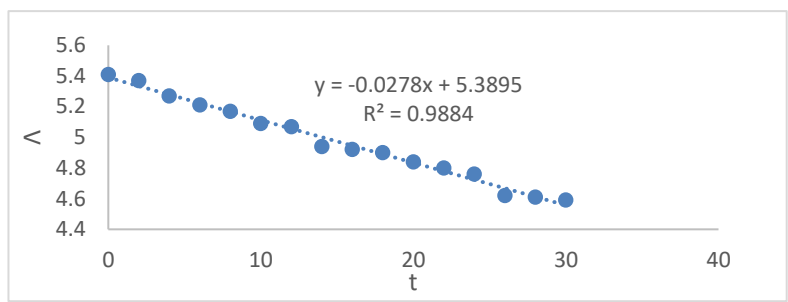
Fig(2) : effect of iodoform oxidation factor at (3x10⁻⁴) molar con.
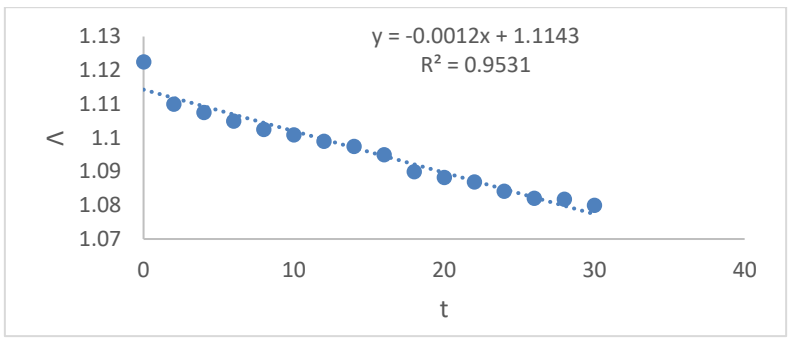
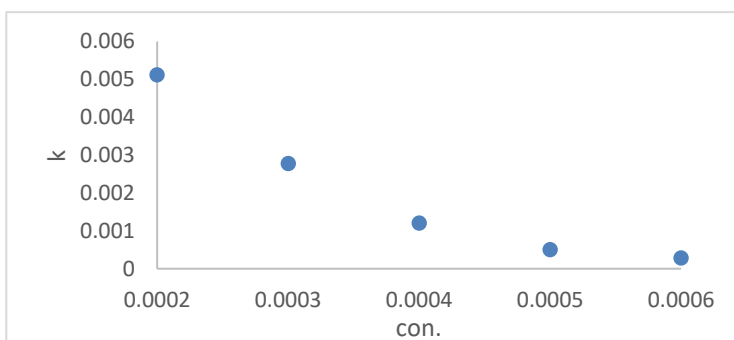**Fig(3) : effect of iodoform oxidation factor at (4x10⁻⁴) molar con.**

Fig(4) : effect of iodoform oxidation factor at (5x10⁻⁴) molar con.

**Fig(5) : effect of iodoform oxidation factor at (6x10^{-4}) molar con.**

The figure (6) show the stability constant (k) decreases with concentration increasing .because the steric effect and the attraction and electrostatic forces .

Fig(6) :k plotted against con.at oxidation process

Table (6)
Molar conductance values and molar values at infinite dilution and dissociation degree

