

*University of Baghdad
College of science
Department of chemistry.*



*Practical Physical Chemistry
For Third Year Chemistry*

Translated and revised by

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Experiment (1)

Studying the kinetics of the hydrolysis of methyl acetate catalyzed by hydrochloric acid.

Aim of the experiment:

Determination of.

- 1- Specific rate constant.
- 2- The pseudo first order rate constant.
- 3- The half-life time.

Apparatus and materials:

Burette 50 ml, pipette 10 ml , volumetric flask, conical flask , measuring cylinder 50ml, Methyl acetate, Hydrochloric acid 0.1 N ,sodium hydroxide 0.05N , Ph. indicator.

Procedure:

- 1- Prepare (0.1M) of HCl solution in 100 ml volumetric flask.
- 2- Prepare (0.05M) of NaOH Solution in 250 ml volumetric flask for titration.
- 3- Mix 5ml (Methyl acetate) with 100 ml (0.1M) HCl Solution.
- 4- In 100 ml round bottom flask, equipped with an air condenser, putting 10 ml of mixture in (step-3) and heating in water bath for one hour; to calculate the V_∞ .
- 5- In conical flask contain 10 ml cold distilled water, titrate 10ml of the mixture in (step-3) against (0.05 M) NaOH solution using few drops of (ph.) as an indicator, repeat this step after (5, 10,15, 25 ,30,35 ,40min.)to calculate V_t .
- 6- After one hour titrate the solution in the round flask after cooling and washing it with 10 ml of cold distilled water then titrate it with 0.05 M of NaOH solution to calculate V_∞ .

Results and calculations:

- 1- Put the results in the following table.

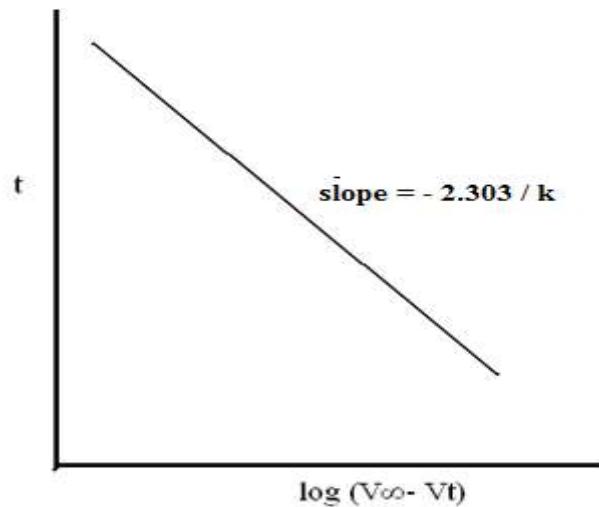
t(sec.)	V_t	$(V_\infty - V_t)$	$\log (V_\infty - V_t)$	$V_\infty / (V_\infty - V_t)$	$\log V_\infty / (V_\infty - V_t)$	k

2-alculate the rate constant according to the following equation for each time t.

$$k = (2.0303/t) \log [V_\infty / (V_\infty - V_t)]$$

$$t = (2.303/k) \log(V_\infty) - (2.303/k) \log(V_\infty - V_t)$$

3-Plot the curve between $\log(V_\infty - V_t)$ against (t) to calculate k practically from the slop then compares it with the value of k theoretically.



4- Calculate the half-life time according to the following equation.

$$t_{1/2} = 0.693/k$$

Experiment (2 A)

Determination of the dissociation constant for weak acid by conductivity measurements.

Aim of the experiment:

Determination the equivalent conductance of acetic acid at several concentrations and calculate the dissociation constant k_a of the acid.

Apparatus and materials:

Volumetric flask 25ml, pipette, beakers, potassium chloride, acetic acid.

Procedure:

- 1- Prepare (0.1M) of potassium chloride in 25 ml volumetric flask (stock solution).
- 2- Prepare (0.2M) of acetic acid from (1M) of concentration and then prepare by dilution the other concentration (0.1, 0.05, 0.01, 0.005 M) in 25 ml volumetric flasks.
- 3- Measure the conductivity for each solution which prepared in (step1, 2).

Results and calculations:

- 1- Calculate the cell constant (K) for (0.1M) of KCl solution as the following

$$K = L / C$$

K = Cell constant

C = Conductivity of 0.1 M KCl solution.

L = Specific conductance of 0.1M of KCl solution, L (0.1M), $KCl = 0.01285 \Omega^{-1} \text{cm}^{-1}$.

- 2- Calculate the specific conductance for all acetic acid solution by the following equation:

$$L = K C$$

- 3- Calculate the equivalent conductance Λ for acetic acid solution as following.

$$\Lambda = 1000 L / C$$

C = Concentration of acetic acid solutions.

- 4- Calculate the degree of dissociation for the solutions as following.

$$\alpha = \Lambda / \Lambda_0$$

Λ_0 = equivalent conductance for acetic acid solution in an infinite dilution.

$$\Lambda_0 = 390 \Omega^{-1} \text{cm}^2 \text{eq}^{-1}$$

5- Calculate the dissociation constant ka as follows.

$$ka = \alpha^2 C / (1 - \alpha)$$

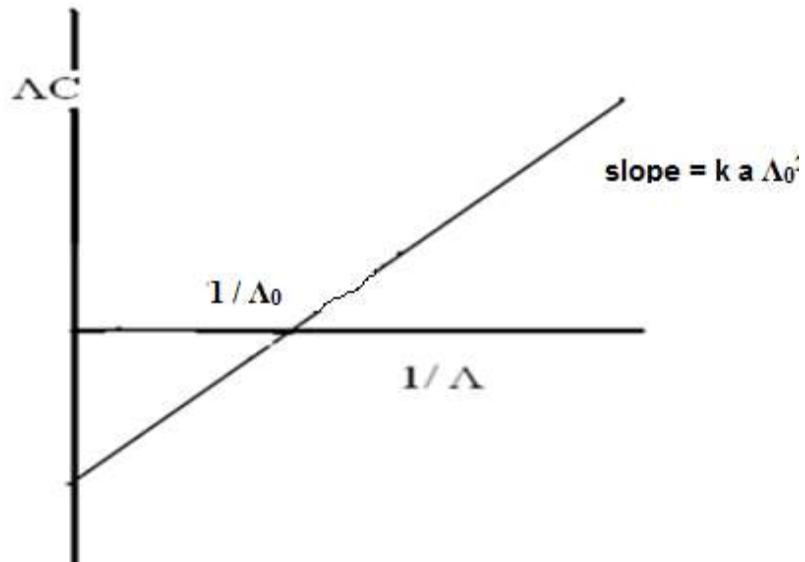
6- Put the results in the following table.

No.of sample	C	C	Λ	ΛC	$1/\Lambda$	$\alpha = \Lambda / \Lambda_0$	$ka = \alpha^2 C / (1 - \alpha)$

7- Plot a curve between ΛC against $1/\Lambda$ for calculate Ka practically as the following equation:

$$\Lambda C = ka [\Lambda_0^2 / \Lambda - \Lambda_0]$$

$$\Lambda C = ka \Lambda_0^2 (1/\Lambda) - Ka \Lambda_0$$



8- Compare it with the theoretical value of ka .

Experiment (2 B)

Determination of standard electrode potential for zinc and copper

Aim of the experiment:

Determine the standard electrode potential for zinc electrode and copper electrode by using calomel electrode as standard electrode.



Apparatus and materials:

Galvanometer, potentiometer, Weston cell, resistance, calomel electrode, zinc electrode, copper electrode, zinc sulphate and copper sulphate.

Procedure:

- 1- Prepare 0.1M of copper sulphate solution in 50 ml volumetric flask.
- 2- Prepare 0.1M of zinc sulphate solution in 50 ml volumetric flask.
- 3- Wash the copper and zinc electrodes by distilled water and dried them before using.
- 4- Put the copper sulphate solution in the beaker and left for 10 minutes for equilibrium, then put the copper electrode in the solution and measure the electromotive force (E_{cell}), repeat the measurement after 5 minutes, for insure the thermo equilibrium.
- 5- Repeat step (4) for the zinc sulphate solution and its electrode.

