
KEEP SILENCE

Home Assignment -

Page No. 0
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Std :- BSC-I Division :- B
Roll No :- 1035 Subject :- Microbiology
College Name :- Shri. Shivaji Mahavidyalay, Barshi.
Paper :- I

5/5 14/5

Q.1] Define procaryotic and Eucaryotic cell with figures, Differentiate between procaryotic and eucaryotic cell structure. [write all differencess]

- prokaryotic cell :- Organisms which contain primitive nucleus are called prokaryotic cells. In these cells DNA or genetic material is very primitive. It is freely suspended in cytoplasm, Nucleolus are absent. It is not true nucleus so we can call it as nucleiod. Nuclear material chromatin body bacterial DNA, Bacterial chromosome, nucloid is circular Double standed DNA (Histone) Basic protiens are absent. Gtc percentage is 28 to 73. There is no distintion between cytoplasm, Neudeoplasm, ER. GA, chlorobium vesides are absent. Ribosomes are only 70^S type Mesosomes, Mongnetosomes. Ribosomes are only 70^S types. vesicles stored food is pressent in the cytoplasm cell wall is very unique, it is made up of peptidoglycon. Cynobacteria are prokaryotic.

- Eukaryotic Cell :- Cell which contain well developed nucleus are eukaryotic cells, Nucleus is bounded by nuclear membrane. Nucleus contain more than one linear chromosomes. histones are present Neuclus are present cytoplasm and nucleoplasm are separated. cytoplasm contain chlorobium vesicles, Vaccules, Endoplasmic Reticulum, Golgi bodies, mitochondria, Ribosome, lysozome etc.
Ribosome are of two types 80^S & 70^S.

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आणि

श्री शिवाजी महाविद्यालय, बार्शी

यांचे संयुक्त विद्यमाने

कर्मवीर डॉ. मामासाहेब जगदाळे

यांच्या ११८ व्या जयंतीनिमित्त आयोजित

निःशुल्क प्रवेश

वर्ष २७ वे

शिवछत्रपती आंतरमहाविद्यालयीन राज्यस्तरीय ऑनलाईन वक्तृत्व स्पर्धा - २०२१

गुरुवार दि. २१ जानेवारी २०२१

प्रथम पारितोषिक ₹ १०,०००/-	द्वितीय पारितोषिक ₹ ७,०००/-
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* विषय : १) ज्ञानतपस्वी : कर्मवीर डॉ. मामासाहेब जगदाळे
२) छत्रपती शिवरायांचा आदर्श राज्यकारभार
३) कोरोना आणि मानवापुढील आव्हाने
४) आत्महत्येने खरेच प्रश्न सुटतात का ?
५) सोशल मिडीया : वास्तव की विपर्यास
६) साऱ्या जगाचा पोशिंदा....सदा राहतो उपाशी
७) वाचूया आनंदे !
८) आजच्या राजकारणाची दशा आणि दिशा
९) महात्मा गांधीजींच्या सहिष्णुतेची समकालीन उपयुक्तता
१०) ऑनलाईन शिक्षण : संधी आणि मर्यादा

* नियम व अटी : १) स्पर्धा ऑनलाईन पद्धतीने झूम ॲपद्वारे घेण्यात याईल. २) प्रत्येक स्पर्धकास ७ मिनिटे (५+२) इतका वेळ देण्यात येईल. ३) स्पर्धा महाराष्ट्रातील सर्व वरिष्ठ महाविद्यालयीन विद्यार्थ्यांसाठी खुली राहील. ४) या स्पर्धेतील भाषण मराठी, हिंदी किंवा इंग्रजी यापैकी कोणत्याही एका भाषेतून करता येईल व पारितोषिके या तिन्ही भाषांत मिळून दिली जातील. ५) प्रत्येक महाविद्यालयातून जास्तीत जास्त दोन स्पर्धकांना सहभाग नोंदवता येईल. ६) सहभागी होऊ इच्छिणाऱ्या विद्यार्थ्यांनी आपली नावे महाविद्यालयामार्फत नोंदवावीत व मा. प्राचार्यांच्या सही व शिक्क्याचे संमतीपत्र आणि स्वतःचे ओळखपत्र गुगल फॉर्म सोबत अपलोड करावे. ७) इच्छुक विद्यार्थ्यांनी मंगळवार दि. १९ जानेवारी २०२१ पर्यंत खालील गुगल फॉर्म लिंकद्वारे नोंदणी करावी.
(https://docs.google.com/forms/d/e/1FAIpQLSeA_KvnGRNubTYi4rRoRXFdG_DMc5IBaXBsAzdQM3p_t9DFzA/viewform?usp=sf_link)

डॉ. रविकांत शिंदे
चेअरमन, शिवछत्रपती वक्तृत्व स्पर्धा

प्राचार्य डॉ. प्रकाश थोरात
श्री शिवाजी महाविद्यालय, बार्शी.

Shri Shivaji Shikshan Prasarak Mandal Barshi's

SHRI SHIVAJI MAHAVIDYALAYA, BARSHI

DEPARTMENT OF CHEMISTRY

B.Sc. II Practical Chart

CHART INDEX

PRACTICAL EXAMINATION PATTERN

Total 100 marks

- **Internal practical examinations** (As per the schedule given by the college) = **20 marks**
- **University practical examinations** (Annual as per the schedule given by the University) = **80 marks**

Sr. No.	Section
1	Physical Chemistry
2	Inorganic Chemistry
3	Organic Chemistry

Chemistry in Everyday life

There is no aspect of our life that is not affected by developments in chemistry.

Chemistry plays a very vital role in our everyday life. Our daily need of food, clothing, shelter, potable water, medicines etc. are connected with chemical compounds, processes and principles.

Chemistry has important contribution for giving us:

- ✓ Life saving drugs
- ✓ Synthetic fibers,
- ✓ Synthetic detergents
- ✓ Variety of cosmetics
- ✓ Preservatives for our food
- ✓ Fertilizers & pesticides
- ✓ Paper
- ✓ Glass
- ✓ Plastics
- ✓ Beautiful paints etc.

JOURNAL INDEX

Sr. No.	Name of experiment	Date	Page No.	Remark
	A) PHYSICAL CHEMISTRY			
	a) Instrumental			
1	**Conductometry No. 1** **Verification of Ostwald's Dilution Law** To determine the degree of dissociation and dissociation constant of acetic acid at various dilutions and to verify Ostwald's dilution law.			
2	**Conductometry No. 2** **Conductometric Titration** To determine the normality of the given strong acid by titrating it against strong alkali conductometrically.			
3	**Conductometry No. 3** **Verification of Onsagar equation** To determine equivalent conductance at infinite dilution of strong electrolyte by using five different dilutions conductometrically (Ex. KCl /NaCl / KNO_3 / HCl) & to verify Onsagar equation.			
4	**Polarimetry** To determine the specific rotation of the given sugar solutions polametrically.			
5	**Refractometry** To determine the specific and molar refractivities of the given liquids A, B and C and hence determine the refractivity of $-CH_2$ group.			
6	**Viscosity** To determine the percentage composition of the given unknown mixture by using viscometer.			
	b) Non-Instrumental			
7	**Chemical Kinetics No. 1** **Relative Strength of Two Acids** To determine the relative strength of acids from the hydrolysis of methyl acetate in presence of 0.5 N HCl and 0.5 N H_2SO_4.			
8	**Chemical Kinetics No. 2** **Effect of Acid Strength** To study the effect of acid strength on hydrolysis of an ester by using 0.5 N HCl and 0.25 N HCl.			
9	**Chemical Kinetics No. 3** **$K_2S_2O_8$and KI (Unequal concentration)** To investigate the reaction between $K_2S_2O_8$ and KI with unequal concentrations of the reactants .			
10	**Chemical Kinetics No. 4** **$KBrO_3$ and KI (Equal concentration)** To determine the order of the reaction between $HBrO_3$ and HI.			
11	**Partition coefficient** To determine partition coefficient of benzoic acid between water and benzene			

	B) INORGANIC CHEMISTRY			
	a) Gravimetric Estimations			
12	**Gravimetric Estimation of Barium** To determine the amount of barium as $BaSO_4$ from the given solution of $BaCl_2 .2H_2O$ and free HCl.			
13	**Gravimetric Estimation of Iron** To determine the amount of Fe as Fe_2O_3 from the given solution of. F.A.S. and free H_2SO_4.			
	b) Volumetric Analysis			
14	**Calibration of Volumetric Apparatus** To calibrate volumetric apparatus-burettes, pipettes and volumetric flasks			
15	**Analysis of Commercial Vinegar** To determine the percentage of acetic acid in commercial vinegar sample.			
16	**Total Hardness of Water** To prepare standard solution of calcium chloride from calcium carbonate and determine the total hardness of given water sample			
	c) Inorganic Preparations			
17	**Preparation of F.A.S. (Mohr's Salt)** To prepare ferrous ammonium sulphate.			
18	**Preparation of Tetraamminecopper(II) sulphate** To prepare tetraamminecopper(II) sulphate.			
19	**Preparation of Chloropentaamminecobalt (III) chloride** To Prepare Chloropentaamminecobalt (III) chloride			
20	**Preparation of Hexaamminenickel(II) chloride** To Prepare of Hexaamminenickel(II) chloride			
	Qualitative Analysis			
21	**Semi – Micro Qualitative Analysis** Give the complete qualitative analysis of the mixture given in capsule marked 'A'.			
	Cations Anions			
	Inorganic Mixture No: 1 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 2 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 3 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 4 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 5 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 6 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 7 1)…… 1) …….. 2)…… 2)……			
	Inorganic Mixture No: 8 1)…… 1) …….. 2)…… 2)……			

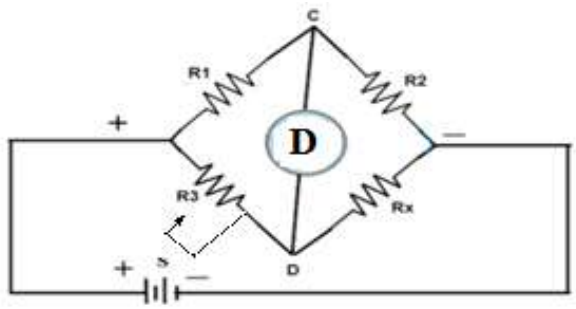
C) ORGANIC CHEMISTRY					
	a) Organic Estimations				
22	**Estimation of Acetone** To estimate the amount of acetone in the given solution by iodometric method.				
23	**Estimation of Ester (Ethyl Benzoate)** To estimate the amount of ethyl benzoate in the given solution.				
24	**Estimation of Ibuprofen from Ibuprofen Tablet** To determine the amount of ibuprofen from given ibuprofen tablet.				
	b) Organic Preparations				
23	**Preparation of m-Dinitrobenzene** To prepare m-dinitrobenzene from given amount of nitrobenzene.				
24	**Preparation of Phthalimide** To prepare phthalimide from given amount of phthalic anhydride.				
25	**Preparation of p-Bromoacetanilide** To prepare p-bromoacetanilide from given amount of acetanilide.				
26	**Preparation of Acetanilide** To prepare acetanilide from given amount of aniline.				
	Organic Qualitative Analysis				
27	**Identification of an Organic Compound** To identify Organic compound given in a container marked (A) bearing your table number.				
	Organic Comp. No.	Name of the compound	Structural formula		
	1				
	2				
	3				
	4				
	5				
	6				
	7				
	8				

1. Conductometry No. 1
Verification of Ostwald's Dilution Law

Aim : To determine the degree of dissociation and dissociation constant of acetic acid at various dilutions and to verify Ostwald's dilution law.

Given : N/10 or N/50 KCl solution, N/10 Acetic acid, conductivity water, 100 ml volumetric flask etc.

Circuit diagram:



Where ,

S= A.C. Source
R_1, R_2, R_3 = Known Resistances
Rx = Unknown Resistance
D = Detector

Wheatstone's Bridge

Part-A

Procedure: Determination of cell constant of the given conductivity cell.

1. Wash the conductivity cell and beaker with conductivity water.
2. Rinse both cell and beaker with N/10 or N/50 KCl
3. Take sufficient quantity of given N/10 or N/50 KCl solution in the beaker, so as to dip the electrodes of cell properly.
4. Connect terminals of the cell to the terminals of the conductometer.
5. Measure the conductance and determine cell constant (X)
6. Ask for specific conductance (k) of N/10 or N/50 KCl.

Observation table:

Solution used	Observed conductance (C)	Specific conductance (k)	Cell constant X = k/C
$\frac{N}{10}$ or $\frac{N}{50}$ KCl	…….. x 10^{-3}		

Part-B

Procedure: Determination of conductance of acetic acid solution of different concentrations

1. Prepare 100 ml of acetic acid solutions of each concentration N/20, N/40 and N/80 by diluting 50, 25 ml and 12.5 ml of the given N/10 acetic acid to 100 ml with conductivity water in separate volumetric flask (100 ml each) and shake well.
2. Take sufficient quantity of solution in the beaker so as to dip the electrodes of the cell properly.
3. Measure the conductance and enter the results in the tabular from.

Observation table:

Sr. No.	Concentra -tion (N)	Observed Conductance (C)	Specific Conductance k = X . C	Equivalent Conductance $\lambda_v \frac{1000\ k}{N}$	Degree of Dissociation $\alpha = \frac{\lambda_v}{\lambda_\infty}$	Dissociation Constant $K = \alpha^2 . N$
1	N/10	-------x 10^{-3}				
2	N/20	-------x 10^{-3}				
3	N/40	-------x 10^{-3}				
4	N/80	-------x 10^{-3}				

Given: λ_∞ = 390.8 Siemens

Calculations: Specific conductance (k) = cell constant x observed conductance.
Show all details of calculations for each concentration.

Results:

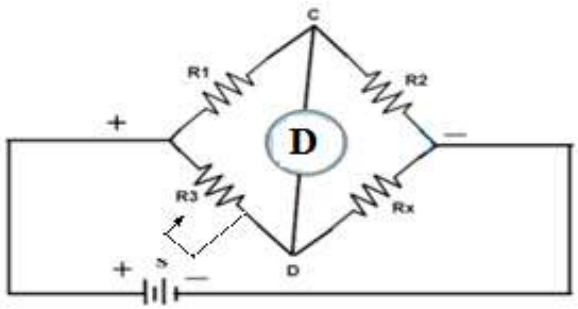
1.	Dissociation constant of acetic acid (Mean)	K= ----------
2.	**Conclusion: -** Here the values of dissociation constant (K), are fairly constant. Hence Ostwald's dilution law is verified.	

2. Conduetometry No. 2
Conductometric Titration

Aim : To determine the normality of the given strong acid by titrating it against strong alkali, conductometrically.

Given : 0.1 N (approximately) acid solution, 0.2 N strong alkali (NaOH), conductivity water, microburette, 10 ml pipette etc .

Circuit diagram:



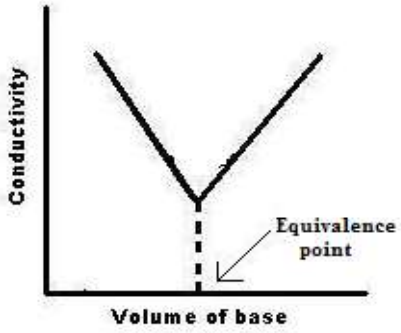
Where ,
S= A.C. Source
R_1, R_2, R_3 = Known Resistances
Rx = Unknown Resistance
D = Detector

Procedure:

1. Wash the beaker and conductivity cell with conductivity water.
2. Take 10 ml of the given 0.1 N acid solution by pipette in 100 ml beaker.
3. Add 50 ml conductivity water by the measuring cylinder and stirr well.
4. Dip the cell in the solution and connect the terminals of the cell to the conductometer.
5. Measure the conductance directly.
6. Then add exactly 0.5 ml of 0.2 N strong alkali solutions from the burette to the solution in the beaker.
7. Stir well and measure the conductance of the solution.
8. Similarly take the readings after every addition of 0.5 ml alkali solution up to 10 ml of alkali.

Observation table:

Sr. No.	Volume of alkali added V ml	Conductance (C) = 1/R ohm^{-1}
1	0	X 10^{-3}
2	0.5	X 10^{-3}
3	1.0	X 10^{-3}
4	1.5	X 10^{-3}
↓	↓	X 10^{-3}
↓	↓	X 10^{-3}
21	10.0	X 10^{-3}



Plot the graph of conductance (C) [on y- axis] against ml of strong alkali added (on x- axis). From graph find out the equivalence point.

Calculations:

Calculate the normality of the given strong acid using relation

$N_1V_1 = N_2V_2$

Acid Vs Alkali

$N_1 \times 10 = 0.2 \times$ equivalence point

$$N_1 = \frac{0.2 \times \text{equivalence point}}{10}$$

= ---------- N

Result:

1.	Equivalence point from the graph	= -------- ml
2.	Normality of the given strong acid	= ------- N

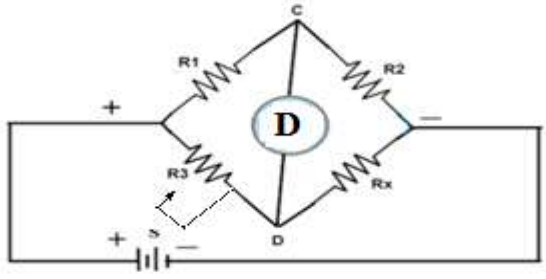
3. Conductometry No. 3

Verification of Onsager equation

Aim : To determine equivalent conductance at infinite dilution of strong electrolyte (HCl) by using five different dilutions conductometrically and to verify Onsager equation

Given : 0.1 N KCl solution, 0.1 N HCl, 0.1 N KNO_3, 0.1 N NaCl, conductivity water, 100 ml measuring flask etc.

Circuit diagram:



Where ,

S= A.C. Source

R_1, R_2, R_3 = Known Resistances

Rx = Unknown Resistance

D = Detector

Part-A

Procedure: Determination of cell constant of given conductivity cell.

1. Wash the conductance cell & beaker with conductivity water and rinse with N/10 KCl.

2. Take sufficient quantity of given N/10 or N/50 KCl solution in the beaker, so as to dip the electrodes of conductivity cell properly.
3. Connect terminals of the cell to terminals of the conductometer.
4. Measure the conductance & determine cell constant (X).
5. Ask for the specific conductance (k) of N/10 or N/50 KCl.

Observation table:

Solution used	Observed Conductance (C)	Specific Conductance (k)	Cell Constant (x)= k/C
$\frac{N}{10}$ or $\frac{N}{50}$ KCl	---------- x 10^{-3}		

Part-B

Procedure: Determination of equivalent conductance (λ_v).

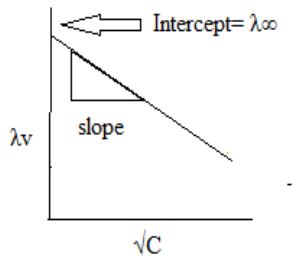
1. Prepare 100 ml of HCl solution of each concentration of 0.075, 0.05, 0.025, 0.01 and 0.005 N solution by diluting 75 ml, 50 ml, 25 ml, 10 ml & 5 ml of the given 0.1 N HCl solution to 100 ml with conductivity water in separate volumetric flasks (100 ml each) & shake well.
2. Measure the conductance of each solution & enter the results in the tabular from.

Observation table:

Conc. of HCl solution (N)	Observed Conductance (C) (ohm $^{-1}$)	Specific Conductance k= X x C (ohm^{-1}cm^{-1})	Equivalent Conductance $\lambda_v = 1000 \times k/N$
0.100			
0.075			
0.050			
0.025			
0.010			
0.005			

Calculations:

Calculate specific conductance (k), equivalent conductance (λ_v), by using formulae and show all details of calculations.
Determination of equivalent conductance at infinite dilution (λ_∞)
$\lambda_v = \lambda_\infty - (AB) \sqrt{C}$ (For HCl $\sqrt{C} = \sqrt{N}$), Where A & B are the constants for particular solvent at particular temperature. Using this equation, plot the graph of λ_v (Y axis) versus $\sqrt{C}$ (X axis) Slope = (A X B), Intercept on Y axis = $\lambda\infty$ i.e. Equivalent conductance at infinite dilution



Result:

1.	Equivalent conductance at infinite dilution (λ_∞)	= …… ohm^{-1} cm^{-2} eq^{-1}

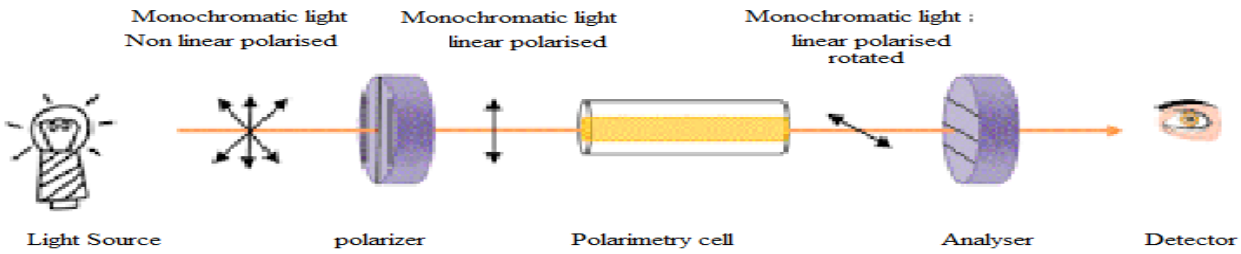
4. Polarimetry

Aim : To determine the specific rotation and concentration of the given cane sugar solution polametrically.

Given : 10 % sugar solution, distilled water, 50 ml measuring flask

Apparatus : 50 ml measuring flask , polarimeter, polarmetric tube, lamp etc .

Diagram :



Procedure:

1. Rotate the polaroid wheel so that the degree meter reads zero.
2. Fill the polarimeter tube with distilled water, remove bubble if any and place the tube in the chamber.
3. Slowly rotate the wheel so that intensity of light is minimum in intensity meter. Rotate the wheel back and forth to arrive at the exact minimum intensity. Use lens for accuracy.
4. Note down the reading on degree meter as (B).
5. From the given 10% sugar solution, prepare five different concentrations as per the instructions.
6. Replace water in the tube by sugar solutions under study, remove bubble if any and place the tube in the chamber.
7. Now rotate the wheel in the direction of decreasing intensity till intensity is minimum, rotate the wheel back and forth for accuracy.
8. At minimum intensity note down reading in the degree meter as (A).
9. (A-B) is the angle of rotation (θ).
10. Plot the graph of angle of rotation (θ) against concentration %.
11. From the graph determine the % of the unknown sugar solution.
12. Calculate the values of angle of specific rotation (α) by using the values of (θ) and calculate the mean value of (α).

Observation table:

Sr. No.	Conc. of sugar solution (C)	Reading for solution (A)	Reading for H_2O (B) or zero reading	Angle of Rotation θ= A – B	Specific angle of rotation (α)
1					
2					
3					
4					
5					
6	Unknown				

θ = Reading of solution (A) – zero reading (B)

Preparation of sugar solutions of different concentrations: (Use 50 ml volumetric flask)

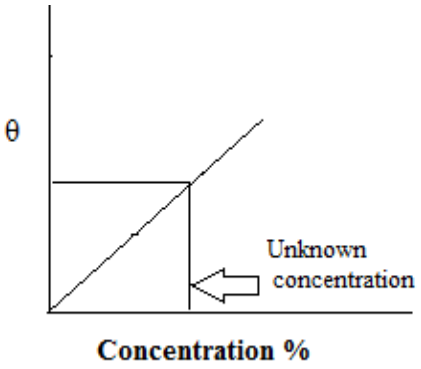
1. Given sugar solution . = 10 %
2. Dilute 40 ml of 10% solution to 50 ml = 8 %
3. Dilute 30 ml of 10% solution to 50 ml = 6 %
4. Dilute 20 ml of 10% solution to 50 ml = 4 %
5. Dilute 10 ml of 10% solution to 50 ml = 2 %

1. Dilute 45 ml of 10% solution to 50 ml = 9 %
2. Dilute 35 ml of 10% solution to 50 ml = 7 %
3. Dilute 25 ml of 10% solution to 50 ml = 5 %
4. Dilute 15 ml of 10% solution to 50 ml = 3 %
5. Dilute 5 ml of 10% solution to 50 ml = 1 %

.**Calculations:** Show all details of calculations.

$$\alpha = \frac{100 \times \theta}{L \times C}$$

where α = Angle of specific rotation
θ = Angle of rotation
L = Length of the polarimetric tube in decimeter
C = Conc. of sugar solution in %



Results:

1.	Specific rotation (Mean)	α = ----------
2.	Concentration of unknown sugar solution from the graph	= --------- %

5. Refractometry

Aim : To determine the specific and molecular refractivities of the given liquids A, B, and C and hence determine the refractivity of -CH_2 group.

Given : Liquids (A, B, C), acetone, cotton, droppers, Abbe's refractometer etc

Part- A (Determination of densities of liquid)

$$\text{Density of liquid} = \frac{\text{weight of liquid}}{\text{weight of water}} \text{ x density of water}$$

Liquid	Wt of empty Sp. gravity bottle = (B)	Wt. of specific gravity bottle + liquid = (A)	Wt. of liquid = A – B	Density of liquid (d)
A				
B				
C				
Dist. water				1.00

Part –B (Determination of refractive index of liquids)

Procedure:

1. Set the instrument in front of a well light window.
2. Open the prism box, clean and dry prism by acetone by using soft cotton.
3. By dropper put some of the liquid sample A on the prism and lock it.
4. Bring the mirror to the proper position.
5. See in the telescope and move the position of prism with the help of adjusting screw.
6. At the proper position colours of two halves will be seen.
7. With the help of the circular device (compensator) remove the colour fringes and bring the fine position of the two halves.
8. Now with the help of the adjusting screw, coincide the line of separation of the two colours with the crosswire.
9. Read and note the refractive index (n) directly from the scale.
10. Repeat the above procedure for remaining liquids B and C.

Observation table:

Liquid	Refractive index (n)	Density (d)	Molecular weight (M)	Specific refractivity (Rs)	Molecular refractivity (Rm)	Refractivity of -CH_2 group (Δ Rm)
A			78			ΔRm_1 =
B			92			ΔRm_2 =
C			106			ΔRm_3 =

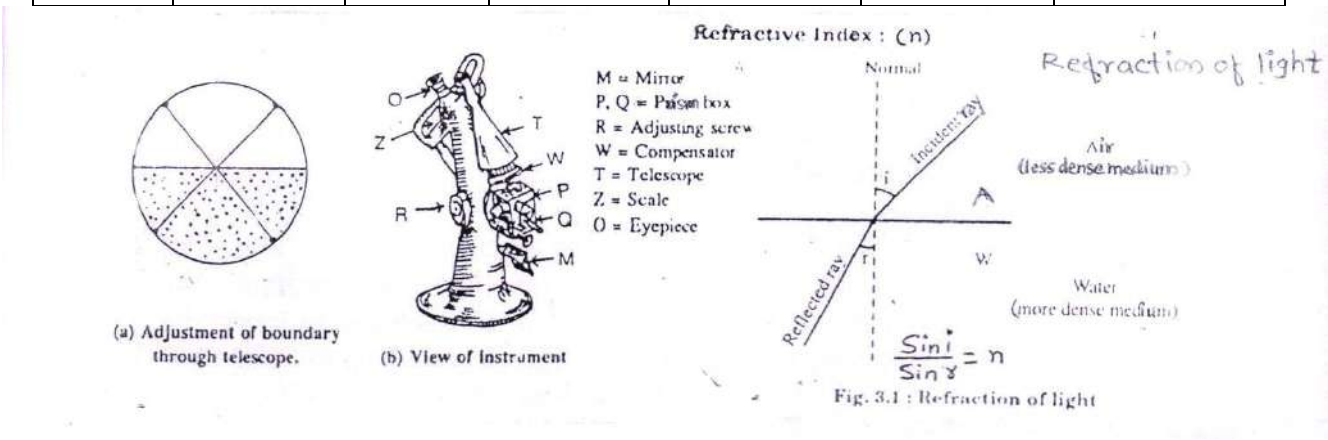


Fig. 3.1 : Refraction of light

Calculations : Calculate the specific and molar refractivities of liquids by using the formulae

1. Specific recfractivity $Rs = \frac{n^2-1}{n^2+2} \times \frac{1}{d}$
2. Molecular refractivity Rm = Rs x M
3. Determination of refractivity of – CH_2 group

   ΔRm_1 = Rm (B) – Rm (A)

   ΔRm_2 = Rm (C) – Rm (B)

   ΔRm_3 = 1/2 [Rm (C) – Rm (A)]

Results:

1.	Refractivity of -CH_2 group	= ----------

6. Viscosity

Aim : To determine the percentage composition of the given unknown liquid mixture by using Ostwald's viscometer.

Aparatus : Ostwald's viscometer, dryer, stopwatch.

Chemicicals: Drying liquid (acetone), pure liquid A and B, their mixtures (C, D, E and F)

Procedure :

1. Wash the viscometer with acetone and dry it by Passing current of air. Attach a piece of rubber tube to the narrow arm and clamp it in a perfectly vertical position.
2. Introduce required volume of liquid A (20 or 25 ml) in to the viscometer and suck the liquid A by means of rubber tube till it rises little above the upper mark.
3. Allow the liquid to flow through the capillary tube in to the lower bulb and measure the time required for the flow of liquid from upper mark to lower mark. Note down the time in seconds.

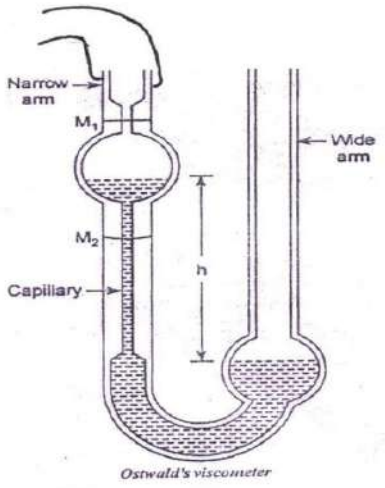
4. Repeat the measurement of time of flow three times and take the mean readings as the time of flow.
5. Remove liquid A from viscometer, rinse it with acetone and dry it.
6. Rinse viscometer with liquid B and introduce the same volume of liquid B and determine the time of flow as explained above .Repeat this procedure for other liquid mixtures C, D, E, and F. Ask the densities of A, B, C, D, E and F.