C	C$^{1/2}$ X10^{-4}	Stability time	Λ	Λ°	α	Kc
2^{x}10^{-4}	0.414	30	13.6		0.956	**4.165 x10^{-3}**
3^{x}10^{-4}	0.732	30	4.59		0.322	**4.58x10^{-5}**
4^{x}10^{-4}	2	30	1.0808	14.22	0.0760	**1.875x10^{-6}**
5^{x}10^{-4}	2.23	30	0.622		0.0437	**9.984x10^{-7}**
6^{x}10^{-4}	2.449	30	0.713		0.0501	**3.0180x10^{-6}**
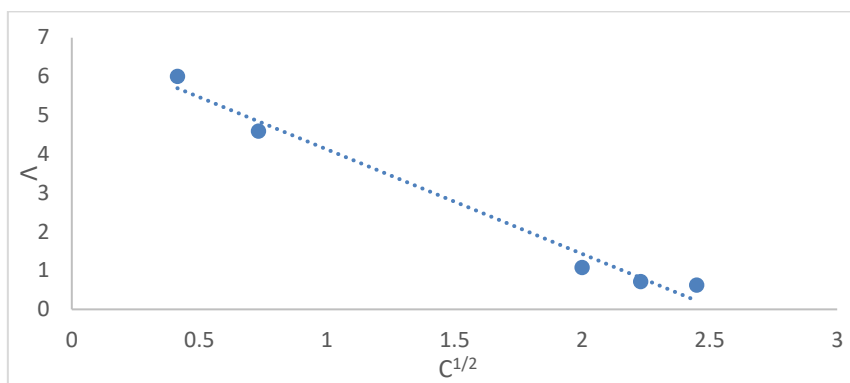
Fig(7) molar conductance values at infinite dilution via kohlrausch equation

The Helmy's equation is :

$$\ln \Lambda = \ln \Lambda_0 - \left(\frac{\alpha + \beta}{\Lambda_0}\right)\sqrt{C}$$

The equation is calculate Λ_0 value for the weak and very weak electroplate from O·nsacers equation $\left(\Lambda - \sqrt{C}\right)$, also the calculate values by which values were calculated more easily than the values calculated by the Kohlrausch'sequations, since both had sometimes given a negative assessment of the equivalent or initial molar conductivity.Figure (8)
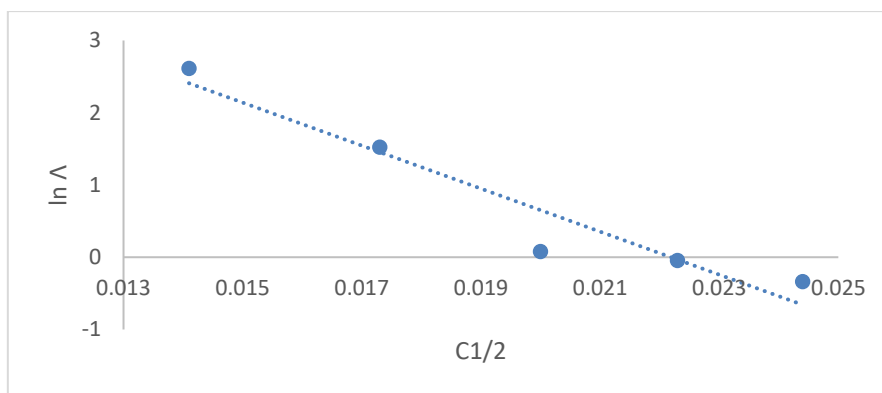

Fig (8) Helmy's theory of oxidation solutions was applied in ethanol for the initial infinity Molaric conductivity Λoat 25°.

2- <u>Results of Kineticexperiments</u>

The kinetic study had been performed at basic medium between ketone drug and iodoformas shown in following results:

Table (7)
absorptivity values and reaction constant rate to oxidation factor at concentration 2×10^{-4}

Time(min)	A	$\log\dfrac{A_\circ}{A_t}$	K(min)
0	0.140	0	
5	0.145	-0.01	
10	0.147	-0.02	
15	0.149	-0.027	
20	0.151	-0.032	$1.5\text{X}10^{-4}$
25	0.154	-0.04	
30	0.155	-0.045	
35	0.159	-0.055	
40	0.163	-0.065	

Table (8)
absorptivity values and reaction constant rate to oxidation factor at concentration 3×10^{-4}

Time(min)	A	$\log\dfrac{A_\circ}{A_t}$	K(min)
0	0.032	0	
5	0.033	- 0.013	
10	0.034	- 0.026	
15	0.035	- 0.039	
20	0.036	- 0.051	
25	0.037	- 0.063	
30	0.038	- 0.074	
35	0.039	- 0.086	
40	0.040	- 0.096	$3.3\text{ x}10^{-5}$
45	0.042	- 0.118	
50	0.043	- 0.126	
55	0.046	- 0.158	
60	0.048	- 0.176	
65	0.051	- 0.202	
70	0.055	- 0.235	
75	0.058	- 0.258	

