

PHYSICAL CHEMISTRY

PRACTICALS

(LAB MANUAL)

G.T.N. ARTS COLLEGE (AUTONOMOUS)

(Affiliated to Madurai Kamaraj University, Madurai)
Dindigul – 624 005

POST GRADUATE AND RESEARCH DEPARTMENT OF CHEMISTRY

EXPERIMENTS IN PHYSICAL CHEMISTRY

PRACTICALS



NAME : _____

REG.No. : _____

CLASS : _____

YEAR : _____

GENERAL INSTRUCTIONS TO THE STUDENTS

I. LABORATORY SAFETY

The following points are important and must be followed to avoid any kind of accidents inside the laboratory:

- a) Do not smoke, drink and eat inside the laboratory.
- b) Try to wear laboratory coat and safety goggles.
- c) Do not perform many unauthorized experiments without the permission of the instructors.
- d) For any kind of chemical burn, wash the area with plenty of water and inform your instructor promptly.
- e) Do not pipette out liquids by mouth use suction bulbs.
- f) Do not try to smell, taste any chemicals without the instructions of the concerned person.
- g) Do not add water to the concentrated acids.
- h) Do not touch any unknown chemicals by hands.
- i) Before leaving the laboratory, wash your hands properly.

II. GENERAL RULES

- a) Try to understand the experiment before you start it. If you have any query, discuss with the instructor.
- b) If some reagent / apparatus is not found, ask your laboratory attendant.
- c) Finish the experiments within the specified time period.
- d) Regular submission of the laboratory note book is mandatory. Late submission will not be entertained for evaluation.
- e) Never use mobile phones inside the laboratory.

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Programme	B.Sc,Chemistry	Programme Code	UCH		
Course Code	20UCHC6R	Number of Hours/Cycle	3		
Semester	VI	Max. Marks	100		
Part		Credit	4		
Core Practical V					
Course Title	Experiments in Physical Chemistry		L	T	P
Cognitive Level		Up to K4			30

Scheme of Evaluation (Max.marks100)

Internal Assessment 40 Marks

Regular Test in the Class	30 Marks
Observation note book	10 marks
Total	40 marks

External Examination: 60 Marks (3 hours)

Viva voce	10 marks
Record Notebook	10 marks
For completion of the experiment	20 marks
Short procedure	10 marks
Result	10 marks
Total	60 marks

Ex.No: 1. TRANSITION TEMPERATURE**DATE:**

AIM:

1. To find out the transition temperature of the given salt hydrate.
2. To find out the molar depression constant for the salt hydrate using the unknown molecular weight of “B”
3. To find out the molecular weight of solute using the known KJ value.

APPARATUS REQUIRED:

Thermometer, glass rod, boiling tube, 250ml beaker,

PRINCIPLE:

The crystalline solids on heating lose their water of crystallization at particular temperature to change into anhydrous form (or) less hydrated form, their transition takes place at constant temperature and is accompanied by absorption (or) evolution of the temperature at which such a transition takes place is called transition temperature.

TABULATION =I

SYSTEM - A		SYSTEM A+B₁		SYSTEM A+B₁+B₂	
Time (Sec)	Temperature (°C)	Time (Sec)	Temperature (°C)	Time (Sec)	Temperature (°C)
0		0		0	
30		30		30	
90		90		90	
120		120		120	
150		150		150	
180		180		180	
210		210		210	
240		240		240	
270		270		270	
300		300		300	
330		330		330	
360		360		360	
390		390		390	
420		420		420	
450		450		450	
480		480		480	
510		510		510	
540		540		540	
570		570		570	
600		600		600	

PROCEDURE:

7 gram of given hydrate are weighted into two pockets 350gram are known molecular weight or solution B are weighted into two pockets 350 gram of unknown molecular weight or the given solute “C” are weighted in two pockets (c1,c2) gram of the given salt hydrate “A” is introduced into a boiling tube is fitted with a stirrer and a thermometer, the bulb of the thermometer should be kept involved into salt hydrate the boiling tube is kept in a water bath slowly raised by using thermometer. The rate of increase in temperature is noted.

The kept uniform by slowly and regular stirring the temperature gradually increase and remaining constant for the same time and again increase apparatus is taken out of water bath. It is allowed to cool, slowly the temperature in noted

$A+B$, $A+B_1+B_2$, $A+C_1+C_2$ system at transition temperature are found out them the result of temperature are found out.

GRAPH:

The graph is plotted between the times. The second in “x” axis as the temperature in “y” axis from the graph. The transition temperatures of the system are found out.

TABULATION =II

SYSTEM - A		SYSTEM A+B ₁		SYSTEM A+B ₁ +B ₂	
Time (Sec)	Temperature (°C)	Time (Sec)	Temperature (°C)	Time (Sec)	Temperature (°C)
0		0		0	
30		30		30	
90		90		90	
120		120		120	
150		150		150	
180		180		180	
210		210		210	
240		240		240	
270		270		270	
300		300		300	
330		330		330	
360		360		360	
390		390		390	
420		420		420	
450		450		450	
480		480		480	
510		510		510	
540		540		540	
570		570		570	
600		600		600	

System -A+B1

Transition temperature of pure solvent A =

Transition temperature of pure solvent B₁ =

Depression of Freezing point ΔT_b =

Weight of solvent A₁W₁ =

Weight of known solute B₁ W₂ =

Molecular weight of known solute m₁ =

Molar depression constant kF₁ = $\frac{W_1 \times \Delta T_b \times m_1}{1000 \times w_2}$

System –A+B₁+B₂

Transition temperature of pure solvent A =

Transition temperature of pure solvent B₂ =

Depression of freezing point ΔT_b =

Weight of solvent A₁W₁ =

Weight of known solute B₂w₂ =

Molecular weight of known solvent m₂ =

System A+C1

Transition temperature of pure solvent A =

Transition temperature of pure unknown solute C₁ =

Depression of freezing point ΔT_b =

Weight of the solvent A₁, W₁ =

Weight of the unknown solute C₁W₂ =

Molecular Weight of unknown solute m₂ = $\frac{W_2 \times 1000 \times KF}{W_1 \times \Delta T_b}$

Using KF₁,

Using KF₂,

Using KF₁,

Using KF₂,

SYSTEM A+C1+C2:

Transition temperature of pure solvent A =

Transition temperature of pure unknown solute C₁ =

Depression of freezing point ΔT_b =

Weight of the solvent A₁, W₁ =

Weight of the unknown solute C₁W₂ =

Molecular Weight of unknown solute m₂ = $\frac{W_2 \times 1000 \times KF}{W_1 \times \Delta T_b}$

Using KF₁,

Using KF₂,

Using KF₁,

Using KF₂,

RESULT:

1. Transition temperature salt hydrate =

2. Molar depression constant of salt hydrate (KF_1) =

3. Molar depression constant of salt hydrate (KF_2) =

4. Molecular weight of unknown solute A+C1

BY using KF_1 =

BY using KF_2 =

5. Molecular weight of unknown solute (A+C1+C2)

By Using KF_1 =

By Using KF_2 =

Ex.No: 2.