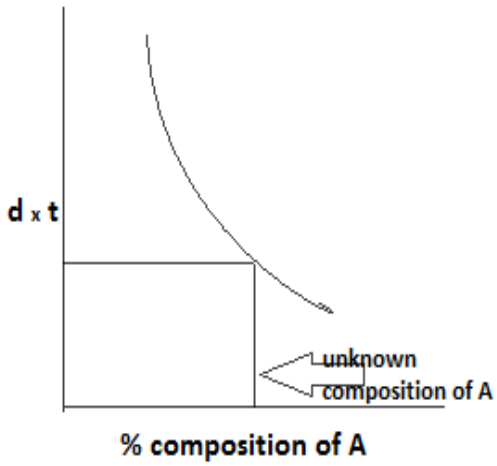
Observation table:

Liquids	% of A	% of B	Time of flow in seconds (t)				Density (d)	d x t
			I	II	III	Mean(t)		
A	100	0						
B	0	100						
C								
D								
E								
F	Unknown	Unknown						

Diagram and Graph:

Plot the graph of (d x t) (Y axis) against percentage of A (X axis). Obtain the smooth curve from the graph and determine the percentage composition of F.





Result : Percentage composition of F from graph

1.	Percentage of A	= ----------%
2.	Percentage of B	= ----------%

7. Chemical Kinetics No. 1
Relative Strength of Two Acids

Aim : To determine the relative strength of two acids from the hydrolysis of methyl acetate in presence of 0.5 N HCl and 0.5 N H_2SO_4.

Given : Methyl acetate, 0.5 N HCl, 0.5 N H_2SO_4, 0.1 N NaOH, phenolphthalein, ice

Procedure : Perform two set of experiments as follows

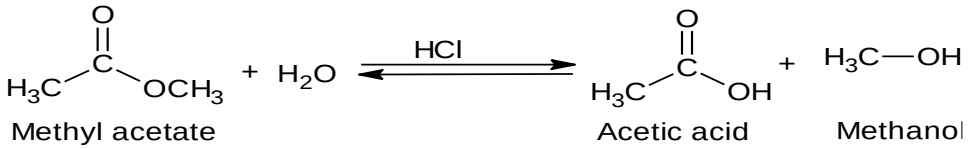
Set – I: Bottle No. 1: 5 ml methyl acetate.
Bottle No. 2: 100 ml 0.5 N HCl

1. Keep these two bottles in water bath to attain the same temperature.
2. Mean while fill the burette with 0.1 N NaOH. Take 3 or 4 ice pieces and 2 to 3 drops of phenolphthalein indicator in a conical flask.
3. Add solution from bottle No. 2 to bottle No. 1 completely and start the stop watch. Shake well and immediately pipette out 5 ml reaction mixture in the conical flask containing ice and indicator. Titrate this reaction mixture against 0.1 N NaOH solution, till colour changes from colourless to faint pink. Note down the burette readings as T_o (zero minute reading).
4. Again fill the burette with 0.1 NaOH. Take ice and indicator in conical flask.
5. Similarly take readings for time intervals of 10, 20, 30 and 40 minutes from the mixing time (Ask for T_∞).

Set – II: Bottle No. 1: 5 ml methyl acetate.
Bottle No. 2: 100 ml 0.5 N H_2SO_4

Follow all the steps in the procedure of the Set – I

Reaction:



Set- I (0.5 N HCl)

Observations:

1. In burette : 0.1 N NaOH
2. In conical flask : Ice + indicator + 5 ml reaction mixture
3. Indicator : Phenolphthalein
4. End point : Colourless to pink

Observation table

Given T_∞ = ---------- ml $a = T_\infty - T_0$ = ---------ml

Time in Min. t	Titration reading T_t ml	$X=T_t - T_o$	a-x	$\frac{a}{a-x}$	$log\frac{a}{a-x}$	k_1 min^{-1}
0	T_0=					
10	T_t=					
20	T_t=					
30	T_t=					
40	T_t=					

Mean k_1 = …… min^{-1}

Set- II (0.5 N H_2SO_4)

Observations:

1. In burette : 0.1 N NaOH
2. In conical flask : Ice + indicator + 5 ml reaction mixture
3. Indicator : Phenolphthalein
4. End point : Colourless to pink

Observation table:

Given T_∞ = ---------- ml $a = T_\infty - T_0$ = ---------ml

Time in Min. t	Titration reading T_t ml	$X=T_t - T_o$	a-x	$\frac{a}{a-x}$	$log\frac{a}{a-x}$	k_2 min^{-1}
0	T_0=					
10	T_t=					
20	T_t=					
30	T_t=					
40	T_t=					

Mean k_2 = …… min^{-1}

Calculation: Formula to be used

$$k = \frac{2.303}{t} log\frac{a}{a-x} \qquad \text{Relative strength of two acids} = \frac{\text{Mean of -}k_2}{\text{Mean of -}k_1}$$

Give all details of calculations.

Results :

1.	Mean of k_1	= ---------- min^{-1}
2.	Mean of k_2	= ---------- min^{-1}
3.	Relative strength of two acids	= ----------

8. Chemical Kinetics No. 2

Effect of Acid Strength

Aim **:** To study the effect of acid strength on hydrolysis of methyl acetate.

Given **:** 0.5 N HCl, 0.25 N HCl, 0.1 N NOH, Methyl acetate, Phenolphthalein, ice etc

Procedure **:** Perform two set of experiments as follows.

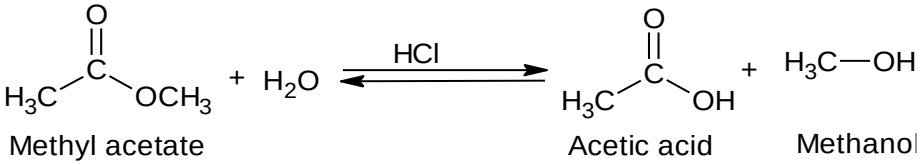
Set – I: Bottle No. 1: 5 ml methyl acetate.
Bottle No. 2: 100 ml 0.5 N HCl

1. Keep these two bottles in water bath to attain the same temperature.
2. Mean while fill the burette with 0.1 N NaOH. Take 3 or 4 ice pieces and 2 to 3 drops of phenolphthalein indicator in a conical flask.
3. Add solution from bottle No. 2 to 1 completely & start the stop watch. Shake well & immediately pipette out 5 ml reaction mixture in conical flask containing ice & indicator. Titrate this reaction mixture against 0.1 N NaOH solution, till colour changes from colourless to faint pink. Note the burette readings as T_o (Zero minute reading).
4. Again fill the burette with 0.1 NaOH. Take ice and indicator in conical flask.
5. Similarly take readings for time intervals of 10, 20, 30 and 40 minutes from the mixing time (Ask for T_∞).

Set – II: Bottle No. 1: 5 ml methyl acetate.
Bottle No. 2: 100 ml 0.25 N HCl

Follow all the steps in the procedure of the Set – I

Reaction:



Set- I (0.5 N HCl)

Observations:

1. In burette : 0.1 N NaOH
2. In conical flask : Ice + indicator + 5 ml reaction mixture
3. Indicator : Phenolphthalein
4. End point : Colourless to pink

Observation table

Given T_∞ = ---------- ml $a = T_\infty - T_0$ = ---------ml

Time in Min. t	Titration reading T_t ml	$X=T_t - T_o$	a-x	$\frac{a}{a-x}$	$log\frac{a}{a-x}$	Velocity constant k_1 min^{-1}
0	T_0=					
10	T_t=					
20	T_t=					
30	T_t=					
40	T_t=					

Mean k_1 = …… min^{-1}

Set- II (0.25 N HCl)

Observations:

1. In burette : 0.1 N NaOH
2. In conical flask : Ice + indicator + 5 ml reaction mixture
3. Indicator : Phenolphthalein
4. End point : Colourless to pink

Observation table:

Given T_∞ = ---------- ml $a = T_\infty - T_0$ = ---------ml

Time in Min. t	Titration reading T_t ml	$X=T_t - T_o$	a-x	$\frac{a}{a-x}$	$log\frac{a}{a-x}$	Velocity constant k_2 min^{-1}
0	T_0=					
10	T_t=					
20	T_t=					
30	T_t=					
40	T_t=					

Mean k_2 = …… min^{-1}

Calculation: Formula to be used

$$k = \frac{2.303}{t} \log\frac{a}{a-x}$$

Give all details of calculations

Results :-

1.	Velocity constant for Set-I	Mean of k_1 = ---------- min^{-1}
2.	Velocity constant for Set-II	Mean of k_2 = ---------- min^{-1}
3.	**Conclusion:** The value of k_2 is less than that of k_1 which shows that velocity constant decreases with decrease in concentration of acid i.e catalyst.	

9. Chemical Kinetics No. 3
$K_2S_2O_8$ and KI (Unequal Concentration)

Aim : To investigate the reaction between $K_2S_2O_8$ and KI with unequal concentrations of the reactants.

Given : 0.1 N $K_2S_2O_8$, 0.1 N KI, 0.002 N $Na_2S_2O_3$, distilled water, ice, starch etc

Procedure : Perform the experiment as follows

Bottle No. 1: 10 ml 0.1 N $K_2S_2O_8$ + 30 ml distilled water
Bottle No. 2: 20 ml 0.1 N KI + 20 ml distilled water

1. Keep these bottles in water bath to attain the same temperature and fill the burette with 0.002 N $Na_2S_2O_3$ solution.
2. Add the solution from bottle No. 2 to 1 and note the time of mixing. Shake well (do not take zero time reading).
3. Take 3 to 4 ice pieces and 8 to 10 drops of starch indicator in a conical flask.
4. Pipette out 10 ml reaction mixture in conical flask containing ice and indicator. Titrate it against $Na_2S_2O_3$ solution at the time interval 10, 15, 20, 25, 30, and 40 minutes from the mixing time till colour changes from blue to colourless.

Observation:

1. In burette : 0.002 N $Na_2S_2O_3$
2. In conical flask : Ice + indicator + 10 ml reaction mixture
3. Indicator : Starch solution
4. End point : Blue to colourless

Observation table:

Time in Min. t	Titration reading 'x' ml	a – x	b – x	$\frac{a-x}{b-x}$	$log\frac{a-x}{b-x}$	k dm^3/min/mol
10						
15						
20						
25						
30						
40						

Reactions:

$$2\,KI + K_2S_2O_8 \longrightarrow 2\,K_2SO_4 + I_2\downarrow$$

$$Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

Calculation :

Initial concentration 'a' and 'b'

For KI

KI (initial) x KI (in mixture)

$N_1V_1 = N_2V_2$

$0.1 \times 20 = N_2 \times 80$

$N_2 = 0.025$

KI (in mixture) against $Na_2S_2O_3$

$N_2V_2 = N_3V_3$

$0.025 \times 10 = 0.002 \times V_3$

V_3 = **125ml = (a)**

For $K_2S_2O_8$

$K_2S_2O_8$ (initial) against $K_2S_2O_8$ (in mixture)

$N_1V_1 = N_2V_2$

$0.1 \times 10 = N_2 \times 80$

$N_2 = 0.0125$

$K_2S_2O_8$ (in mixture) against $Na_2S_2O_3$

$N_2V_2 = N_3V_3$

$0.0125 \times 10 = 0.002 \times V_3$

V_3 = **62.5ml = (b)**

Calculate the values of k using formula

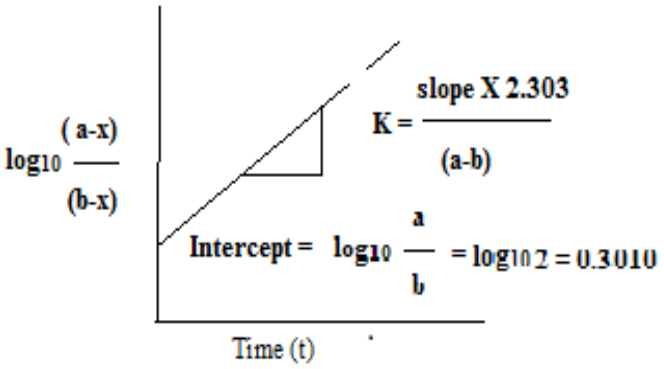
$$k = \frac{2.303}{t(a-b)} \log \frac{b(a-x)}{a(b-x)}$$

Give all details of calculation

.

Plot the graph of log (a – x) / (b-x) against time 't' Calculate the value of k from graph

$$k = \frac{2.303 \text{ x slope}}{a-b}$$



Result:

1.	Velocity constant (Mean k) by calculation	= -------- dm^3/min/mol
2.	Velocity constant (k) by graph	= ------- dm^3/min/mol
3.	**Conclusion:** Since values of k are fairly constant, it is a bimolecular or second order reaction	

10. Chemical Kinetics No. 4
$KBrO_3$ and KI (Equal Concentrations)

Aim : To determine the order of the reaction between $HBrO_3$ and HI.

Given : 0.1 N $KBrO_3$, 0.1 N KI, 0.01 N $Na_2S_2O_3$, starch indicator, 0.1 N HCl, distilled water, ice .

Procedure : Perform the experiment as follows.

Bottle No. 1: 25 ml 0.1 $KBrO_3$

Bottle No. 2: 25 ml 0.1 N KI + 100 ml 0.1 N HCl + 100 ml distilled water.

1. Keep the two bottles in a water bath to attain the same temperature and fill the burette with 0.01 N $Na_2S_2O_3$.
2. Add solution from bottle No. 2 to bottle No. 1 completely and note the time of mixing the mixture is stirred and placed in water bath throughout the experiment. It is shaken occasionally (do not take zero reading).
3. At intervals of 5, 10, 15, 20, 25 and 30 minutes from the mixing time, pipette out 25 ml of the reaction mixture in the conical flask containing 2 or 3 ice pieces and 8-10 drops of starch indicator. Titrate this reaction mixture against 0.01 N $Na_2S_2O_3$.The end point is blue to colourless.
4. Calculate the values of 'a' and 'b'.
5. Calculate the values of k using the given formula. Plot a graph of 1/ (a- x) against time (t). Calculate two values of k from the graph (k= slope).

Observation:

1. In burette : 0.01 N $Na_2S_2O_3$
2. In conical flask : Ice + indicator + 25 ml reaction mixture
3. Indicator : Starch (8 to 10 drops)
4. End point : Blue to colourless

Observation table:

Time in minutes (t)	**Titration reading 'x' ml**	**a - x**	$\frac{1}{a-x}$	$k = \frac{x}{t.a\,(a-x)}$ $dm^3/min/mol$
5				
10				
15				
20				
25				
30				

Mean k=…… lit mol^{-1} min^{-1}

Calculations:

Initial concentrations 'a' and 'b'

Normality of $KBrO_3$ in mixture

$KBrO_3$ (original) = $KBrO_3$ (in mixture)

$N_1V_1 = N_2V_2$

$0.1 \times 25 = N_2 \times 250$

$N_2 = (0.1 \times 25) / 250$

$N_2 = 0.01N$

Initial concentrations of $KBrO_3$ (a) in terms of 0.01 N $Na_2S_2O_3$

$N_2V_2 = N_3V_3$

$V_3 = (0.01 \times 25) / 0.01$

$V_3 = 25$ ml

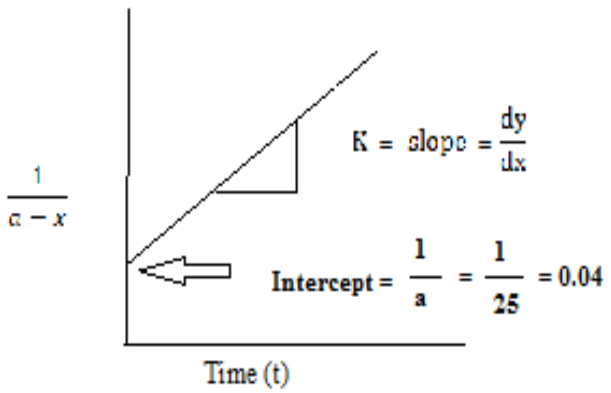
$\therefore a = V_3 = 25$ ml

As the normality and volume of KI used is same as that of $KBrO_3$ the concentration of KI in terms of 0.01 N $Na_2S_2O_3$ is 25 $\therefore a=b=25$ ml.

Calculate the values of k using formula
Give all details of calculations

$$k = \frac{x}{t.a\,(a-x)}$$

Plot 1/ (a-x) versus time and calculate k from graph,



k =slope.

Results:

1.	Velocity constant (Mean k) by calculation	= -------- dm^3/min/mol
2.	Velocity constant (k) by graph	= ------- dm^3/min/mol
3.	**Conclusion:** The graph is straight line intersecting on Y axis. Therefore reaction is bimolecular i.e. Order of reaction is 2.	

11. Partition Coefficient

Aim **:** To determine partition coefficient of benzoic acid between water and benzene

Given **:** Glass stoppered bottles of small diameter, beakers, pipette, benzoic acid, 0.1 N NaOH, 0.01 N NaOH, phenolphthalein, benzene etc.

Procedure **:**

1. Take three clean stoppered bottles and number them as a 1, 2 and 3. Weigh 1, 2 and 3 gram of benzoic acid and transfer these amounts in bottles 1, 2 and 3 respectively. To each bottle add 50 ml benzene and 50 ml water.
2. Stopper the bottles tightly and shake them vigorously for 30 minutes from time to time and allow to stand at room temperature. The content will separate in two layers, the lower layer will be the aqueous and upper layer will be benzene.

Part-I

3. Pipette out 10 ml of lower aqueous layer from bottle no. 1 in a conical flask containing 20 ml distilled water.
4. Titrate it with 0.01 N NaOH using phenolphthalein as indicator. End point is colourless to faint pink. During the titration, the contents should be shaken vigorously. Take two more readings for the same bottle and find out constant burette reading (C.B.R.).
5. Similarly, repeat the procedure in points 3 and 4 for remaining two bottles.

Part-II

6. Pipette out 10 ml of upper benzene layer from bottle no. 1 in a conical flask containing 20 ml distilled water.
7. Titrate it with 0.1 N NaOH using phenolphthalein as indicator. End point is colourless to faint pink. During the titration, the contents should be shaken vigorously. Take two more readings for the same bottle and find out constant burette reading (C.B.R.).
8. Similarly, repeat the procedure in points 6 and 7 for remaining two bottles.

Part-I (Lower Aqueous Layer)

Observation:

1. Solution in burette **:** 0.01 NaOH solution
2. Solution in conical flask**:** 10 ml aqueous layer
3. Indicator **:** Phenolphthalein
4. End point **:** Colourless to faint pink

Observation Table:

Bottle No.	Burette Reading in ml			C. B. R. ml
	I	II	III	
1				
2				
3				

Calculations:

Determine the concentration of aqueous layer ($C_{aq.}$) for each bottle by using following formula

Aqueous layer ≡ NaOH

$N_1 V_1 = N_2 V_2$

$N_1 \text{ X } 10 = 0.01 \text{ X C.B.R.}$

$$N_1 = \frac{0.01 \text{ X C.B.R.}}{10}$$

Part-II (Upper Benzene Layer)

Observation:

1. Solution in burette **:** 0.1 NaOH solution
2. Solution in conical flask**:** 10 ml benzene layer
3. Indicator **:** Phenolphthalein
4. End point **:** Colourless to faint pink

Observation Table:

Bottle No.	Burette Reading in ml			C. B. R. ml
	I	II	III	
1				
2				
3				

Calculations:

Determine the concentration of benzene layer ($C_{ben.}$) for each bottle by using following formula

Benzene layer ≡ NaOH

$N_1 V_1 = N_2 V_2$

$N_1 \times 10 = 0.1 \times C.B.R.$

$$N_1 = \frac{0.1 \times C.B.R.}{10}$$

Calculations of partition coefficient:

Bottle No.	Concentration of aqueous layer ($C_{aq.}$)	Concentration of benzene layer ($C_{ben.}$)	Partition coefficient $K = \frac{\sqrt{C_{ben.}}}{C_{aq..}}$
1			
2			
3			
		Mean K	

Result: Partition coefficient of benzoic acid between water and benzene (Mean K) = ----------

--

……X……

B. INORGANIC CHEMISTRY

a) Gravimetric Estimations

12. Gravimetric Estimation of Barium

Aim : To determine the amount of Barium as $BaSO_4$ from given solution of $BaCl_2.2H_2O$ and free HCl

Chemicals : Given sol^n of barium chloride in 250 ml volumetric flask, conc.HCl, 2 N H_2SO_4

Apparatus : Beakers, 25 ml pipette, 250 ml volumetric flask, glass rod, tripod stand, washing bottle, silica crucible, drying cone, asbestos sheet, desiccators, digital balance etc

Procedure :

1. **Dilution:** Dilute the given barium chloride solution to 250 ml with distilled water and shake well.
2. **Precipitation:** Pipette out 50 ml diluted solution in 500 ml beaker + 100 ml distilled water +2 ml conc. HCl and boil it on wire gauze. Keep beaker on asbestos sheet & add hot 2 N H_2SO_4 (about ¾ t.t.) with constant stirring. White precipitate of $BaSO_4$ is formed.
3. **Digestion:** Digest the precipitate of $BaSO_4$ on sand both for about 30 min. (Now test whether precipitation is completed or not by adding drop of 2 N H_2SO_4 with the help of glass rod in contact with inner side of beaker).
4. **Filtration and Washing:** Now filter the supernatant liquid through Whatman filterpaper No.42. Wash the precipitate for 3-4 times **in beaker** by hot distilled water and decant the supernant liquid through the same Whatman filter paper. Now transfer the white ppt. of $BaSO_4$ to Whatman filter paper by using washing bottle. Then Wash precipitate with distilled water until the fresh filtrate free from SO_4^{--} & Cl^- [Test with $Ba(NO_3)_2$ and $AgNO_3$].
5. **Drying and Ignition:** Dry the precipitate on drying cone and ignite in a weighed crucible. Heat the crucible for 45 minute. Cool and weigh the crucible. Find out constant weight of residue.

Reaction:

$$BaCl_2 + H_2SO_4 \longrightarrow BaSO_4\downarrow + 2HCl$$

$$BaSO_4 \xrightarrow{\text{Ignition}} \underset{\textbf{Residue}}{BaSO_4\downarrow}$$

Observation:

1. Weight of empty crucible	**W_1 = ……g**
2. Weight of empty crucible + residue	
On first heating (45 min) a = ……g	
On second heating (10 min) b = ……g	
On third heating (10 min) c = ----------g	
∴ Constant weight of crucible + residue	**W_2 = ……g**
3. Weight of residue = (W_2-W_1)	**W = ……g**

Calculation :

Constant weight of residue, W = ……g

$BaSO_4$	:	Ba	:	$BaCl_2.2H_2O$
233.4	:	137.4	:	244.28
1	:	0.5883	:	1.04469

a) Quantity of Ba:

Hence, 1 g $BaSO_4$ = 0.5883 g Ba

$\therefore$ W g $BaSO_4$ = W x 0.5883 g Ba

i.e. A = …….. g Ba

Thus the quantity of Ba present in 50 ml diluted solution is = A =……g

$\therefore$ Quantity of Ba present in 250 ml i.e. in given solution is = A x 5 = B =…….. g

b) Quantity of $BaCl_2.2H_2O$:

Now 0.5883 g Ba = 1.0469 g $BaCl_2.2H_2O$

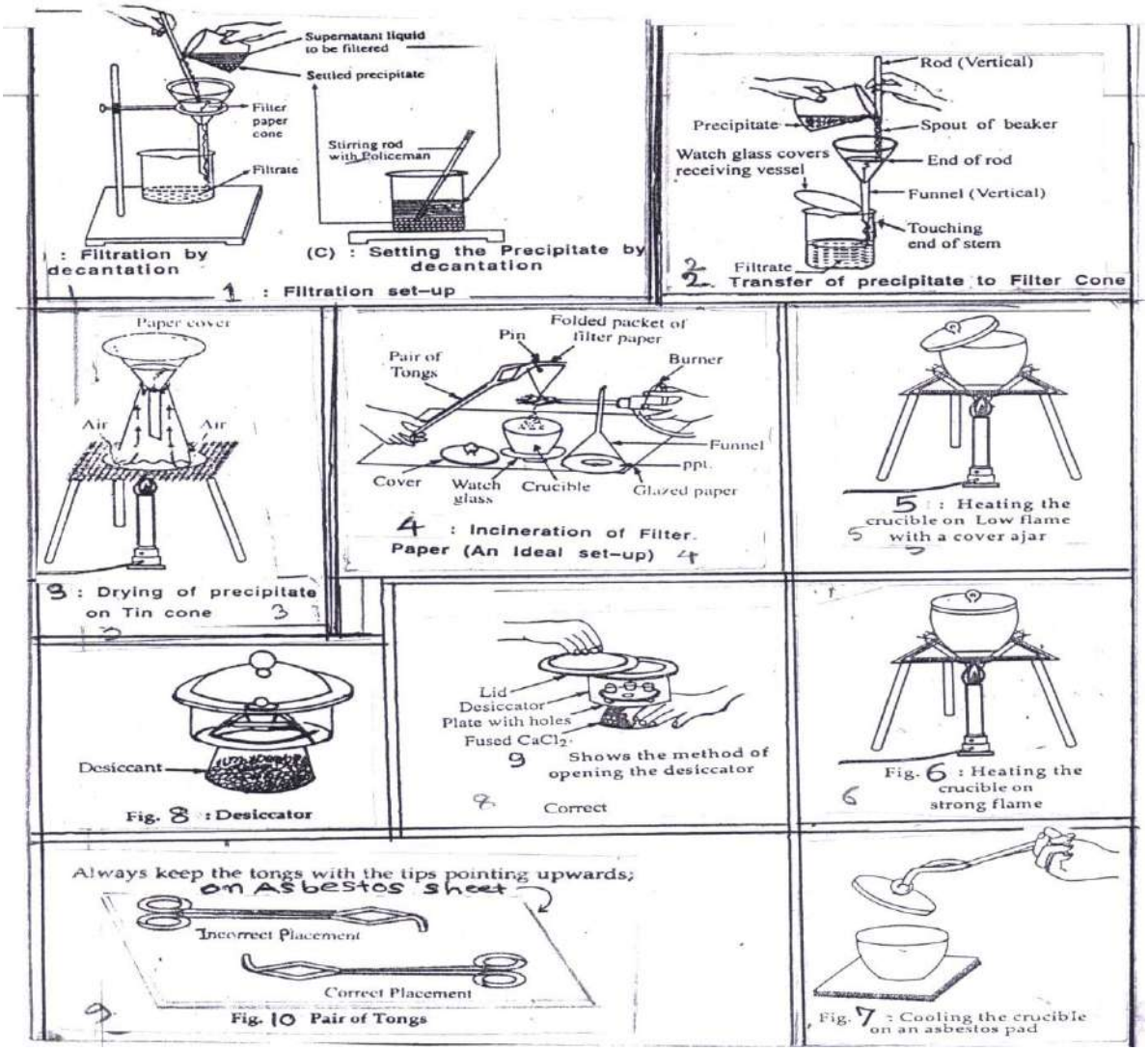
$\therefore$ B g Ba = $\frac{B \times 1.0469}{0.5883}$ g $BaCl_2.2H_2O$

i.e C = …….. g $BaCl_2.2H_2O$

Results:

1.	50 ml of the diluted solution gave residue (weight of $BaSO_4$residue)	(W) = ……g	=…….x 10^{-3}Kg
2.	Quantity of metal (Ba) in given solution	(B) =……..g	=…….x 10^{-3}Kg
3.	Quantity of metal salt ($BaCl_2.2H_2O$) in the given solution	(C) =……..g	=…….x 10^{-3}Kg

Techniques in Gravimetric Analysis



13. Gravimetric Estimation of Iron

Aim : To the determine the amount of Fe as Fe_2O_3 from a given solution of ferrous ammonium sulphate (F.A.S.) and free sulphuric acid .

Chemical : Given sol^n of F.A.S. in 250 ml volumetric flask, 1:1 ammonia sol^n, conc. HNO_3 dil. H_2SO_4, solid NH_4Cl, 1% Ammonium Nitrate

Apparatus : Beakers, 25 ml pipette, 250 ml volumetric flask, tripod stand, washing bottle, silica crucible, drying cone, asbestos sheet, desiccators, digital balance etc.

Procedure :

1. **Dilution:** Dilute the given solution to 250 ml with distilled water and shake well.
2. **Precipitation:** Pipette out 50 ml diluted solution in 500 ml beaker + 100 ml distilled water and boil it on wire gauze + 10 ml dil. H_2SO_4 + 5 ml conc. HNO_3 + about 2 g solid ammonium chloride**. Observe colour (it should be intense yellow).** Stop heating and add 1:1 ammonia solution with constant stirring until there is a distinct smell of ammonia to the solution. A dark brown gelatinous precipitate of Fe $(OH)_3$ is formed . Boil the solution on wire gauze.
3. **Filtration and Washing:** Immediately filter the ppt. through Whatman Paper No. 41and wash the precipitate with distilled water and then hot 1% ammonium nitrate solution until the fresh filtrate is free from SO_4^{--} and Cl^- [test with $Ba(NO_3)_2$ and $AgNO_3$ solution].
4. **Drying and Ignition:** Dry the precipitate on drying cone and ignite in the weighed crucible. Heat the crucible for 45 minutes on blue flame. Now cool the crucible and weigh. Find out constant weight of residue (Fe_2O_3).

Reaction:

$$FeSO_4(NH_4)_2SO_4 \cdot 6H_2O \xrightarrow{\text{Dissolution}} FeSO_4 + (NH_4)_2SO_4 + 6H_2O$$

$$6FesO_4 + 3H_2SO_4 + 2HNO_3 \xrightarrow{\text{Oxidation}} 3Fe_2(SO_4)_3 + 4H_2O + 2NO\uparrow$$

$$Fe_2(SO_4)_3 + 6NH_4OH \xrightarrow{\text{Precipitation}} 2Fe(OH)_3\downarrow + (NH_4)_2SO_4$$

$$2Fe(OH)_3 \xrightarrow{\text{Ignition}} Fe_2O_3 + 3H_2O$$

Observation:

1. Weight of empty crucible	**W_1 = ……g**
2. Weight of empty crucible + residue	
On first heating (45 min) a = ……..g	
On second heating (10 min) b = ……..g	
On third heating (10 min) c = ……...g	
∴ Constant weight of crucible + residue	**W_2 = ……g**
3. Weight of residue = (W_2-W_1)	**W = ……g**

Calculation:

Constant weight of residue, W = ----------g

Fe_2O_3	:	2Fe	:	$2FeSO_4(NH_4)_2SO_4.6H_2O$
160	:	112	:	784.26
1	:	0.6994	:	4.9092

a) Quantity of Fe:

Hence, 1 g Fe_2O_3 = 0.6994 g Fe

$\therefore$ W g Fe_2O_3 = W x 0.6994 g Fe

i.e. A = ……. g Fe

Thus the quantity of Fe present in 50ml diluted solution is = A = …… g Fe

Quantity of Fe present in 250 ml i.e. in given solution is = A x 5 = …… g Fe

i.e. B = ……g Fe

b) Quantity of F.A.S.:

0.6994 g Fe = 4.9092 g F.A.S.