Table (9)
absorptivity values and reaction constant rate to oxidation factorat concentration4×10^{-4}

Time(min)	A	$\log\dfrac{A_\circ}{A_t}$	K(min)
0	0.224	0	
5	0.228	-0.007	
10	0.231	-0.012	
15	0.232	-0.015	
20	0.234	-0.018	1.0×10^{-4}
25	0.237	-0.024	
30	0.242	-0.033	
35	0.243	-0.035	
40	0.246	-0.040	

Table (10)
absorptivity values and reaction constant rate to oxidation factor at concentration 5×10^{-4}

Time(min)	A	$\log\dfrac{A_\circ}{A_t}$	K(min)
0	0.314	0	
5	0.315	-0.0017	
10	0.317	-0.0043	
15	0.318	-0.0056	
20	0.319	-0.0070	3×10^{-5}
25	0.320	-0.0083	
30	0.321	-0.0096	
35	0.322	-0.010	
40	0.323	-0.021	

Table (11)
absorptivity values and reaction constant rate to oxidation factor at concentration 6×10^{-4}

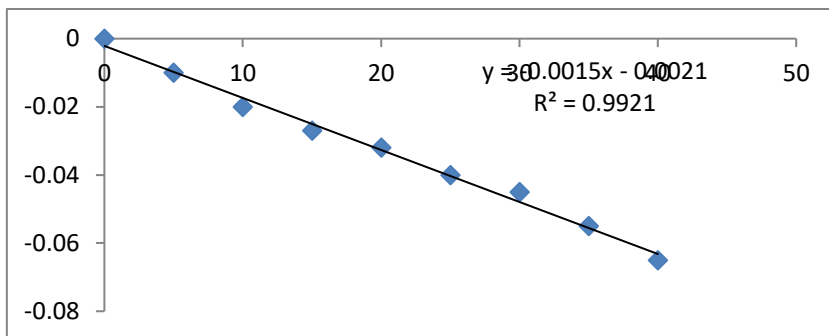| Time(min)	A	$\log\dfrac{A_\circ}{A_t}$	K(min)
0	0.177	0	
5	0.176	0.0024	
10	0.172	0.0124	
15	0.166	0.027	
20	0.161	0.041	2.2×10^{-4}
25	0.157	0.052	
30	0.153	0.063	
35	0.149	0.074	
40	0.145	0.086	

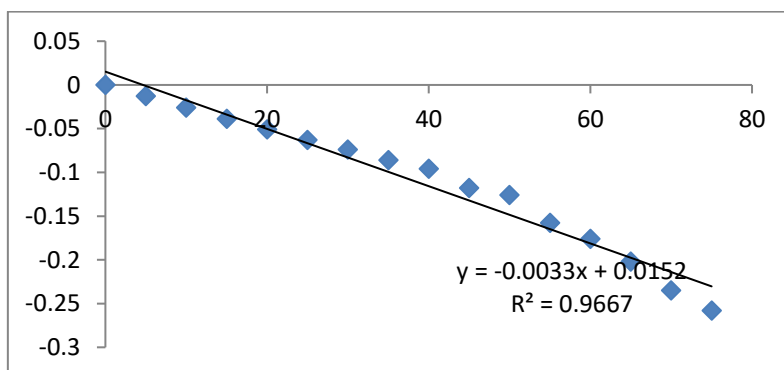**Fig (9) the relationship of first order reaction between drug ketone and iodoform at 2x10^{-4}**