RAST MACRO METHOD

DATE:

AIM :

- i) To find the motor depression constant of “X” by using known molecular weight of solute “Y”
- ii) To find out the molecular weight of unknown solute “Z” using the solvent “X”

APPARATUS REQUIRED:

Thermometer, glass rod, Boiling tube, 250ml beaker,

PRINCIPLE:

When volatile non electrolyte dissolves in a non-electrolyte solvent A decreasing depression in greeting point is directly related to molarity of the solution

$$\Delta T_y = K F m$$

$$\Delta T F = K F$$

$\Delta T F$ = It is depression in freezing point

$K F$ = Molar freezing point constant

m_2 = Molecular masses of the solute

w_2 = weight of solute

w_1 = weight of solvent

System	Melting point of the system
$\mathbf{X}$ $\mathbf{X} + \mathbf{Y}_1$ $\mathbf{X} + \mathbf{Y}_1 + \mathbf{Y}_2$ $\mathbf{X} + \mathbf{Z}_1$ $\mathbf{X} + \mathbf{Z}_1 + \mathbf{Z}_2$	

PROCEDURE:

Exactly 7 gram of solvent weighted in two pockets 0.35g of known solvent y are weighted into pockets y1 and y2 0.35gram of unit solute z are weighted into two pockets are z1 and z2.

7 gram of solvent in introduced into the pure boiling tube is filled with thermometer are stirrer the tube is placed into a water bath. The temperature of the water is slowly increased then the solid melts and clear liquid is obtained the tube is taken off from the water bath cooled slowly in the air.

At the temperature appears in crystalline stands the freezing temperature is needed. It is the melting point of the system (x1,y1) the process is repeated until get a concordant value.

0.35 gram of known molecular weight of y2 is added to the boiling tube containing x1,y1 the melting point of x+y1+y2 system is found of the another dry boiling tube the melting point of x1+z1+z2 is found out. The resulted are observed from the result of motor depression constant of the solvent x and molecular mass of unknown solute are calculator

For system (x+y₁):

1. Melting point of the pure solvent **x(T₁)** =
2. The depression of freezing point ΔTF =
3. Melting point of the system(**x+y₁**) =
4. Weight of pure solvent **x (w₁)** =
5. Weight of pure solute **y₁ (w₂)** =
6. Molecular weight of known solute (**m₂**) =
7. Molar depression constant **kF₁** = $\frac{W_1 \times \Delta TF \times m_2}{1000 \times w_2}$

Using KF₁

For system (x+y₁+y₂)

1. Melting point of the pure solvent **x(T₁)** =
2. The depression of freezing point ΔT_F =
3. Melting point of the system **(x+y₁+y₂)** =
4. Weight of pure solvent (x) (w₁) =
5. Weight of pure solute **y₁+y₂** (w₂) =
6. Molecular weight of known solute (m₂) =
7. Molar depression constant kF₂ = $\frac{W_1 \times \Delta T_F \times m_2}{1000 \times w_2}$

Using KF₂

For system (x+z₁)

1. Melting point of the pure solvent **x(T₁)** =
2. Melting point of the system(**x +z₁**) =
3. The depression of freezing point ΔTF =
4. Weight of pure solvent (x) **w₁** =
5. Weight of known solute (**z₁**) **w₂** =
6. Molecular weight of unknown solute (**m₂**) = $\frac{W_2 x 1000 x \Delta K_F}{W_1 x \Delta TF}$

Using KF1,

Using KF2,

For system (x+z₁+z₂)

1. Melting point of the pure solvent **x(T₁)** =
2. Melting point of the system(**x +z₁+ z₂**)**T₂** =
3. The depression of freezing point **Δ TF** =
4. Weight of pure solvent (x) **w₁** =
5. Weight of unknown solute (**z₁ + z₂**) **w₂** =
6. Molecular weight of unknown solute (**m₂**) = $\frac{W_2 \times 1000 \times \Delta K_F}{W_1 \times \Delta T_F}$

Using KF1

Using KF2

RESULT

1. Melting point of the pure solvent (T_1) =
2. Molar depression constant for the system ($x+y_1$) =
3. Molar depression constant for the system ($x+y_1+y_2$) =
4. Molecular weight of unknown solute,
 - a) By using value for ($x+z_1$) =
 - b) By using value for ($x+z_1$) =
5. Molecular weight of unknown solute
 - a) By using KF_1 value for ($x+z_1+z_2$) =
 - b) By using KF_2 value for ($x+z_1+z_2$) =

**Ex.No: 3 PHASE DIAGRAM FOR A COMPUND SYSTEM-
DATE: SIMPLE EUTECTIC**

AIM :

To construct a phase diagram for a binary mixture and determine the eutectic point of the compound A and B and interpret the result.

PRINCIPLE:

When a binary mixture is i) correct in a solid separate out and the composition of solid depends upon the nature of the substance of the compounds are chemically dissimilar to each other. The freezing point, a eutectic system is obtained the temperature at which the separation results is known as eutectic temperature.