$\therefore$ B g Fe = $\frac{\text{B x 4.9092}}{0.6994}$ g F.A.S.

i.e. C = …… g F.A.S.

Results:

1.	50 ml of the diluted solution gave residue (weight of Fe_2O_3 residue)	(W) = ……g	=……x 10^{-3}Kg
2.	Quantity of metal (Fe) in given solution	(B) =……..g	=……x 10^{-3}Kg
3.	Quantity of metal salt $[FeSO_4(NH_4)_2SO_4.6H_2O]$ in the given solution	(C) =……..g	=……x 10^{-3}Kg

b) Titrimetric Analysis

14. Calibration of Volumetric Apparatus

Aim : To calibrate volumetric apparatus- burette, pipettes and volumetric flasks.

Appratus : 50 ml burettes, 5, 10& 25 ml pipettes, 100 & 250 ml volumetric flasks.

Calibration : For most of analytical purposes class A and class B glass apparatus are manufactured and are used without calibration. But for the highest accuracy all the glass apparatus (burette, pipettes, volumetric flasks etc) are need to be calibrated.

Cleaning of apparatus:

1. Saturated solution of powdered sodium or potassium dichromate in conc. sulphuric acid used in the form of Cleaning Mixture.
2. More efficient cleaning solution is a mixture of concentrated sulphuric acid and fuming nitric acid. This cleaning solution is used only when glass apparatus is very dirty and greasy, but must be handled with extreme caution.
3. Effective degrasing agent which is much quicker in action then a ‘cleaning mixture’ is obtained by dissolving 100g of potassium hydroxide in 50 ml of distilled water and after cooling making up to 1 liter with industrial methylated spirit .
4. For cleaning the apparatus, it is filled with cleaning mixture and allows standing for several hours preferably overnight. Cleaning mixture is then poured off, then the apparatus is thoroughly rinsed with distilled water and allow to drain off until dry.

The calibration process can be carried out by two procedures

i. Direct or Absolute Calibration Method &

ii. Relative Calibration Method

i. **Direct or Absolute Calibration Method:** This Method involves weighing of water at 20^0c as there is variation in density of water at different temperature. Therefore there is a variation in the volumes of water at various temperatures. It should be also noted that, there is little expansion / contraction of glass at different temperature. Due to this errors are arises in the measurement of volumes. To avoid this volume and weight of deionized water is noted at respective temperatures which different in different seasons & it is compared with standard volume of water at various temperature (Table No.1).

Table: - 1: Volume of 1 g water at various temperatures

Temp (^{0}C)	Volume (ml)		Temp (^{0}C)	Volume (ml)		Temp (^{0}C)	Volume (ml)		Temp (^{0}C)	Volume (ml)
10	1.0013		20	1.0028		25	1.0040		30	1.0054
12	1.0015		21	1.0030		26	1.0043		31	1.0056
14	1.0018		22	1.0033		27	1.0045		32	1.0059
16	1.0021		23	1.0035		28	1.0048		34	1.0065
18	1.0024		24	1.0037		29	1.0051		35	1.0068

ii. Relative Calibration Method

A. In this method relationship between two items of glassware determined without knowing the absolute volume or standard volume of any one.

B. Glassware's to be calibrated are washed / degreased and rinsed with distilled water.

C. Set of pipettes, burettes and volumetric flasks are calibrated by relative calibration method as given below -

1. Clamp the labeled (for e.g.No.1) burette perfectly vertical position to the burette stand.

2. Fill the burette with distilled water up to lower mark i.e.50 ml mark (at bottom side) by taking care that, there should not be any drop of liquid coming out from the burette (or there should not be any leakage).

3. Here burette volume is supposed to be standard volume and hence its volume is compared with pipettes (25ml, 10ml & 5ml) and volumetric flasks (100ml &250 ml).

4. Calibration of pipettes.

a) 25 ml pipette :-i) Take clean 25 ml pipette. ii) Suck by mouth the distilled (deionised) water carefully until it is well above the volume mark on stem. iii) Close the mouth of stem by index finger and hold pipette in the vertical position. iv) Allow the solution to come down slowly by releasing index finger. v) Press the index finger again when the lower limit of the meniscus is just against the volume mark on stem. See that the mark on the stem is inline with the level that of the eye. vi) Insert the pipette into burette which already contains water up to 50 ml mark. vii) Now release the index finger and deliver the distilled water in to burette. During the release of water touch the jet of pipette to the inner side of the burette to drain it completely. viii) Transfer last traces of water completely from pipette in to burette by usual procedure (do not blow pipette by mouth) ix) Now see the volume occupied by distilled water in burette, it should be exactly 25 ml. If not then try another 25 ml pipette, till the matching volume pipette is obtained. x) Now label this matched pipette as that of burette label (for e.g. No: 1)

b) 10 ml pipette: i) Take clean 10 ml pipette and calibrate it against the same burette, by using similar procedure mentioned above. ii) Label this10 ml pipette as that of the burette (for e.g. No.1).

c) 5 ml pipette :- i) Take clean 5 ml pipette and calibrate it against the same burette, by using similar procedure mentioned above. ii) Label this 5 ml pipette as that of the burette (for e.g. No.1).

5. Calibration of Volumetric flasks:-

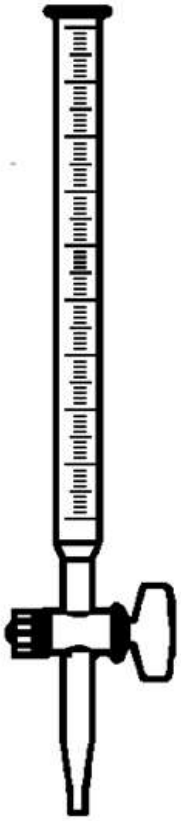
a) 250 ml Volumetric flasks: - i) Prepare clean 50 ml burette (for e.g. No.1) and dry 250ml volumetric flasks ii) Fill the 50 ml burette up to the mark with distilled water and drain it five times (50 ml volume each time) in to the volumetric flasks. iii) If the 250 ml mark on the neck of flask coincides with the meniscus of the distilled water, then the 250 ml volumetric flask is said to be calibrated with respect to burette and pipettes. If the 250 ml water level **does not match to the mark on the neck of flask then make a new marking of 250ml on the neck of the flask** & label this flask accordingly (for e.g. No.1).

b) 100 ml Volumetric flask: - By using same burette, calibrate a clean 100 ml volumetric flask as mentioned above and label it as usual (for e.g. No.1)In this way the apparatus burette, pipette and volumetric flask required for volumetric analysis are calibrated and labeled set of apparatus prepared. Similarly required number of sets of calibrated apparatus is prepared.

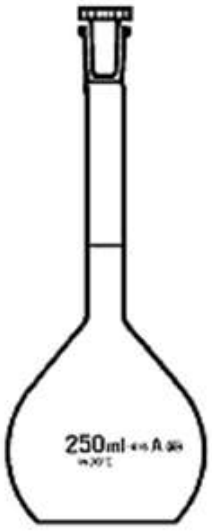
Result: - All the apparatus required for volumetric analysis like burette, pipettes and volumetric flask are calibrated by using relative calibration method and 1 or 2 or 3 or …….sets are prepared.

Volumetric Apparatus

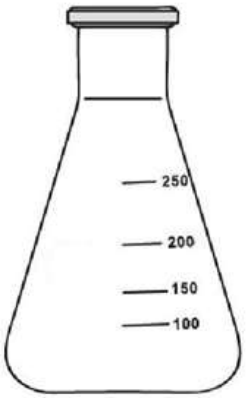
Volumetric pipette



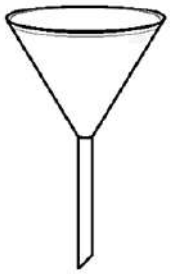
Volumetric Burette



Volumetric flask



Conical flask



Funnel

15. Analysis of Commercial Vinegar

Aim : To determine the percentage of acetic acid in commercial vinegar sample.

Chemicals : Given vinegar sample in 250 ml volumetric flask, app. 0.1 N NaOH solution phenolphthalein indicator etc.

Apparatus : 250 ml volumetric flask, burette, pipette, conical flask etc

Theory : Commercial vinegar sample usually contains 4-5 % acetic acid. This acetic acid in vinegar solution is titrated against standard solution of NaOH and content of acetic acid in vinegar is determined.

Procedure :

Part –I: Preparation of standard sol^{n} of oxalic acid and standardization of NaOH

1. Weigh accurately 1.575 g of oxalic acid on a watch glass and transfer it in beaker, Dissolve it in minimum distilled water and transfer this solution to 250 ml volumetric flask. Dilute the contents up to the mark with distilled water and shake well. It gives standard 0.1 N oxalic acid solution (**Primary standard**).
2. Take 25ml of 0.1N oxalic acid solution in conical flask. Add two drops of phenolphthalein indicator and titrate this solution against supplied NaOH solution from the burette.
3. Take three readings and find out constant burette reading as ‘X ’ml.
4. Calculate the exact normality of NaOH solution. (**Secondary standard**).

Part-II: Determination of % of acetic acid commercial sample of Vinegar

1. Dilute the given sample of commercial Vinegar to 250 ml with distilled water.
2. Pipette out 25 ml of this diluted solution of Vinegar in 250 ml conical flask.
3. Add two drops of phenolphthalein indicator in conical flask.
4. Add standardized NaOH from burette till colour changes from colourless to just pink.
5. Take two more readings as above and find out CBR as ‘Y’ ml.

Observations and Observation table:

Part –I: Preparation of standard sol^n of oxalic acid and standardization of NaOH

Observations:

1.	Weight of empty watch glass	W_1 = ……g
2.	Weight of oxalic acid	W = 1.575 g
3.	Weight of watch glass + sample	W_2 = ……g

Observations :

1. In Burette : App. 0.1 N NaOH sol^n
2. In conical flask : 25 ml 0.1 N oxalic acid solution
3. Indicator : Phenolphthalein (2-3drops)
4. End point : Colorless to Pink

Observation Table:

Burette level	**Burette Readings in ml**			**CBR**
	I	**II**	**III**	
Final level				
Initial level	0.0	0.0	0.0	X= ….. ml
Difference				

Part-II: Determination of % of acetic acid commercial sample of Vinegar

Observations :

1. In Burette : Z N NaOH solution (**Secondary Standard**)
2. By Pipette : 25 ml diluted vinegar solution
3. Indicator : Phenolphthalein (2-3drops)
4. End point : Colorless to Pink

Observation Table:

Burette level	**Burette Readings in ml**			**CBR**
	I	**II**	**III**	
Final level				
Initial level	0.0	0.0	0.0	Y= …..ml
Difference				

Reaction: $CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$

Calculations:

1) To determine exact Normality of NaOH solution:

NaOH Oxalic acid

$N_1V_1 = N_2V_2$

$$N_1 = \frac{0.1 \times 25}{V_2}$$

$$N_1 = \frac{0.1 \times 25}{X}$$

i.e Z = …… N NaOH

2) To determine percentage of acetic acid in Vinegar :

1 ml 0.1 N NaOH = 0.006 g acetic acid

1 ml Z N NaOH = $\frac{Z \times 0.006}{0.1}$ g acetic acid

i.e A = …… g acetic acid

Thus

1 ml Z N NaOH = A g acetic acid
∴ Y ml Z N NaOH = A x Y g acetic acid in 25 ml dil. solution
i.e. B = …… g acetic acid in 25 ml dil. solution

Now

25 ml diluted solution = B = ……. g of acetic acid .
∴ 250 ml diluted solution = B x 10 = ……. g acetic acid
i.e. C =…… g acetic acid

Given: Volume of Vinegar = D = …….. ml (**Ask for D)**

Now

∴ D ml given solution contains = C g acetic acid
∴ 100 ml given solution ≅ $\frac{C \times 100}{D}$ % acetic acid
i.e.E =……% acetic acid

Results:

1.	Exact normality of given NaOH	Z =…… N of NaOH
2.	Percentage of acetic acid in Vinegar	E = …….. %

16. Total Hardness of Water

Aim : To prepare standard solution of calcium chloride from calcium carbonate and determine total hardness of given water sample.

Apparatus : Burette, pipette, conical flask, measuring flask, watch glass, beakers, fractional weight box etc.

Chemicals : Given sample of hard water, EDTA (0.01M), $CaCO_3$, buffer solution (pH 10) Eriochrome black –T indicator etc.

Principle : Hardness of water is due to presence of Ca and Mg salts. EDTA reacts quantitatively with Ca and Mg to produce soluble stable chelates;

M + In ⟶ M-In

M-In + EDTA ⟶ M-EDTA + In

Where, M = Metal ion, In = Indicator.

Procedure :

Part I: Preparation of standard $CaCl_2$ solution from $CaCO_3$

Weigh accurately 0.250 g of $CaCO_3$ on watch glass and dissolve in minimum amount of conc. HCl and distilled water (About 5 ml Conc. HCl and 50 ml distilled water). Transfer this solution to 250ml volumetric flask by taking usual precautions. Dilute it up to the mark with distilled water and shake well.

Part II: Standardisation of EDTA solution:-

Pipette out 25 ml of standard 0.01 M $CaCl_2$ solution in conical flask. Add to it 5 ml buffer solution (pH10) and Eriochrome black – T indicator. Titrate it against given EDTA solution till colour changes from wine red to sky blue. Find out CBR as X ml.

Part III: Determination of total hardness of water sample:-

Dilute the given hard water sample to 250 ml with distilled water & shake well Pipette out 25 ml of this diluted sample solution in conical flask . Add to it 5 ml buffer solution (pH 10) and Eriochrome black –T indicator. Titrate it against 0.01M EDTA solutions till colour changes from wine red to sky blue. Find out CBR as Y ml.

Part I

Observations:-

1.	Weight of empty watch glass	W_1 = …… g
2.	Weight of $CaCO_3$	W = 0.250 g
3.	Weight of watch glass + $CaCO_3$	W_2 = …….. g

Part II

Observations:-

1. Solution in burette **:** Given 0.01 EDTA solution
2. Solution conical flask **:** 25ml standard $CaCl_2$ solution by pipette + 5ml buffer solution (pH 10).
3. Indicator **:** Eriochrome black –T (Pinch of solid or 3-4 drops liquid)
4. End point **:** Wine red to sky blue.

Observation Table

Burette level	**Burette reading ml**			**C.B.R** ml
	I	II	III	
Final level				X=……..ml
Initial level	0.0	0.0	0.0	
Difference				

Part III

Observations:-

1. Solution in burette **:** Given 0.01 EDTA solution
2. Solution conical flask **:** 25ml standard $CaCl_2$ solution by pipette + 5ml buffer solution (pH 10).
3. Indicator **:** Eriochrome black –T (Pinch of solid or 3-4 drops liquid)
4. End point **:** Wine red to sky blue.

Observation Table:- Draw as above & CBR = Y ml

Reactions: -

$$Ca^{2+} + Na_2H_2EDTA \longrightarrow 2Na^{+} + [\ CaEDTA\ ]^{-2} + 2H^{+}$$

Calculations :-

A) Standardisation of Given EDTA

EDTA	**Vs**	**$CaCl_2$**
N_1V_1	=	N_2V_2

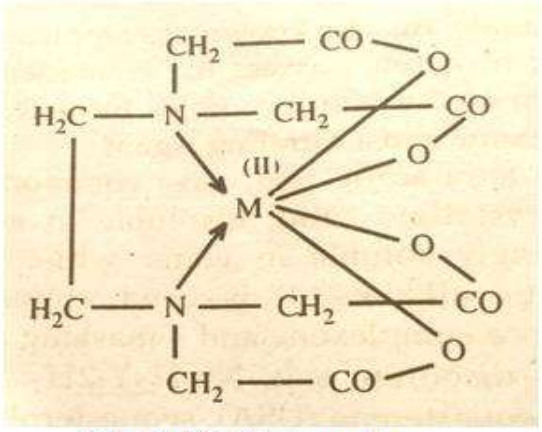
$$N_1 = \frac{N_2 \times V_2}{V1}$$

$$N_1 = \frac{0.01 \times 25}{X}$$

i.e. Z = …….. M EDTA

∴ Molarity of EDTA = Z =

Structure of Metal-EDTA Complex:-



Metal-EDTA complex

B) Hardness of water sample:-

We have

1 ml of 0.01 M EDTA = 0.001 g $CaCO_3$

∴Y ml of Z M EDTA = $\frac{Z \times Y \times 0.001}{0.01}$ g $CaCO_3$

A = ……. g $CaCO_3$

Thus, 25 ml of diluted water sample = A g $CaCO_3$

250 ml of diluted water sample = A x 10 g $CaCO_3$

i.e B = ……. g $CaCO_3$

Hardness in ppm:

Now, 250 ml of sample = B g $CaCO_3$

10^6 ml of sample = $\frac{B \times 10^6}{250}$ g $CaCO_3$

i.e C = ……. ppm

∴ Total hardness of water as parts per million (ppm) of $CaCO_3$ = C = …… ppm

Results

1	25ml of standard calcium chloride require	X = ……ml of given EDTA sol^n
2	25 ml of diluted sample of water	Y = …… ml of given EDTA sol^n
3	Total hardness of given sample of water	C =……ppm

c) Inorganic Preparations

17. Preparation of Ferrous Ammonium Sulphate (Mohr's Salt)

Aim **:** To prepare ferrous ammonium sulphate (F.A.S.) from ferrous sulphate and ammonium sulphate.

Chemicals **:** Ferrous sulphate ($FeSO_4.7H_2O$), ammonium sulpahte $(NH_4)_2\ SO_4$, ethyl alcohol etc.

Apparatus **:** 250 ml Beakers, glass rod, measuring cylinder etc.

Procedure **:**

1. Weigh 10 g of Ferrous Sulphate and transfer in to beaker. Add in it about 60ml distilled water and 5 ml dil. H_2SO_4, dissolve by boiling the solution.
2. Add to it 5g of ammonium sulphate with constant stirring.
3. Add a bright iron nail to maintain iron content.
4. Boil the solution till the crystallization points is just reached (avoid formation of crystal masses).
5. Cool and add about 10 ml ethyl alcohol. Faint green coloured crystals of F.A.S. are obtained.
6. Now filter the product on Buchner funnel and wash the product with little alcohol. Dry and weigh the product.

Observations:

i. Ferrous sulphate $FeSO_4.7H_2O$ = …….. g
ii. Ammonium sulpahte $(NH_4)_2\ SO_4$ = …… g

Reaction:

$$FeSO_4 + (NH_4)_2\ SO_4 + 6H_2O \longrightarrow FeSO_4\ (NH_4)_2SO_4 \cdot 6H_2O$$

Simple salt Simple salt Double salt (F.A.S.)

Calculations:

a) Theoretical yield	**b) Percent % yield**
From chemical reaction we get $FeSO_4.7H_2O$ = $FeSO_4(NH_4)_2SO_4.6H_2O$ 278 392 Now , 278 g Ferrous sulphate = 392 g F.A.S $\therefore$ 10 g Ferrous sulphate = $\frac{10 \times 392}{278}$ g F.A.S. = 14 g $\therefore$ Theoretical yield of product (A) = 14 g	Weight of the product = X = ……g $\therefore$ 14 g product F.A.S = 100 % yield $\therefore$ X g product F.A.S = $\frac{X \times 100}{14}$ % i.e. B = ……%

Results:

1.	Colour of the product F.A.S.	Faint green	
2	Weight of the product F.A.S.	X = …… g	=…….. x 10^{-3}Kg
3.	Theoretical yield of product	A =……. g	=…….. x 10^{-3}Kg
4.	Practical % yield of product	B =……. %	

18. Preparation of Tetraamminecopper(II) sulphate

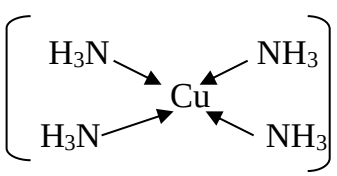
Aim : To Prepare Tetramminecopper(II) sulphate. $[Cu(NH_3)_4]SO_4.H_2O$

Chemicals : Copper sulphate, liquor ammonia, ethyl alcohol, dil. H_2SO_4

Apparatus : 250 ml beakers, glass rod, measuring cylinder, etc.

Procedure :

1. Weigh 5 g of copper sulphate and dissolve in 20 ml distilled water (add 1-2 drops of dil. H_2SO_4 if necessary).
2. Add liquor ammonia (1 T.T.) with constant stirring, until the bluish precipitate of $Cu(OH)_2$ formed first, completely dissolves to give a clear deep blue solution and there is a distinct smell of ammonia over the beaker.
3. Cool the beaker in water bath.
4. Add ethanol slowly from a common burette with constant stirring until the blue colour is nearly discharged (about 50 ml).
5. Heat the beaker carefully on water bath at about 70^0C for 15 minutes by keeping watch glass on beaker.
6. Allow it to stand and cool on an asbestos sheet to room temperature.
7. Filter the deep purple coloured crystals on Buchner funnel, wash with little alcohol and dry in oven and weigh the product.

Observations :-	**Structure :**
i) Copper sulphate : 5 g ii) Liquor ammonia : 1 T.T iii) Ethyl alcohol : 50 ml	 $SO_4^{2-}.H_2O$

Reaction:

$$CuSO_4 + 4\,NH_3 + H_2O \xrightarrow{\text{Ethyl alcohol}} [Cu(NH_3)_4]SO_4.H_2O$$

Calculations:

a) Theoretical yield	**b) Practical % yield of product**
From chemical reaction , We have : $CuSO_4{\cdot}5H_2O$ = $[Cu(NH_3)_4]SO_4{\cdot}H_2O$ 249.68 245.74 ∴ 249.68 g Copper Sulphate = 245.74 g complex ∴ 5g Copper Sulphate = $\frac{5 \times 245.74}{249.68}$ g complex . = 4.92 g complex ∴ Theoretical yield of Complex (A) = 4.92 g	Weight of the product = X = …….g ∴ 4.92 g of Complex = 100 % yield ∴ X g Complex = $\frac{X \times 100}{4.92}$ % i.e. B =……. %

Results:

1.	Colour of the product	Deep purple	
2.	Weight of the product	X = …… g	=…….. x 10^{-3}Kg
3.	Theoretical yield of product	A =…… . g	=…….. x 10^{-3}Kg
4.	Practical % yield of product	B =…….%	

19. Preparation of Chlorpentamminecobalt(III) chloride

Aim **:** To Prepare chloropentaamminecobalt (III) chloride. $[Co(NH_3)_5Cl]Cl_2$

Chemicals :1) Cobaltous chloride, 2) Ammonium chloride , 3) Ammonia.
4) Hydrogen peroxide (20 Volume) 5) Hydrochloric acid.

Procedure :

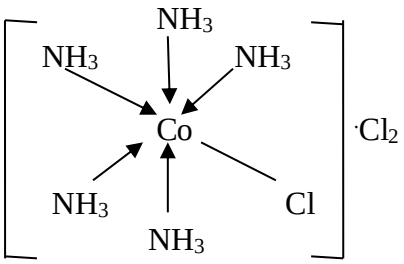
i) Take 5 g cobaltous chloride in a 250 ml beaker and dissolve it in minimum distilled water. ii) Weigh 10 g ammonium chloride and dissolve in 40 ml liquor ammonia. iii) Add this ammonical ammonium chloride solution to the cobaltous chloride solution with constant stirring. Cool the solution in a water bath. iv) Add by a burette 2 ml H_2O_2 at a time with constant stirring until the addition of 25 ml H_2O_2 is complete v) Continue the stirring of solution till the effervescence O_2 ceases (it take about 15 minutes.) vi)Then neutralize this solution with Conc. HCl. (Test it by litmus paper, both blue and red litmus should remain unaffected.) vii) Then add 10 ml conc. HCl in excess (Blue litmus paper should turn red). viii) Heat the solution gently to boiling. ix) Allow it to cool to room temperature when purple coloured crystals separate out. x) Filter the product and wash with alcohol. Dry and record the yield.

Observation:

i)	Cobaltous chloride	=	5.0 g
ii)	Ammonium chloride	=	10.0 g
iii)	Ammonia	=	40 ml
iv)	H_2O_2	=	25 ml
v)	Con. HCl	=	….. ml.

Reactions: i) $H_2O_2 \longrightarrow H_2O + (O)$

ii) $2CoCl_2 + 2NH_4Cl + [O] \longrightarrow 2CoCl_3 + 2NH_3 + H_2O$

iii) $CoCl_3 + 5NH_3 \longrightarrow$ 

Calculations :

1) Theoretical yield

From the chemical reaction we get,

$CoCl_2.6H_2O$ = $[Co(NH_3)_5Cl]Cl_2$.

237. 93 = 250. 43

Now, 237.93 g $CoCl_2.6H_2O$ = 250.43 g of complex

∴ 5.g $CoCl_2.6H_2O$ = $\frac{5 \times 250.43}{237.93}$ g of complex

i.e. A = 5.269 g of the complex

2) Practical percentage yield

Wight of Product = X = …..g

Now, 5.26 g of complex = 100%

∴ 'X' g of complex = $\frac{\text{'X' x 100 \%}}{5.26}$

i.e. B = ……. %

Result :

i)	Weight of product	= X = …….. g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

.20. Preparation of hexamminenickel (II) chloride

Aim : To Prepare of Hexaamminenickel(II) chloride. $[Ni(NH_3)_6]Cl_2$

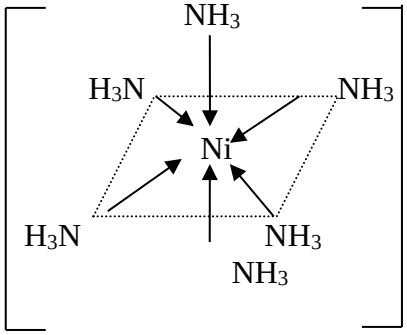
Chemicals : 1) Nickel Chloride 2) Ammonia Buffer (pH 10) 3) Ethyl alcohol

Procedure :

i) Weigh 5 g Nickel Chloride and dissolve it in 20 ml warm distilled water by constant stirring ii) keep it in a ice bath for about 30 min. iii) Add 30 ml ammonia buffer with constant stirring until the blue ppt of Nickel hydroxide first formed is dissolves iv) Cool the solution in ice bath.The crystals of Hexaamminenickel(II) chloride separates out v) Add 50 ml ethyl alcohol. Filter the product on Buckner funneland wash with little alcohol. v) Dry the product and weigh it on rough balance

Reactions:

$NiCl_2 + 6NH_3 \longrightarrow [Ni(NH_3)_6]Cl_2$



Observations:

i) Nickel Chloride = 5.0 g

ii) Ammonia Buffer (pH 10) = 30 ml

iii) Ethyl alcohol = 50 ml

Calculations :

1) Theoretical yield	2) Practical percentage yield
From the chemical reaction we get, $NiCl_2.6H_2O \equiv [Ni(NH_3)_6]Cl_2$ 337.7 ≡ 231.71 Now, 337.7 g $NiCl_2.6H_2O \equiv$ 231.71 g complex $\therefore$ 5 g $NiCl_2.6H_2O = \frac{5 \times 231.71}{337.7}$ g complex = 4.87 g of the complex i.e. A = 4.87 g complex	Weight of the product = X = …….g Now, 4.87 g of complex = 100% yield $\therefore$ 'X' g of complex = $\frac{'X' \times 100}{4.87}$ % i.e B = ……... %

Result :

i)	Weight of product	= X = …….. g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

Inorganic Qualitative Analysis

21. Semi –Micro Qualitative Analysis

Aim: Give the complete qualitative analysis of the mixture given in capsule mark A.

N. B.

1. Credit will be given for neat and systematic work and intelligent interpretation of observation.
2. Observation as soon as they are made should be entered in answer book.
3. Dry and Preliminary test must be done and examiner's signature must be obtained before preparing the solution.
4. Positive radicals must be confirmed by use of spot test wherever possible.

Analysis of Inorganic Binary Mixture is performed by following Tests:-

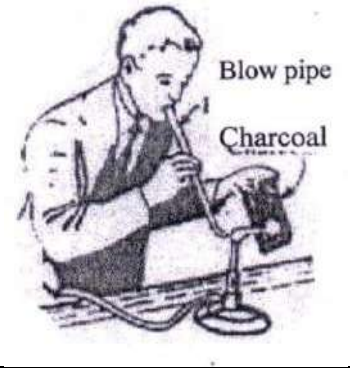
A] Preliminary Tests
B] Dry Test for Basic Radicals (Cations)
C] Dry Tests for Acidic Radicals (Anions)
D) Detection and Confirmation of Acidic Radicals
   i. Preparation of O.S.
   ii. Detection of Groups
   iii. Group Analysis

Inorganic Mixture No…….