The results and calculations:

- 1- Calculate the electrode potential for zinc electrode as following :

$$E_{\text{cell}} = E_{\text{cal}} - E_{\text{zn}} \dots \text{the cell is (zinc- calomel)}$$

- 2- Calculate the electrode potential for copper electrode as following :

$$E_{\text{cell}} = E_{\text{cu}} - E_{\text{cal}} \dots \text{the cell is (copper- calomel)}$$

- 3- Calculate the electrode potential for calomel electrode from this equation:

$$E_{\text{cal}} = 0.244 - 0.007(t - 25)$$

t = the temperature of laboratory during the experiment (in $^{\circ}\text{C}$).

4- Calculate the standard electrode potential E^0 , for zinc electrode E_{zn}^0 and copper electrode E_{cu}^0 by the following equation:

$$E_{\text{cell}} = E_{\text{cal}} - (E_{\text{zn}}^0 + RT/nf \ln m \text{Zn}^{+2} \cdot \gamma^{-+})$$

$$E_{\text{cell}} = (E_{\text{cu}}^0 + RT/nf * \ln 1/(m \text{Cu}^{+2} \cdot \gamma^{-+}) - E_{\text{cal}})$$

$n = 2$ equivalent number

$F = 96500$ coulomb / mole.

$R = 8.314$ J/ K .mol.

γ^{-+} = activity coefficient for 0.1M (ZnSO₄) solution = 0.161

γ^{-+} = activity coefficient for 0.1M (CuSO₄) solution = 0.158.

Experiment (3 A)

The hydrolysis of ethyl acetate by sodium hydroxide (equal conc.) (Second-order reaction)

Aim of the experiment:

- 1- Determine the second- order rate constant of the hydrolysis of ethyl acetate by Sodium Hydroxide.
- 2-Calculate the half life time for the reaction.

Apparatus and materials:

Two volumetric flask 100 ml. , burette 50 ml ,two conical flasks for titration , round bottom flask for mixing , pipette 10ml., stop watch clock, ethyl acetate, hydrochloric acid, sodium hydroxide, phenolphthalein indicator (ph. ph.) .

Procedure:

- 1- Prepare (0.1M)of HCl solution in 100 ml volumetric flask, then prepare from it (0.025M) in 100 ml. volumetric flask to stop the reaction.
- 2- Prepare (0.1M) of NaOH in 100ml volumetric flask then prepare from it (0.05M)in 50 ml. volumetric flask for mixing.
- 3- Prepare (0.05M) ethyl acetate in 50ml volumetric flask for mixing.
- 4- Prepare (0.025M) of NaOH in 100 ml volumetric flask for titration.
- 5- Mix ethyl acetate in step (3) with (0.05M) NaOH in round flask (clean and dry) and recorded the time of mixing.
- 6- After 5mindraw 10ml of the mixture in (step -5) and put it in the conical flask contained 10ml. of distilled water and 10ml. of (0.025M) HCl then titrate with (0.025M) of NaOH solution using few drops of ph.ph. as an indicator.
- 7- For an hour repeat (step 6) at (10, 20, 30, 40, 50, 60) min.

Results and Calculations:

- 1- Calculate the rate constant(k) for each time (t) from the following equation:

$$t = 1 / (k \cdot a) * x / (a - x)$$

a = The initial concentration of the base or ester at time of mixing (t = 0).

x = The concentration of the reactant base or ester at(t = 5,10,20,30)(different reaction time).

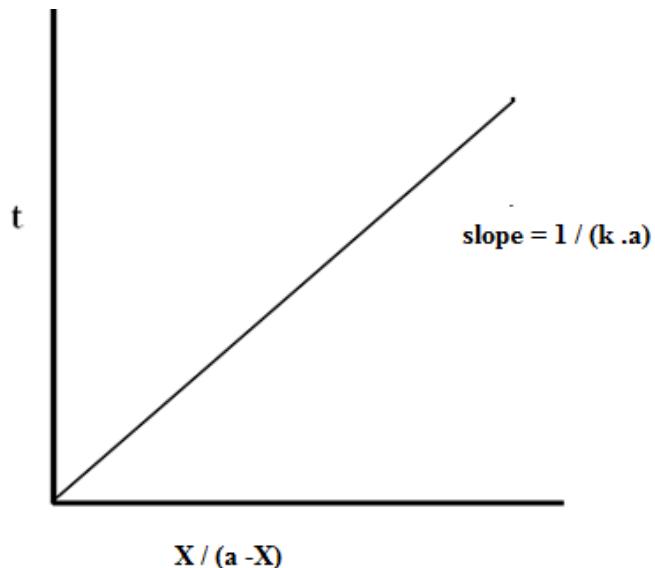
a-x= The concentration of the remaining ester or base (the unreacted concentration).

2- put the results in the following table.

No. of sample.	t	V for NaOH	X	(a-x)	x / (a-x)	k

3-Calculate the average of **k** (the value of **k** theoretically).

4-Plot $x / (a-x)$ against (t) for calculate **k** practically.



5-Calculate the half life time($t_{1/2}$) from the following equation:

$$t_{1/2} = 1 / k \cdot a$$

Experiment (3 B)

The hydrolysis of ethyl acetate by sodium hydroxide (unequal conc.)

Aim of the experiment:

- 1- Determine the second- order rate constant of the hydrolysis of ethyl acetate by sodium hydroxide.

Apparatus and materials:

Two volumetric flask 100 ml. , burette 50 ml, two conical flasks for titration , round flask for mixing , pipette 10ml., stop clock, ethyl acetate, hydrochloric acid, sodium hydroxide, ph. Ph. as indicator.

Procedure:

- 1- Prepare (0.1M) of HCl solution in 100 ml volumetric flask, then prepare from it (0.0125M) in 100 ml. volumetric flask to stop the reaction.
- 2- Prepare (0.1M) NaOH in 100ml. volumetric flask and Prepare from it (0.025M) in 50ml. vol.flask for mixing.
- 3- Prepare (0.05M) ethyl acetate in 50ml vol. flask for mixing.
- 4- Prepare (0.0125M) NaOH in 100ml. volumetric flask from 0.1MNaOH solution for titration.
- 5- Mix ethyl acetate in (step -3) with (0.025M)NaOH in clean and dry round flask and record the time of mixing.
- 6- After 5 min. take 10 ml. of mixture and put it in the conical flask contained 10ml. of distilled water and 10 ml. of (0.0125M) HCl and then titrate the excess acid against (0.0125M) NaOH with few drops of ph.ph.as an indicator.
- 7- Repeat step 6 at the following time :(10, 15, 20, 25, 30, 35, min.)

The Results and Calculations:

- 1- Calculate the rate constant (k) for each time (t) from the following equation:

$$k = 2.303 / (t (a - b)) * \log(b (a - x) / a (b - x))$$

b = The initial conc. of the base after mixing.

a = The initial conc.of the ethyl acetate after mixing.

x = The conc. of the reactant base after each time (t).

a-x = The conc. of the remaining ester.

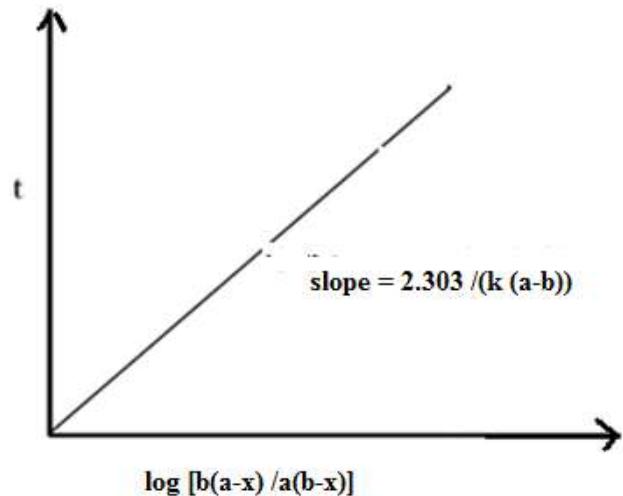
b-x = The conc. of the remaining base.