System	Weight of A (g)	Weight of B (g)	Weight of A/Weight of A+B x100	Melting point of system 0°C
A	2	0	100	
A+B₁	2	0.2	90.90	
A+B₁+B₂	2	0.4	83.33	
A+B₁+B₂+B₃	2	0.6	76.92	
A+B₁+.....B₄	2	0.8	71.92	
A+B₁+.....B₅	2	1.0	66.66	
A+B₁+.....B₆	2	1.2	62.50	
A+B₁+.....B₇	2	1.4	58.82	
A+B₁+.....B₈	2	1.6	55.55	
A+B₁+.....B₉	2	1.8	52.63	
A+B₁+.....B₁₀	2	2.0	50.00	

PROCEDURE:

Exactly 7 gram of weighed pocket 0.7 gram of compound A is also weighted in 10 more pocket exactly 7 gram of compound B is also weighted into pockets 0.7gram of compound B is also weighted into 10 more pockets. Then 7 g of compound A is transferred into a clean boiling tube and substance should never set to the side of the tube. It is closed with a cork containing a sensitive thermometer and a stirrer. The arrangement is kept in a water and boiled over a burned the heating is continued until the mixture i) completely melted and the temperature is above 10ohigher than melting point.

Now the melt is stirred well to allow the cool slowly, the turbidity just appear and study the temperature i) noted is as the melting point of “A” and “B” in the boiling tube and the melting point of a system A+B is noted. Repeat this process until the addition of (B2,B3,.....B10) to A+B system of boiling tube. The melting point of various system of boiling tube is calculated.

Similarly , 7g of compound B is found out Repeat the process by the addition of compound (A1,A2.....A10) to the boiling tube which contains pure compound B the melting point of various systems are found out.

System	Weight of B (g)	Weight of A (g)	Weight of B/Weight of A+B x100	Melting point of system 0°C
B	2	0	100	
B₁+A₁	2	0.2	90.90	
B₁+A₁+A₂	2	0.4	83.33	
B+A₁+A₂+A₃	2	0.6	76.92	
B+A₁+.....+A₄	2	0.8	71.92	
B+A₁+.....+A₅	2	1.0	66.66	
B+A₁+.....+A₆	2	1.2	62.50	
B+A₁+.....+A₇	2	1.4	58.82	
B+A₁+.....+A₈	2	1.6	55.55	
B+A₁+.....+A₉	2	1.8	52.63	
B+A₁+.....+A₁₀	2	2.0	50.00	

GRAPH :

A graph is plotted between melting point of the system in X=axis and the composition of A and B in Y=axis from the graph eutectic temperature of the composition and recorded.

RESULT :

1. Melting point of “A” T_1 =
2. Melting point of “B” T_2 =
3. Eutectic Temperature (E) =
4. Eutectic composition (C) =

DATE: _____ **COMPUND FORAMTION**

To construct the phase diagram for the system including two compounds A and B under the residue.

Thermometer, glass rod, Boiling tube, 250ml beaker,

There are numerous system including two compounds metal organic compounds and inorganic compounds salt dissolved in water which fall under this conditions the two compounds of a system contain stage enter into chemical condition with another chemical is stable come the number formed hen two compound are metals similarly the several sample compound are formed. When two compound salt and water numbers of salt hydrates are warmed such as two kindly in depending upon whether the compound formed has controlled.

System	Weight of A (g)	Weight of B (g)	Weight of A/Weight of A+B x100	Melting point of system 0°C
A	2	0	100	
A+B₁	2	0.2	90.90	
A+B₁+B₂	2	0.4	83.33	
A+B₁+B₂+B₃	2	0.6	76.92	
A+B₁+.....B₄	2	0.8	71.92	
A+B₁+.....B₅	2	1.0	66.66	
A+B₁+.....B₆	2	1.2	62.50	
A+B₁+.....B₇	2	1.4	58.82	
A+B₁+.....B₈	2	1.6	55.55	
A+B₁+.....B₉	2	1.8	52.63	
A+B₁+.....B₁₀	2	2.0	50.00	

PROCEDURE:

Exactly 2 gram of compound is weighted a pocket 0.2g of a compound a weight 10 more pockets. Exactly 2g of compound B is weighted in a pocket and 02.g of same compound is weighted 10 more pocket 0.2g of compound A is transferred into a bright boiling tube and group should not stick the sides of the test tube. It is used a stirrer containing sensitive thermometer and stirrer

This arrangement is kept in a water bath and heated in a water bath with help of burner the heating is continued until the minute is completely need and temperature of minute mixture is about higher than melting point and stirred well to make uniform concentration slowly.

When a turbidity first appears the temperature is needed are reach melting point of A and B added to boiling tubes and melting point of system A+B is needed repeated the process until the addition of B1,B2,..... B10 to A+B system of same boiling tube.

The melting point of substance of continuous various system is calculated similarly 2g of compound B is introduced into another boiling tube, the melting point of pure compound B is needed, Repeat the process by adding A1,A2,..... A10 to boiling tube with contain the pure compound B, the melting point to various systems is found out.

System	Weight of B (g)	Weight of A (g)	Weight of B/Weight of A+B x100	Melting point of system 0°C
B	2	0	100	
B₁+A₁	2	0.2	90.90	
B₁+A₁+A₂	2	0.4	83.33	
B+A₁+A₂+A₃	2	0.6	76.92	
B+A₁+.....+A₄	2	0.8	71.92	
B+A₁+.....+A₅	2	1.0	66.66	
B+A₁+.....+A₆	2	1.2	62.50	
B+A₁+.....+A₇	2	1.4	58.82	
B+A₁+.....+A₈	2	1.6	55.55	
B+A₁+.....+A₉	2	1.8	52.63	
B+A₁+.....+A₁₀	2	2.0	50.00	

RESULT :

1. Melting point of pure “A” =
2. Melting point of pure “B” =
3. Eutectic temperature T_1 =
4. Eutectic temperature T_2 =
5. Eutectic temperature C_1 =
6. Eutectic temperature C_2 =
7. Percentage of “C” =
8. Melting point of “C” =

Ex.No: 5 CRITICAL SOLUTION TEMPERATURE OF
DATE: PHENOL - WATER-SYSTEM

AIM :

To determine the CST of phenol- water system and to find composition of phenol at CST.

APPARATUS REQUIRED:

Thermometer, glass rod, Boiling tube, 250ml beaker,

PRINCIPLE:

When two partially miscible liquids pairs such as phenol to water are mixed two mutually immiscible layer in the solution of water in the upper layer is the solution of water in phenol and lower layer is the phenol in water. At only given temperature the composition of two layer is fixed however, such as a system becomes completely miscible.