Test	Observation	Inference
A] Preliminary Tests:		
1. Colour	1. Blue	Cu^{++} may be present
	2. Bluish green or Green	Cu^{++} , Cr^{+++} , Ni^{++} may be present
	3. Faint pink	Mn^{++} may be present
	4. Coloured	Cu^{++}, Fe^{+++} , Cr^{+++} , Ni^{++} may be present
	5. White	Zn^{++}, NH_4^+, K^+ may be present
2. Nature or Appearance	1. Crystalline	Water soluble salts generally containing Cl^-, Br^-, I^-, NO_3^-, SO_4^{--}, NH_4^+ and K^+ may be present
	2. Hygroscopic (moist)	Chlorides of Cu^{++}, Fe^{++}, Cr^{+++}, Zn^{++}, Mn^{++}, Mg^{++}etc may be present
	3. Amorphous	Water insoluble salts generally containing CO_3^{--} may be present

B] Dry Test for Basic Radicals (Cations):

1. Action of Heat : Heat small quantity of the Mixture in a clean and dry test tube at first gently and then strongly.	1. Substance decrepitates		Crystalline salts like $Pb(NO_3)_2$, KBr , K_2SO_4 etc. may be present
	2. Substance fuses and water vapours condense on the cooler part of the test tube.		Salts of Ba^{++} , Ca^{++} ,K^+ and nitrates of other radicals containing water may be present
	3. Brown fumes		NO_3^- , Br^- may be present
	4. Violate fumes		I^- may be present
	5. White sublimate		NH_4^+ and Cl^- may be present.
	6. Colour changes		
	When Hot	**When Cold**	
	Yellow	White	Zn^{++} may be present
	Brown	Yellow	Pb^{++} may be present
	Brown	Brown	Cd^{++} may be present

	Black	Reddish brown	Fe^{++} may be present
	Green	Green	Cr^{++} may be present
	Black	Black	Carbonates of Cu^{++}, Ni^{++}, Mn^{++} may be present
	7. Coloured infusible residue		Cu^{++},Fe^{+++}, Cr^{+++}, Ni^{++}, Mn^{++} may be present
	8. White infusible residue		Al^{+++}, Ba^{++}, Ca^{++}, Mg^{++} may be present
2. Charcoal Test : Heat small quantity of a mixture in a freshly prepared cavity on a charcoal.  	1. Decrepitation		Crystalline salts like $Pb(NO_3)_2$, KBr , K_2SO_4 etc may be present
	2. **Deflagration:**Charcoal glows due to supply of O_2 from a comp. in the mixture		NO_3^- may be present
	3.Fusion (Substance fuses and sinks in the cavity)		Salt of Ba^{++} , Ca^{++}, K + may be present
	4. Brown gas and brown encrustation		Cd^{++} may be present
	5. Coloured infusible residue		Cu^{++} , Fe^{+++}, Cr^{+++}, Ni^{++} Mn^{++} may be present
	6. White infusible residue		Al^{+++} , Ba^{++}, Ca^{++}, Zn^{++} , Mg^{++} may be present
3. Cobalt Nitrate Test : This test is to be performed only when a white infusible residue is obtained in the charcoal cavity.			
Moisten white residue with 2 drops of cobalt nitrate solution & heat on a charcoal.	1. Blue residue 2. Green residue 3. Grey residue		Al^{+++} may be present Zn^{++} may be present Ba^{++} may be present
4. Action of NaOH :			
Mixture + NaOH solution warm	Evaluation of NH_3 gas turing moist turmeric paper red / brown		NH_4^+ may be present
C] Dry Tests For Acidic Radicals (Anions):			
1.Action of H_2SO_4 Mixture + dil H_2SO_4 (heat if necessary)	1. Brisk effervescence of CO_2 gas turning fresh lime water milky. (Collect these fumes in a second dry test tube by holding it on the mouth of first test tube + lime water)		CO_3^{--} may be present

2. Action of conc. H_2SO_4 Mixture + conc. H_2SO_4 (warm carefully)	1. Reddish brown fumes of NO_2 or Br_2 evolved (Note: Generally Br_2 is immediately evolved in cold while NO_2 is evolved on heating)	Br^- or NO_2^- present
	2. Violet fumes of I_2 evolved turning starch paper blue.	I^- present
	3. Colourless pungent smelling gas (HCl) giving white fumes when a rod dipped in NH_4OH solution is brought near the mouth of the test tube.	Cl^- present
	4. No evaluation of coloured or colourless fumes.	Cl^-, Br^-, I^- & NO_3^- absent
3 **<u>Action of MnO_2 &</u> conc. H_2SO_4** Mix + MnO_2 + conc. H_2SO_4 (warm carefully)	1. Reddish brown fumes	Br^- Present
	2. Violet vapors turning moist starch paper blue- black	I^- Present
	3. Pungent smelling yellowish green Cl_2 gas turning moist blue litmus paper first red and then white**(i.e. bleaching action)**	Cl^- Present

D) Detection and Confirmation of Acidic Radicals:

For convenience acidic radicals (anions) are classified in following four groups:

Group I : CO_3^{--}
Group II : NO_3^-
Group III : Cl^-, Br^-, I^-
Group IV : SO_4^{--}

Test	Observation	Inference
Group I : CO_3^{--}		
1. Mix. + dil. H_2SO_4 warm if necessary	Brisk effervescence of CO_2 turning lime water milky. (Collect these fumes in a second dry test tube by holding it on the mouth of first test tube + lime water).	CO_3^{--} present
C.T. for CO_3^{--} 1. Mix + dil. HCl warm if necessary 2. Mix + $K_2Cr_2O_7$ + dil. H_2SO_4	Effervescence of CO_2 gas turning lime water milky. Effervescence of CO_2 gas turning lime water milky	CO_3^{--} present CO_3^{--} present

Preparation of Water Extract:-

100 mg Mixture in T.T. + half T.T. distilled water – Boil for few minutes and filter. Filtrate is uses as **<u>water extract</u>** for further tests.

Preparation of Sodium carbonate extract (Na_2CO_3 extract):

100 mg mixture + 200 mg anhydrous sodium carbonate + half T.T. distilled water. Boil for minutes and filter. Filtrate is used as '**Sodium carbonate extract**'.

Group II : NO_3^-		
10 mg mixture + 2-3 Pieces of Cu fillings + 5 drops of conc. H_2SO_4. Heat on water bath	Evaluation of pale brown gas seen directly from mouth of the test tube	NO_3^- Present
C.T. for NO_3^- 5 drops of Na_2CO_3 extract + few drops of Conc. H_2SO_4 cool well and from the side of the test tube add carefully with the dropper freshly prepared $FeSO_4$ solution	Brown ring is formed at the junction of two layers	NO_3^- Confirmed
Group III: Cl^- , Br^- , I^-		
1. <u>Action of MnO_2 & H_2SO_4</u> Mix. + MnO_2 + conc. H_2SO_4 (warm carefully)	1.Pungent smelling colourless gas giving white fumes when a rod dipped in NH_4OH solution is brought near the mouth of the test tube.	Cl^- present
	2. Brownish red vapors turning moist starch paper yellow	Br^- present
	3. Violet vapors turning moist starch paper blue-black.	I^- present
2. Confirmatory Tests Water extract + Fresh Chlorine water + $CHCl_3$ shake well and observe colour of lower layer	1. Pink or violet $CHCl_3$ layer 2. Brown or yellowish brown $CHCl_3$ layer 3. Both aqueous and $CHCl_3$ layer remains colourless	I^- present & confirmed Br^- present & confirmed Cl^- confirmed

Distinction between Br- & NO_3:

Use two dry test tubes for this test. In first test tube take mixture + MnO_2 + Conc. H_2SO_4, warm carefully, evaluation of red brown fumes. Collect these fumes in a second dry test tube by holding it on the mouth of first test tube. When sufficient fumes get collected in the second test tube, take it away and add about 1 ml distilled water and shake well.

If distilled water becomes **yellow in colour, it indicates the presence of Br**. If it remains colourless, it indicates presence of NO_3^-.

Group IV : SO_4^{--}		
Na_2CO_3 extract + dil. HCl + $BaCl_2$ solution	White ppt. insoluble in conc. HCl	SO_4^{--} present and confirmed

Given mixture contains Acidic Radicals: 1. ……
2. …….

E) Detection of Basic Radicals:

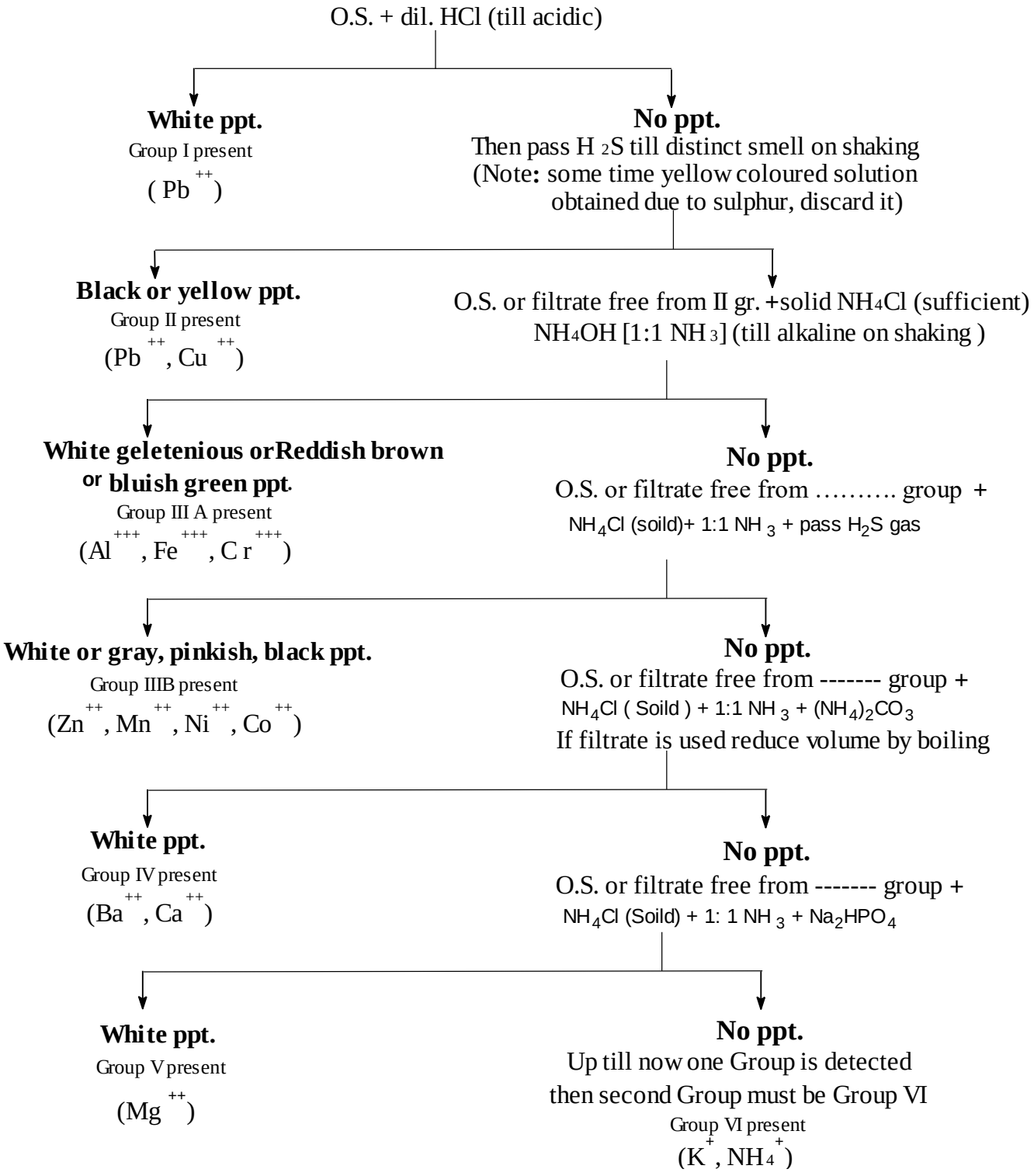
i) Preparation of Original Solution (O.S.):

Try the solubility of small quantity of the substance in the following solvent , first in cold then on warming and then on boiling i) water ii) dil. HCl iii) Conc. HCl
Thus prepare one test tube O.S. from about 1 g of mixture (filter if necessary).

∴ **O.S. prepared in ……**

Note: If mixture is soluble in conc. HCl dilute it with water carefully.

ii) Detection of Groups:



∴ **Basic radicals of the mixture belong to …… and ……. groups.**

iii) Group Analysis
(Separation, Detection and Confirmation of Basic Radicals)

Analysis of Group II : Cd^{++} & Cu^{++}

Test	Observation	Inference
1. Observe colour of ppt	1. Yellow 2. Black	Cd^{++} present Cu^{++} present
2. Black ppt. of group II + 1:4 HNO_3 and boil	ppt. dissolves	Say this solution (a)
3. Solution (a) + NaOH or NH_4OH	Bluish ppt. soluble in excess NH_4OH forming a deep blue solution	Cu^{++} present
C.T. For Cd^{++} :		
1. Yellow ppt. of Cadmium + dil. HNO_3 heat with Stirring in water bath.	ppt. dissolves	Solution (b)
2. Solution (b) + $K_2Cr_2O_7$	Yellowish ppt	Cd $^{++}$confirmed
3. **Spot Test :** Solution (b) + NH_4CNS till sol^n become colourless + crystals of KI + 2 drop of diphenyl carbazide.	Violet coloration	Cd^{++} confirmed
C.T. for Cu^{++} :		
Solution (a) + dil. acetic acid + $K_4[Fe(CN)_6]$ solution	Reddish brown or chocolate red ppt.	Cu $^{++}$confirmed
Spot Test : 1. Drop of solution (a) + drop of rubeanic acid & expose to ammonia	Olive green colour	Cu $^{++}$confirmed
2. One drop of sol^n (a) or O.S. on filter paper + one drop cupron and expose to ammonia	Green colour	Cu $^{++}$confirmed

Analysis of Group III A: Al^{+++} & Fe^{+++} & Cr^{+++}

Test	Observation	Inference
1. Observe colour of ppt.	1. Reddish brown 2. Bluish green 3. White gelatinous	Fe^{+++} present Cr $^{+++}$ present Al^{+++} present
2. ppt. of IIIA + dil. HCl	ppt. dissolves	Solution (a)
C.T. or Fe^{+++} :		

Solution (a) + KCNS or NH_4CNS	Blood red ppt.	Fe^{+++}confirmed
Spot test : One drop of solution (a) in a spot plate + $K_4[Fe(CN)_6]$ solution	Deep blue ppt.	Fe^{+++}confirmed
C.T. for Cr^{+++} :		
Solution (a) + dil. acetic acid till acidic + drop of lead acetate	Yellow ppt.	Cr^{+++}confirmed
Spot test : Two drops of solution (a) on spot plate + 2 drops of 1:1 H_2SO_4 + 2 drops of diphenyl carbozide	Violet colour	Cr^{+++} confirmed
C.T. for Al^{+++} :		
Solution (a) + NaOH	White gelatinous ppt. soluble in excess NaOH	Al^{+++} confirmed
Spot test : 1. Drop of solution (a) on filter paper + drop of alizarin & expose to NH_3.	Violet colour	Al^{+++} confirmed
2. Two drops of solution (a) or O.S. on spot plate + drop of dil. HCl + 4 drops of ammonium acetate + 3 drops of Aluminon wait for 5 min.+ excess of ammonical $(NH_4)_2CO_3$	Red colour or ppt.	Al^{+++} confirmed

Analysis of Group III B: Zn^{++}, Mn^{++}, Ni^{++} & Co^{++}

TEST	OBSERVATION	INFERENCE
1. Observe colour of ppt.	1.White or gray insoluble in acetic acid	Zn^{++} present
	2. Pinkish or flesh ppt. soluble in acetic acid. Say this **solution (a)**	Mn^{++} present
	3. Black ppt. insoluble in dil. HCl	Ni^{++} / Co^{++} present
i) White or Grey ppt. of III B + conc. HCl boil	White or Grey ppt. dissolves	Solution (b)
ii) Black ppt. of Ni^{+++} / Co^{++} + (HCl + HNO_3. 1:4) warm	Black ppt. dissolves	Solution (c)
	<u>Observe colour of solution (c)</u> i. Pink solution ii. Green solution	 Co^{++} present Ni^{++} present
C.T. for Zn^{++} :		
1.Solution (b) + $K_4[Fe(CN)_6]$	White / bluish white ppt. insoluble in dil. HCl	Zn^{++} confirmed

2. Solution (b) + NaOH	Bluish white ppt. insoluble in excess& reappears by H_2S gas	Zn^{++} confirmed
Spot Test : 1. Solution (b) or a drop of O.S. in spot plate + a drop of copper acetate + 3-4 drops of ammonium mercuric thiocynate and rub with glass rod	Immediate blue ppt. or Violet colour	Zn^{++} confirmed
2. Solution (b) or a drop of O.S. in spot plate + a drop of potassium ferricyanide + a drop of diphenylamine in glacial acetic acid.	Green ppt.	Zn^{++} confirmed
C .T. for Mn^{++} :		
1. Solution (a) + NaOH	White ppt. soluble in excess but turns brown on expose to air	Mn^{++} confirmed
Spot Test : 1. Drop of Solution (b) on spot plate + solid sodium bismuthate. Stir well	Purple or Violet colour	Mn^{++} confirmed
2. Sol^n (a) or drop of O.S. on filter paper + a drop of NaOH + a drop of Sodium tartrate + a drop of Benzidine	Blue colour (fades on standing)	Mn^{++} confirmed
C.T. for Co^{++} Drop of Solution (c) on filter paper + drop of dil. HCl + one drop of α-nitroso β- naphthol	Red brown colour	Co^{++} confirmed
C.T. for Ni^{++}:		
Solution (c) + NaOH	Pale green ppt.	Ni^{++} confirmed
Spot Test : 1. Drop of Solution (c) on filter paper + drop of Dimethyl glyoxime and expose to ammonia	Red / pink colour	Ni^{++} confirmed
2. Drop of Solution (c) on filter paper + Rubeanic acid and expose to ammonia	Blue colour	Ni^{++} confirmed

Analysis of IV Group: Ba^{++} & Ca^{++}

Test	Observation	Inference
i) White ppt.	Dissolve the white ppt. of IV group in dil. acetic acid by warming	say this Solution (a)

ii) Solution (a) + $CaSO_4$ solution	1. White ppt. immediately obtained	Ba^{++} present
	2. No ppt. or turbidity even on warming the solution	Ca^{++} present
C.T. for Ba^{++} :		
1. Solution (a) + K_2CrO_4 i.e. Potassium chromate	Yellow ppt. insoluble in acetic acid	Ba^{++} confirmed
2. Prepare paste of mixture in conc. HCl and take flame test	Green flame	Ba^{++} confirmed
C.T. for Ca^{++} :		
Solution (a) + Ammonium oxalate solution	White ppt. insoluble in acetic acid	Ca^{++} confirmed
Spot Test : Drop of solution (a) on filter paper + drop of NH_4OH + drop of sodium rhodizonate + 2 drops of NaOH solution	Violet colour	Ca^{++} confirmed

Analysis of Group V: Mg^{++}

Test	Observation	Inference
1. Colour of ppt.	White	Mg^{++} present
2. ppt. of group V + dil. HCl	Soluble	Solution (a)
C.T. for Mg^{++} :		
Solution (a) + Sodium hypoiodide solution (add iodine solution to NaOH drop by till yellow colour is just obtained)	Reddish brown ppt.	Mg^{++} confirmed
Spot Test : 1. Solution (a) or 5drops O.S. on spot plate + Mangeson–I. Stir with glass rod and add NaOH till alkaline.	Blue ppt. or colour	Mg^{++} confirmed
2. Solution (a) in spot plate + 2 drop of titan yellow + 4 drop of NaOH	Red ppt. or colour	Mg^{++} confirmed

Analysis of Groups VI: K^+ & NH_4^+

Note: Use water extract (W. E) for detection

Test	Observation	Inference
W.E. + NaOH solution and heat	1. Evolution of NH_3 turning moist Turmeric paper red	NH_4^+ is present
	2. Evolution of NH_3 gives white fumes to HCl rod when kept near mouth of T.T	NH_4^+ present
	3. No evolution of NH_3	K^+ is present
C.T. for NH_4^+ :		
W.E. + Nessler 's reagent	Brown ppt.	NH_4^+ confirmed
Test for K^+ : 1. Water extract + picric acid solution and rub with glass rod. 2. Water extract + perchloric acid ($HClO_4$) solution. 3. Flame Test: - Prepare paste of mixture in evaporating dish with conc. HCl + ethanol, take flame test.	 Crystalline yellow ppt. Crystalline white ppt. Violet flame by naked eye and crimson red seen through cobalt glass.	 K^+ Present and confirmed K^+ confirmed K^+ confirmed.

Preparation of Nessler's reagent:

Few drops of $HgCl_2$ + Add drop by drop KI solution till scarlet red ppt. formed which just redissolved + equal volume of dil. NaOH.

Results: The given mixture contains:

Positive Radicals / Basic Radicals	Negative Radicals / Acidic Radicals
1)	1)
2)	2)

……

C. ORGANIC CHEMISTRY

a) Organic Estimations

22. Estimation of acetone

Aim : To estimate the amount of acetone in the given solution by iodometric method.

Apparatus: Burette, pipette, conical flask, 100 ml measuring flask, beakers etc.

Chemicals: Acetone solution (Given in 100 ml measuring flask), 0.1 N (approx.) iodine solution, 0.1 N $Na_2S_2O_3$ solution, 1N KOH solution, 2N HCl, fresh starch solution

Procedure:

Part-I: Back Titration

1. Dilute the given solution of acetone in 100 ml measuring flask up to mark carefully, with distilled water and shake well.
2. Pipette out 10 ml of this diluted solution in a stopper bottle. Add 25 ml 1 N KOH solution by common burette and 25 ml 0.1 N (approx.) iodine solution by pipette.
3. Shake well and keep it for 15 minutes (shake the flask occasionally).
4. Add 25 ml, 2N HCl solution (2 T.T.) by measuring cylinder (to liberate free I_2).
5. Shake well and titrate liberated I_2 immediately against 0.1N $Na_2S_2O_3$ solution by using starch (1 ml) as an indicator.
6. The end point is blue to colourless (white precipitate). Take three accurate readings. Say the C.B.R. as Y ml.

Part-II: Blank Titration: Standardization of Iodine

1. Pipette out 25 ml of 0.1 N (approx.) Iodine solution in a conical flask.
2. Titrate this against 0.1N $Na_2S_2O_3$ solution by using Starch (1 ml) as an indicator.
3. The end point is blue to colourless. Take three accurate readings Say the C.B.R. as X ml.

Note: Add starch when solution becomes pale yellow (i.e. near the end point)

Reactions :

1. $6KOH + 3I_2 \rightarrow 3KI + KOI + 3H_2O$.
2. $CH_3COCH_3 + 3KOI \rightarrow CH_3COCI_3 + 3KOH$
3. $CH_3COCI_3 + KOH \rightarrow CH_3COOK + CHI_3$
4. $KI + KOI$ (unused) $+ 2HCl \rightarrow 2KCI + H_2O + I_2$
5. I_2 (excess) $+ 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

Observations and Observation table:

Part-I: Back Titration

Observations:

1. In Burette **:** 0.1N $Na_2S_2O_3$ sol^n
2. By pipette **:** 10 ml acetone sol^n + 25 ml iodine sol^n + 25 ml KOH sol^n
3. Indicator **:** Starch (1 ml)
4. End point **:** Blue to Colourless

Observation Table :

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				Y= …ml
Initial Level				
Difference				

Part-II: Blank Titration: Standardization of Iodine

Observations:

1. In Burette : 0.1N $Na_2S_2O_3$ sol^n
2. By pipette : 25 ml iodine sol^n
3. Indicator : Starch (1 ml)
4. End point : Blue to Colourless

Observation Table:

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				X= …ml
Initial Level				
Difference				

Calculations : 1. Back Titration Reading = Y = ------- ml

2. Blank Titration Reading = X = ------- ml

3. X – Y = Z = -------- ml

1. Amount of I_2 consumed by the given Acetone solution

i.e. Z ml is the amount of I_2, in terms of $Na_2S_2O_3$ consumed by 10 ml dil. acetone sol^n

∴ 100 ml diluted or for given solution requires = Z x 10 ml = …… ml acetone sol^n

2. Amount of acetone in the given solution

From chemical reactions we get

$6KOH \equiv 3I_2 \equiv 3KOI \equiv CH_3COCH_3$

i.e . $3I_2 \equiv 6I \equiv 6M\ Na_2S_2O_3 \equiv 1M$ acetone $\equiv 58$ g acetone

i.e. 6 KOH $\equiv$ 58 g acetone

∴ 6000 ml 1 N $Na_2S_2O_3 \equiv$ 58 g acetone

i.e. 60,000ml 0.1 N $Na_2S_2O_3 \equiv$ 58 g of acetone

∴ Z ml of 0.1 N $Na_2S_2O_3$ = $\frac{58 \times Z}{60,000}$ g of acetone

i.e. A = ------ g of acetone present in 10 ml of diluted solution

∴ Amount of acetone present in 100 ml in given solution = A x 10 = -------- g

i.e = B = ----------g

Results:

1. Amount of I_2 consumed in terms of 0.1 N NaS_2O_3 solution by the given acetone solution	= Z x 10	=……. ml
2. Amount of acetone in the given solution	= B =……. g	= -------- x 10^{-3} kg

23. Estimation of Ester (Ethyl benzoate)

Aim : To estimate the amount of ethyl benzoate in the given solution.

Apparatus : Burette, pipette , conical flask , 250 ml measuring flask , round / flat bottom flask (R. B. flask), water condenser etc

Chemicals : Ethyl benzoate solution (Given in flat bottom flask), 0.1 N HCl, 1N (approx.) KOH solution, phenolphthalein indicator

Procedure :

Part-I: Back Titration

1. To the given solution of ethyl benzoate in R.B. flask, add carefully 25 ml KOH solution by common burette.
2. Add 2-3 pieces of porcelain.
3. Heat the reaction mixture on water bath using **water condenser** for one and half hour.
4. Cool the reaction mixture and transfer the contents to 250 ml flask with washing and dilute it up to mark with distilled water and shake well.
5. Pipette out 25 ml of this diluted solution and titrate it against 0.1 N HCl solution using phenolphthalein as an indicator.
6. The end point is pink to colourless. Take three accurate readings. Find out C.B.R. & say it as Y ml.

Part-II: Blank Titration

1. From burette, take 25 ml of 1 N (approx.) KOH solution in a volumetric flask.
2. Dilute it up to mark with distilled water and shake well.
3. Titrate 25 ml of this diluted KOH solution against 0.1 N HCl solution using Phenolphthalein as an indicator. 4) The end point is pink to colourless. Take three accurate readings. Say the C.B.R. as X ml.

Reactions:

1. $C_6H_5COOC_2H_5 + KOH \xrightarrow{\text{Hydrolysis}} C_6H_5COOK + C_2H_5OH$
2. $KOH + HCl \xrightarrow{\text{Neutralization}} KCl + H_2O$

Observations & Observation table:

Part-I (Back Titration)

Observations:

1. In Burette **:** 0.1N HCl solution
2. By pipette **:** 25 ml dilute reaction mixture
3. Indicator **:** Phenolphthalein
4. End point **:** Pink to colourless

Observation Table :

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				Y= -- ml
Initial Level				
Difference				

Part-II (Blank Titration)

Observations:

1. In Burette : 0.1N HCl solution
2. By pipette : 25 ml dil. KOH sol^n
3. Indicator : Phenolphthalein
4. End point : :Pink to Colourless

Observation Table :

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				X= -- ml
Initial Level				
Difference				

Calculations: 1. Blank Titration Reading = X = ---------- ml

2. Back Titration Reading = Y = ---------- ml

3. X-Y = Z = ---------- ml

1. Amout of KOH solution consumed during the hydrolysis of ethyl benzoate in the given solution:-

i.e. Z ml is the amount of KOH consumed, in terms of 0.1 N HCl, during hydrolysis of ethyl benzoate for 25 ml dilute sol^{n}.

$\therefore$ For 250 ml diluted solution or for given solution = Z x 10 ml

From chemical reaction we know

1mole of KOH Solution $\equiv$ 1 mole of ethyl benzoate

i.e. 1000 ml of KOH Solution $\equiv$ 150 g of ethyl benzoate

1000 ml of 1 N KOH solution $\equiv$ 150 g of ethyl benzoate

i.e. 10,000 ml of 0.1 N KOH Solution $\equiv$ 150 g of ethyl benzoate

Now 10,000 ml 0.1 N HCl Solution $\equiv$ 150 g of ethyl benzoate

$\therefore$ Z ml of .0.1 N HCl Solution $\equiv$ $\frac{150 \times Z}{10,000}$ g of ethyl benzoate

i.e. A $\equiv$ -------- g of ethyl benzoate present in 25 ml of diluted solution .

$\therefore$ Amount of ethyl benzoate present in 250 ml i.e. in the given sol^{n} = A x 10 g

= -------- g

i.e. = B = --------g

Results:

1. Volume of 0.1 N HCl equivalents to KOH solution consumed during the hydrolysis of ethyl benzoate in the given solution.	= Z x 10	=…… ml
2. Amount of ethyl benzoate in the given solution	= B =……. g	= ……x 10^{-3} kg

24. Estimation of Ibuprofen

Aim : To determine the amount of ibuprofen from given ibuprofen tablet.

Apparatus : Burette, pipette, conical flask, 250 ml measuring flask, 100 ml measuring flask, beakers, watch glass etc.

Chemicals : Given ibuprofen tablet, oxalic acid (solid), 0.1N (approx.) NaOH Solution, chloroform, phenolphthalein indicator etc.

Procedure :

Part I: Preparation of standard solution of oxalic acid

1. Weigh accurately 1.575 g of oxalic acid on a watch glass or glazed paper and transfer it in a beaker.
2. Dissolve it in minimum distilled water and transfer this solution to 250 ml measuring flask. Dilute the contents up to the mark with distilled water and shake well.

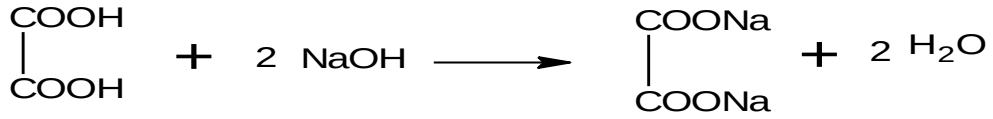
Part II: Standardization of NaOH solution

1. Fill the burette with 0.1N (approx.) NaOH solution.
2. Pipette out 25 ml 0.1N oxalic acid solution in conical flask and add phenolphthalein indicator. Titrate with NaOH solution till colour changes from colorless to faint pink. Say the C.B.R. as 'a' ml.
3. Calculate the normality of NaOH solution. Let the normality be 'Z' N.

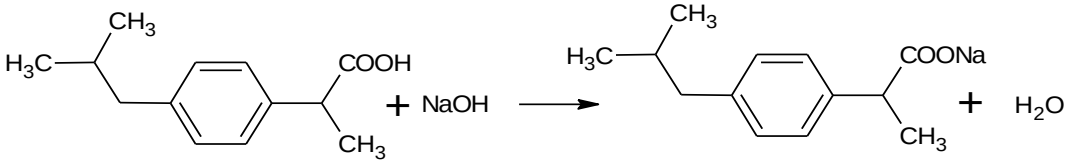
Part III: **Estimation of Ibuprofen**

1. Powder the given ibuprofen tablet and weigh accurately but not exactly 0.8 to 1 gm on a clean watch glass.
2. Transfer it to conical flask and extract it with 30 ml chloroform and filter it using Whatman paper no. 1.
3. Unfiltered residues of tablet are again extracted with 15 ml chloroform two times and finally wash the residue with 15 ml of chloroform. Collect the filtrate and washing into 250 m l conical flask and evaporate the filtrate to dryness by gentle heating and further it is allow to air dry.
4. Dissolve the residue in ethyl alcohol, transfer it carefully in 100 ml measuring flask and dilute up to mark with ethyl alcohol.
5. Titrate 25 ml of this diluted solution against Z N NaOH solution using phenolphthalein as an indicator.
6. The end point is colourless to pink. Say the C.B.R. as 'b' ml.