2- put the results in the following table:

No. of sample.	t	V for (NaOH)	X	(a-x)	(b-x)	$\log b (a-x) / a(b-x)$	k

3- Calculate the average value of **k** (theoretically).

4- Plot $\log (b (a-x) / a (b-x))$ against (t) for calculating **k** practically.



Experiment (4A)

Determination of the equivalent conductance for a strong electrolyte by conductivity measurement.

Aim of Experiment:

- 1- Examine the validity of the Onsager theory as limiting law at infinite dilutions.
- 2- Calculation of the equivalent conductance for strong electrolyte.

Apparatus and materials:

Conductivity cell, conductivity meter, pipette, volumetric flask (25 ml), potassium chloride, sodium chloride and beakers.

Procedure:

- 1- Prepare (0.1M) of KCl solution in 25 ml volumetric flask to calculate cell constant (K).
- 2- Prepare (0.1M) NaCl solution in 50ml. volumetric flask and then prepare by dilution the following conc. (0.05, 0.01, 0.005, and 0.0025) min 25ml. vol. flasks.
- 3- Measure the electric conductance for all solutions which prepared above.

Results and Calculations:

- 1- Calculate the cell constant (K) from the following equation:

$$K = L / C$$

C=Electric conductance (Ω^{-1})

L= The specific conductance for 0.1MKCl solution is $0.01285 \Omega^{-1}cm^{-1}$.

- 2- Calculate the specific conductance L for NaCl solutions according the following equation:

$$L = K \cdot C$$

- 3-Calculate the equivalent conductance (Λ) for all solutions as following:

$$\Lambda = 1000L / C$$

C= the concentration of the solutions.

- 3- Calculate the equivalent conductance at infinite dilution (Λ_0) by Onsager equation.

$$\Lambda_0 = \Lambda + 60.2\sqrt{C} / (1 - 0.229\sqrt{C})$$

4- Put the results in the following table:

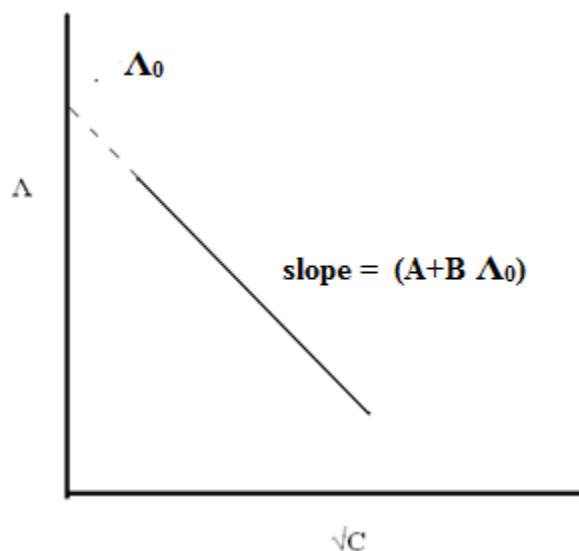
No. of sample.	C	\sqrt{C}	\bar{C}	L	Λ	Λ_0

5- Calculate the average value of Λ_0 (theoretically).

6- Plot Λ against \sqrt{C} for calculate Λ_0 experimentally according this equation.

$$\Lambda = \Lambda_0 - (A + B \Lambda_0) \sqrt{C}$$

$$\Lambda = \Lambda_0 - (60.2 + 0.229 \Lambda_0) \sqrt{C}$$



Experiment (4B)

Determination of the pH value using Quinohydrone electrode.

Aim of the experiment:

Determination the pH value for a solution by using Quinohydrone electrode.

Apparatus and materials:

Electric cyrclecontaine: Galvanometer, potentiometer, stander cell (Weston cell), Calomel electrode, Quinohydrone electrode, hydrochloric acid (HCl), sodium acetate (CH_3COONa).

Procedure:

- 1- Prepare solution contain 5 ml from(1 M) CH_3COONa and unknown volume of (1M)HCl in 25ml volumetric flask and complete the volume with distilled water.
- 3- Prepare another solution as in step (1) with another volume of (1 M) HCl.
- 4- Put each sample above in beaker and saturate them with quinohydrone until the color of solution become green and then dip the platinum electrode in the solution and leave it for 15min. for reach the equilibrium between the electrode and its solution.
- 5- Connect the quinohydrone electrode with Calomel electrode for measure E_{cell} .

The Results and calculations:

- 1- Calculate E_{cal} from the following equation:

$$E_{\text{cal}} = 0.244 - 0.007(t - 25)$$

t = the temp. of the laboratory

- 2- Calculate the electromotive force for the cell (E_{cell}) for each solution.

$$E_{\text{cell}} = E_{\text{QH}_2} - E_{\text{cal}}$$

- 3-Calculate the value of pH from this equation:

$$E_{\text{QH}_2} = E_{\text{QH}_2}^{\circ} - 0.059\text{pH}$$
$$E_{\text{QH}_2} = 0.6994 - 0.059 \text{ pH}$$

Experiment (5)

Determination the effect of acid concentration on the rate of inversion of sucrose.

Aim of Experiment:

1-Determination the effect of Hydrochloric acid on the rate of the inversion of sucrose.

2- Determination the half-life time for this reaction.

Apparatus and materials:

Polarimeter, Sodium lamp, Conical flask 100 ml., Volumetric flask 25 ml., burette 25 ml., stop clock, concentrated hydrochloric acid, sucrose.

Procedure:

1-Weight 5 g of sucrose and dissolve in 25 ml volumetric flask by distilled water.

2-Prepare (2 N) of HCl solution from 5N in 25 ml. volumetric flask.

3-Set up the polarimeter and determine the zero by full the cell or tube with distilled water.

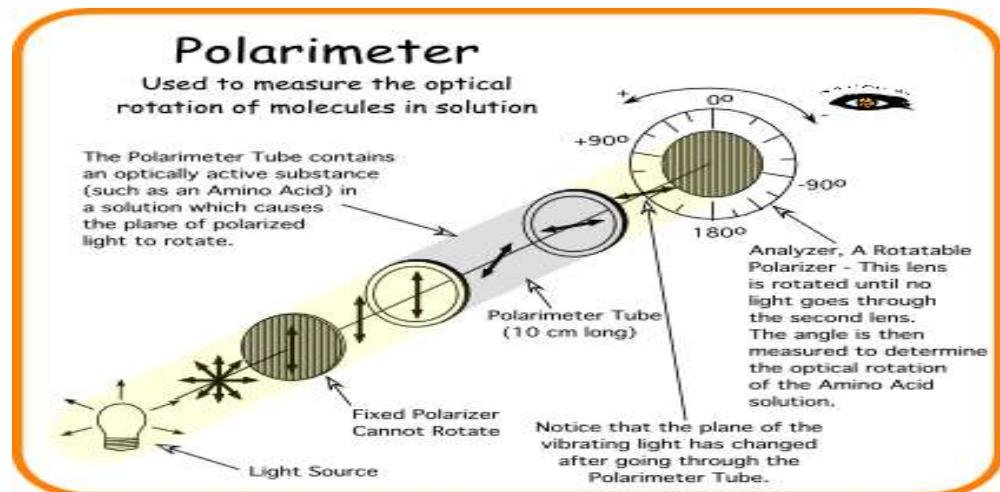
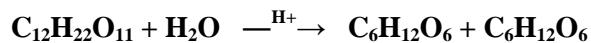
4-Mix the sucrose solution with 2N HCl solution in conical flask and record the time of mixing.