PROCEDURE:

Exactly 3 gram of water of phenol is taken in a boiling tube. It is filled with a thermometer and stirrer. A burette is filled with distilled water is added into a boiling tube. From the burette the boiling tube is placed on the water bath and slowly heated, the mixture is stirred well. At a particular temperature, at which the turbidity appears is heated this temperature two layers are separated this process is repeated by adding 4,5,6..... 25 ml of water. The correspond turbidity temperature in noted.

S.NO	VOLUME OF PHENOL	VOLUME OF WATER	WEIGHT OF PHENOL	WEIGHT OF WATER	% OF PHENOL = $\frac{\text{WEIGHT OF PHENOL}}{\text{WEIGHT OF PHENOL} + \text{WEIGHT OF WATER}} \times 100$	TURBIDITY OF TEMPERATURE(°C)
1	3	3	3.213	3	51.71	
2	3	4	3.213	4	44.54	
3	3	5	3.213	5	34.12	
4	3	6	3.213	6	34.37	
5	3	7	3.213	7	31.45	
6	3	8	3.213	8	28.65	
7	3	9	3.213	9	26.30	
8	3	10	3.213	10	24.31	
9	3	11	3.213	11	22.60	
10	3	12	3.213	12	21.12	
11	3	13	3.213	13	19.81	
12	3	14	3.213	14	18.66	
13	3	15	3.213	15	17.64	
14	3	16	3.213	16	16.72	
15	3	17	3.213	17	15.89	
16	3	18	3.213	18	15.14	
17	3	19	3.213	19	14.46	
18	3	20	3.213	20	13.84	
19	3	21	3.213	21	13.26	
20	3	22	3.213	22	12.7	

GRAPH:

The graph is plotted turbidity temperature on the y-axis of percentage of phenol on the x-axis of maximum of curve indicated CST of the phenol of water system, the corresponding percentage of CST of phenol

RESULT :

- 1) Critical solution temperature of phenol water system =
- 2) Percentage of phenol critical solution temperature =

Ex.No: 5

EFFECT OF IMPURITY ON (CST)

DATE:

PHENOL – NaCl –System

AIM:

To study the effect of NaCl impurities on critical solution temperature of phenol sodium chloride system.

APPARATUS REQUIRED:

Thermometer, glass rod, boiling tube, 250ml beaker,

PRINCIPE:

The critical solution temperature of phenol water system is greatly influenced by the addition of an electrolyte (NaCl to KCL) the water solubility of phenol and water are affected in the presence of an electrolyte. If a substance is dissolved only in one layer it results in the increase of a system. A substance dissolving in both the layers effectively decreases the CST of the system, this effect has been used to determine the concentration of a given electrolyte solution.

S.NO	NORMALITY OF NaCl (N)	TURBIDITY OF TEMPERATURE(°C)
1	0.01	
2	0.02	
3	0.03	
4	0.04	
5	0.05	
6	0.06	
7	Unknown	

PROCEDURE:

0.1 N of NaCl solution is prepared by dissolving 1.45 gram of sodium chloride crystal in dissolved water made up to 250 ml of the constant are shaken well. The pipette 10 ml, 20 ml, 0 ml 60 ml of 0.1 N of NaCl solution into size separated 100 ml SMF. This concentration of 0.01 N, 0.02 N, ..., 0.06 N of NaCl heatly the given unknown concentration solution is made up to 10 ml in the seventh SMF exactly 3 ml of phenol is taken in a boiling tube. It is fitted with a thermometer and stirrer, 5 ml of 0.01 N of NaCl solution is placed inside the water boiling tube and then it is placed inside the water bath and slowly heated. The mixture of which two liquid becomes completely miscible. The mixture is taken out from it and similarly, 0.02 N, 0.03 N, 0.06 N and unknown concentration solution is added separately with 3 ml of phenol and do the process repeatedly. The impurities temperature corresponding concentration are needed.

GRAPH:

A Graph is plotted to turbidity temperature on Y axis and concentration NaCl on X axis. From the graph, we can find out the normality of NaCl Solution corresponding to its turbidity temperature.

RESULT:

- 1) The Durability temperature of phenol –water system containing the unknown concentration of sodium chloride =
- 2) The concentration of given sodium chloride =

Ex.No: 6

HEAT OF SOLUTION

DATE:

AIM:-

To determine the heat of solution of ammonium oxalate by titrating with standard KMnO_4 .

APPARATUS REQUIRED:-

Burette, pipette, beaker, thermometer, standard measuring flask, and tiles

CHEMICAL REQUIRED:-

Ammonium oxalate, KMnO_4 , 2N H_2SO_4 .

PRINCIPLE:

$$\Delta H_{\text{water}} = \text{mass}_{\text{water}} \times \Delta T_{\text{water}} \times \text{specific heat}_{\text{water}}$$

Where

ΔH = heat change

mass water = sample mass

ΔT = temperature difference

Specific heat = $0.004184 \text{ kJ/g}^\circ\text{C}$.

The quantity of heat evolved when molecule of substance in KMnO_4 quantity of water this is known quantity of water solid is bring solid compound which small amount of called the solute the solid point in the solid to different grams 10^{-1} m position the sodium.

Si.No	Temperature (C)	Temperature (K)	1/T	1/T X 10 ⁻³	VOLUME OF KMnO ₄	Log (s)
1.	52	325	3.0769 x 10 ⁻³	3.076		
2.	49	322	3.1053 x 10 ⁻³	3.105		
3.	46	319	3.1347 x 10 ⁻³	3.134		
4.	43	316	3.1645 x 10 ⁻³	3.164		
5.	40	313	3.1948 x 10 ⁻³	3.194		
6.	37	310	3.2256 x 10 ⁻³	3.225		

PROCEDURE:

- ✓ First titration:- ammonium oxalate against KMnO_4
- ✓ Indicator:- KMnO_4 acts as a self indicator
- ✓ End point:- Appearance of light pink colour
- ✓ Prepared by saturated solution of 20g ammonium oxalate in water is taken in the 20ml beaker the constant are slowly heated. The thermometer rates the 70°C the beaker is kept a constant are asked to cooled by constant string when the temperature rates 52.5°C pipette cut 5 ml of the clear liquid of the and make up to 100ml of in the SMF at 52°C similarly when up to the temperature rate 49°C ., 46°C ., 43°C ., 42°C ., 39°C ., respectively the solution shake well pipette out 10ml of makeup solution and half a test tube full dil. 2N H_2SO_4 . Is added and slowly added standard KMnO_4 Appearance of light pink colour result is calculated.