Reaction:



oxalic acid — sodium salt of oxalic acid



Ibuprofen — Sodium salt of Ibuprofen

<u>Observations & Observation table:</u>

Part I: Preparation of standard solution of oxalic acid

Observations:

1.Weight of empty watch glass	M_1	= ---------- g
2.Weight of oxalic acid	M	= ---------- g
3. Weight of watch glass + oxalic acid	$M_2 = M_1 + M$	=……g

Part II: Standardization of NaOH solution

Part III: Estimation of Ibuprofen

Observations I:

1. Weight of empty watch glass	W_1	= ----------g
2. Weight of empty watch glass + ibuprofen tablet	W_2	= ---------- g
3. Weight of ibuprofen tablet	$W = W_2 - W_1$	= ……g

Observations II:

1. In Burette : Z N NaOH solution
2. By pipette : 25 ml diluted ibuprofen tablet solution
3. Indicator : Phenolphthalein
4. End point : Colourless to Pink

Observation Table :

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				Y=-- ml
Initial Level				
Difference				

Calculations:

1. Normaility of NaOH

Oxalic acid NaOH

$N_1V_1 = N_2V_2$

$0.1 \times 25 = N_2 \times X$

Observations:

1. In Burette : NaOH solution
2. By pipette : 25 ml 0.1N Oxalic acid solution
3. Indicator : Phenolphthalein
4. End point : Colourless to Pink

Observation Table :

Burette Level	Burette Reading in ml			C.B.R.
	I	II	III	
Final Level				X=--- ml
Initial Level				
Difference				

$$N_2 = \frac{0.1 \times 25}{X}$$

i.e. N_2 = Z = --------- N

Thus exact normality of NaOH = Z = -------- N

2. Amount of ibuprofen in given tablet

From chemical reaction we know

1 Mole of NaOH solution ≡ 1 mole of ibuprofen

1000 ml of 1M ≡ 206 g

∴ Y ml of Z N NaOH Solution ≡ $\frac{Y \times Z \times 206}{1{,}000 \times 1}$ g of ibuprofen

i.e A ≡ ---- g of ibuprofen

Now 25 ml of diluted ibuprofen tablet = A g of ibuprofen

∴ 100 ml of ibuprofen tablet solution contain = A x 4 = ----- g of ibuprofen

i.e. B = ------- g of ibuprofen

2. Percentage of ibuprofen in given tablet:

Weight of ibuprofen tablet = W = --------- g

$\therefore$ W g of ibuprofen tablet = B g ibuprofen

$\therefore$ 100 g of ibuprofen tablet = $\frac{100 \times B}{W}$ % ibuprofen

i.e. C = ---------- % ibuprofen

i.e. Percentage of ibuprofen in given tablet C = ----------% ibuprofen

Results:

1. Exact normality of NaOH solution	= Z =……. N	
2. Amount of ibuprofen in the given tablet	= B =……. g	= ------- x 10^{-3} kg
3. Percentage of ibuprofen in given tablet	= C =……..%	

b) Organic Preparations

25. Preparation of m-Dinitrobenzene

Aim : Preparation of m-dinitrobenzene from given amount of nitrobenzene

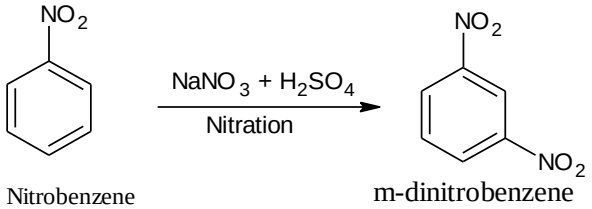
Apparatus : Flat bottom flask, air condenser, water bath, beakers, measuring cylinder

Chemicals : Sodium nitrate (powder), conc. H_2SO_4, nitrobenzene (given), ethyl alcohol

Procedure :

1. Weigh 8 g sodium nitrate (powder) and place in a clean and dry flat bottom flask.
2. Add to it 13 ml conc. H_2SO_4 in a small portions with constant stirring & cool the flask and keep it in water bath.
3. Add given nitrobenzene (4/5/6 ml) to the reaction mixture in a small portion with constant stirring.
4. Fit up air condenser and heat the flask in a boiling water bath for one hour.
5. After completion of reaction, pour the reaction mixture on a crushed ice in a 400 ml beaker with constant stirring, wash the flask one or two times with cold water and transfer reaction mixture completely to beaker. Here yellow solid separates out.
6. Filter the product on a buckner funnel & wash with cold water. Dry and weigh the product.
7. Recrystallise about 1 g of the product from ethyl alcohol. Dry and determine its M.P.
8. **Method of Recrystalisation:** In a big test tube, add about 1 g crude product and half test tube ethyl alcohol. Heat the test tube in a **boiling water bath** and dissolve the crude product by stirring with glass rod. Filter the hot dissolved product solution through a cotton plug in another test tube and cool it. Shining crystals of m-Dinitrobenzene separate out. Filter through ordinary filter paper and dry it.

Reaction:



Nitrobenzene m-dinitrobenzene

Result:

1. Weight of the crude product	= ---------- g	= ---------- x 10^{-3} kg
2. M.P. of recrystallized product	= ----------^{0}C	= ---------- K

26. Preparation of Phthalimide

Aim **:** Preparation of Phthalimide from given amount of phthalic anhydride

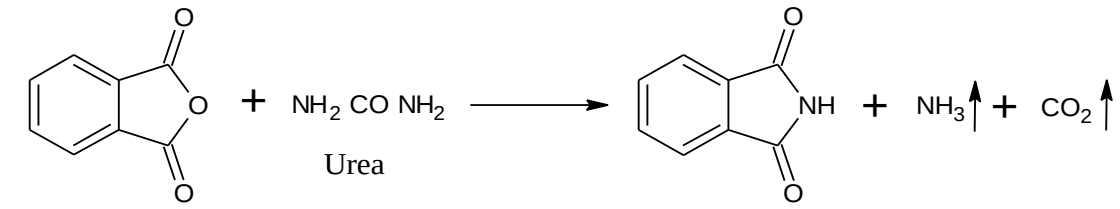
Apparatus : Conical flask, sand bath, beakers, measuring cylinder, evaporating dish etc.

Chemicals : Phthalic anhydride, urea, ethyl alcohol etc.

Procedure :

1. Take given amount of phthalic anhydride (4/6/8 gm) and 3 g urea separately. Mix these two compounds in a clean evaporating dish.
2. Transfer the mixture in a clean and dry conical flask. Place funnel on that flask & heat it on a sand bath at 130-135 ^{0}C carefully.
3. The reaction begins with frothing of the mass at temperature 160 ^{0}C.
4. When frothing subsides, stop heating and cool the contents at room temperature
5. Now add two T.T. water & stir the contents with glass rod. Here yellow solid separates out.
6. Filter the product on a Buckner funnel and wash with cold water. Dry and weigh the product.
7. Recrystalize about 1g of the product from ethyl alcohol. Dry and determine its M.P.
8. **Method of Recrystalisation** : In a big test tube, add about 1 g crude product and half test tube ethyl alcohol . Heat the test tube in a boiling water bath and dissolve the crude product by stirring with glass rod. Filter the hot dissolved product solution through a cotton plug in another test tube. Cool the test tube. Shining white crystals of phthalimide separate out. Filter through ordinary filter paper and dry it.

Reactions:



Phthalic anhydride Phthalamide

Results:

1.Weight of the crude product	= ---------- g	= ---------- x 10^{-3} kg
2. M.P. of recrystallized product	= ----------^{0}C	= ---------- K

27. Preparation of p-Bromoacetanilide

Aim : Preparation of p-bromoacetanilide from given amount of acetanilide.

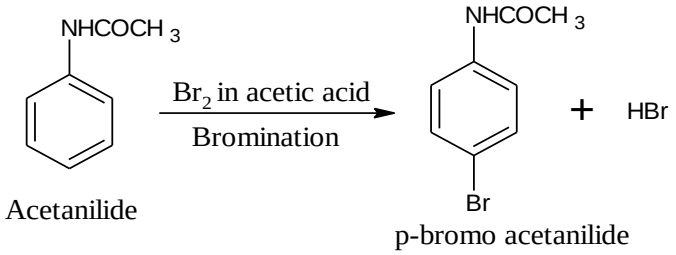
Apparatus : Conical flask, beakers, measuring cylinder etc.

Chemicals : Acetanilide (given), glacial acetic acid, bromine in acetic acid, ethyl alcohol etc.

Procedure :

1. Transfer the given acetanilide (4/6/8 gm) in conical flask and dissolve in 25 ml glacial acetic acid.
2. Take 20 ml Bromine in acetic acid in two T. T. and add it to reaction mixture drop wise slowly with constant stirring till slight orange color is obtained.
3. Place the glass funnel on the mouth of the flask and allow reaction mixture to stand at room temperature for 20 minutes.
4. Now pour the reaction mixture on crushed ice taken in beaker. White ppt. is obtained.
5. Filter the product on Buckner funnel and wash with cold water. Dry & weigh the product.
6. Recrystalise about 1g of the product from 50% ethyl alcohol. Dry & determine its M.P.
7. **Method of Recrystalisation :** In a big test tube, add about 1 g crude product and half test tube 50% ethyl alcohol. Heat the test tube in a **boiling water bath** and dissolve the crude product by stirring with glass rod. Filter the hot dissolved product solution through a cotton plug in another test tube. Cool the test tube. Yellow crystals of p-Bromoacetanilide separate out. Filter through filter paper and dry it.

Reactions:



Acetanilide p-bromo acetanilide

Results:

1. Weight of the crude product	= ---------- g	= --------- x 10^{-3} kg
2. M.P. of recrystallized product	= ----------^{0}C	= ---------- K

28. Preparation of Acetanilide

Aim : Preparation of acetanilide from given amount of aniline

Apparatus : Flat bottom flask, air condenser, measuring cylinder, sand bath, funnel etc

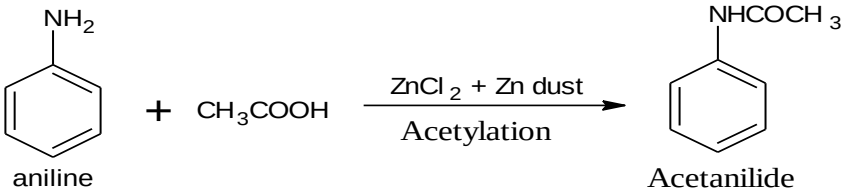
Chemicals : Aniline, glacial acetic acid, $ZnCl_2$ (anhydrous), Zn dust, ethyl alcohol

Procedure :

1. To the given volume of aniline (8 / 10 / 12 ml) in flat bottom flask or conical flask, add 10 ml glacial acetic acid, 1 g $ZnCl_2$ (anhydrous) and pinch of Zn dust.
2. Fit up air condenser to the flask and heat the flask on sand bath for two hours.

3. After completion of reaction, pour the reaction mixture on crushed ice taken in beaker with vigorous stirring with glass road. White precipitate of acetanilide is obtained.
4. Filter the product on a buckner funnel & wash with cold water. Dry and weigh the product.
5. Recrystalise about 2g of the product from hot water. Dry and determine its M.P.
6. **Method of Recrystalisation:** Place about 1 g crude product in a big test tube and add about 10 ml distilled water. Heat test tube on wire gauze carefully till most of the product dissolves in it. Filter this hot solution through cotton plug. Collect the filtrate in another test tube. Cool the filtrate in a water bath. White shining crystals of acetanilide separate out. Filter through filter paper and dry it.

Reactions:



Results:

1. Weight of the crude product	= ---------- g	= --------- x 10^{-3} kg
2. M.P. of recrystallized product	= ----------^{0}C	= ---------- K

Organic Qualitative Analysis

29 Identification of an Organic compound

Aim: To identify the organic compound, given in a container marked (A) bearing your table number.

Note: i) M.P. / B.P. of the compound should be detected before identification and the examiner's signature should be obtained .
ii) Wherever possible special tests should be shown to the examiner.
iii) Reactions for positive tests are expected.

Organic Compound No. ------

A) Physical Constant:

M.P. / B.P. of the Organic compound = ---------- ^{0}C = ---------- K

B) Preliminary test:

Sr. No.	Test	Observation	Inference
1	Colour	i) White solid	Phthalic acid, succinic acid, aspirin urea, acetalnilide, naphthalene, anthracene may be present.
		ii) Colourless liquid	CCl4, bromobenzene , methyl acetate, acetophenone, ethyl methyl ketone may be present
		iii) Dark brown solid	α- napthol may be present
		iv) Yellow coloured solid	o or p-nitrophenol, o/m/p-nitroaniline may be present .
		v) Yellow coloured liquid	Nitrobenzene may be present.
		vi) Yellow brown liquid	N, N–dimethyl aniline may be present
2	Odour	i) Pleasant	CCl_4, ethyl methyl ketone, acetophenone may be present
		ii) Pungent and irritating	Bromobenzene may be present
		iii) Fruity smell	Methyl acetate may be present
		iv) Fishy smell	N, N-dimethyl aniline may be present
		v) Odour of bitter almond	Nitrobenzene may be present
		vi) Moth ball like smell	Naphthalene may be present
		vii) Odourless	Sumlinic, Phthalic, salicylic acid, aspirin, o or p- nitrophenol, o/m/p-nitroaniline, anthracene etc. may be present
		viii) Phenolic	α- napthol may be present
3	Cu foil test: Heat small amount of compound on cu foil or on glass rod	i) Sooty flame	Compound is aromatic
		ii)Non –sooty flame	Compound is aliphatic
		iii) Green flame after the initial sooty flame has vanished	Compound containing halogen is present (exception –urea)
4	$KMnO_4$ Test : Org. compd + dil. $KMnO_4$ solution (faint violet coloured)	i) Decolourisation of $KMnO_4$ solution	Unsaturated compound is present
		ii) No Decolourisation of $KMnO_4$ solution	Saturated compound is present

C) Type of the Organic Compound:

Sr. No.	Test	Observation	Inference
1	Organic compound + water, shake well	i) Soluble	Water soluble compound is present

		i) Insoluble	Water insoluble compound is present

Type of the Water Soluble Organic compound

1	Org. comp .+ water, shake well	Compound dissolves completely	Original solution (O.S.) Use O.S. for further tests
2	Test O.S. with blue litmus paper	Blue litmus paper turns red	Water soluble acid is present
3	Test O.S. with red litmus paper	Red litmus paper turns blue	Water soluble base is present
4	O.S. neutral $FeCl_3$ solution	Violet colour	Water soluble phenol is present
5	All above tests are absent. Therefore compound is neutral.		

Type of the Water insoluble Organic compound

1	a) Org.comp.+$NaHCO_3$ sol^n shake well & filter	Effervescence of CO_2	Acid is present.
	b) Filtrate + 1:1 HCl	White ppt.	Acid is confirmed
2	a) Org. comp.+ 10% NaOH sol^n, shake well & filter	Compound dissolves	Phenol is present
	b) Filtrate + 1:1 HCl	White ppt. or yellow ppt.	Phenol is conformed
3	a)Org.comp.+1:1 HCl shake well and rub with glass rod and filter	Compound dissolves	Base is present
	b) filter + 30% NaOH solution	Yellow ppt.	Base is conformed
4	All above tests are absent. Therefore compound is neutral.		

Type of the organic compound is …….

D) Detection of Elements (Lassaigne's Test)

Take a dry piece of freshly cut sodium (Na) metal in a fusion tube. Heat the tube gently to melt the Na metal. Cool and add little amount of organic compound to it. For liquid compound use 5 ml pipette for dropping the liquid in fusion tube. Heat it first slowly and then strongly until tube is red hot. Drop it while red hot in about 15ml distilled water (about 1 t.t.) taken in an evaporating dish. Repeat the procedure 3 to 4 times.

Boil contents of evaporating dish for 5 minute (till half of the distilled water in evaporating dish evaporates) and filter through ordinary filter paper in a large test tube. This filtrate is known as sodium extract. Use this filtrate /extract for detection of elements.

Sr. No.	Test	Observation	Inference

<table>
<tr><td rowspan="2">1</td><td>Test for Nitrogen
1ml sodium extract + 2ml freshly prepared $FeSO_4$ Sol^n + 1 drop NaOH , boil well and cool then add excess conc. HCl</td><td>Blue ppt. or green colouration</td><td>Nitrogen (N) is present</td></tr>
<tr><td colspan="3">Reactions:
$Na + C + N \longrightarrow NaCN$
$2\,NaCN + FeSO_4 \longrightarrow Fe(CN)_2 + Na_2SO_4$
$Fe(CN)_2 + 4\,NaCN \longrightarrow Na_4[Fe(CN)_6]$
$3\,Na_4[Fe(CN)_6] + 4\,FeCl_3 \xrightarrow{HCl} Fe_4[Fe(CN)_6]_3\downarrow + 12\,NaCl$
Ferric ferrocyanide (prussian blue)</td></tr>
<tr><td rowspan="2">2</td><td>Test for sulphur
2ml sodium extract + 1drop NaOH + few drops of sodium nitroprusside sol^n</td><td>Purple or violet colour</td><td>Sulphur (S) is present</td></tr>
<tr><td colspan="3">Reactions:
$2\,Na + S \longrightarrow Na_2S$
Sodium sulphide
$Na_2S + Na_2[Fe(CN)_5\,NO] \longrightarrow Na_4[Fe(CN)_5NOS]$
Sodium nitroprusside — purple colour</td></tr>
<tr><td rowspan="4">3</td><td rowspan="2">Test for Halogens
(<u>Precaution</u> :Wash the T.T. with distilled water twice) 1 ml sodium extract + two drops of conc. HNO_3 boil and cool well + $AgNO_3$ solution</td><td rowspan="2">White or yellow ppt.</td><td rowspan="2">Halogen is present</td></tr>
<tr></tr>
<tr><td colspan="3">If above test is positive, carry out following test</td></tr>
<tr><td rowspan="2">1ml sodium extract + $CHCl_3$ or CCl_4 + chlorine water, shake well and observe colour of lower layer</td><td>i) colourless lower layer</td><td>Cl is present</td></tr>
<tr><td>ii) pale brown or yellow layer</td><td>Br is present</td></tr>
<tr><td colspan="3">Reactions :
$Na + X \longrightarrow NaX$ (X = Cl, Br or I)
Sodium halide
$NaX + AgNO_3 \longrightarrow AgX\downarrow + NaNO_3$
Silver halide</td></tr>
</table>

The given organic compound contains the C, H, (O) ---------- &------- elements.

E) Detection of functional group & Identification of Organic Compound

I) Acids having elements C, H & (O)

Sr. No.	Test	Observation	Inference
1	Org. comp. + saturated $NaHCO_3$ solution.	Slow effervescence of CO_2	Acid is present (-COOH)
	Reactions: $R - COOH + NaHCO_3 \longrightarrow R\text{-}COONa + H_2O + CO_2\uparrow$		
2	Org .comp. + distilled water, shake well	a) comp. is soluble	Succinic acid is present
		b) comp. is insoluble	Salicylic acid, phthalic acid, aspirin etc. is present

Preparation of neutral solution: In a 250 ml beaker take about 1 g of organic compound. Add to it about half T.T. dist. water, heat if necessary + 1 or 2 drops of phenolphthalein + dil . NaOH solution till pink colour just develops + dil. HCl till pink colour just disappears. Add slight excess of dilute ammonia solution and boil to remove the excess ammonia. Test with moist turmeric paper. Turmeric paper should not turn brown. If solution is faint pink coloured, add one drop of dil. HCl, it becomes colourless. Resulting solution is neutral solution.

Sr. No.	Test	Observation	Inference
3	Neutral solution + $FeCl_3$ solution	i) violet colour in cold disappearing by dil. HCl	Salicylic acid or aspirin is present
		ii) reddish brown or buff ppt. soluble in dil. HCl	Succinic acid or phthalic acid is present
4	Neutral solution + $CaCl_2$ solution	white ppt. on boiling , soluble in acetic acid	Succinic acid is present
	C.T. for Salicylic acid		
	Org. comp. + water shake well then add $FeCl_3$ solution	violet colour	Salicylic acid is confirmed
	C.T. for Aspirin (acetyl salicylic acid)		
	i) Org. comp.+ dil. HCl, boil then add neutral $FeCl_3$ solution	violet colour	aspirin is confirmed
	ii) Org. comp. + water shake well then add $FeCl_3$ solution	No violet colour	aspirin is confirmed
	C.T. for Succinic acid		
	i) Org .comp .+ water shake well + dil. HCl + neutral $FeCl_3$ solution	buff ppt.	Succinic acid is confirmed
	ii) In a dry T.T. take Org. comp. + double amount of resorcinol + 1ml conc. H_2SO_4, heat gently till mixture becomes red brown colour, cool well, pour it in water taken in a beaker. Then add excess of NaOH solution	Yellow green fluorescence	Succinic acid is confirmed

	C.T. for Phthalic acid		
	In a dry T.T. take Org .comp. + double amount of resorcinol +1ml conc. H_2SO_4, Heat gently till mixture attains red brown colour cool well .Pour it in water taken in a beaker. Then add excess of NaOH solution	Orange green fluorescence	Phthalic acid is confirmed

II) Phenols

a) Phenols having elements C, H & (O)

1	Org. comp .+ water & boil, then add $FeCl_3$ solution in excess	White ppt. slowly changes to violet	Phenol is present (Ar-OH)
	Reactions : $6Ar\text{-}OH + FeCl_3 \longrightarrow [(Ar\text{-}O)_6Fe]^{3-} + 3HCl + 3H^+$ (complex ion, violet colour)		
	C.T. for α – naphthol		
	Org .comp.+ NaOH shake well + CCl_4 + Cu fillings & warm	Blue colouration	α -naphthol is confirmed

b) Phenols having elements C,H,(O) & N (Nitrophenols)

1	**Test for NO_2 group by neutral reduction** Org.comp. + pinch of zinc dust + 50% ethyl alcohol + $CaCl_2$ solution Add 1or 2 pieces of porcelain. Heat to vigorous boiling and filter in to Tollen's reagent	Black or grey ppt.	NO_2 group is present

	Preparation of Tollen's reagent : 5ml $AgNO_3$ solution + 1 drop NaOH + NH_4OH solution till the ppt. formed just dissolves. **Reactions :** $Ar\text{-}NO \xrightarrow[\text{Reduction}]{Zn / C_2H_5OH} Ar\text{-}NHOH$ $Ar\text{-}NHOH + 2\,Ag(NH_3)_2OH \longrightarrow Ar\text{-}NO + 2\,Ag\downarrow + 4\,NH_3\uparrow + 2\,H_2O$ (Tollen's reagent)		
2	Org. comp. + NaOH shake well	Compound dissolves	Phenolic – OH group is present
	Reactions: $Ar\text{-}OH + NaOH \longrightarrow Ar\text{-}ONa + H_2O$ Sodium phenoxide (soluble)		
3	Observe colour of the Org. comp.	Bright / dark yellow	o- Nitrophenol
		Pale yellow / colourless	p- Nitrophenol

	C.T.for o- Nitrophenol		
	i) Org. comp. + 60% HNO_3 warm gently	Compound dissolves with evolution of brown gas and reappears again on cooling as 2,4 dinitrophenol	o- Nitrophenol is confirmed
	ii) Org .comp.+ NaOH	Orange red colour	o- Nitrophenol is confirmed
	C.T. for p- Nitrophenol		
	i) Org. comp. + bromine in acetic acid , warm gently	Dark brown coloured solid or solution	p- Nitrophenol is confirmed
	ii) Org. comp. + NaOH	Yellow colour	p- Nitrophenol is confirmed

III) Base or Amines having elements C, H, (O) and N

Distinction between primary, secondary and tertiary amines

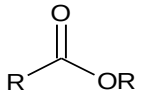
Sr. No.	Test	Observation	Inference
1	Org. comp. + 3 times conc. HCl + few drops of distilled water, cool thoroughly in ice cold water + few drops of $NaNO_2$ solution (Test tube 1)	i) yellow oil or solid separates	Secondary amine (>N-H) is present
		ii) red colour and on adding NaOH green solid is obtained	Tertiary amine (>N-) is present N, N dimethyl aniline is present
2	If observations (i) & ii) are absent, To the solution of β – napthol in NaOH, add 1 ml of solution from test tube 1.	Orange dye stuff	Primary amine i.e.o, m or p-Nitroaniline is present (Functional group Ar-NH_2)
	Reactions : $Ar\text{-}NH_2 \xrightarrow[\text{Low Temp.}]{NaNO_2 + HCl} \underset{\text{Benzene diazonium chloride}}{Ar\text{-}N{=}N\text{-}Cl} + NaCl + 2\,H_2O$		

a) Primary amines			
1	Colour of the organic compound	Yellow coloured solid	o, m or p- nitroaniline is present
	Tests for o, m or p-nitro anilines		
2	**Diazotization test** Use two test tubes (T.T.) for this test First T.T: Org comp. + 3 times conc. HCl cool and dilute with water + $NaNO_2$ sol^n, cool in ice cold water. Second T.T:	Orange dye stuff	. Primary amine (Ar-NH_2) group is present

	β -napthol + NaOH, shake well and cool in ice cold water. Add about 1ml sol^n from first T.T. to second T.T.		
3	**Neutral Test for NO_2 group** Org. comp.+ pinch of Zn dust + 50% ethyl alcohol + $CaCl_2$ sol^n + few pieces of porcelain, heat to vigorous boiling and filter in to Tollen's reagent	Black or grey ppt.	NO_2 group is present
4	Distinction between o, m or p-Nitroaniline is done from their m. p. Theoretical m. p. of : o-Nitroaniline is 70 ^{0}C m-Nitroaniline is 114 ^{0}C p-Nitroaniline is 147 ^{0}C	Practical m. p. of given nitro aniline is-------- ^{0}C	∴ …. - nitro aniline is present and confirmed

b) Tertiary amines			
1	Org .comp.+ 3 times conc. HCl few drops of distilled water – cool thoroughly in ice cold water + few drops of $NaNO_2$ solution	Red colour &on adding NaOH green solid is obtained	Tertiary amine i.e. N, N-dimethyl aniline is present
	C.T. for N,N-dimethyl aniline		
	2 ml saturated sol^n of picric acid in ethyl alcohol + few drops of Org. comp , shake vigorously	Yellow ppt.	N, N-dimethyl aniline is confirmed

IV) Neutral Compound having C, H &(O) elements

a) Esters (Liquid only)			
1	Use two T.T. for this test First T.T.- 1ml NaOH + 1 drop of phenolphthalein – pink colour obtained, disappear after some time. Again add one drop of phenolphthalein-pink colour appears. Wait for some time. If pink colour disappears, again add one drop of phenolphthalein. By this way stabilize the pink colour. Now add water so that faint pink coloured solution is obtained. Second T.T.- In a second T.T. take organic liquid and add faint pink coloured solution from T.T. No.1, shake well. If necessary heat	Pink colour disappears	Ester i.e. methyl acetate is present and confirmed  functional group
If given organic compound is solid , mention the ester test as follows –			
	All esters are liquids but given compound is solid	--	Ester is absent

b) Ketones – (Liquid only)			
1	Org. comp. + few drops of sodium nitroprusside + NaOH solution	Red colour	Methyl ketone group (CH_3CO-R) is present .i.e. ethyl methyl ketone and acetophenone is present
	C. T. for Ethyl methyl ketone		
	Org. liquid + solid m-dinitrobenzene, shake well + NaOH in excess	Violet colour which fades slowly	Ethyl methyl ketone is confirmed
	C. T. for acetophenone		
	Org .liquid + few drops of NaOH + sodium nitroprusside To the above solution add acetic acid	Wine red colour changes to blue	Acetophenone is confirmed CH_3CO-Ph
If given organic compound is soild , mention the ketone test as follows			
2	All ketones are liquids , but given compound is solid	--	Ketone is absent (-CO-)
c) Hydrocarbons			
1	Org. comp. does not show tests of acid, phenol, ester & ketones.	--	Compound is hydrocarbon i.e. naphthalene, anthracene is present
	C. T. for Naphthalene		
	i) Org. comp. is white solid. It is insoluble in water. It has smell of moth balls and it is aromatic in nature	--	Naphthalene is present & confirmed
	ii) Use two dry T.T. for this test <u>First T.T.-</u> Org. comp. + 2 ml benzene and shake well <u>Second T.T.-</u> Picric acid + 2 ml benzene (saturated solution) and shake well. Mix these two solution in a clean and dry evaporating dish. Keep it for 5 minutes.	Golden yellow ppt. of picrate	Naphthalene is confirmed
	C. T. for Anthracene i) Org.comp.is yellowish white solid, insoluble in water .It has no particular odour, it is aromatic in nature.	--	Anthracene is present & confirmed

	ii) Use two dry T.T. for this test First T.T.- Org. comp. + 2 ml benzene and shake well . Second T.T.- Picric acid + 2 ml benzene and shake well Heat these two T.T. in a water bath for few minutes. Mix these two solutions in a clean and dry evaporating dish. keep it for 5 minutes.	Red ppt. of picrate	Anthracene is present & confirmed

V) Neutral Compound having C, H, (O) & N elements

a) Nitrohydrocarbons :			
1	Org. comp. pinch of Zn dust +50% ethyl alcohol + $CaCl_2$ solution. Add 1 or 2 pieces of porcelain .Heat to vigorous boiling and filter in to Tollen's reagent.	Black or gray ppt	Nitrohydrocarbon i.e. Nitrobenzene is present (functional group $–NO_2$)
	Preparation of Tollen's reagent: refer previous procedure.		
	C.T. for Nitrobenzene		
	Organic liquid + conc. HCl snCl2 + 1 or 2 pieces of porcelain. Heat	Fishy smell of aniline	Nitrobenzene is confirmed
b) Amides			
1	Org. comp. + NaOH, boil	Evolution of NH_3 gas turning turmeric paper brown.	Amide group ($-CONH_2$) is present i.e. urea is present.
	Reaction: $RCONH_2 + NaOH \xrightarrow{\triangle} R\text{-}COONa + NH_3\uparrow$		
	C. T. for urea		
	Heat small amount of org. comp. in a dry test tube. Compound sublimes and NH_3 is evolved. Cool the T.T. and dissolve the residue in 2 ml distilled water + 1 drop of $CuSO_4$ + 2 ml NaOH	Violet colour	Urea is confirmed

c) Anilide			
1	Use two dry T.T. for this test First T.T.- Org .comp. + conc. HCl + 1 or 2 pieces of porcelain boil for 5 minutes & cool well + few drops of water +$NaNO_2$ solution, cool in ice cold water Second T.T.- β-napthol in NaOH solution, shake well and cool in ice cold water. Add about 1ml solution from first T.T. to second T.T.	Orange dye stuff	Anilide group (-$NHCOCH_3$) is present i.e. Acetanilide is present
	Reactions- $Ar\text{-}NH\text{-}COCH_3 + H_2O \xrightarrow[\text{Hydrolysis}]{HCl} Ar\text{-}NH_2 + CH_3COOH$ $Ar\text{-}NH_2 \xrightarrow[\text{Low Temp.}]{NaNO_2 + HCl} Ar\text{-}N{=}N\text{-}Cl + NaCl + 2\,H_2O$ Benzene diazonium chloride		
	C. T. for Acetanilide		
	Take org. comp. in dry T.T. + conc. H_2SO_4 stir with glass rod + powdered $K_2Cr_2O_7$	Red colour immediately changes to green.	Acetanilide confirmed.