5-Full the cell or tube with mixture and read the rotation angle α_t in the following time: (5, 15, 25, 35, min.)So on for one hour.

6-Heat the remaining mixture for 30 minute in 50 C° water bath to determine α_{∞} .

7-Measure the final rotation angle α_{∞} .

8-Repeat the steps (1-7) for (4N) HCl solution.



Results and Calculations:

1-If α represents the initial angle, α_t is the angle during the time of inversion and α_∞ the final angle of rotation .Then:

$$\alpha_t = \alpha_0 - \alpha_\infty$$

$$\alpha_\infty = \alpha_0 - \alpha_t$$

2-Put the result in the following table:

No.of sample	t	α_t	$\alpha_0 - \alpha_t$	$\alpha_t - \alpha_\infty$	$\log \alpha_t - \alpha_\infty$	$\log \alpha_\infty / (\alpha_t - \alpha_\infty)$	k

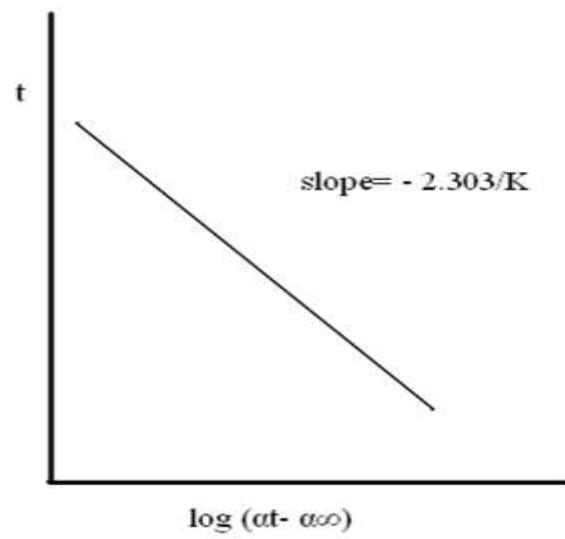
3-Calculate the rate constant K from this equation:

$$k = 2.303/t \log [\alpha_0 - \alpha_\infty / \alpha_t - \alpha_\infty]$$

And then calculate the average of k (theoretically).

4-Plot the value of $\log (\alpha_t - \alpha_\infty)$ against (t) for calculates practically.

$$t = 2.303/k \log (\alpha_0 - \alpha_\infty) - 2.303/k \log (\alpha_t - \alpha_\infty)$$



5- Calculate the half- time of reaction from this equation:

$$t_{1/2} = 0.693 / k$$

Experiment (6A)

Determination of the decomposition potential for some electrolytes.

Aim of the experiment:

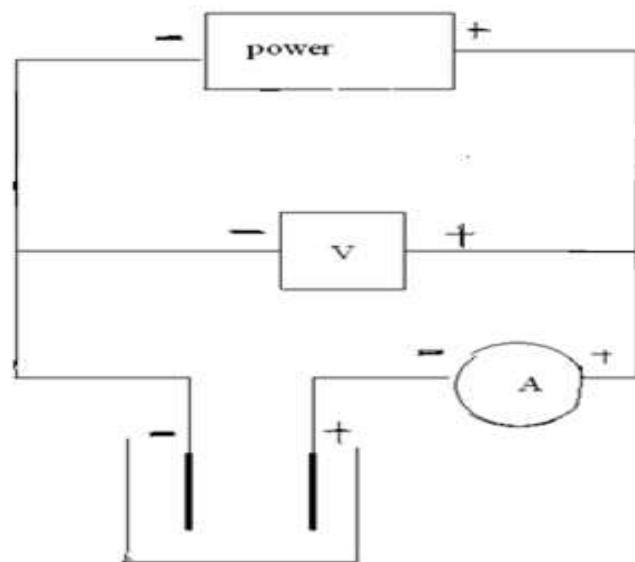
- 1- Determine the decomposition potential for dilute solution of sulphuric acid and copper sulfate.
- 2-Determine current – voltage curves for these dilute solutions.

Apparatus and materials:

Voltmeter, Ammeter, power supply, variable resistance, electric cell consist two platinum electrode, volumetric flask 50ml, pipette 10 ml ,sulphuric acid (1M), copper sulphate, beakers.

Procedure:

- 1- Prepare 0.1M H₂SO₄ in 50ml volumetric flask.
- 2- Prepare 0.1M CuSO₄ in 50ml volumetric flask.
- 3- Leave the solutions with electrode in the beakers for the equilibrium.
- 4- Measure the voltage and current by changing the resistance until numerous numbers of bubbles appear through the experiment.

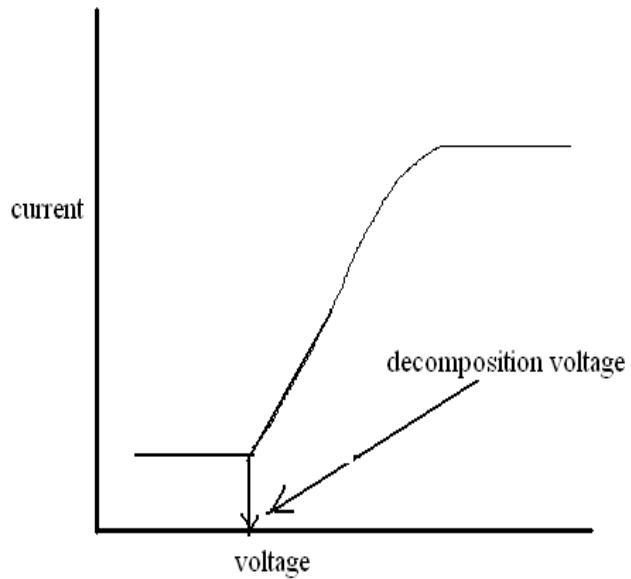


Results and calculations:

1- Arrange the results of I and V for the solutions in tables as the following .

CuSO ₄ solution		H ₂ SO ₄ solution	
I	V	I	V

2- Plot the curve between the values of I and V for H₂SO₄ solution and CuSO₄ solution and determine the decomposition voltage for these solutions.



Experiment (6B)

Determination of the solubility of sparingly soluble salt.

Aim of the experiment:

Measuring the equivalent conductance for sparingly soluble salt and determination the solubility.

Apparatuses and materials:

Filter paper, funnel, conduct meter, beakers 100ml, volumetric flask 25ml, heater, calcium sulphate, calcium carbonate, potassium chloride.

Procedure:

- 1- Prepare 0.01MKCl solution in 25ml volumetric flask.
- 2- Dissolve small amount of CaSO_4 salt in 50ml of distill water in small beaker then heat the solution and filter it and then leave the solution to cool.
- 3- Repeat the step 2 with CaCO_3 salt.
- 4- Measure the conductivity for two solutions.
- 5- Measure the conductivity for distilled water.

Results and Calculations:

- 1-Determine the cell constant K for 0.01MKCl solution as follows:

$$K = L / \bar{C}$$

The Specific conductance L for 0.01MKCl solution is $0.00143 \text{ ohm}^{-1}\text{cm}^{-1}$.

\bar{C} = Electric conductance (ohm^{-1}).

- 2-Calculate the Specific conductance for CuSO_4 solution, CaCO_3 solution and distilled water from this equation:

$$L = K * \bar{C}$$

- 3-Calculate the equivalent conductance at infinite dilution Λ_0 from the following.

$$\Lambda_0 = 1/2 [\lambda^\circ \text{Ca}^{2+} + \lambda^\circ \text{CO}_3^{2-}]$$

$$\Lambda_0 = 1/2 [\lambda^\circ \text{Ca}^{2+} + \lambda^\circ \text{SO}_4^{2-}]$$

If you know:

$$\lambda^\circ \text{SO}_4^{2-} = 159.6, \quad \lambda^\circ \text{Ca}^{2+} = 119, \quad \lambda^\circ \text{CO}_3^{2-} = 128$$

λ° = is the ionic equivalent conductance in an infinity dilution.