GRAPH:

A graph is plotted between logs (title values) in x axis and an $1/7 \times 10^{-3}$ by axis A strength line is obtained from the slope of the heat of the solution is calculated by the equation.

$$\Delta H = \frac{dy}{dx} \times 2.303$$

RESULT:

The heat of solution given salt $\Delta H =$

CALCULATION:

Ex.No: 7 DETERMINATION OF THE RATE CONSTANT FOR THE
DATE: ACID-CATALYZED HYDROLYSIS OF METHYL ACETATE

AIM:

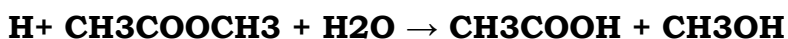
To determine the rate constant for the acid-catalyzed hydrolysis of methyl acetate.

MATERIALS REQUIRED

Methyl acetate, HCl, NaOH, 0.1N standard Oxalic acid, phenolphthalein, thermostat, reagent bottles, conical flask, burette, pipette (10 ml, 5 ml,), ice

PRINCIPLE:

Methyl acetate undergoes hydrolysis, in the presence of an acid (HCl, for example), to give acetic acid and methyl alcohol.



In the presence of an acid, this reaction should be of second order, since two molecules are reacting. But, it is found to be first order. This may be explained in the following way: The rate of the reaction is given by

$$dx / dt = k [\text{CH}_3\text{COOCH}_3] [\text{H}_2\text{O}]$$

Where k is the rate constant (or specific rate constant).

Since water is present in large excess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to :

$$dx / dt = k [\text{CH}_3\text{COOCH}_3]$$

OBSERVATIONS AND CALCULATIONS

Room Temperature = ... °C ; V_{∞} = ... ml ; V_o = ... ml ; $(V_{\infty}-V_o)$ = ... ml ; $\log(V_{\infty}-V_o)$ = ...

Mean Value of Rate Constant (k_1) = _____

Sl.NO	TIME	Volume of ACIDIC ESTER	Volume of NaOH	$(V_{\infty} - V_o)$ ml	$(V_{\infty} - V_t)$ ml	Log $(V_{\infty} - V_o)$ ml	Log $(V_{\infty} - V_t)$ ml	Log $\frac{V_{\infty} - V_o}{V_{\infty} - V_t}$	K= 2.303/ T Xlog $\frac{V_{\infty} - V_o}{V_{\infty} - V_t}$
1.	0	10ml							
2.	600	10ml							
3.	1200	10ml							
4.	1800	10ml							
5.	2400	10ml							
6.	3000	10ml							
7.	3600	10ml							
8.	∞	10ml							

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only).

Hence, the reaction is first order. Such reactions are also referred to as pseudo first order reactions.

The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definite intervals of time, cooling it in ice, and titrating the acetic acid formed against alkali, which has already been standardized.

The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction. The amount of acetic acid formed (x), at definite intervals of time (t), can be obtained.

The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration (a) of the ester. Suppose the volumes of the sodium hydroxide solution required for neutralization of 5 ml of the reaction mixture are:

- (i) at the commencement of the reaction is V_0
- (ii)** (ii) after time (t) is V_t
- (iii)** (iii) at the end of the reaction is V_∞

Then:

x (amount of acetic acid formed after time t) is proportional to $(V_t - V_0)$.

a (initial concentration of ester) is proportional to $(V_\infty - V_0)$.

$[a - x]$ (Concentration of ester present after time t) is proportional to $(V_\infty - V_0) - (V_t - V_0) = (V_\infty - V_t)$

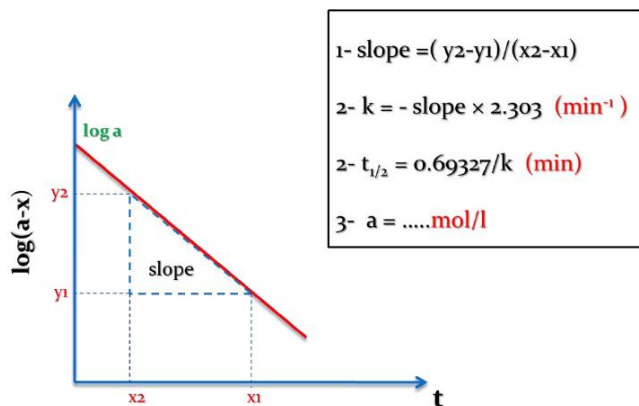
The first order rate expression given by :

$$\text{LOG } (A-X) = -K T / 2.303 + \text{LOG } A$$

Hence, the rate constant (k) could be calculated.

GRAPH:

A graph is plotted between $\log (V_{\infty}-V_0)$ of Y Axis and time (s) on X axis from the separate constant 'K' calculation.



PROCEDURE:-

100ml of the given hydrochloric acid is taken and placed in the thermostat the ester is the also kept in a thermostat. The for 10 mints the 5ml of the given ester preparation and pipette out in to clean 150 ml beaker it is also containing given HCL and simultaneously a stop clock is started pipette 100ml of acid mixture in to a clean conical flask the solution is the titration against sodium hydroxide solution using phenolphthalein indicator. The end is just appearance of pale pink colour permanent end colour. The titration represented the volume of sodium hydroxide 2ml of given by acid mixture is titrated is result of constant K can be calculation.

Step I: Standardization of NaOH using standard Oxalic acid (0.1N)

- ✓ 1. 10ml of given 0.1N standard Oxalic acid is pipette out into a 100ml conical flask.
- ✓ 2. This solution is titrated against the given unknown concentration of NaOH using phenolphthalein indicator until the end point is colorless to pale pink.
- ✓ 3. Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied NaOH solution.

Step II: Standardization of HCl using NaOH solution.

- ✓ 2 ml of given HCl is pipette out into a 100ml conical flask.
- ✓ This solution is titrated against the NaOH using phenolphthalein indicator until the end point is colorless to pale pink.
- ✓ Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied HCl solution.