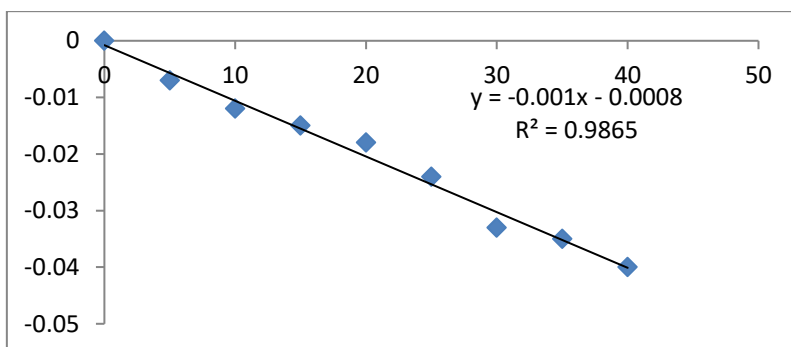**Fig (10) the relationship of first order reaction between drug ketone and iodoform at 3x10^{-4}**

Fig (11) the relationship of first order reaction between drug ketone and iodoform at 4x10^{-4}
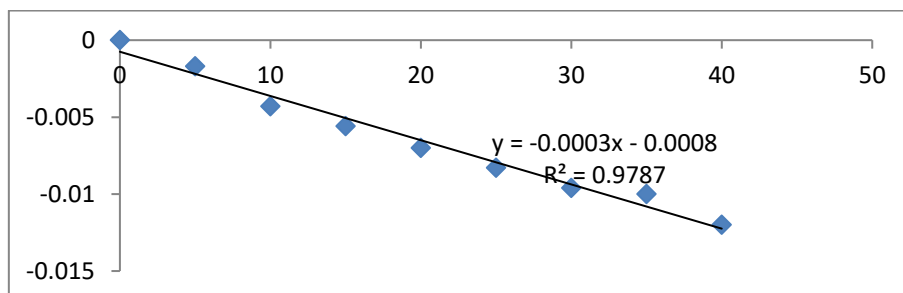

Fig (12) the relationship of first order reaction between drug ketone and iodoform at 5x10^{-4}

According to data obtained in tables (7-11) and figures (9- 12) it is clear that the mechanical reaction , taking place , is first order reaction during k values obtained from the following equation $k = \frac{2.303}{t} \log \frac{A_\circ}{A_t}$ and clear correlation factor in all figures , having the values m ranged (0.9921-0.9667) , wave lengths of the prepared solution , were measured according to the wavelength of drug ketone and iodoform oxidized factor and also the kind of used solvent , the peaks wave (325nm) and(363nm) respectively , this change to new peak at 498nm as indication to obtain the electronic transition in this reaction as (n-π*) , which is transferring the electrons between the drug ketone and oxidized factor in basic medium [16-19] . According to mechanism of reaction , it is showed that presence of cage effect and solvation effect which are effecting to the absorption peaks directly . These factors basically depend on nature of material and solvent used to solve the material , so this leads to combine solvent molecules round the reacted material which can be explained as surrounding for obstical the molecules to interact with each other , which can be noticed in case of decreasing the absorptivity , changing charge for dissimilar of ions that can positively and negatively effecting to the stability of formed compounds[20] .

In conclusion we can say that the solvent has great effect m which can explain the contrast in the values of absorption , exclusively in polar solvents as aversely to ability of the compound to interaction with polar solvents because they have functional groups like carbonyl one and that is because of cage effect .

Previous studies have the In present investigation we are studied the kinetics and mechanism of oxidation of ester by potassium dichromate in acid medium. In this study we reported the effect of oxidant $K_2Cr_2O_7$, effect of substrate (ester), effect of sulphuric acid and effect of temperature on oxidation of ester.The reaction was first order according to oxidant and substrate, as temperature increases rate of the reaction also increases. and second research is show the Permagnetic oxidation of 3-Ethoxy-4- Hydroxybenzaldehyde has been studied at different temperatures using spectrophotometer under acidic conditions. The effect of variation of substrate (3-E-4-HB), oxidant (KMnO$_4$) andH$_2$SO$_4$ was studied under pseudo first order reaction conditions. The effect of different salts on oxidation of 3-E-4-HB was also studied. The reaction was found to be first order with respect to oxidant, substrate and H2SO4. Asuitable mechanism is also suggested for the oxidation reaction.

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