VI) Neutral Compound having C, H & (O) and halogens elements

1	Org. liquid + NaOH solution , boil well + about 1ml dil HNO_3 and add $AgNO_3$ solution	i) white or yellow ppt.	Aliphatic halide , CCl_4 is present
		ii) No ppt	Aromatic halide i.e. bromobenzene is present
	C. T. for CCl_4		
	i) 1ml org. liquid + 2ml alcoholic KOH solution + 2ml aniline, warm carefully . Note: To the reaction mixture conc. HCl should be added before it is poured into sink	Disagreeable odour of carbylamine	CCl_4 is confirmed
	ii) Org. liquid + Cu fillings + solution of α –naphthol in NaOH, warm	Blue colour	CCl_4 is confirmed
	C.T. for Bromobenzene		
	i) Sodium extract+ CCl_4 +Cl_2 water in excess , shake well	Lower layer is yellow or yellowish brown	Bromobenzene is confirmed

	ii) In a dry T.T. take 1ml conc. HNO_3 + conc. H_2SO_4 + 0.5 ml org. liquid +1 or 2 pieces of porcelain. Heat for 1 min and pour in to water	Yellow ppt.	Bromobenzene is confirmed

F) Summery of Organic Compound

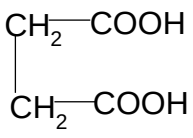
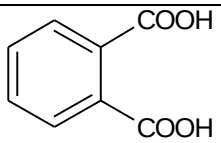
1.	State	Solid / Liquid
2.	Nature	Aliphatic /Aromatic
3.	Type	Acid / Phenol / Base / Neutral
4.	Elements	C, H, (O) &--------
5.	Functional group / groups	---------
6.	Physical constant :	a) Theoretical M.P. / B.P. = ---- ^{0} C = ------K
		b) Practical M.P. / B.P. = ---- ^{0}C =------- K

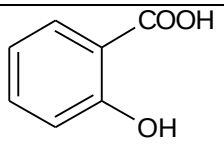
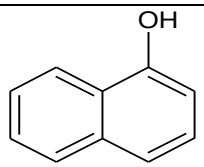
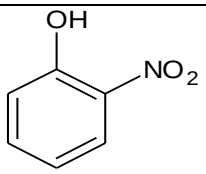
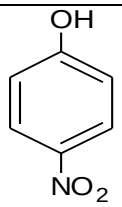
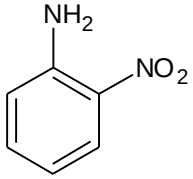
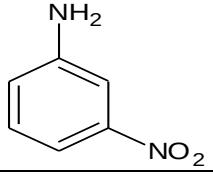
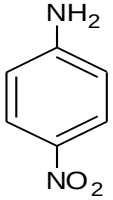
Therefore given organic compound is ----------

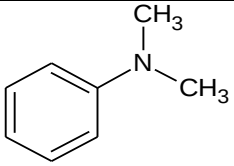
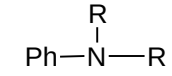
G) Results:

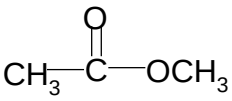
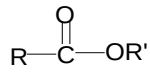
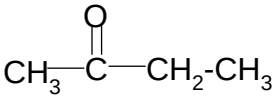
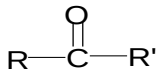
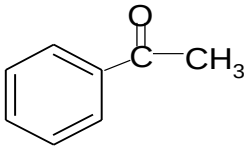
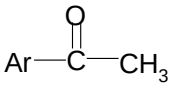
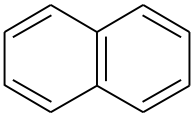
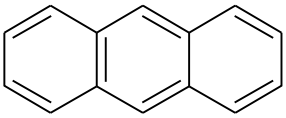
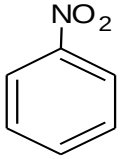
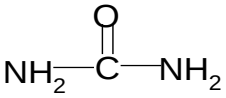
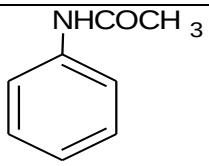
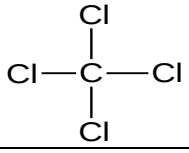
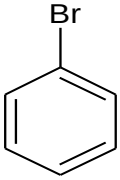
Name of Organic Compound	Structural formula	M.P / B.P.

H] Consultation with Literature:

Sr. No	Name of the organic compound & its molecular formula	Structural formula	M.P. or B.P.	Functional group
	I) Acids having element C, H & (O)			
1	Succinic acid $C_4H_6O_4$		M.P. 183^{0}C (461 K)	-COOH
2	Phthalic acid $C_8H_6O_4$		M.P. 193^{0}C (466 K)	-COOH

3	Salicylic acid $C_7H_8O_4$		M.P. 158 ^{0}C (431K)	-COOH Ar–OH
	II)Phenols			
	a) Phenols having element C, H &(O)			
4	α – naphthol $C_{10}H_7OH$		M.P. 94 ^{0}C (367 K)	Phenolic –OH
	b)Phenols having element C, H, (O)& N (Nitro phenols)			
5	o-Nitrophenol OH $(C_6H_4)NO_2$		M.P. 45 ^{0}C (318 K)	Phenolic- OH –NO_2
6	p-Nitrophenol OH $(C_6H_4)NO_2$		M.P. 114 ^{0}C (387 K)	Phenolic- OH –NO_2
	III) Base or Amines having elements C, H, (O) and N			
7	o-Nitroaniline $NH_2(C_6H_4)NO_2$		M.P. 71 ^{0}C (344 K)	Primary amine (Ar-NH_2) and –NO_2
8	m-Nitroaniline NH_2 $(C_6H_4)NO_2$		M.P. 114 ^{0}C (387 K)	Primary amine (Ar-NH_2)and –NO_2group
9	p-Nitroaniline NH_2 $(C_6H_4)NO_2$		M.P. 147 ^{0}C (420 K)	Primary amine (Ar-NH_2)and –NO_2group

10	N,N dimethyl aniline $CH_6N(CH_3)_2$		B.P. 193^{0}C (466 K)	Tertiary amine group 
	IV) Neutral Compound having C, H, & (O) elements			
	i) Esters			

11	Methyl acetate $C_3H_6O_2$		B.P. 57^0C (330 K)	Ester group 
	ii) Ketones			
12	Ethyl methyl ketone C_4H_8O		B.P. 80^0C (330 K)	Ketone group 
13	Acetophenone $C_6H_5.COCH_3$		M.P. 202 ^{0}C (475 K)	Ketone group 
	iii) Hydrocarbons			
14	Naphthalene $C_{10}H_8$		M.P. 80 ^{0}C (353 K)	CH
15	Anthracene $C_{14}H_{10}$		M.P. 217 ^{0}C (490 K)	CH
	V) Neutral Compound having C, H, (O) & N elements			
16	Nitrobenzene		B.P. 209^0C (482 K)	- NO_2
17	Urea $H_2N.CO.NH_2$		M.P. 132 ^{0}C (405 K)	-$CONH_2$ Amide group
18	Acetanilide $C_6H_5.NHCOCH_3$		M.P. 114 ^{0}C (387 K)	-$NHCOCH_3$ Amide group
	VI) Neutral Compound having elements C, H, & (O) and halogens			
19	Carbon tetrachloride CCl4		B.P. 78^0C (351 K)	Aliphatic-Cl
20	Bromobenzene C_6H_5Br		M.P. 155 ^{0}C (428 K)	Ar -Br

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INDEX

19.	Estimation of Sodium by ion exchange method		34

I. GRAVIMETRIC ESTIMATIONS

1. Gravimetric Estimation of Iron.

Aim : Gravimetric estimation of Iron as Fe_2O_3 from the given solution of ferrous ammonium sulphate, copper sulphate and free sulphuric acid

Chemicals :
1) Given sol^n of F.A.S,
2) Ammonia 1:1
3) Conc. HCl
4) Conc. HNO_3
5) 1% Ammonium nitrate sol^n
6) H_2S gas,
7) Distilled water.

Apparatus :
i) Pipette 25 ml
ii) Beaker 250 ml/500ml
iii) Desiccator
iv) Silica crucible

Procedure :

A) Removal of Copper as CuS : -

1) Dilute the given solution up to mark (100 ml or 250 ml) with distilled water and shake well. **2)** Pipette out 50 ml diluted solution (by using 25 ml pipette) in 250 ml beaker. Add to it about 100 ml distilled water with measuring cylinder and 2-3 ml of conc. HCl. **3)** Boil the solution on wire gauze and pass H_2S gas to precipitate copper as CuS. (Use separate gas passing tube.) **4)** Filter the solution through ordinary filter paper and wash the ppt. and beaker with hot distilled water. **5)** Collect the filtrate and washing in 500ml beaker. Reject the ppt. of CuS (Do not waste single drop of filtrate). Boil off H_2S gas. Test vapors with lead acetate paper. Use this filtrate for estimation of iron.

B) Estimation of Iron.

6. **Precipitation**: To the filtrate free from Copper add 10 ml dil. H_2SO_4 solution and 5 ml conc. HNO_3 and boil the solution. While heating add 2 g of NH_4Cl.Observe colour –Colour of solution is intense yellow.

7. Keep the beaker on asbestos sheet and precipitate Fe as Fe $(OH)_3$ by adding 1:1 ammonia solution. Add ammonia solution till distinct smell of ammonia to the solution (Check carefully).

8. **Filtration and Washing**: Filter the ppt. of Fe $(OH)_3$ in hot condition by using Whatman filter paper No. 41.Wash the ppt and beaker with hot 1% Ammonium nitrate solution (use small amout of sol^n each time). Continue washing of ppt until the fresh filtrate is free from Cl^- and SO_4^{2-} (Test with Ag NO_3 and $BaCl_2$ sol^n respectively)

9. **Drying** : Dry the ppt. on drying cone .

10. **Ignition and weighing:** Ignite the ppt. in previously weighed crucible. Heat crucible on **blue flame** of burner for about 45 min.

11. Cool the crucible on **tripod stand first for 10 min.** and transfer it to desiccators when it cool to room temperature & weight it on **Digital balance**.

12. Again heat the crucible on blue flame for 10 min. and after cooling weigh it accurately. Repeat the procedure till constant weight is obtained. From the weight of residue obtained, find out amount of Fe and F.A.S. in the given sol^{n}**.**

Observations:

1)	Weight of empty Crucible	W_1 = ……g.
2)	Weight of Crucible + Residue On first heating (45 Min) a = ……g. On second heating (10 Min) b = ……g. On third heating (10 Min) c = ……g. ∴ Constant weight of crucible + Residue	 W_2 = ……g.
3)	Weight of Residue W_2-W_1	W = …… g.

Reactions :

i) $CuSO_4 + H_2S \longrightarrow CuS\downarrow + H_2SO_4$

ii) $6FeSO_4\,(NH_4)_2SO_4 + 2HNO_3 + 3\,H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + 6\,(NH_4)_2SO_4 + 4H_2O + 2\,NO$

iii) $Fe_2(SO_4)_3 + 6\,NH_4OH \longrightarrow 2Fe\,(OH)_3\downarrow + 3(NH_4)_2SO_4$

iv) $2Fe(OH)_3 \xrightarrow[1000^0C]{\text{Ignition}} Fe_2O_3 + 3H_2O\uparrow$

Calculation:

$Fe_2O_3 \equiv 2Fe \equiv 2\,FeSO_4\,.(NH_4)_2SO_4.\,6H_2O$

$159.69 \equiv 111.60 \equiv 784.26$

$1 \equiv 0.6994 \equiv 4.911$

Now,

a) Quantity of Fe_2O_3 in the given solution

50ml diluted sol^{n} FAS ≡ W g. of Fe_2O_3

∴ Quantity of Fe_2O_3 in 100 ml sol^{n} = $\frac{100 \times W}{50}$

= 2 x W g of Fe_2O_3

i.e. A = ……g of Fe_2O_3

50ml diluted sol^{n} FAS ≡ Wg. of Fe_2O_3

∴ Quantity of Fe_2O_3 in 250 ml sol^{n} = $\frac{250 \times W}{50}$

= 5 x W g of Fe_2O_3

i.e. A = ……g of Fe_2O_3

b) Quantity of Fe in the given solution

From eq^{n}, 1 g of Fe_2O_3 ≡ 0.6994 g of Fe

∴ A g of Fe_2O_3 ≡ A x 0.6994 g of Fe

i.e. B = ……g of Fe

c) Quantity of $FeSO_4$. $(NH_4)_2SO_4$. $6H_2O$:

∴ 1 g of Fe_2O_3 ≡ 4.911 g of $FeSO_4$. $(NH_4)_2SO_4.6H_2O$.

∴ 'A' g of Fe_2O_3 ≡ A x 4.911 g of F.A.S.

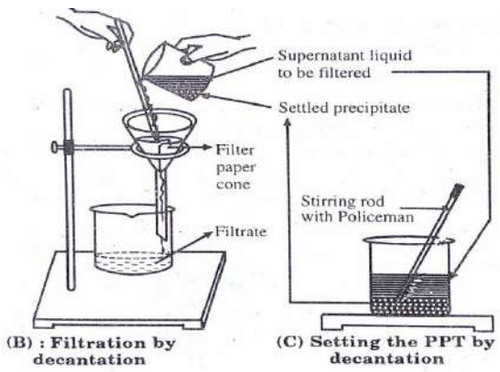
i.e. C = …… g of F.A.S.

Results :

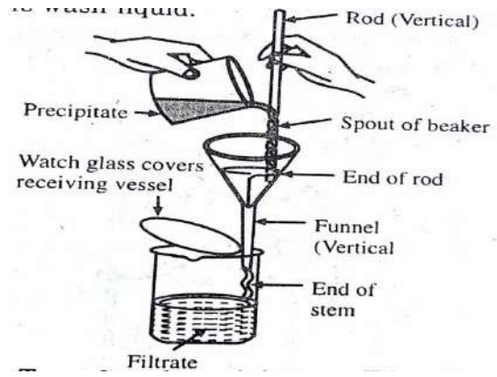
1.	50 ml diluted solution gave	W = …….. g Fe_2O_3

2.	Quantity of Fe in the given solution	B = ……g
3.	Quantity of $FeSO_4$. $(NH_4)_2SO_4$. $6H_2O$ in the given sol^n	C = ……g

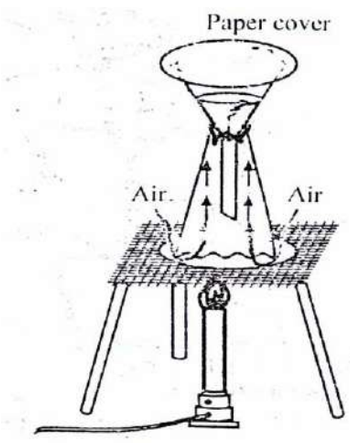
TECHNIQUES IN GRAVIMETRIC ANALYSIS



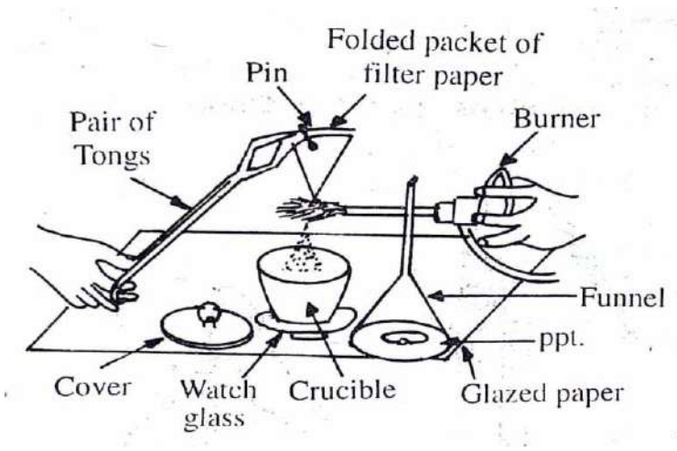
1.Filtration



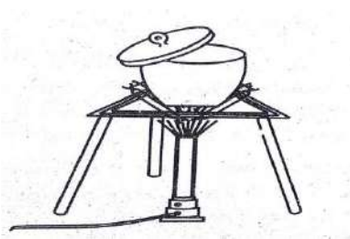
2.Transfer of Precipitate to Filter Cone



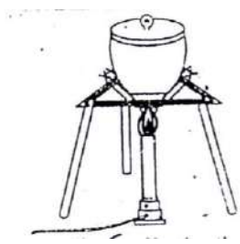
3.Drying of ppt on Metal Cone



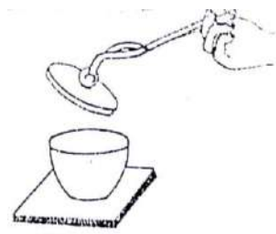
4. Ignition / Incineration of filter paper



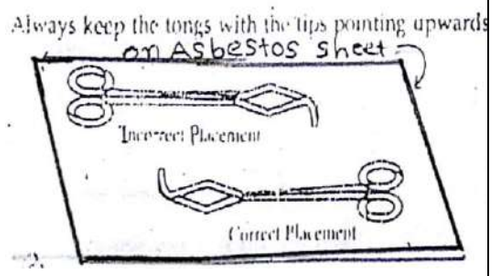
5. Heating the Crucible



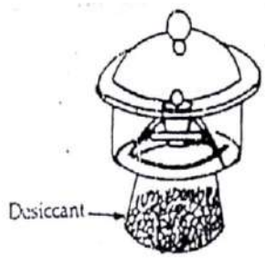
6.Heating the Crucible



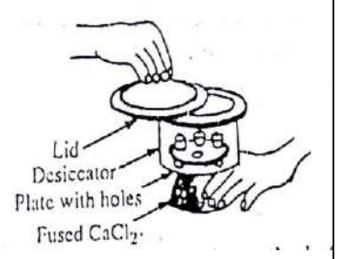
7.Cooling the crucible on asbestos Sheet



8. Correct position of Pair of tong



9.Desiccetor



10.Method of opening the Desiccetor

2. Gravimetric Estimation of Barium

Aim : Gravimetric estimation of Barium as $BaSO_4$ from the given solution of barium chloride, ferric chloride and free HCl.

Chemicals :
1) Given sol^n of barium chloride
2) 2 N H_2SO_4
3) Conc. HCl
4) Conc. HNO_3
5) 1:1 NH_3 solution
6) Solid NH_4Cl
7) Distilled water.

Apparatus : ***As Gravimetric Estimation No. 1***

Procedure:

A) Removal of IRON :

1. Dilute the given solution to up to mark (100ml or 250ml) with distilled water and shake well.
2. Pipette out 50 ml (by using 25 ml pipette) diluted solution in 250 ml. beaker. Add to it about 2 ml of conc. HCl, about 100 ml of distilled water and about 1-2 ml of conc. HNO_3. Heat the solution up to boiling and add about 1g of solid NH_4Cl. Observe colour-colour of solution is intense yellow.
3. Now stop heating and precipitate Fe as $Fe(OH)_3$ by adding 1:1 Ammonia solution. Continue addition of ammonia until distinct smell of ammonia to the solution in beaker (Check carefully).
4. Quickly filter the ppt. $Fe(OH)_3$ through ordinary filter paper in hot condition. Wash the ppt. and beaker with hot 1% ammonium nitrate or hot distilled water. Collect filtrate and washing in a 500ml beaker. Reject the ppt. of $Fe(OH)_3$.
5. Boil off excess ammonia, test vapors with turmeric paper. Use this filtrate for estimation of Barium.

B) Estimation of Barium :

6. **Precipitation:** To the filtrate free from ammonia. add 1 test-tube hot 2N H_2SO_4 drop by drop with constant stirring.
7. **Digestion:** Digest the ppt of $BaSO_4$ on sand bath for about 30 min. by covering the beaker with watch glass. Now check whether the precipitation of Barium is complete or not by adding hot 2N H_2SO_4 from side of beaker. (If precipitation is not complete add few ml of 2N H_2SO_4)

8. **Filtration & washing:** Filter the digested ppt. of $BaSO_4$ through Whatman No. 42 and wash with hot distilled water. Test the fresh filtrate with $AgNO_3$ and $BaCl_2$ or $Ba(NO_3)_2$ solution for removal of Cl^- and SO_4^{2-} respectively.

9. **Drying:** Dry the ppt. on drying cone.

10. **Ignition and Weighing**: Ignite the ppt. of $BaSO_4$ in previously weighed crucible. Heat the crucible on pipe clay triangle for about 45 min strongly on blue flame.

11. Cool the crucible on tripod stand first for about 10 min. and then transfer in to desiccators and Weigh it on the digital balance, as a weight of Crucible + Residue.

12. Heat the crucible once again for 10 min. Cool and weigh again. Repeat heating, cooling and weighing till constant weight is obtained.

From the weight of $BaSO_4$ obtained, find out the amount of Ba and $BaCl_2.2H_2O$ in the given solution.

Reactions:

$FeCl_3 + 3NH_4OH \longrightarrow Fe(OH)_3\downarrow + 3NH_4Cl$

$BaCl_2 + H_2SO_4 \longrightarrow BaSO_4\downarrow + 2HCl$

$BaSO_4 \xrightarrow{\text{Ignition}} BaSO_4$

Observations:

1) Weight of empty Crucible	W_1 = ----------g.
2) Wight of Crucible + Residue On first heating (45 Min) a = ----------g. On second heating (10 Min) b = ----------g. On third heating (10 Min) c = ----------g. ∴ Constant weight of crucible + Residue	 W_2 = ----------g.
3) Weight of Residue W_2-W_1	W = ----------g.

Calculation :

$BaSO_4 \equiv Ba \equiv BaCl_2.2H_2O$

$233.39 \equiv 137.34 \equiv 244.28$

$1. \equiv 0.5884 \equiv 1.0467$

Now

a) Quantity of $BaSO_4$ in the given solution

50ml diluted sol^n $BaCl_2$ $2H_2O \equiv$ Wg. of $BaSO_4$

∴ Quantity of $BaSO_4$ in 100 ml sol^n = $\frac{100 \times W}{50}$

= 2 x W g of $BaSO_4$

i.e. A = ……g of $BaSO_4$

50ml diluted sol^n $BaCl_2$ $2H_2O \equiv$ Wg. of $BaSO_4$

∴ Quantity of $BaSO_4$ in 250 ml sol^n = $\frac{250 \times W}{50}$

= 5x W g of $BaSO_4$

i.e. A = ……g of $BaSO_4$

b) Quantity of Ba in the given solution

∴ 1 g of $BaSO_4$ ≡ 0.5887g of Ba

∴ A g of $BaSO_4$ = A x 0.5887 g of Ba.

i.e. B = ……..g. of Ba.

c) Quantity of $BaCl_2$. 2 H_2O in the given solution.

∴ 1 g of $BaSO_4$ ≡ 1.0467 g of $BaCl_2$. $2H_2O$.

∴ A g of $BaSO_4$ = A x 1.0467 g of $BaCl_2$. $2H_2O$.

i.e. C = ……. g of $BaCl_2$. $2H_2O$

Results :

1.	50 ml diluted solution contains amount of $BaSO_4$	W = …….. g
2.	Quantity of Ba in the given solution	B = ……g
3.	Quantity of $BaCl_2.2H_2O$ in the given solution	C = ……g

3. Gravimetric Estimation of Manganese

Aim: Gravimetric estimation of Manganese as Manganese ammonium phosphate from the given solution containing manganese sulphate,copper sulphate and free sulphuric acid

Theory : The $MnNH_4PO_4$ is a complex salt formed due to reaction between Mn & $(NH_4)_2HPO_4$. This salt is formed at a controlled pH 6.7 to 6.9 and is very unstable. On over heating, it gets converted into $Mn_2P_2O_7$. So temperature is to be controlled to retain it as desired complex. The direct heating of the precipitate is avoided by drying it in a sintered glass (Gooch) crucible at 383 to 388 K (110 to 115° C) in an electrical oven.

Chemicals : 1) Conc. HCl 2) H_2S gas 3) Methyl red indicator
4) 1:1 NH_3 5) 2 N CH_3COONH_4 6) 10% $(NH_4)_2 HPO_4$

Apparatus : 1-3 as gravimetric estimation of Iron,
4. Sintered glass crucible (Gooch crucible)

Procedure :

***Weighing of sintered Glass crucible*:** You are supplied with clean sintered Glass Crucible.Wash it with Distilled water and dry it in electric oven for about 20 min at 110° C and weigh after cooling

A) Removal of copper as Cus :

1 – 5 : As gravimetric estimation of Iron.

B) Estimation of Manganese -

6. **Precipitation:** To the filtrate free from iron add 2 drops of 1% methyl red indicator solution, which turns pink. Add one test tube (20 ml.) each of 2N CH_3COONH_4 and 10% $(NH_4)_2 HPO_4$ reagent with constant stirring. If any precipitate forms, then add a few drops of cons. HCl to dissolve it. Heat the solution to boiling. Add 1:1, NH_3 to the hot solution drop wise with constant stirring till precipitate of $MnNH_4PO_4$ begins to form and, solution turns yellow.
7. **Digestion :**Stop addition of ammonia and keep the beaker in boiling water bath. Stir the solution vigorously for 15 minutes to obtain the crystalline $MnNH_4PO_4$.

Precipitate becomes silky in appearance, solution remains yellow. Allow the precipitate to cool, keep at room temperature for about 30 minutes.

8. **Filtration:** Filter the precipitate through a weighed sintered glass crucible.
9. **Washing:** Wash the precipitate with 1% NH_4NO_3 solution till the filtrate is free from Cl^- and SO_4^{--} and PO_4^{3-} ions.
10. **Drying and Weighing:** Dry the precipitate in an electric oven at 110°C for about 45 minutes and weigh as $MnNH_4PO_4$ after cooling. Repeat the procedure of heating, cooling and weighing till constant weight is obtained.

Observations:

1) Weight of empty Gooch Crucible	W_1 = …….g.
2) Wight of Gooch Crucible + Residue On first heating (45 Min) a = …….g. On second heating (15 Min) b = …….g. On third heating (15 Min) c = …….g. ∴ Constant weight of Gooch crucible + Residue	 W_2 = …….g.
3) Weight of Residue W_2-W_1	W = ……. g.

Reactions : 1) $CuSO_4 + H_2S \longrightarrow CuS\downarrow + H_2SO_4$

2) $2\,MnSO_4 + 2(NH_4)_2\,HPO_4 \longrightarrow 2MnNH_4PO_4 + (NH_4)_2\,SO_4 + H_2SO_4$

Calculations:

We know from reaction

$MnNH_4\,PO_4.\,H_2O \equiv Mn \equiv MnSO_4.\,4H_2O$

185.74 ≡ 54.93 ≡ 223.06

i.e. 1 ≡ 0.2958 ≡ 1.2009

1) Quantity of $MnNH_4\,PO_4.\,H_2O$ in the given solution

∴ 50ml diluted sol^n ≡ Wg. of $MnNH_4\,PO_4.H_2O$	∴ 50ml diluted sol^n ≡ Wg. of $MnNH_4\,PO_4.H_2O$
∴ 100 ml of Sol^n = $\frac{100 \times W}{50}$ g of $MnNH_4\,PO_4.H_2O$	∴ 250 ml of Sol^n = $\frac{250 \times W}{50}$ g of $MnNH_4\,PO_4.H_2O$
i.e. A = 2 x W g $MnNH_4\,PO_4.H_2O$	i.e. A = 5 x W g $MnNH_4\,PO_4.H_2O$

2. Quantity of Mn in the given solution

If 1 g of $MnNH_4\,PO_4.H_2O$ ≡ 0.2958 g of Mn

∴ 'A'g of Complex ≡ A x 0.2958 g of Mn.

i.e. B = …….g Mn

3. Quantity of $MnSO_4.\,4H_2O$ in the given solution

∴ 1 g $MnNH_4\,PO_4.H_2O$ ≡ 1.2009 g of $MnSO_4.\,4H_2O$

∴ A g $MnNH_4\,PO_4.H_2O$ ≡ A x 1.2009 g $MnSO_4.\,4H_2O$

i.e. C = …….g $MnSO_4.\,4H_2O$

Results:

1.	50ml diluted solution contains	W = …….. g $MnNH_4PO_4$
2.	Quantity of Mn in the given solution	B = ……g
3.	Quantity of $MnNH_4PO_4$ in the given solution	C = ……g

4. Gravimetric Estimation of Nickel

Aim : Gravimetric estimation of Nickel as bis (dimethlylglyoximato) nickel (II) i.e. [$Ni(dmg)_2$] from the given solution containing nickel sulphate, ferrous ammonium sulphate and free sulphuric acid.

Chemicals : 1) Given sol^n of Nickel sulphate 2) Cone. HNO_3
3) 1% Alcoholic Dimethylglyoxime sol^n 4) Solid NH_4Cl
5) 1:1 NH_3 sol^n 6) 2 N Acetic acid sol^n 7) Distilled water.