- 4- Calculate the Specific conductance for each solute from this equation:

$$L_{\text{solute}} = L_{\text{solution}} - L_{\text{H}_2\text{O}}$$

- 5- Calculate the solubility S from this equation:

$$S = 1000 L / \Lambda_0$$

- 6- Calculate the solubility constant as the following equation.

$$k_{\text{sp}} = [S / 2]^2$$

Experiment (7)

Salt effect on the reaction rate.

Aim of the experiment:

Study the influence of ionic strength on the reaction between Persulphate ions and iodide ions.

Apparatus and materials:

Five round flat bottom flasks 250ml , conical flask, pipette (25ml) , burette, measuring cylinder, stop watch clock, volumetric flasks (250ml, 100ml), potassium chloride, potassium persulphate, potassium Iodide, sodium thiosulphate, starch as an indicator.

Procedure:

- 1- Dry five round flask.
- 2- Prepare (0.04M) Potassium persulphate solution in 250ml volumetric flask.
- 3- Prepare (0.04M) Potassium Iodide solution in 250 ml volumetric flask.
- 4- Prepare (0.01M) sodium thiosulphate solution in 50 ml volumetric flask.
- 5- Prepare (0.4M) Potassium chloride solution in 100 ml volumetric flask.
- 6- Mix the solutions in round flasks as the table:

The mixture materials	1 st mixture material	2 nd . mixture material	3 ^{ed} mixture material	4 th . Mixture material	5 th . mixture material
H ₂ O	50	40	30	20	10
KCl	----	10	20	30	40
KI	25	25	25	25	25

- 7- Put 25ml of K₂S₂O₈ solution to the first flask and wait 5 minutes then put 25ml of K₂S₂O₈ solution to the second flask and soon until flask number 5 and wait 10 minutes after flask 5.
- 8- After 30 minutes take 25ml from the first flask and put it in conical flask contain few drops of starch and then titrate it with 0.01M Sodium thiosulphate solution until the color changes from blue to colorless, (calculate the time of titration t_2).
- 9- Take 25ml from the second mixture after 5 minutes and repeat step 8 and soon for all mixtures.

Results and Calculations:

- 1- Calculate the concentration of Potassium Iodide and Potassium persulphate in the mixture.

2- Calculate the concentration of Potassium chloride in each mixture.
 3- Calculate the ionic strength for all mixtures from the following equation:

$$\mu = \frac{1}{2} \sum (M Z^2)$$

4- Calculate the time of reaction t for each mixture from this equation:

$$t = t_1 + (t_2 / 2)$$

t_1 = 30 minutes.

t_2 = the time of titration.

5- Calculate the rate of reaction k from the following equation:

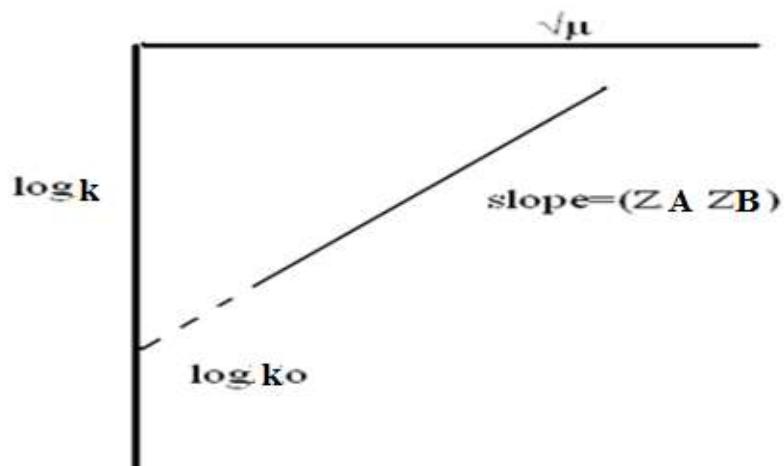
$$t = 1/k * [X / 0.01(25 - X)]$$

6- Put the results in this table:

No. of sample	$X(V_{Na_2S_2O_3})$	k	$\log k$	μ	$\sqrt{\mu}$	t

7- Plot $\log k$ against $\sqrt{\mu}$ for calculate k and $Z_A Z_B$ from this equation:

$$\log k = \log k_0 + (Z_A Z_B) \sqrt{\mu}$$



Experiment (8)

Determination of activity coefficient from solubility of weak electrolyte.

Aim of the experiment:

- 1- Determination the activity coefficient for benzoic acid as weak electrolyte.
- 2- Study the effect of ionic strength on the activity coefficient.

Apparatus and materials:

Burette 50 ml. ,pipette 10ml., volumetric flask (50ml.) , beakers 25ml., stop watch clock, Benzoic acid, sodium Hydroxide solution 0.03M, sodium Chloride solution 1M, (ph.ph.) as indicator.

Procedure:

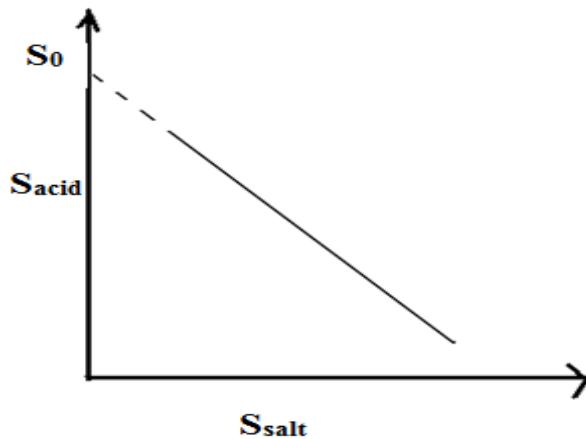
- 1- Prepare (1M) of sodium Chloride solution in 50 ml. volumetric flask as salt.
- 2- Prepare (0.02M) of Sodium Hydroxide solution in 100 ml. volumetric flask for titration.
- 3- Prepare five solutions from 1M sodium Chloride solution by dilution the following concentration in 25ml volumetric flask 1M, 0.5M, 0.25M, 0.1M, 0.05M.
- 4- Wash and dry six 50ml. conical flask and weight 0.1g from benzoic acid and put it in these flasks.
- 5- Drop 12.5 ml. of distilled water in first flask and 12.5ml. From 0.05M sodium Chloride solution in second flask and so on for the other flask according the concentration.
- 6- Shake the flasks for five minutes about one hour.
- 7- Filter the solutions and with draw 10ml. from each filtrate solution and titrate it with 0.02 M Sodium Hydroxide solution withph.ph. As indicator and record the volume.

Result and Calculation:

- 1- Determine the solubility S_0 of benzoic acid in pure distilled water (first flask).
- 2- S_{salt} is the concentration of the sodium Chloride salt solution.
- 3- S_{acid} is the concentration of Benzoic acid solution.
- 4- Put the results in the following table:

No.of sample	V volume of NaOH	S_{acid}	S_{salt}

- 5- Plot the curve between S_{salt} and S_{acid} to determine S_0 .



6-Calculate the ionic strength for all solutions from this equation:

$$\mu = \frac{1}{2} \sum (M Z_i^2)$$

M is the concentration in molar.

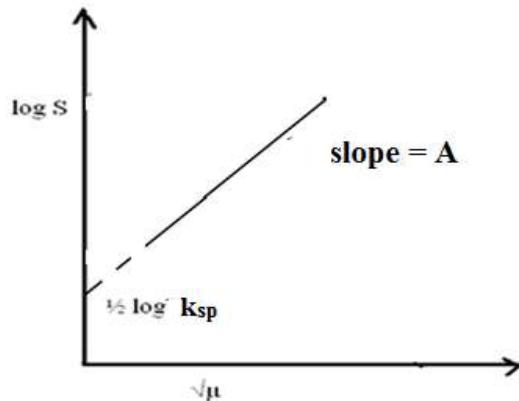
Z_i is the charge of ions.