Step III: Determination of rate constant (k_1) for the acid-catalyzed hydrolysis of methyl acetate.

- ❖ 100 ml of given HCl (whose strength is determined in step II) solution is taken in a stopper reagent bottle.
- ❖ 5 ml of methyl acetate solution is added to the HCl solution. Note the time when half of the methyl acetate solution is added. The mixture is shaken well. 3. Pipette out 5 ml of the reaction mixture and discharge it into 50 ml of ice cold water kept in a conical flask.
- ❖ Titrate the reaction mixture against NaOH solution using phenolphthalein as indicator. This titrate value corresponds to V_o .
- ❖ Steps 3 and 4 are repeated at intervals of 5, 10, 15, 20, 30, 45, 60 minutes. Each titrate value corresponds to V_t .
- ❖ Take 10 ml only of the remaining solution and leave it for 24 hours and repeat step 3&4 and record $V_{NaOH} = [a]$
- ❖ The remaining solution is taken in a stopper conical flask and heated to 60°C , and kept at this temperature for 5 minutes.
- ❖ The solution is allowed to cool to room temperature.
- ❖ Repeat Steps 3 and 4. This titrate value corresponds to V_∞ till concurrent values are obtained.
- ❖ Plot a graph of $\log (V_\infty - V_t)$ versus time (t) and determine the slope.
- ❖ Report the theoretical and graphical value of rate constant (k).

RESULTS:

1. Strength of NaOH Solution = _____

1. Strength of HCl Solution = _____

2. Rate Constant (k_1) for the acid-catalyzed hydrolysis of methyl acetate
($\text{CH}_3\text{COOCH}_3$)

By Calculation Value = _____

By Graphical Value = _____

3. Comment on the nature of the graph

Ex.No: 8 ESTIMATION OF MIXTURE OF ACID [NaOH + HCl]**DATE:**

AIM:-

To determine the concentration of hydrochloric acid by conductometric titration.

APPARATUS REQUIRED:-

Conductivity cell, conductivity meter, standard measuring flask,

CHEMICAL REQUIRED:-

HCl, NaOH, NH_4Cl .

PRINCIPLE:

According to Kohlrausch law, the electrical conductivity of a solution depends upon the number of ions present and their mobility. So conductivity measurements can be employed to find the end point of acid, alkali and other type of titrations. Let us consider the titration of strong acid like HCl vs strong base like NaOH.

A known volume of HCl is taken In the conductivity cell. A known volume of NaOH is added and after each addition the conductance is determined. The conductance of the solution is plotted vs the volume of alkali added. Two straight lines are obtained. The point of intersection is the end point and the volume of alkali corresponding to this point is the volume required to neutralize

TITRATION -I:-

TITRATION OF HYDROCHLORIC ACID VS SODIUM HYDROXIDE:

S.NO	VOLUME OF NaOH (ml)	CONDUCTANCE $C \times 10^{-3} \text{ ohm}$	$V + \gamma/V$	ACTUAL CONDUCTANCE $V + \gamma/V \times$ $C \times 10^{-3} \text{ mho}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				

The known volume of the acid. Before the addition of alkali the conductivity is due to the presence of H^+ and Cl ions.

The conductance is mainly due to the H^+ ions which have very high mobility. As alkali is added the H^+ ions are removed as feebly ionized water molecules. The conductivity of the solution decreases due to replacement of H^+ ions by slow moving Na^+ ions. Once the end point is reached, after the neutralization, there will be an increase in conductance due to increase in concentration of OH^- ions because the OH^- ions have the high mobility. The point of minimum conductivity will therefore be the end point of the titration.

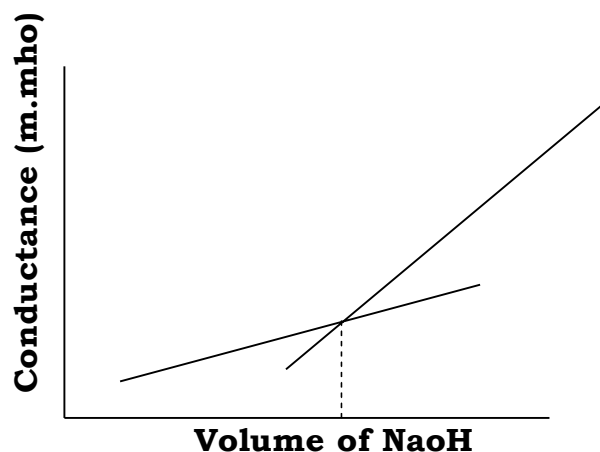
PROCEDURE:-

Pipette out 40ml of 0.02 N HCl into a 150 ml beaker so that the electrodes are completely immersed in the solution. Sodium hydroxide solution of strength 0.1 N is added in 0.5ml portions. The solution is stirred well and allowed to attain equilibrium. The conductance is noted and is plotted vs the volume of NaOH added. The point of intersection of two straight lines gives the end point.

CALCULATION:

TITRATION -I:-

DETERMINATION OF STRENGTH OF HYDROCHLORIC ACID FROM GRAPH:-



From graph:-

Volume of Sodium hydroxide (V_1) = 20ml

Normality of Sodium hydroxide (N_1) =

Volume of Hydrochloric Acid (V_2) =

Normality of Hydrochloric Acid (N_2) = V_1N_1/V_2

RESULT:

The strength of Hydrochloric acid is _____ N.

Ex.No: 9

POTENTIOMETRIC REDOX TITRATION

DATE:

[HYDROCHLORIC ACID VS SODIUM HYDROXIDE]

AIM :

To determine the strength of hydrochloric acid potentiometrically by titration with sodium hydroxide.

APPARATUS REQUIRED :

Potentiometer, saturated calomel electrode, platinum electrode, pipette, burette.

SOLUTION REQUIRED:

0.2 Hydrochloric acid, 0.2 Sodium Hydroxide, Quinhydrone powder, Solution, 2N-H₂SO₄, KCl Solution

PRINCIPLE:

Potentiometric titration is a laboratory method to determine the concentration of a given analyzed. It is used in the characterization of acids. In this method, there is no use of a chemical indicator. Instead, the electric potential across the substance is measured.