Apparatus : As previous Experiment

Procedure :

***Weighing of sintered Glass crucible*:** You are supplied with clean sintered Glass Crucible Wash it with Distilled water and dry it in electric oven for about 20 min at 110^o C and weigh after cooling

A) Removal of IRON:

1. Dilute the given solution up to mark [100ml or 250ml] with distilled water and shake well.
2. Pipette out 50ml (Two 25ml pipette) diluted solution in 250 ml Beaker & add to it about 100 ml distilled water. Add about 5ml dilute sulphuric acid 5 ml of conc. HNO_3 Sol^n. Boil the Sol^n .+ 1g solid NH_4Cl observe colour (Intense yellow)
3. Now precipitate Fe as $Fe(OH)_3$ by adding 1:1 Ammonia solution. Add ammonia till a distinct smell of it to the solution.
4. Filter the ppt. of Fe $(OH)_3$ through ordinary filter paper in hot condition.Wash the ppt. and beaker with hot distilled water and then with hot 1% NH_4NO_3sol^n. Collect filtrate and washing in 500ml beaker without wasting.Reject the ppt of Fe $(OH)_3$.
5. Boil of excess ammonia [Reduce the volume to half] test vapors with turmeric paper. Use this filtrate for estimation of nickel.

B) Estimation of Nickel:

6. **Precipitation:** To the filtrate free from ammonia add about 30 ml alcoholic solution of dimathylglyoxime. Immediately add 1:1 ammonia solution with constant stirring till there is distinct smell of ammonia to the solution.
7. **Digestion :** Digest the scarlet red ppt of Ni-DMG complex on boiling water bath for about 50 min. Now test the supernatant liquid with reagent D.M.G. and ammonia to ensure the completion of precipitation of Nickel.
8. **Filtration:** Cool the solution and filter the ppt.through previously weighed sintered glass crucible .
9. **Washing:** Wash the ppt. and beaker with hot distilled water. Continue the washing of ppt until fresh filtrate is free from Cl^- and SO_4^{2-} ions. Finally wash ppt. with 5-10 ml of ethanol.
10. **Drying and Weighing:** Dry the ppt. at about 120^o C in an elective oven for about 45 min. Cool the crucible on asbestos sheet and transfer it to desiccators and weigh the residue. Repeat the procedure of heating and cooling and weighing till constant weight is obtained. From the weight of Ni-DMG obtained, find out the amount of $NiSO_4.7H_2O$ in the given solution using following equations.

Observation:

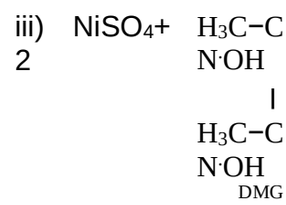
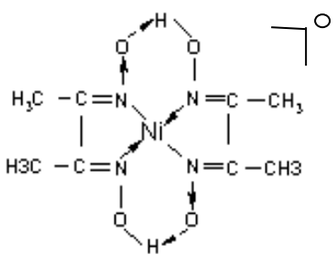
1) Weight of empty Gooch Crucible	W_1 = ----------g.
2) Wight of Gooch Crucible + Residue	

On first heating (45 Min) a = ----------g. On second heating (15 Min) b = ----------g. On third heating (15 Min) c = ---------- g. ∴ Constant weight of Gooch crucible + Residue	W_2 = ----------g.
3) Weight of Residue [$Ni(dmg)_2$] W_2-W_1	W = ----------g.

Reactions :

i) $6FeSO_4(NH_4)_2SO_4 + 3H_2SO_4 + 2\,HNO_3 \xrightarrow{\text{Oxidation}} 3Fe_2(SO_4)_3 + 6(NH_4)_2SO_4 + 4H_2O + 2\,NO$

ii) $Fe_2(SO_4)_3 + 6NH_4OH \longrightarrow 2Fe(OH)_3 \downarrow + 3(NH_4)_2SO_4$

iii) $NiSO_4 + 2$  $+2NH_4OH \rightarrow$  $+2H_2O + (NH_4)_2SO_4$

DMG

Calculations :

We know from reaction,

$Ni(C_2H_7O_2N_2)_2 \equiv Ni \equiv NiSO_4 . 7H_2O$

i.e 288.92 ≡ 58.70 ≡ 280.87

i.e. 1 ≡ 0.2032 ≡ 0.9721

1) Quantity of $Ni(dmg)_2$ in the given solution

∴50ml diluted sol^n ≡ Wg.of $[Ni(C_4H_7O_2N_2)_2]$	∴50ml diluted sol^n ≡ Wg.of $[Ni(C_4H_7O_2N_2)_2]$
∴100ml of Sol^n = $\frac{100 \times W}{50}$ g of $[Ni(C_4H_7O_2N_2)_2]$	∴250ml of Sol^n = $\frac{250 \times W}{50}$ g of $[Ni(C_4H_7O_2N_2)_2]$
= 2 x W g $[Ni(C_4H_7O_2N_2)_2]$	= 5 x W g [$Ni(C_4H_7O_2N_2)_2$]
i.e. A = ……. g $[Ni(C_4H_7O_2N_2)_2]$	i.e. A = ……. g $[Ni(C_4H_7O_2N_2)_2]$

2) Quantity of Nickel in the given solution: -

∴ 1 g of [Ni $(C_4H_7O_2N_2)_2$] = 0.2032 g of Ni.

∴ A g of complex = A x 0.2032 g of Ni

i.e. B = ……. g of Ni.

3) Quantity of $NiSO_4$. $7H_2O$ in the given solution: -

1 g of $[Ni(C_4H_7O_2N_2)_2]$ = 0.9721 g of $NiSO_4$. $7H_2O$

∴ A g of $[Ni(C_4H_7O_2N_2)_2]$ = $\frac{A \times 0.9721}{1}$ g of $NiSO_4$. $7H_2O$

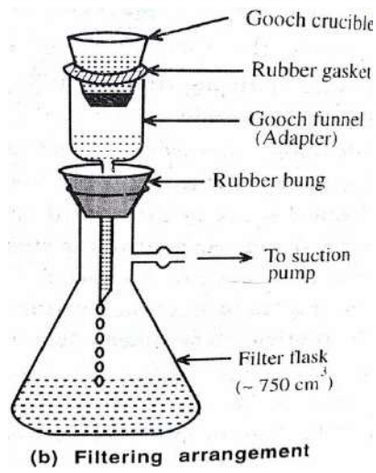
i.e. C = ……. g of $NiSO_4$. $7H_2O$

Results :

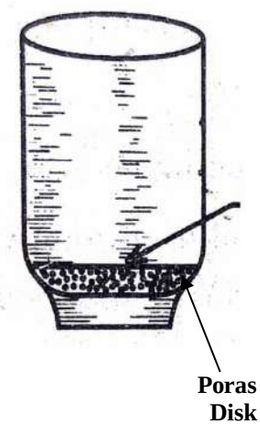
1. 50 ml diluted solution contains amount of $[Ni(dmg)_2]$	W = ……..g

2. Quantity of Ni in the given solution	B = ……g
3. Quantity of $NiSO_4.7H_2O$ in the given Solution	C = ……g

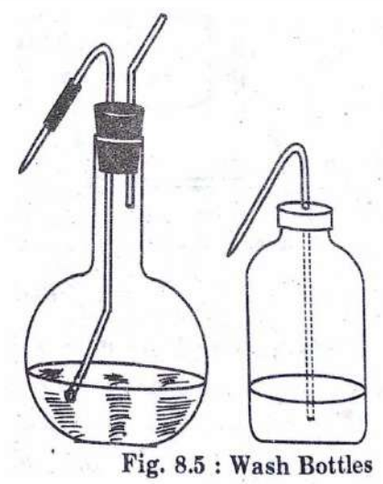
……



Filteration by Using Vaccum pump



Gooch crucible



Wash Bottes

II. INORGANIC PREPARATIONS

5. Preparation of Potassium trioxalatoaluminate (III)

Aim : To prepare potassium trioxalatoaluminate(III) $K_3[Al(C_2O_4)_3]\cdot 3H_2O$

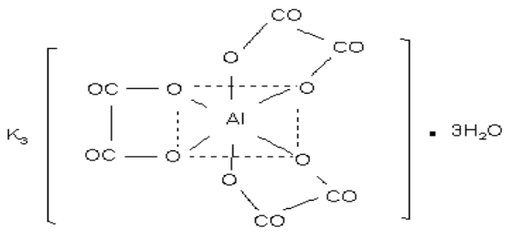
Chemicals : 1) Aluminum shavings or wire or foil 3) Oxalic acid
2) Potassium hydroxide (20%) 4) Ethanol.
5) 1:1 NH_3

Procedure :

i) Weigh 1 g 'Al' metal (foil / Shaving) on **Digital balance** ii) Take 30 ml 20% KOH solution in conical flask. Keep the flask on asbestos sheet & add to it 10ml boiled water. iii) Divide 'Al' foil / shavings in four small portions & add one portion to flask and shake. Aluminum gets dissolved with the vigorous effervescence of H_2. After complete dissolution add remaining portions one by one & dissolve Aluminum carefully. iv) Then filter in hot condition through glass wool / cotton plug & heat the solution to boiling v) Weigh 14 g of oxalic acid and add it in small portions to hot solution until the precipitate of hydrated alumina $Al_2O_3\cdot 3H_2O$ formed first, is just redissolved on continued boiling vi) Cool to room temperature vii) Add small piece of litmus in solution. Neutralize the solution by adding 1:1 ammonia solution from **common burette** drop by drop with constant stirring till litmus becomes just blue. Viii) Boil off excess ammonia (Test) & Cool to room temperature. ix) Add 50 ml ethanol with stirring x) Cool beaker in ice-cold water & filter the product through Buchner funnel, dry & weigh.

Observations: i) Aluminum Foil – 1.0 g iii) Ethanol – 50 ml.
ii) 20% KOH – 30 ml iv) Oxalic acid -- 14.0 g

Reactions :

i) $2\,Al + 6\,KOH \xrightarrow{Heat}$	$2K_3AlO_3 + 3H_2\uparrow$
ii) $K_3AlO_3 + 3\,H_2C_2O_4 \longrightarrow$	

Calculations :

1) Theoretical yield	2) Percent yield
From the chemical reactions we get Al = $K_3[Al(C_2O_4)_3]\,3H_2O$ 26.68 = 462 Now 26.68 g Al metal = 462 g complex ∴ 1 g Al metal = $\frac{1 \times 462}{26.68}$ g complex i.e. A = 17 g. complex	Weight of Product = (X) = ……g Now, 17 g of complex = 100% yield ∴ X g of complex = $\frac{X \times 100}{17}$ % i.e. B = ……. %

Result:

i)	Weight of product	= X = …….. g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..x 10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

6. Preparation of Tris (ethylenediamine) nickel (II) thiosulphate

Aim : To prepare Tris (ethylenediamine) nickel (II) thiosulphate. $[Ni\,(en)_3]\,S_2O_3$

Chemicals : i) Nickel nitrate ii) Sodium thiosulphate
iii) Ethylenediamine (Hydrated) iv) Ethanol

Procedure :

i) Weigh out 5 g Nickel nitrate and dissolve in about 25 ml distilled water & add 6 ml ethylenediamine in a beaker with vigorous stirring and then boil. ii) Weigh 5 g sodium thiosulphate and dissolve it in 50 ml distilled water and boil it. iii) Add hot solution of sodium thiosulphate to boiled nickel nitrate solution with constant stirring. Boil resulting solution for a minute & stir well. iv) Cool and filter the faint violet product on Buchner funnel. Wash the product with cold water 2-3 times and finally with little alcohol. v) Dry the product at about 100^oC and weigh.

Observation: i) Nickel nitrate – 5.0 g ii) Sodium thiosulphate – 5.0 g
iii) Ethylene diamine – 6 ml iv) Alcohol – 10 ml

Reactions :

i) $Ni(NO_3)_2 + 3C_2H_4(NH_2)_2 \xrightarrow{Dissolution} [Ni(en)_3](NO_3)_2$

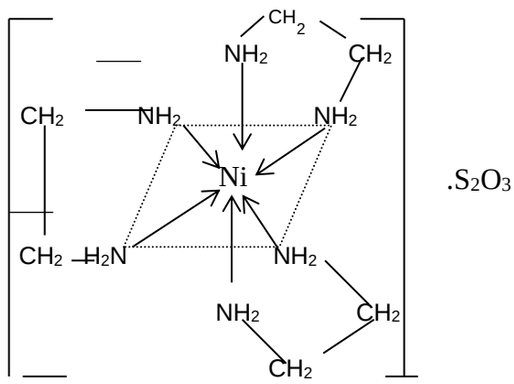
ii) $[Ni(en)_3](NO_3)_2 + Na_2S_2O_3 \xrightarrow[\text{Cool}]{\text{Boil}} [Ni(en)_3]S_2O_3 + 2NaNO_3$

Calculations:

1) Theoretical yield	2) Percent yield
From the chemical reactions we get $Ni(NO_3)_2.6H_2O = [Ni(en)_3]S_2O_3$ 291 = 351 i.e. 291 g $Ni(NO_3)_2.6H_2O$ = 351 g complex $\therefore$ 5 g $Ni(NO_3)_2.6H_2O = \frac{5 \times 351}{291}$ g complex i.e. A = 6.0 g complex	Weight of the product (X) = …… g Now, 6 g of complex = 100% yield $\therefore$ X g of complex $= \frac{X \times 100}{6}$ % i.e. B = ……%

Result :

i)	Weight of product	= X = - - - - g =…….x10^{-3} Kg
ii)	Theoretical yield	= A = - - - g =…….x10^{-3} Kg
iii)	Practical % yield	= B % = - - %



$.S_2O_3$

Tris (Ethylenediamine) Nickel (II) thiosulphate

7. Preparation of Reinecke's Salt

Aim : To Prepare ammonium diamminetetrathiocyanatochromate (III), (Reineck's Salt)

$NH_4[Cr(CNS)_4.(NH_3)_2].H_2O$.

Chemicals :i) Ammonium thiocyanate, ii) Ammonium dichromate, 3) Ice etc.

Procedure :

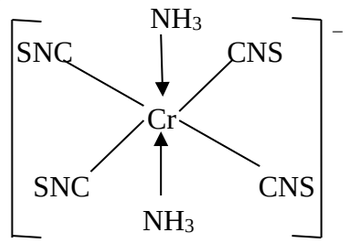
i) Weigh 10 g. Ammonium thiocyanate in evaporating dish. ii) Fuse 10 g of ammonium thiocyanate at 150^0 in an evaporating dish. Stir the fused mass iii) To this melt add 2 g of crushed powder of ammonium dichromate, in small lots (200 mg) with constant stirring. Violent reaction occurs with evolution of NH_3.iv) Heat the dish carefully until brisk effervescence stop. Cool the purple colored melt and allow it to solidify. v) Powder the solid mass with metal spatula & transfer it to a beaker vi) Dissolve out unreacted NH_4CNS and $(NH_4)_2Cr_2O_7$ by shaking the impure product with 20 ml of ice cold water. vii) Filter the solution by suction. Wash solid with alcohol followed by cold distilled water.

Recrystallization: Dissolve the crude product in about 25 ml of hot distilled water at about 70^oC. Filter the solution while hot. Cool the filtrate in ice-bath and allow the Reineck's salt to recrystallize as glistering red solid. Filter the solution by suction, dry the product in sunlight and record its yield. Calculate theoretical and practical percentage yield of the complex.

Observation: i) Ammonium Thicyanate : 10.0 g

ii) Ammonium Dichromate : 2.0 g

Reactions :

$(NH_4)_2Cr_2O_7 + 8NH_4CNS \rightarrow 2NH_4^+$ 

$^- + NH_3 + 4\,H_2O + 3/2\,O_2$

Calculations :

1) Theoretical yield	**2) Practical yield**
From the chemical reaction we get, $(NH_4)_2Cr_2O_7 = 2NH_4[Cr(CNS)_4.(NH_3)_2].\,H_2O.$ 252.063 = 2 x 354 Now, 252.063 g $(NH_4)_2Cr_2O_7$ = 2 x 354 g complex ∴ 2 g $(NH_4)_2Cr_2O_7$ = $\frac{2 \times 354 \times 2}{252.063}$ g complex i.e. A = 5.62 g complex	Weight of the product = X= ……g Now 5.62 g complex = 100 % yield ∴ X g complex = $\frac{X \times 100}{5.62}$ % i.e. B = ... %

Result :

i)	Weight of product	= X = …….. g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..x 10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

8. Preparation of Chloropentaamminecobalt (III) Chloride

Aim : To Prepare Chloropentaamminecobalt (III) chloride. $[Co(NH_3)_5Cl]Cl_2$

Chemicals : 1) Cobaltous chloride, 2) Ammonium chloride , 3) Ammonia.
4) Hydrogen peroxide (20 Volume) 5) Hydrochloric acid.

Procedure :

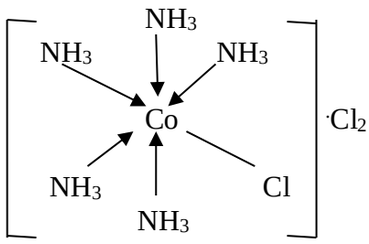
i) Take 5 g cobaltous chloride in a 250 ml beaker and dissolve it in minimum distilled water. ii) Weigh 10 g ammonium chloride and dissolve in 40 ml liquor ammonia. iii) Add this ammonical ammonium chloride solution to the cobaltous chloride solution with constant stirring. Cool the solution in a water bath. iv) Add by a burette 2 ml H_2O_2 at a time with constant stirring until the addition of 25 ml H_2O_2 is complete v) Continue the stirring of solution till the effervescence O_2 ceases (it take about 15 minutes.) vi)Then neutralize this solution with Conc. HCl. (Test it by litmus paper, both blue and red litmus should remain unaffected.) vii) Then add 10 ml conc. HCl in excess (Blue litmus paper should turn red). viii) Heat the solution gently to boiling. ix) Allow it to cool to room temperature when purple coloured crystals separate out. x) Filter the product and wash with alcohol. Dry and record the yield.

Observation:

i) Cobaltous chloride = 5.0 g
ii) Ammonium chloride = 10.0 g
iii) Ammonia = 40 ml
iv) H_2O_2 = 25 ml
v) Con. HCl = ….. ml.

Reactions: i) $H_2O_2 \longrightarrow H_2O + (O)$

ii) $2CoCl_2 + 2NH_4Cl + [O] \longrightarrow 2CoCl_3 + 2\,NH_3 + H_2O$

iii) $CoCl_3$ + 5 NH_3 ⟶ 

Calculations :

1) Theoretical yield

From the chemical reaction we get,

$CoCl_2.6H_2O$ = $[Co(NH_3)_5Cl]Cl_2$.

237. 93 = 250. 43

Now, 237.93 g $CoCl_2.6H_2O$ = 250.43 g of complex

∴ 5.g $CoCl_2.6H_2O$ = $\frac{5 \times 250.43}{237.93}$ g of complex

i.e. A = 5.269 g of the complex

2) Practical percentage yield

Wight of Product = X = …..g

Now, 5.26 g of complex = 100%

∴ 'X' g of complex = $\frac{\text{'X' x 100}}{5.26}$ %

i.e. B = ……. %

Result :

i)	Weight of product	= X = …….. g	= …….x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

9. Preparation of Tris(thiourea)cuprous sulphate

Aim : To Prepare of tris (thiourea) cuprous sulphate. $[Cu.3tu]_2SO_4.2H_2O$

Chemicals : 1) Copper sulphate , 2) Thiourea, 3) 1 N H_2SO_4 4) 5 % Thiourea

Procedure :

i) Weight 5 g thiourea and dissolve it in 30 ml hot distilled water by constant stirring ii) Weight 5 g of copper sulphate and dissolve in 30 ml. water. iii) Cool both the solution to room temperature. Add slowely the solution of copper sulphate to the thiourea solution with constant stirring. iv) Cool the mixture under a running tap water until the separated oil adheres to the wall of the beaker. Decant the mother liquour. v) Dissolve 4 g thiourea in 40 ml water, add it to oily layer with constant stirring. Keep the beaker in ice-cold water till the crystallization is complete. vi) Filter the solution and wash the crystals with distilled water.

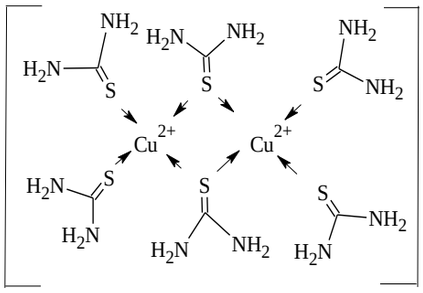
vii) **Recrystallition**: Dissolve the product in 50 ml solution of 5 % thiourea in a flat bottom flask add 1 ml of 1 N H_2SO_4. Heat the solution in water bath for 30 minutes (The solution may be heated to maximum of 75° C to dissolve the product) Cool the solution in ice bath. Filter & dry the product and record the weigh.

Observations: i) Copper Sulphate = 5.0 g

ii) Thiourea = 5.0g

iii) H_2SO_4, 1 N = 1.0 ml

Reactions:

$2CuSO_4 + (NH_2)_2SC + 2H_2O \longrightarrow$  $SO_4 \cdot 2H_2O$

Calculations :

1) Theoretical yield	2) Practical percentage yield
From the chemical reaction we get, $2CuSO_4.5H_2O \equiv [Cu.\ 3tu]_2\ SO_4.\ 2H_2O$ 499.366 ≡ 715.867 Now, 499.366 g ≡ 715.867 g complex ∴ 5 g $CuSo_4$ = $\frac{5 \times 715.867}{499.366}$ g complex = 7.167 g of the complex i.e A = 7.167 g complex	Weight of the product = x = …….g Now, 7.167 g of complex = 100% yield ∴ 'X' g of complex = $\frac{\text{'X' x 100 \%}}{7.167}$ i.e B = …….. %

Result :

i)	Weight of product	= X = …….. g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

10. Preparation of Hexaamminenickel(II) chloride

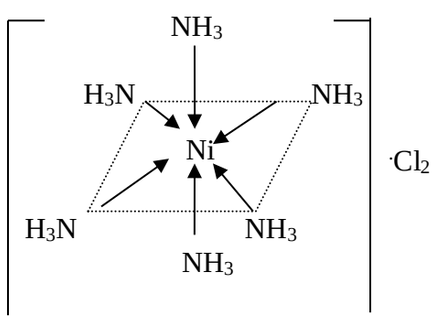
Aim : To Prepare of Hexaamminenickel(II) chloride. $[Ni(NH_3)_6]\ Cl_2$

Chemicals : 1) Nickel Chloride 2) Ammonia Buffer (pH 10) 3) Ethyl alcohol

Procedure :

i) Weigh 5 g Nickel Chloride and dissolve it in 20 ml warm distilled water by constant stirring ii) keep it in a ice bath for about 30 min. iii) Add 30 ml ammonia buffer with constant stirring until the blue ppt of Nickel hydroxide first formed is dissolves iv) Cool the solution in ice bath.The crystals of Hexaamminenickel(II) chloride separates out v) Add 50 ml ethyl alcohol. Filter the product on Buckner funneland wash with little alcohol. v) Dry the product and weigh it on rough balance

Reactions:

$NiCl_2 + 6NH_3 \longrightarrow$  $\cdot Cl_2$

Observations:

i) Nickel Chloride = 5.0 g
ii) Ammonia Buffer (pH 10) = 30 ml
iii) Ethyl alcohol = 50 ml

Calculations :

1) Theoretical yield	**2) Practical percentage yield**
From the chemical reaction we get, $NiCl_2.6H_2O \equiv [Ni(NH_3)_6]Cl_2$ 337.7 ≡ 231.71 Now, 337.7 g $NiCl_2.6H_2O \equiv$ 231.71 g complex ∴ 5 g $NiCl_2.6H_2O = \frac{5 \times 231.71}{337.7}$ g complex = 4.87 g of the complex i.e. A = 4.87 g complex	Weight of the product = X = …….g Now, 4.87 g of complex = 100% yield ∴ 'X' g of complex = $\frac{'X' \times 100}{4.87}$ % i.e B = ……... %

Result :

i)	Weight of product	= X = ……... g	= ……..x 10^{-3} kg
ii)	Theoretical yield	= A = ……..g	= ……..10^{-3} kg
iii)	Practical % yield	= B %	= ……..%

CHARACTERISTICS OF INORGANIC PREPARATIONS

Sr. No.	Characteristics	5.Potassiumtrioxalato aluminate(III)	6.Tris(en)nickel(II) thiosulphate	8.Chlopentaminecobalt(III) chloride	9.Tristhioureacuprous sulphate	10.Hexaminenickel(II) chloride
1.	Nature	Crystalline	Crystalline	Crystalline	Crystalline	Crystalline
2.	Color of the compound	White	Faint violet	Purple	White	Blue / Violet
3.	Central metal	Al	Ni	Co	Cu	Ni
4.	Oxidation state of metal ion	+3	+2	+3	+1	+2
5.	Nature of ligand	Bidentate Oxalato chelating	Bidentate Ethylenediammine Chelating	Monodentate Strong field=5, Weak field=1	Monodentate	Monodentate
6.	Nature of bonding	Covalent	Coordinate	Coordinate, Covalent	Coordinate	Coordinate
7.	Type of hybridization	sp^3d^2	sp^3d^2	d^2sp^3	sp^3	sp^3d^2
8.	Inner/ Outer orbital complex	Outer	Outer	Inner	-	Outer
9.	Geometry	Octahedral (See Page No.11)	Octahedral (See Page No.12)	Octahedral (See Page No.14)	Tetrahedral (See Page No.15)	Octahedral (See Page No.16)
10.	Magnetic property	Dimagnetic	Paramagnetic	Dimagnetic	Dimagnetic	Paramagnetic

III. TITRIMETRIC ESTIMATIONS

A) PERCENTAGE PURITY

11. Percentage Purity of Ferrous ammonium sulfate (Mohr's Salt)

Aim : To determine the percentage purity of the given sample containing crystalline Ferrous ammonium sulfate by using standard solution of Potassium dichromate.

Chemicals :

1) Given crystalline sample of Mohr's salt (FAS). 2) 0.1N $K_2Cr_2O_7$ solution.
3) Masking reagent (H_2SO_4 + H_3PO_4 mixture) 4) Diphenylamine indicator
5) Distilled water.

PROCEDURE : Part –I : Preparation of Sample solution : -

i) Weigh accurately ***about but not exactly*** **5.000 g** (4.800 g to 5.200 g) of the given sample of F.A.S., on a watch glass. ii) Transfer the sample to 250 ml beaker & wash the watch glass with distilled water in same beaker. Add about ½ T.T. dil. H_2SO_4 and dissolve. iii) Transfer the content of beaker to 250 ml measuring flask without wasting single drop of solution. Wash the beaker 2-3 times with distilled water. iv) Dilute the solution up to mark & shake well.

Part –II : Determination of % Purity : -

i) Pipette out 25 ml of diluted solution in a conical flask. ii) Add to it one test tube masking reagent carefully. iii) Then add to it 2-3 drops of Diphenylamine indicator and titrate against 0.1 N $K_2Cr_2O_7$ solution. End point is pale green to violet blue. iv) Repeat the procedure and find out CBR. Say X ml.

Part –I : Preparation of Sample solution

Observations :–

1)	Weight of empty watch glass	W_1 = …….g.
2)	Weight of sample	W = …….g.
3)	Weight of watch glass + sample (W_1+W)	W_2 = …….g.

Part –II : Determination of % Purity

Observations:-

1) In burette : 0.1 N $K_2Cr_2O_7$ solution

2) In conical flask : 25 ml dil. sample sol^n by pipette + (1 T.T. masking reagent.)

3) Indicator : Diphenylamine (2-3 drops)

4) End point : Pale green to violet blue.

Observation Table:-

Burette level	**Burette Reading in ml**			**C.B.R.**
	I	II	III	
Final level				X = …….ml
Initial level				

Difference				

Reactions :-

$K_2Cr_2O_7+6FeSO_4.(NH_4)_2SO_4+7H_2SO_4 \rightarrow K_2SO_4+ Cr_2(SO_4)_3+ 3Fe_2(SO_4)_3+ 6(NH_4)_2SO_4+7H_2O.$

Calculations:

We have

I) Weight of sample taken = W = ……g

II) C.B.R. X = ……ml.

1. Calculation of % purity –

1 ml of 0.1 N $K_2Cr_2O_7$ ≡ 0.0392 g FAS

∴ X ml of 0.1 N $K_2Cr_2O_7$ ≡ 0.0392 x X g FAS.

i.e. A = …….. g FAS.

Amount of FAS present in the 25 ml dil. sol^n = A g.

∴ Amount of FAS present in 250ml sol^n = A x 10 g

i.e. B = …….. g FAS.

Now B g of F.A.S. present in 250 ml diluted sample sol^n i.e. in the 'W'g of sample.

Then, we write

∴ W g of sample. = B g of FAS

∴ 100 g of sample = $\frac{B \times 100}{W}$ %

i.e. Percentage purity of F.A.S. = P = ……..%

2. Calculation of Volume of 0.1 N $K_2Cr_2O_7$ sol^n equivalent to 0.500 g of given sample

As $\frac{W}{10}$ g of sample requires = X ml of 0.1 $K_2Cr_2O_7$ sol^n

∴ $\frac{5.000}{10}$ OR 0.500 g of sample requires = $\frac{X \times 0.500 \times 10}{W}$ ml of 0.1 N $K_2Cr_2O_7$

i.e. V = …… ml of 0.1 N $K_2Cr_2O_7$

Results:

1	Weight of sample taken	W = ……. g
2	Volume of 0.1 N $K_2Cr_2O_7$ solution required for 25 ml diluted sample solution	X = ……ml
3	Volume of 0.1 N $K_2Cr_2O_7$ solution equivalent to 0.5 g of the given sample	V = ……ml
4	Percentage Purity of the given sample	P= ……..%

12. Percentage Purity of Tetraamminecopper (II) sulphate

Aim : To determine the percentage purity of the given sample containing crystalline Tetraaminecopper (II) sulphate monohydrate by redox titration using standard $Na_2S_2O_3$ Solution.

Chemicals : 1) Given sample of $[Cu(NH_3)_4]\ SO_4.H_2O$. 2) 1:1 NH_3

3) Standard 0.05 N $Na_2S_2O_3$ solution 4) 2 N Acetic acid

5) 10% KI solution 6) Starch Indicator

Procedure : Part – I :Preparation of sample Solution

i) Weigh accurately about ***but not exactly*** **1.500 g** (1.300 g to 1.700 g) of the given solid sample on watch glass. ii) Transfer it completely to 250 ml beaker and wash watch glass with distilled water in same beaker. iii) Add to it ½ T.T. 2N Acetic acid and dissolve the complex. iv) Now transfer the solution from beaker to 250 ml volumetric flask without wasting a single drop of solution. Dilute the solution up to mark carefully with distilled water & Shake well.