7-Record the result in the table:

No. of sample	S _{acid}	Log S _{acid}	μ_{BaCl_2}	$\sqrt{\mu}$

8- Plot log S against $\sqrt{\mu}$ to calculate water constant A at 25°C according the following equation:

$$\text{Log } S = \frac{1}{2} \log k_{\text{sp}} + A \sqrt{\mu}$$



Calculate the activity coefficient (f) according the following equation:

$$k_{\text{sp}} = S_0^2 f^2$$

k_{sp} is the solubility constant.

Experiment No. (9)

Anodizing Aluminum (Honeycomb Nonporous Al₂O₃)

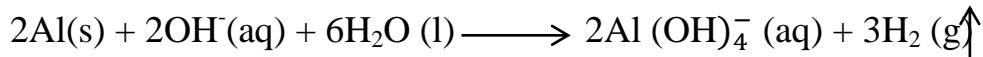
1-Theory

Untreated aluminum has a layer of oxide about 10⁻⁸m thick. This explains its apparent lack of reactivity. Anodizing, invented in 1923, is used commercially to thicken this layer to 10⁻⁵m to improve the metal's corrosion resistance. The relevant equations are:

Cleaning:



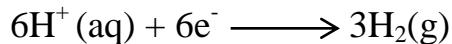
Once the oxide is removed:



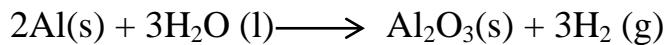
Electrolysis at the anode:



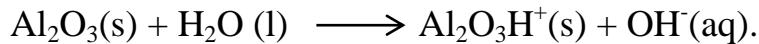
Electrolysis at the cathode:



Electrolysis overall:



The oxide coating develops a positive charge by the reaction:



Thus it attracts dyes that contain colored anions. These are absorbed in the pores of the sponge-like oxide layer, where they can be trapped by heating the oxide to form an Al₂O₃.H₂O seal.

2- Experimental

In this experiment, the student will anodize a piece of aluminum and partially color it with an organic dye. By taking quantitative measurement of the current flow, he will be able to calculate the thickness of the anodizing layer.

Problem: How is aluminum anodized and colored?

Apparatus:

- Safety goggles.
- Connecting wires,
- Crucible tongs,
- Beakers,
- DC power supply,
- Ammeter

Materials:

- Acetone,
- 3.3 M Sulphuric acid,
- ret Fabric Dye,
- Aluminum foil,
- Aluminum metal.

Procedure:

1. Obtain a clean 400 ml beaker, and line it with aluminum foil.
2. Obtain a piece of aluminum metal to be anodized. Clean first with a detergent, and then rind with water. De-grease the aluminum strip by rubbing with a tissue soaked in acetone and allowing drying. From now on hold the aluminum by the top few cm only (where it will not be anodized). Dip the bottom half of the aluminum strips into the sodium hydroxide solution in a beaker. Leave it until it brings to effervesce, indicating that the surface layer of oxide has been removed. (This will take about one minute). Now remove the strip and dip the cleaned portion of it into the nitric acid for a few seconds to neutralize the alkali. Then rinse away the acid with water. Handle the aluminum piece by its edges only; do not touch its surface with your fingers. After cleaning dry it with acetone, lay the piece down on a clean, dry paper towel.
3. Add 250 ml of 3.3 M sulphuric acid to your aluminum lined beaker. Make sure about 2 cm of foil are above the surface of the sulphuric acid. The aluminum foil will act as a cathode and is to be connected to the negative side of your power source. Set up the power supply and ammeter as shown in the illustration below. DO NOT connect the negative terminal to the aluminum foil at this time. The piece of aluminum to be anodized is to be suspended in the sulphuric acid solution in the center of the beaker. Attach a burette clamp to a ring stand above the beaker. The lead from the positive terminal of the power supply should be tied or wrapped around the burette clamp so that your piece of aluminum will be suspended perfectly in the beaker held by the alligator clamp.

4. Have your teacher check your set up before you make the final connections, when ready, note the time to the nearest second at which you connect the negative lead to the aluminum foil. Record this time in the data table.
5. Let the cell operate for 20 minutes. At the start of the cell operation, note and record the current as indicated by the ammeter. Have ready a second 400 ml beaker three-quarters filled with distilled water to rinse the piece of aluminum when it has been anodized.
6. While the cell is operating, we will prepare the dye bath. In a 100 ml beaker, add 10 ml of red, green, or yellow Rite fabric dye. Add 40 ml of distilled water to this. Set the beaker on a piece of wire gauze on a ring stand, and begin heating with your Bunsen burner or (hotplate). When the solution begins to boil, turn down the heat to just maintain boiling.
7. After the aluminum piece has been anodized for 20 minutes, again note and record the ammeter reading. Then, disconnected the negative lead, noting the exact time when you do so. Record this value.
8. Use the crucible tongs to remove the piece of aluminum from the sulphuric acid solution. Disconnect the alligator clip. Quickly rinse the aluminum in the beaker of distilled water, and then immediately place it in the hot dye bath. Let it remain in the dye bath for 10 minutes, keeping the dye solution just at or near the boiling point.
9. After 10 minutes have elapsed, remove the aluminum from the hot dye with your crucible tongs, rinse it well, and dry it with a paper towel. In the observation section, write a complete description of the piece of anodized aluminum.
10. With a metric ruler, measure the length and width of the anodized portion of your aluminum piece. Record these values. From your data and the instructions in the calculations section, calculate the thickness of the anodized layer.

Data:

Beginning time: _____ Ending time: _____

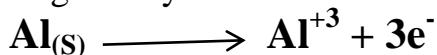
Beginning Current: _____ Ending Current: _____

Anodized aluminum: Length: _____ Width: _____

Observation: Describe the anodized piece of aluminum: _____

3. Calculations:

In order to calculate the thickness of the anodized layer, we first need to determine the amount of aluminum oxide that formed. The half-cell reaction that occurred at the anode is given by:



The equation tells us that for every mole of electrons that flowed, one-third of a mole of aluminum was oxidized. A mole of electrons carries a charge of 96,500 coulombs. A coulomb is a unit of electrical charge. The flow of one coulomb per second is called an ampere. Therefore, we can find the number of coulombs your cell used while you were anodizing the aluminum by multiplying the current in amperes times the time in seconds.

From your beginning and ending readings of current
Taken from the ammeter calculate the average value of
the current:

From your beginning and ending times, calculate the
number of seconds that the current was flowing:

Now multiply the amperes times the seconds: This will
give you coulombs:

Divide this value by 96,500 coulombs/mole to find moles
of electrons

Divide this by 3 to find moles of aluminum that were
oxidized:

Aluminum oxide has the formula Al_2O_3 . Since it takes two
moles of Al to form one mole of Al_2O_3 divide the moles
of Al reacted by 2

Multiply this by 102 g/mole, the molecular mass of
aluminum oxide.

The density of Al_2O_3 is 3.97 g/cm³. Divide the grams of
aluminum oxide by its density in order to calculate the
volume of oxide formed:

Now, from your data, calculate the area of the aluminum
piece that was anodized:

If the volume of aluminum oxide is divided by the area
anodized, this will yield the thickness of the anodized
layer.