The end point of the neutralization titration may be found by direct measurement of the emf of the solution after each addition of alkali to acid. The change of emf for the addition of a given amount of titrate is maximum at



TITRATION-I

TITRATION OF HYDROCHLORIC ACID VS SODIUM HYDROXIDE:

Volume of NaOH (ml)	E_{obs} Volt $\times 10^{-3}$
0	
2	
4	
6	
8	
10	
12	
14	
16	
18	
20	
22	
24	
26	
28	
30	
32	
34	
36	
38	
40	

The equivalence point and so the later can be identified. The potential of any hydrogen electrode is given by $E = E^\circ - 0.059 \log a_{H^+}$. The change in electrode potential or the emf of a cell made up of the hydrogen and a standard electrode is thus proportional to the change in ΔE Ph during titration the end point is maximum, where is the change in potential (or) emf resulting from the addition of a definite volume (eg. 0.1ml) of titrate. The accuracy with which this point can be deducted depends on the magnitude of the inflexion in the pH neutralization curve.

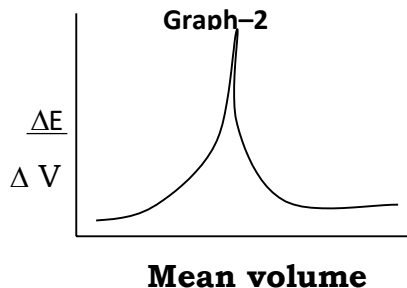
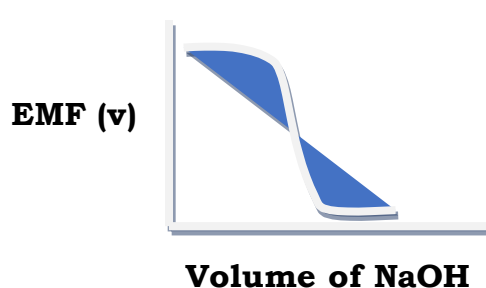
PROCEDURE:

An approximately 0.05N HCl is prepared, 10ml of which is pipette out in to a beaker. A pinch of quinhydrone is added. A platinum electrode is dipped into the solution and it is connected to reference electrode (Saturated calomel electrode). A standard solution of (0.05N) sodium hydroxide is taken in burette and added in 1 cc portions. emf is measured and the range of equivalence point is noted. The exact end point is found out by noting the emf value for each addition of 1 cc portion of the titrate and nearing the equivalence point 0.5cc, 0.2cc and 0.1cc portions. The following graphs are drawn.

1. The value of emf Vs Volume of NaOH
2. The value of $\Delta E / \Delta V$ Vs mean volume of NaOH

CALCULATION:

DETERMINATION OF STRENGTH OF HYDROCHLORIC ACID VS SODIUM HYDROXIDE FROM GRAPH:-



From graph :-

Volume of Sodium hydroxide (V_1) = 20ml

Normality of Sodium hydroxide (N_1) =

Volume of Hydrochloric Acid (V_2) =

Normality of Hydrochloric Acid (N_2) = V_1N_1/V_2

RESULT:

The strength of hydrochloric acid is_____

Ex.No: 10 TITRATION OF FERROUS SULPHATE VS $K_2Cr_2O_7$

DATE:

AIM:-

To determine the strength of ferrous sulphate potentiometrically by titrating with potassium dichromate.

APPARATUS REQUIRED:-

Potentiometer, saturated calomel electrode, platinum electrode, pipette, burette.

PROCEDURE:

A standard solution of 0.05N $K_2Cr_2O_7$ is taken in the burette. 10 ml of approximately 0.05N ferrous sulphate is taken in the beaker and about 10ml of dilute H_2SO_4 is added. A platinum electrode is dipped in the solution and reference electrode is also placed in the beaker. Both are connected to potentiometer.

The $K_2Cr_2O_7$ solution is added from the burette in 1ml portions to fix the range of equivalence point. It is followed by fair titration by adding 0.5ml, 0.2ml and 0.1ml, so that the equivalence point can be determined.

The following two graphs are drawn.

(i) The value of emf v_s volume of $K_2Cr_2O_7$.

(ii) The value of ΔE v_s mean volume of $K_2Cr_2O_7$.

TITRATION OF FERROUS SULPHATE VS POTASSIUM DICHROMATE:

Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (ml)	E_{obs} Volt x 10^{-3}
0	
2	
4	
6	
8	
10	
12	
14	
16	
18	
20	
22	
24	
26	
28	
30	
32	
34	
36	
38	
40	

From graph:-

Volume of potassium dichromate (V_1) = 20ml

Normality of potassium dichromate (N_1) =

Volume of ferrous sulphate (V_2) =

Normality of ferrous sulphate (N_2) = V_1N_1/V_2

Result:

The strength of Ferrous sulphate = _____N

Ex.No: 10 DETERMINATION OF P^H OF THE BUFFER**DATE: SOLUTION****AIM:-**

Determination of concentration of HCl solution by titrating it against standard NaOH solution using a pH-meter.

APPARATUS REQUIRED:-

pH meter, burette, pipette, beakers, measuring cylinder

CHEMICAL REQUIRED:-

HCl, N/10 NaOH, Solutions of pH = 4 & 9

THEORY:

When an alkali is added to an acid solution, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated by normality equation.

PROCEDURE:

First standardize the pH meter with a buffer of pH 4 & 9. Take 50ml of HCl solution in a 100 ml. beaker so that the glass electrodes is completely dipped. Note the pH of pure acid solution. Now add 2 ml of N/10 NaOH from the burette in the beaker. Stir the contents well.