Current (average): _____

Time for current: _____

Coulombs: _____

Moles of e^{-} : _____

Moles of Al reacted: _____

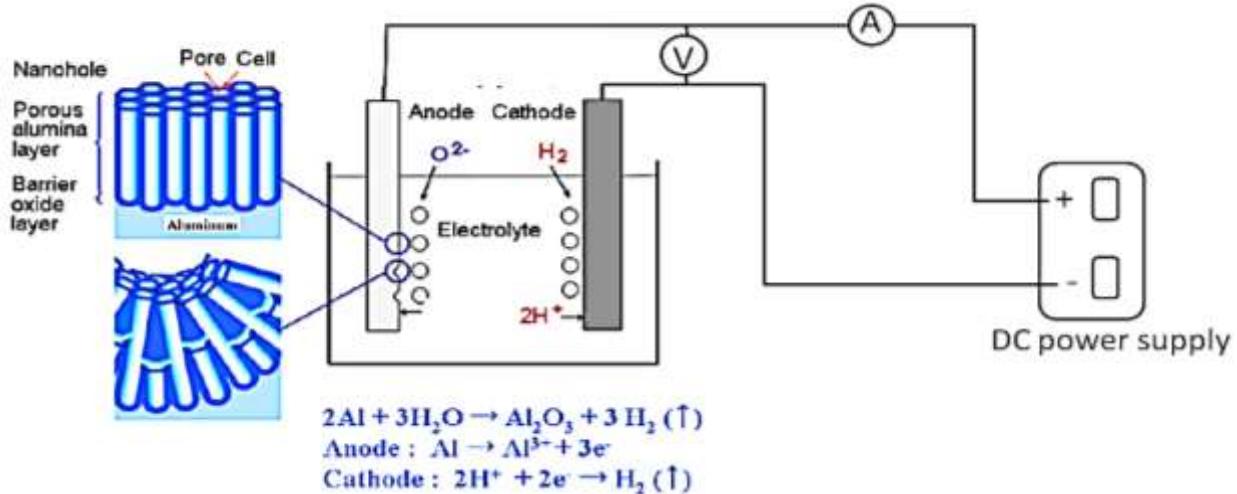
Moles of Al_2O_3 : _____

Grams of Al_2O_3 : _____

Volume of Al_2O_3 : _____

Area anodized: _____

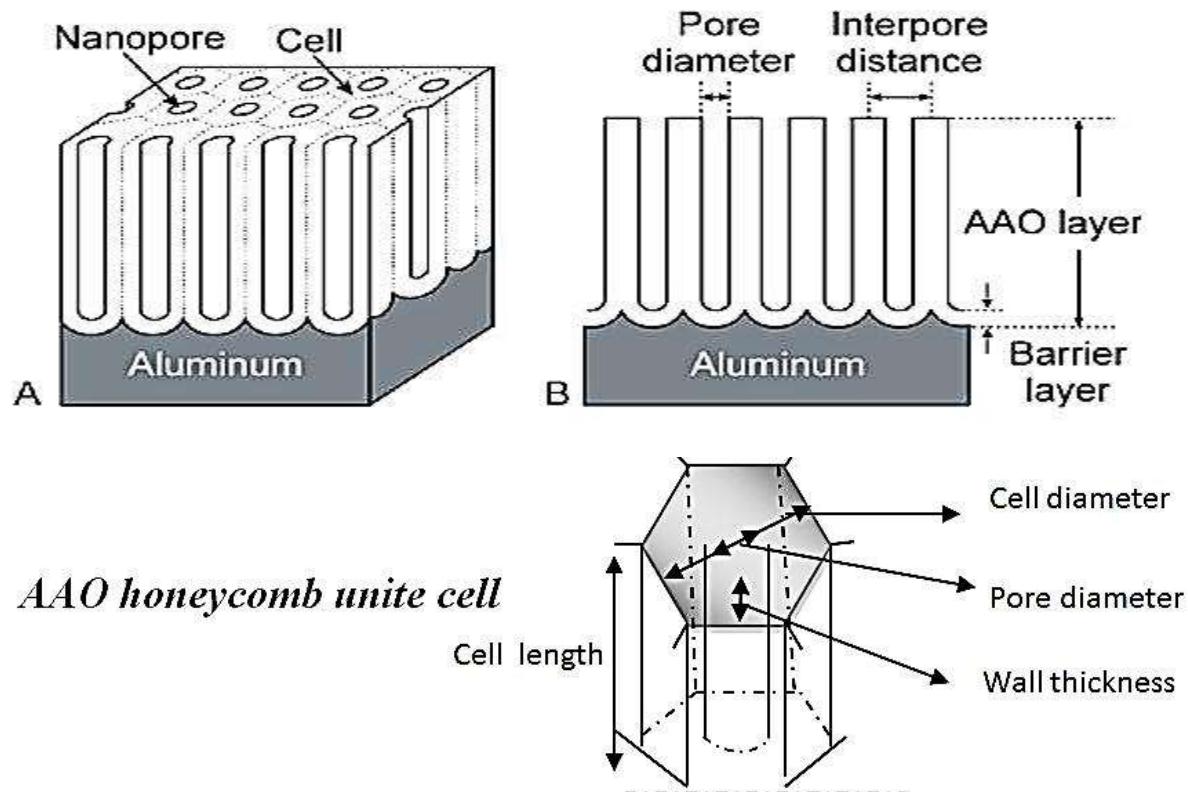
Thickness of anodized
layer: _____



4. Extensions:

There are a great many variables in this experiment such as: electrolysis time, voltage, current density, concentration and types of electrolyte, temperature of electrolyte, temperature of dye bath and type of dye. Investigations of some of these could form interesting projects.

It is possible to measure the gain in mass of the anode by rinsing the aluminum strip with acetone and weighing it immediately before and immediately after electrolysis.



Experiment No. (11)

Synthesis and Characterization of Nano Dye Sensitive solar Cell (DSSC) Grätzel cell

Introduction:

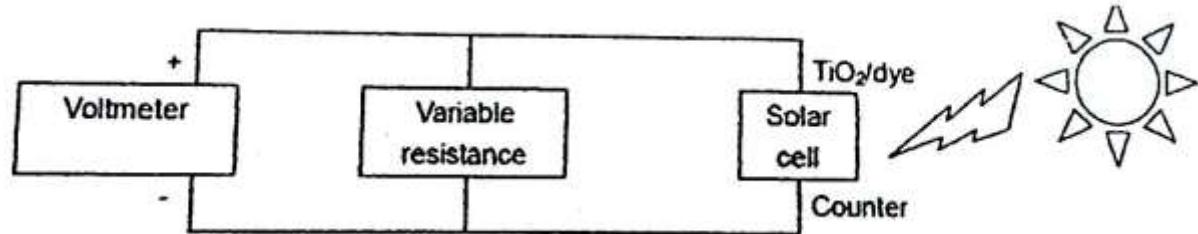
A dye-sensitized solar cell (DSSC, DSC or DYSC) is a low-cost solar cell belonging to the group of thin film solar cells. It is based on a semiconductor formed between a photo-sensitized anode and an electrolyte, a photo electrochemical system. The modern version of a dye solar cell, also known as the Grätzel cell, was originally co-invented in 1988 by Brian O 'Regan and Michael Grätzel.

The dye sensitized solar cell is composed of two surfaces of transparent conductor (mostly a conducting oxide on glass (FTO or JTO), onto one of which a few μm thick film of wide band gap semiconductor has been deposited in the form of a self-connected network of nm-sized particles, with a network of similarly or large sized self-connected pores in between the particles. Energy conversion in a DYSC is based on the injection of an electron from a photo excited state of the sensitizer dye into the conduction band of the nanocrystalline semiconductor (TiO_2 or ZnO are by far the most employed oxide semiconductor), these cells also employ a liquid electrolyte (usually an iodide/triiodide redox-active couple dissolved in an organic solvent) to reduce the dye cation (viz., regenerate the ground state of the dye). Regeneration of iodide ions, which are oxidized in this reaction to triiodide is achieved at a catalyzed counter electrode;



S: represents the dye sensitizer.

1. Connect solar cell, variable resistance and voltmeter with cables.



2. Shine the light from the side of TiO_2/dye electrode.

(Note; light intensity has to be known in advance to calculate power conversion efficiency. It can be measured by a power meter or a calibrated photodiode.)