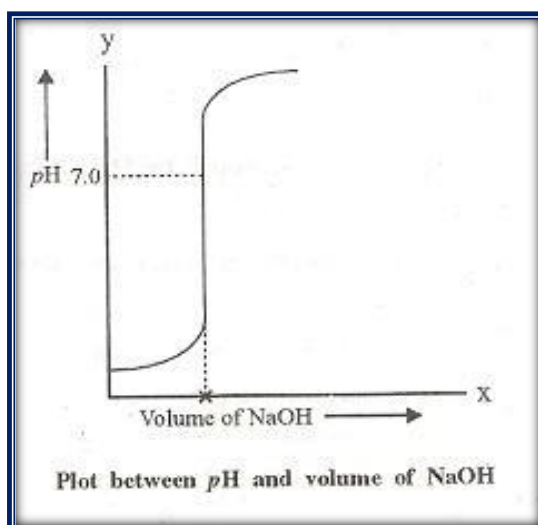
OBSERVATION:

Volume of HCl taken = 50ml

Normality of NaOH solution=N/10

SNO	Vol. of N/10 NaOH	pH	SNO	Vol. of N/10 NaOH	pH
1	0.0		14	26.0	
2	2.0		15	28.0	
3	4.0		16	30.0	
4	6.0		17	32.0	
5	8.0		18	34.0	
6	10.0		19	36.0	
7	12.0		20	38.0	
8	14.0		21	40.0	
9	16.0		22	42.0	
10	18.0		23	44.0	
11	20.0		24	46.0	
12	22.0		25	48.0	
13	24.0		26	50.0	

Note the pH of the solution. Now go on adding NaOH solution from the burette and note the pH of the solution after each addition of 2 ml of N/10 NaOH. After equivalence point there will be sudden change in pH value ie it will jump to basic range. After completion of experiment draw a graph between volumes of NaOH added vs pH values. Join two curves and find the volume of N/10 NaOH required to neutralize at pH=7 which is equivalence point

GRAPH:

Volume of N/10 NaOH Solution used from burette (ml) to neutralize 50 ml of HCl solution (From Graph) = v_2 ml.

CALCULATIONS:

$$N_1V_1 = N_2V_2$$

(HCl sample) (NaOH solution)

$$N_1 = (1/10 \times v_2 \text{ ml}) / 10 \text{ ml}$$

$$\begin{aligned} \text{Strength HCl solution} &= N_1 \times 36.5 \text{ gm/liter} \\ &= N_1 \times 36.5 \times 1000 \text{ mgm/liter} \\ &= N_1 \times 36.5 \times 1000 \text{ ppm} \end{aligned}$$

RESULT:

The strength of the given HCl solution = ...ppm.

Ex. No: 11 ADSORPTION OF ACETIC ACID ON ACTIVATED
DATE: CHARCOL

AIM:

- i) To study the adsorption of oxalic acid on activated charcoal and to Verify (a) Freundlich adsorption isotherm and (b) Langmuir adsorption isotherm.
- ii) To determine the strength of unknown oxalic acid using Freundlich adsorption

APPARATUS REQUIRED:

Burette, conical flask, funnel, beaker, filter paper, shaking bottles.

SOLUTION REQUIRED:

Oxalic acid, acetic acid, sodium hydroxide, charcoal.

PRINCIPLE:

Solids especially in porous and finely divided state have the property of holding for an molecule on the surface. This phenomenon is known as adsorption. The two types of adsorption generally recognized.

- i. Physical (or) Vander wall's adsorption
- ii. Chemical adsorption.

The physical adsorption is characterized by low heat of adsorption and the rate at which is reversible between the mass of the adsorbate. Adsorbed on adsorbent and equilibrium concentration of adsorbate at a constant temperature is called the adsorption isotherm and empirical adsorption range was derived by Freundlich. This relates the ratio of the mass adsorbate.

Volume of NaOH V_1 = 20ml

Normality of NaOH N_1 =

Volume of oxalic acid V_2 =

Normality of oxalic acid N_2 =

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 N_1 / V_2$$

To prepare 0.05N NaOH =

TITRATION-I:-

BOTTLES	VOLUME OF ACETIC ACID (ml)	VOLUME OF H₂O (ml)	$C_i = V_{acid} \times N_{acid} / 100$
A			
B			
C			
D			
Unknown1			
Unknown 2			

Adsorbed to that of adsorbate in the solution as $x/m = K (ACOH)^{1/n}$. Thus a plot of $\log x/m$ vs $\log (ACOH)_{aq}$ is linear from this plot K and n and unknown concentration of the adsorbate can be calculated.

PROCEDURE

The standard solution of oxalic acid was prepared and used this oxalic acid and NaOH solution are standardize. The standard NaOH used for the equation. The standard solution of acetic acid was prepared. Six corning bottles were cleaned with chromic acid. Washed and dried. They were labeled from A and F. Exactly 1gm of charcoal was weighed out into each of the type of 6 corning bottle. 80, 60, 40, 20 ml of stock acetic acid solution were run down from the burette into bottles A to D and the total volume is adjusted to 100 ml in all the bottles with water using the burette. In the bottle E and F acetic acid of well known concentration will be supplied. The bottles were stopped and shaken in a mechanical shaker for about 20 minutes.

After the adsorption equilibrium was established the solution in the bottles was filtered through filter paper. Titrated against 0.05N NaOH by pipette out 5 ml from bottle and 10 ml from C.D.E and F.

TITRATION –II:

BOTTLES	VOLUME OF ACETIC ACID (ml)	VOLUME OF NaOH (ml)	$C_e = V_{NaOH} \times N_{NaOH} / V_{(acid)}$
A			
B			
C			
D			
Unkown 1			
Unkown 2			

BOTTLES	AMOUNT OF CHARCOAL	INITIAL CONCENTRATION OF ACID C_i/lit	EQUIVALENT CONCENTRATION OF ACID C_e/lit	AMOUNT OF ACID ABSORBENT $(C_e - C_i) = x$	x/m	$Log\ x/m$	$Log_x\ u$
A							
B							
C							
D							
Unkown 1							
Unkown 2							

CALCULATION:**UNKNOWN: 1:-****From graph I:-**

$$\text{Log } x/m =$$

$$x/m =$$

$$x =$$

$$C_i = x_i + C_e$$

$$C_i =$$

UNKNOWN: II:**From graph II :-**

$$\text{Log } x/m =$$

$$X /m =$$

$$x =$$

$$C_i = x_i + C_e$$

$$C_i =$$

Adsorption of co efficient:

$$n = 1/\text{slope}$$

$$1/n =$$

$$N =$$

Equilibrium constant:

$$\text{Log } K =$$

$$K = A.\log$$

RESULT:

The frendlich adsorption isotherm is verified from the straight line obtained in the graph.

- i. Equilibrium constant K =
- ii. Adsorption constant n =
- iii. Unknown strength of oxalic acid ,

Bottle 1 = N

Bottle 2 = N