3. Set the resistance at maximum value and record voltage.
4. Decrease the resistance and record voltage.
5. Repeat 4 until the voltage reaches nearly zero and turn the light off.
6. Calculate corresponding current at each recorded point.

$$I = V/R$$

7. Plot voltage (X-axis) vs. current (Y-axis).
8. Plot voltage (X-axis) vs. power (Y-axis) to find P_m .
9. Calculate fill-factor (ff).

$$ff = \frac{I_m \times V_m}{I_{sc} \times V_{oc}} = \frac{P_m}{I_{sc} \times V_{oc}}$$

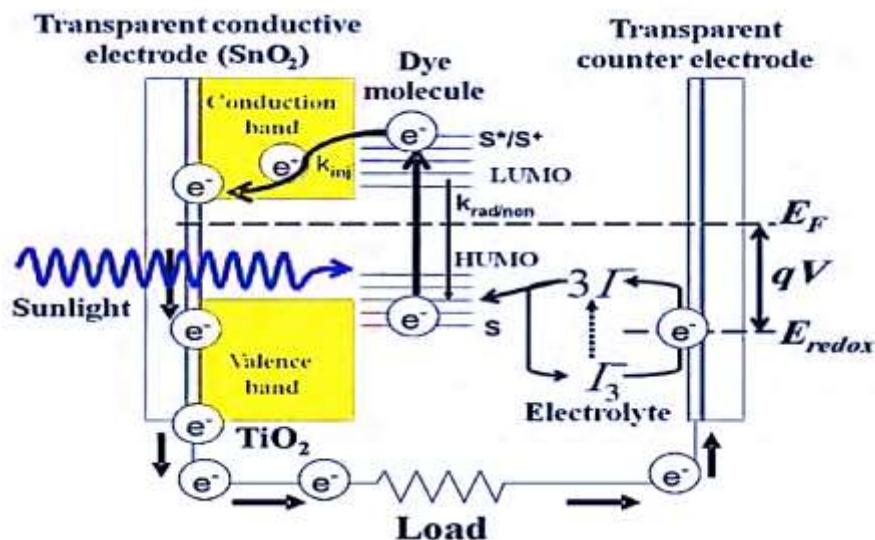
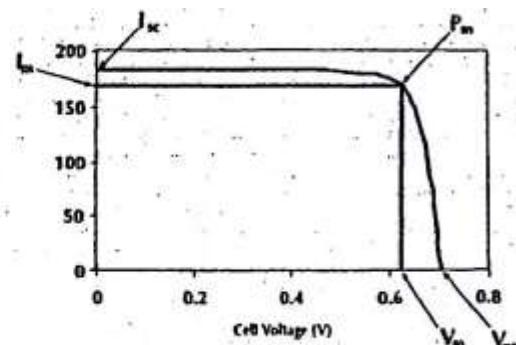
10. Calculate power conversion efficiency (η).

$$\text{Power conversion efficiency } (\eta) = \frac{\text{Output power}}{\text{Input power}} \times 100 \text{ (%)}$$

$$= \frac{P_m \text{ (mW/cm}^2\text{)}}{\text{Light intensity (mW/cm}^2\text{)}} \times 100 \text{ (%)}$$

$$= \frac{I_{sc} \text{ (mA/cm}^2\text{)} \times V_{oc} \text{ (V)} \times ff}{\text{Light intensity (mW/cm}^2\text{)}} \times 100 \text{ (%)}$$

J_{sc} : the photocurrent density measured at short circuit.
 V_{oc} : the open circuit photo-voltage.
 ff : the fill factor of the DSSC.
 I_{in} : the intensity of the light of the system, in mW cm^{-2} .
 P_{max} : maximum output power density of the DSSC.
 η : the total-light-to-electricity conversion efficiency.



Operating principle of dye sensitized solar cell

The efficiency of a DSSC depends on four energy levels of the component: the excited state (approximately LUMO) and the ground state (HOMO) of the photosensitizer, the Fermi level of the TiO_2 electrode and the redox potential of the mediator (I/I_3^-) in the electrolyte.

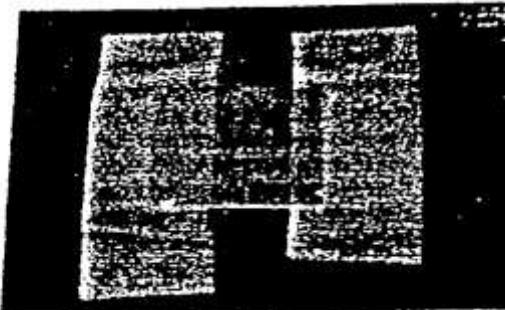
Materials and chemicals

- Florid-doped tin dioxide (FTO) or Indium Tin Oxide (ITO) conductive glass (≥ 2 plates).
- A Source of organic dye (ex. raspberries).
- Titanium Dioxide nanocrystals.
- Acetic acid.
- Water.
- Glass stirring Rod.
- Scotch tape.
- Graphic Pencil.
- Liquid electrolyte solution.

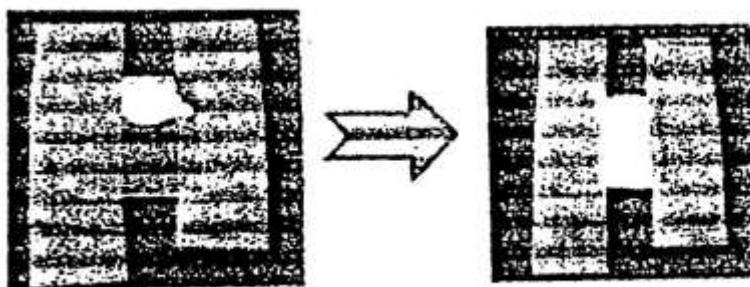
- Binder clips.
- Wires.
- A Multimeter.
- Tungsten Halogen Lamp.
- Testing Apparatus.

Experimental procedure:

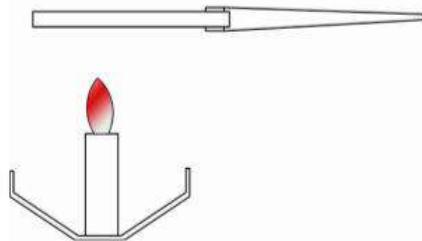
1. Determine the conductive side of glass by touching both of protruding leads of the multi-meter with one side of the glass. The conductive side could be identified with average resistance from 20-38 ohms.
2. Fix two sides of the plate using tape with the conductive sides facing up.



3. Prepare the titanium dioxide paste by adding a few drops of very dilute acetic acid (0.035M) to 1 gram TiO_2 . The resulting mixture was grinded in a mortar and pestle until a colloidal suspension with a smooth consistency (like cake icing) was observed.
4. Add 2-3 drops of the TiO_2 suspension onto the conductive side and spread out the TiO_2 evenly on the surface of the plate with glass rod. Carefully remove the tape without perturbing the TiO_2 layer.



5. Dry the glass with TiO_2 under room temperature and put this electrode on top of a hot plate and heat it at approximately 150 C° for 10 minutes.
6. While heating, light the candle and coat the conductive side of the other piece of glass with graphite over 45 sec.



7. Cool both plates to room temperature.
8. Submerge the plate with TiO_2 face-down in the dye solution and take out quickly but carefully without cracking the TiO_2 . Pomegranate juice, rich in anthocyanine, is a great source of dye.
9. Wash the dye layer with ethanol carefully.
10. Clamp both the plates together and apply 2 drops of KI/I_2 electrolyte solution onto the interface between the two plates.
11. Allow the electrolyte to cover the surface of TiO_2 .
12. Measure the photo-voltage under the UV and fluorescent light after forming a circuit using a multimeter.



Evaluation of dye-sensitized solar cells

Equipment

- Light source (OHP, Halogen lamp, etc.)
- Cables
- Variable resistance (or switchable fixed resistance ex. 50, 100, 200, 500, 800, 1k, 2k, 5k, 10k, 20k, 40k, 80k, 100k, 200k, 400k and 800k Ω).