Part – II: Determination of % Purity

i) Pipette out 25 ml of diluted solution in conical flask. ii) Add 1:1 ammonia drop by drop till bluish precipitate appears. Now add drop-by-drop 2N Acetic acid till the precipitate formed first is just dissolves. iii) Now add 1 T.T. 10% KI solution and shake well. iv) Titrate liberated iodine against given 0.05 N $Na_2S_2O_3$ solution by using starch indicator. End point is blue to colorless (White ppt.) v) Repeat the procedure and find out CBR.

Observations – Part – I: Preparation of Sample Solution

1)	Weight of empty watch glass	W_1 = …….g.
2)	Weight of sample	W = …….g.
3)	Weight of watch glass + sample (W_1+W)	W_2 = …….g.

Part – II : Determination of Percentage purity

1) In burette : 0.05N $Na_2S_2O_3$ solution.

2) In conical flask : 25 ml diluted sample solution by pipette + [1:1 NH_3 till bluish ppt + 2 N Acetic acid sol^n till ppt dissolves + 1 T.T. 10% KI Solution]

3) Indicator : Starch (1 ml)

4) End point : Blue to colorless

Observation Table :

As previous experiment C.B.R. = X ml.

Reactions

1. $[Cu(NH_3)_4]SO_4 + 4CH_3COOH \rightarrow CuSO_4 + 4\,CH_3COONH_4$
2. $2CuSO_4 + 4KI \rightarrow 2CuI + 2K_2SO_4 + I_2$
3. $2\,Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$

Sodium tetra thionate

Calculations: We have

1) Weight of sample taken (W) = …….g.
2) CBR (X) = …….ml.

1) % Purity

As 1 ml of 0.05 $Na_2S_2O_3$ sol^n = 0.01229 g Complex

∴ X ml of 0.05N $Na_2S_2O_3$ sol^n = X x 0.01229 g complex

i. e. A = ……. g of complex

Now,

25 ml of diluted sol^n = A g complex

∴250 ml of diluted sol^n = Ax10 g of complex

i.e B =…… g of complex

The 250 ml diluted sol^n means W g of sample sol^n contains B g of complex .

We write

∴ W g of sample of $[Cu(NH_3)_4]SO_4.H_2O$ = B g of complex

∴ 100 g of sample of complex = $\frac{B \times 100}{W}$ g of complex

i.e. Percentage purity of complex of $[Cu(NH_3)_4]SO_4.H_2O$ = P =…….%.

2) Volume of 0. 05N $Na_2S_2O_3$ sol^n equivalent to 0.150 g of sample.

As $\frac{W}{10}$ g of sample = X ml of 0.05N $Na_2S_2O_3$ sol^n

∴ $\frac{1.500}{10}$ or **0.150** g of sample = $\frac{0.150 \times X \times 10}{W}$ ml of 0.05N $Na_2S_2O_3$ sol^n

i.e V = …… ml of 0.05N $Na_2S_2O_3$ sol^n

Results:

1	Weight of sample taken	W = ……. g
2	Volume of 0.05 N $Na_2S_2O_3.5H_20$ sol^n required for 25 ml diluted sample solution	X = ……ml
3	Volume of 0.1 N $Na_2S_2O_3.5H_20$ Solution equivalent to 0.150 g of the given sample	V = ……ml
4	Percentage Purity of the given sample	P= ……..%

13. Percentage Purity of Potassium trioxalatoaluminate(III)

Aim : To determine the percentage purity of the given sample containing crystalline $K_3[Al(C_2O_4)_3.\ 3H_2O$.

Chemicals : 1) Given sample of $K_3[Al(C_2O_4)_3.3H_2O$

2) Standard sol^n of 0.1 N $KMnO_4$

PROCEDURE **Part –I:Preparation of Sample solution: -**

i) Weigh accurately about ***but not exactly*** **1.450 g** (1.250 g to 1.650 g) of the given sample of crystalline Potassium trioxalatoaluminate (III) on a watch glass. ii) Transfer the sample to 250ml beaker and wash the watch glass with distilled water in same beaker. iii) Dissolve the solid and add ½ T.T. 2N H_2SO_4. iv) Now transfer the contents to 250ml volumetric flask without wasting a single drop of solution. v) Wash the beaker 2-3 times with distilled water and transfer to flask & dilute the solution up to mark with distilled water. Shake well the solution.

Part - II: Determination of % Purity

i) Pipette out 25 ml of diluted solution in a conical flask. ii) Add to it about one T.T. 2 N sulphuric acid. iii) Heat the content of the flask to about 80^o C **on boiling water bath** & titrate this solution against 0.1 N $KMnO_4$ sol^n. End point is colorless to permanent faint pink. iv) Repeat the procedure and find out C.B.R. say 'X'ml.

Observations – Part – I: Preparation of sample solution

1)	Weight of empty watch glass	W_1 = …….g.
2)	Weight of sample	W = …….g.
3)	Weight of watch glass + sample (W_1+W)	W_2 = …….g.

Part - II: Determination of % Purity

1) In burette : 0.1 N $KMnO_4$ solution
2) In conical flask : 25 ml diluted sample solution by pipette +
[1 T.T 2N H_2SO_4 + Heat to 80^{o}C on boiling water bath]
3) Indicator : $KMnO_4$ itself
4) End point **:** Colorless to permanent faint pink

Observation Table:

As previous experiment CBR = X ml

Reactions : i) $2K_3[Al(C_2O_4)_3] + 6H_2SO_4 \rightarrow 3K_2SO_4 + Al_2(SO_4)_3 + 6H_2C_2O_4$

ii) $2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$

Calculations:. 1) Weight of sample taken (W) = …….g. 2) CBR =X = …..g.

1) % Oxalate group :

$\therefore$ 1ml of 0.1 N $KMnO_4$ sol^n = 0.0044 g of oxalato group

$\therefore$ X ml of 0.1 N $KMnO_4$ sol^n = X x 0.0044 g of oxalato group

i.e. A = …….. g of oxalato group

Now, 25 ml of diluted sample sol^n = A g of oxalato group

$\therefore$ 250ml of diluted sample sol^n = A x 10 g of oxalato group

i.e. B = ……..g of oxalato group

Hence,

W g of sample = B g of oxalato group

$\therefore$ 100 g of sample $= \frac{100 \times B}{W}$ %

i.e. P = ……..%

2) Calculation of Purity of $K_3[Al(C_2O_4)_3] . 3H_2O$ Complex

a) % Oxalato group (Theoretically).

$K_3[Al(C_2O_4)_3] . 3H_2O = 3 (C_2O_4)$

462 = 264

i. e. 462 g of complex = 264 g of oxalato group.

$\therefore$ 100 g of complex $= \frac{264 \times 100}{462}$ g of oxalato group

= 57.15 %.

Thus theoretical % of oxalato group in complex = 57.15%

b) Percentage purity of complex $K_3[Al(C_2O_4)_3] . 3H_2O$

$\therefore$57.15 % oxalato group in complex = 100% Purity of complex

$\therefore$ P % oxalato group in given complex = $\frac{P \times 100}{57.15}$ %

i.e. C =……..%

3) Calculation of Volume of 0.1 N $KMnO_4$ Equivalent to 0.145 g of the given sample of $K_3[Al(C_2O_4)_3] . 3H_2O$

$\therefore$ $\frac{W}{10}$ g sample = X ml of 0.1 N $KMnO_4$

$\therefore$ $\frac{1.450}{10}$ **or** 0.145 g of sample = $\frac{0.145 \times X \times 10}{W}$ ml

i.e. V =…….. ml

Results :

1	Weight of sample taken	W= ……. g
2	Volume of 0.1 N $KMnO_4$ sol^n required for 25 ml diluted sample solution	X = ……ml
3	Volume of 0.1N $KMnO_4$ sol^n equivalent to 0.145 g of the sample	V = ……ml
4	Percentage of the oxalato group in the given sample	P= ……..%
5	Percentage purity of the given complex	C=……%

B) ANALYSIS OF COMMERCIAL SAMPLE

14. Percentage of Magnesium in Talcum Powder

Aim **:**To determine the percentage of magnesium in the given sample of talcum powder by complexometric titration, using standard 0.01M EDTA solution.

Chemicals : 1) Given sample of talcum powder 2) 0.01M EDTA solution

3) Buffer Solution of pH-10 4) Eriochrome Black – T indicator,
. 5) Distilled water

Composition of Talcum powder : Zinc oxide, Magnesium oxide, Boric Acid, Scent, Sandal / Starch Antifungal & antibacterial agents

Procedure : Part – I : Preparation of Solution

i) Weigh accurately about ***but not exactly*, 1.5 g** (1.400 g – 1.800 g) of the given sample of talcum powder on a watch glass. ii) Transfer sample completely to 100 ml (small) beaker. iii) Wash the watch glass with water and add 10 ml conc. HCl in same beaker. iv) Heat the solution till volume reduces to about 2 ml. v) Now cool beaker on asbestos sheet and add again 2 T.T. distilled water and boil again. vi) Cool well, and filter the solution through Whatman paper No.1 or ordinary paper in a 250 ml measuring flask. vii) Wash the beaker as well as filter paper and collect washing in same measuring flask. viii) Finally dilute the solution up to mark with distilled water carefully & shake well the solution.

Part - II : Determination of % Magnesium

i) Pipette out 25ml diluted sample solution is a conical flask. ii) Add about 2 T.T. distilled water and about 1/3 T.T.(5ml) buffer solution of pH 10. iii) Titrate this solution against given standard 0.01 M EDTA solution by using Eriochrome Black –T indicator. End point is wine red to sky blue. iv) Repeat the procedure and find out C.B.R. Say it is 'X' ml.

Observations: Part – I : Preparation of Solution

1)	Weight of empty watch glass	W_1 = …….g.
2)	Weight of sample	W = …….g.
3)	Weight of watch glass + sample (W_1+W)	W_2 = …….g.

Part – II: Determination of % Magnesium

1) **In burette :** 0.01M EDTA solution.
2) **In conical flask:** 25 ml diluted sample + [2T.T.Dist.H_2O+1/3 T.T.Buffer Sol^n of pH 10]
3) **Indicator :** Eriochrome Black-T [3-4 drops or pinch of solid]
4) **End point :** Wine red to sky blue.

Reactions : i] $MgO + 2HCl \rightarrow MgCl_2 + H_2O$

ii] $MgCl_2 + Na_2H_2$ EDTA $\rightarrow Na_2$ [Mg EDTA] + 2HCl

Observation Table : Same as in Volumetric Analysis – 1, CBR = Xml.

Calculations : We have

1. Weight of sample taken (W) = …….g.
2. C.B.R . (X) = ……ml.

1) % of Magnesium Then, 1 ml of 0.01 M EDTA= 0.0002431 g Mg ∴Xml of 0.01N M EDTA=0.0002431xXg Mg. i. e. A = ……g of Mg. Now, 25 ml of diluted sample sol^n = A g of Mg.	Thus, W g of sample = B g of Mg. ∴ 100 g of sample = $\frac{B \times 100}{W}$ g of Mg. i.e. P =……% of Mg.

∴ 250 ml of diluted sol^n = Ax10 g of Mg. ∴ B = ……. g of Mg.	

3) Volume of 0.01M EDTA equivalent to 0.150 g of talcum powder.

As $\frac{W}{10}$ g of sample talcum powder = X ml 0.01 M EDTA

∴ $\frac{1.500}{10}$ or 0.150 g of talcum powder = $\frac{0.150 \times X \times 10}{W}$ ml 0.01 M EDTA

i.e V =…… ml

Results:

1	Weight of sample taken	W= ……. g
2	Volume of 0.01 M EDTA solution required for 25 ml diluted sample solution	X = ……ml
3	Volume of 0.01 M EDTA solution equivalent to 0.150 g of the sample	V = ……ml
4	Percentage of Magnesium in the given talcum powder	P= ……..%

15. Amount of Aluminum in Potash Alum

Aim : To determine the amount of Aluminum in potash alum by complexmetric titration (Indirect method) using standard solution of EDTA and Zinc Sulphate

Chemicals: 1) Given sample solution of potash alum in 250 ml volumetric flask

2) 0.01M EDTA 3) 0.01 M $ZnSO_4$, sol^n 4) Ammonia (1:1)

5) Eriochrome Black – T 6) Distilled water

Procedure : A) Back Titration:

i) Dilute the given solution of potash alum to 250 ml distilled water and Shake well. I) Pipette out 25ml of this diluted solution in conical flask. iii)Add to it 25 ml 0.01 M EDTA solution by pipette. Maintain pH between 7 - 8 by addition of 1:1 ammonia (5 ml) test with red litmus paper it should turn blue. iv) Boil the solution for few min. v) Cool the solution to room temperature. vi) Add again 5ml 1:1 Ammonia solution and Eriochrome Black –T indicator and shake well vii) Titrate this solution against 0.01 M Zinc sulphate solution. End point is sky blue to wine red. viii) Repeat the procedure and find out CBR say it is 'Y' ml.

B) Blank Titration :

i) Pipette out 25 ml of standard 0.01 M EDTA sol^n in a conical flask. ii) Add 5 ml of 1:1 Ammonia to adjust pH of sol^n 7-8. iii) Then add 2-3 drops of Eriochrome Black-T indicator.

iv) Titrate this sol^n against 0.01 M Zinc Sulphate solution. End point is sky blue to win red. v) Repet the procedure and find CBR say it is 'X' ml.

Observations

A) Back Titration:	**B) Blank Titration :**
In burette : 0.01M Zinc sulphate sol^n	**In burette :** 0.01M Zinc sulphate sol^n
In conical flask : 25ml diluted Potash alum sol^n + 25 ml 0.01MEDTA + 5 ml Ammonia(1:1) sol^n $\xrightarrow{\text{Boil \& Cool}}$ + 5ml 1:1 NH_3 sol^n	**In conical flask :** 25 ml 0.01M EDTA + 5 ml Ammonia(1:1) sol^n
Indicator : Eriochrome Black-T (5-6 drops OR Pinch of solid)	**Indicator :** Eriochrome Black-T (5-6 drops OR Pinch of solid)
End point : Sky blue to Wine red.	**End point :** Sky blue to Wine red.

Observation Table :

A) Back Titration : As usual : C.B.R.= Y =……ml

B) Blank Titration : As usual : C.B.R.= X =……ml

Reactions: i) $Al^{+3} + Na_2H_2EDTA \rightarrow Na[Al\ EDTA] + Na^+ + 2H^+$

ii) $Zn^{++} + Na_2H_2EDTA \rightarrow Na_2[Zn\ EDTA] + 2H^+$

Calculations :

A] CBR of Back = Y =……ml.

B] CBR of Blank = X =……. ml

∴ **X-Y = Z ml of 0.01 M $ZnSO_4$ i.e. Z ml EDTA required for complexation of Al.**

1) Amount of Aluminium:

As 1ml of 0.01M $ZnSO_4$ (i.e. EDTA) = 0.0002698 g Al.

∴ Z ml of 0.01 M $ZnSO_4$ sol^n = 0.0002698 x Z g Al

i.e. A = … ……. g Al

Now, 25 ml diluted potash alum sol^n = A g Al

∴ 250 ml of diluted potash alum sol^n = A x 10 g Al

i.e. B = ……g Al.

2) **Amount of Potash alum:**

As 1ml of 0.01M $ZnSO_4$ (i.e. EDTA) = 0.004744 g of Potash alum.

∴ Z ml of 0.01 M $ZnSO_4$ sol^n = 0.004744 x Z g of Potash alum

i.e. C =…… g of Potash alum.

Now ,25 ml diluted sol^n = C g of Potash alum.

∴ 250 ml of diluted sol^n = C x10 g of Potash alum

i.e D =…… g of Potash alum

Results :

1	Blank titration reading	(X)	=…...ml	
2	Back titration reading	(Y)	= ……ml	

3	Difference between blank and back titration readings	(X-Y)=Z	=…….ml	
4	Quantity of Aluminum in the given sample	(B)	= ……g	=……x10^{-3} kg
5	Quantity of Potash Alum in the given sample	(D)	= ……g	=…….x10^{-3} kg

16. Titrable Aciditv of Milk/ Lassi

Aim : Determine titrable acidity of the supplied sample of milk / lassi by acid base titration.

Chemicals : 1) $H_2C_2O_4$ crystals 2) 0.1 N NaOH
3) Phenolphthalein 4) Sample of milk etc.

Theory :

Freshly drawn milk is amphoteric i.e. litmus turns blue and vice versa. It shows acidity as determined by titration with an alkali like NaOH using phenolphthalein. This acidity i.e. tritrable acidity (T.A.) as it is determined by titration is known as “natural’ (or apparent) acidity and is caused by presence of casein, acid phosphates, citrates in milk. The higher the solids but not fat content in milk, the higher the natural acidity (N.A.) and vice versa. The T.A. of cow milk is **0.13 to 14 %** and for buffalo milk is **0.14 to 0.15 %.** Developed or real acidity is due to lactic acid formed as a result of bacterial action on lactose in milk. Hence T.A. of stored milk is equal to the sum of natural acidity and developed acidity. It is expressed as a percentage of lactic acid.

Procedure: Part – I: Standardization of NaOH

1) Weigh exactly 1.575 g Oxalic acid crystals on a watch glass and transfer them in a beaker. Dissolve it in minimum quantity distilled water and transfer this solution to 250 ml volumetric flask. Rinse the beaker 2-3 times using distilled water and collect the washing in same volumetric flask. Dilute the content up to the mark with distilled water and shake well. It gives standard 0.1N Oxalic acid solution.

2) Pipette out 25 ml of 0.1 N $H_2C_2O_4$ in conical flask add two drops phenolphthalein indicator and titrate this solution against supplied 0.1 N NaOH solution from the burette till colour changes from colourless to faint pink.

3) Take three readings and note the C.B.R. as ‘X’ ml.

4) Form this calculate the exact normality of NaOH using $N_1V_1 = N_2V_2$ relation

Part – II: Titrable Acidity of Milk/ Lassi (Industrial Method)

1) Take **two** different volume, of milk/Lassi by common burette (Wg) :

10	15	20	25	30	35	ml of milk / lassi

Add to it one test tube **hot distilled water**.

2) Add phenolphthalein indicator to the milk sample taken in the conical flask.

3) Titrate this solution against standardized NaOH solution from burette till colur change from colorless to faint pink. Take more readings using the same procedure and note down CBR as V_1 ml for **Set I**

4) Repeat the experiment for another volume of milk or lassi by using same procedure and note down CBR as V_2 ml for **Set II**

Note: i] Titration should be completed within 20 sec. with constant stirring.

ii] Assume the density of Milk /Lassi=1.0

Observations and Observation Tables:

Part – I: Standardisation of NaOH

Observations [a] :

1)	Weight of empty watch glass	W_1 = …….g.
2)	Weight of sample	W = …….g.
3)	Weight of watch glass + sample (W_1+W)	W_2 = …….g.

Observations [b]

In burette : 0.1N NaOH

In conical flask : 25ml 0.1 N $H_2C_2O_4$

Indicator :Phenolphthalein

End point **:**Colourless to pink

Observation Table

Burette Level	Burette Reading in ml			CBR
	I	II	III	
Final Level				X =…..ml
Initial Level				
Difference				

Reactions: $H_2C_2O_4 + 2NaOH \rightarrow Na_2C_2O_4 + 2H_2O$

Part – II: Titrable Acidity of Milk/ Lassi (Industrial Method)

OBSERVATIONS &OBSERVATION TABLE

Set:I

Observations

In burette : 0.1N NaOH

In conical flask : …..ml milk / lassi

Indicator :Phenolphthalein

End point **:**Colourless to pink

Observation Table

Burette Level	Burette Reading in ml			CBR
	I	II	III	
Final Level				V_1=…..ml
Initial Level				
Difference				

Set:II

Observations

In burette : 0.1N NaOH

In conical flask : …..ml milk / lassi

Indicator :Phenolphthalein

End point **:**Colourless to pink

Observation Table

Burette Level	Burette Reading in ml			CBR
	I	II	III	
Final Level				V_2=…..ml
Initial Level				
Difference				

Calculations:

Part : I: Standardisation of NaOH sol^{n}

NaOH V/S $H_2C_2O_4$

N_1V_1 = N_2V_2

$N_1 \times X$ = 0.1×25

N_1 = $\frac{0.1 \times 25}{X}$

= ……N NaOH

Part – II: Titrable Acidity of Milk/ Lassi (Industrial Method)

We have

Set	Volume / Mass of milk	Titration Reading
I	W =……g	V_1 =……ml
II	W =……g	V_2 =……ml

Calculation of Titrable Acidity Of Milk/Lassi -

Calculate the titrable acidity of milk/ lassi in terms of % of Lactic acid using the relation -

1000 ml ≡ 1 N NaOH ≡ 90.0 g lactic acid

Set : I :	Set : II :
∴ Tritable acidity = $\frac{9 \times V_1 \times N_1}{W}$ % Lactic acid	∴ Tritable acidity = $\frac{9 \times V_2 \times N_1}{W}$ % Lactic acid
Where: V_1 = Titration reading Set –I N_1 = Normality of NaOH W= Mass of milk/lassi in g.	Where: V_2 = Titration reading Set –II N_1 = Normality of NaOH W= Mass of milk/lassi in g.

Result :

1	Volume of supplied NaOH required for 25 ml 0.1N $H_2C_2O_4$	X=……ml
2	The Titrable acidity of Milk / Lassi	Set I = …… % lactic acid Set II = …… % lactic acid

17. Chemical Oxygen Demand of Industrial Effluent

Aim : Determination of Chemical Oxygen Demand (C.O.D.) of the given Sample of industrial effluent using 0.1 N $FeSO_4(NH_4)_2SO_4$ solution

Chemicals: **1.Given** Sewage/ Industrial effluent sample,
2. Conical flask with reflux condenser
3.$HgSO_4$ crystal
4. Ag_2SO_4 crystal
5. Conc. H_2SO_4
6. 0.25 N $K_2Cr_2O_7$ sol^{n}
7. Standard 0.1N F. A.S.
8. Ferroin indicator

Theory: Chemical Oxygen Demand **(**COD) is a measure of oxygen consumed during the oxidation of the **oxidisable organic matter** by a strong oxidizing agent, $K_2Cr_2O_7$ in presence of H_2SO_4 which is generally used as an oxidizing agent in determination of C.O.D.

The sample is refluxed with $K_2Cr_2O_7$ and H_2SO_4 in presence of mercuric sulphate to neutralize the effect of chlorine and silver and Ag_2SO_4 (catalyst). The excess of

$K_2Cr_2O_7$ is titrated against ferrous ammonium sulphate using **ferroin as an indicator**. The amount of $K_2Cr_2O_7$ used is proportional to the oxidisable organic matter in sample. C.O.D. of water or industrial effluent is calculated in terms of grams or ppm of oxygen.

Procedure: A) Back Titration :

1) To the sample of industrial effluent supplied in 250 ml conical flask add 10 ml of 0.25N $K_2Cr_2O_7$ solution and a few glass beads. 2) Then add slowly and in small portions at a time, with shaking 30 ml of conc. H_2SO_4. 3) Now add 10 mg H_gSO_4 and 10 mg Ag_2SO_4 crystals and shake well. 4) Reflux the flask for two hours using water condenser. 5) Cool & add 50 ml distilled water and 3-4 drops of ferroin indicator. Shake well. 6) Titrate this whole solution containing excess of $K_2Cr_2O_7$ against 0.1 N $FeSO_4$ $(NH_4)_2SO_4$ solution. 7) End point is blue green to wine-red. 8) Note this reading as 'Y' ml.

B) Blank Titration:

1) Pipette out 25 ml distilled water in another 250 ml conical flask 2) Add 10 ml 0.25 N $K_2Cr_2O_7$ solution and add a few glass beads. 3) Then add slowly and in small portions at a time with shaking, 30 ml of conc. H_2SO_4. 4) Now add 10 mg $HgSO_4$ and 10 mg Ag_2SO_4 crystals and shake well. 5) Reflux the flask for two hours using water condenser. 6) Cool & add about 50ml distilled water and 3-4 drops of ferroin indicator & Shake well. 7) Titrate this whole solution against 0.1 N $FeSO_4$. $(NH_4)_2SO_4$ solution. 8) End point is blue-green to wine-red. 9) Note this reading as X ml. 10) Ask for volume of supplied sample V ml and its dilution factor (Df).

Observations :

Back titration :

1) In burette : 0.1 N $FeSO_4.(NH_4)_2SO_4$ solution
2) In conical flask : Refluxed sample solution
3) Indicator : Ferroin
4) End point **:** Blue-green to wine-red.

Reaction :

$$K_2Cr_2O_7+6FeSO_4.(NH_4)_2SO_4+7HgSO_4 \rightarrow K_2SO_4+Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 6(NH_4)_2SO_4 + 7H_2O.$$

Blank titration :

1) In burette : 0.1 N $FeSO_4$. $(NH_4)_2SO_4$ solution
2) In conical flask : Refluxed sample solution
3) Indicator : Ferroin
4) End point : Blue-green to wine-red.

Reaction : As in back titration

Observations:

1	Sample dilution factor (Df)	: ……
2	Volume of sample supplied	: V =……ml
3	Back titration reading	: Y = ……ml
4	Blank titration reading	: X =…….ml

5	Difference of Blank and Back readings	: (X-Y)=……. ml 0.1N $FeSO_4$ $(NH_4)_2SO_4$
6	Given sample	: = ……. ml.

Calculations : Calculate C.O.D of the sample using following relation.

1 ml 0.1 N $FeSO_4$. $(NH_4)_2SO_4$ solution ≡ 0.8 mg of oxygen

$$\therefore \text{C.O.D of given sample} = \frac{(X-Y) \times \text{Equivalent wt of } O_2 \times \text{Normality of FAS} \times 1000 \times Df.}{\text{ml of sample take (V)}}$$

$$= \frac{(X-Y) \times 8 \times 0.1 \times 1000 \times Df.}{V}$$

$$= \frac{(X-Y) \times 800 \times Df.}{V} \quad mg/dm^3$$

Results :

1)	Given sample requires(X-Y)	=……ml 0.1N $FeSO_4$ $(NH_4)_2SO_4$ solution
2)	C.O.D. of sample	= ……. mg/dm^3

C) ION EXCHANGE METHOD OF SEPERATION

18. ESTIMATION OF MAGNESIUM AND ZINC BY ION EXCHANGE METHOD

Aim : To determine the amount of magnesium and zinc in the given solution containing $[Mg^{2+} + Zn^{2+}]$ using anion exchange resin by complexometric titration.

Chemicals :

1. 0.01 M EDTA solution
2. Eriochrome black –T indicator
3. Ammonia buffer, pH 10
4. 2 N NaOH
5. 2N HCl
6. 0.25N HNO_3
7. Strong base anion exchange resin Amberlite IR- 400 provided in ion exchange column in chloride form.
8. 100 ml volumetric flask containing given solution of $[Mg^{2+} + Zn^{2+}]$ in 2N HCl.

Apparatus : 1. Ion exchange column 2. Pipettes (10 ml,25 ml) 3. Conical flask 4. Volumetric flask 250 ml 5. Measuring cylinder 50 ml/100 ml 6.Volumetric flask 250 ml

Theory : Ion exchange chromatography is technique of separation in which exchange of ions having similar charges takes place between solid phase, exchange resin and solution. Ions are separated on the basis of their differential migration and estimated separately. When solution of $[Mg^{2+} + Zn^{2+}]$ ions is passed through anion resin column, Zn^{2+} ions are adsorbed on resin column while Mg^{2+} ions remain in effluent which are estimated titramitcally with EDTA.

Here the element Magnesium is belonging to alkaline earth metal, while Zinc is transition metal. In weak chloride medium (2M HCl) alkaline earth metals will be present in the solution in cationic forms [e.g. Mg^{2+}], whereas the transition

metals will be present in anionic forms [e.g. $ZnCl_4^{2-}$].Hence only Zn^{2+} ions are adsorbed on anion resin, while Mg^{2+}passes through the effulant.

Procedure :

Part I: Separation of Mg^{2+} & Zn^{2+}

SEPARATION OF Mg^{2+}

i) Dilute the given solution to 100 ml with 2N HCl.

ii) Wash the ion exchange column with (a) 50 ml distilled water
(b) 50 ml of 0.25 N HNO_3
(c) 50 ml 2N HCl and
(d) 50ml distilled water.

Drain the liquid from the column to almost bed level, keeping it about 1 cm above the surface of resin (Throw away washing)

iii) Adjust a clean 250 ml volumetric flask to collect the effluent of Mg^{2+}.

iv) Now pipette out 10 ml of diluted [Mg^{2+} + Zn^{2+}] solution and transfer it to the top of resin column.

v) Adjust the flow of effluent at the rate of 3-5 ml per minute and collect it in 250 ml volumetric flask. Wash the column with about 50 ml 2N HCl and entire effluent in the same measuring flask at the same rate and thus get all the Mg^{2+} separated out.

vi) Dilute this effluent to 250 ml with distilled water shake well and use this solution to estimate Mg^{2+}.

SEPARATION OF Zn^{2+}

vii) Adjust clean 250 ml standard 250 volumetric flask to collect next effluent of Zn^{2+} ions.

viii) Now wash the column with 50 ml distilled water and then with 50 ml of 0.25 N HNO_3 (elute) and collect the all effluent [washing + elute] at the rate of 3 to 5 ml per minute in same 250 ml volumetric flask.

ix) Dilute the effluent solution to 250 ml with distilled water and use this solution to estimate Zn^{2+}.

Part II: Estimation of Magnesium

i) Pipette out 25 ml of diluted effluent in conical flask, add one piece of red litmus and neutralize excess HCl by adding drop wise 2N NaOH by **common burette** with shaking till litmus becomes just blue.

ii) Now add ammonia buffer (3/4 T.T.) and 3-4 drops or pinch of Eriochrome black-T indicator and titrate this solution against 0.01 M EDTA till the colour changes form wine red to sky blue.

iii) Take three reading and note C.B.R. as X ml.

Part III : Estimation of Zinc

i) Pipette out 25 ml diluted effluent in conical flask, add piece of red litmus and neutralize excess HNO_3 by adding drop wise 2N NaOH by **common burette** with shaking till litmus becomes just blue.

ii) Now add ammonia buffer (3/4 T.T.) and 3-4 drops or pinch of Eriochrome black-T indicator and titrate with 0.01M EDTA till colour changes form wine red to sky blue.

iii) Take three reading and note C.B.R. as Y ml.

OBSERVATIONS & OBSERVATION TABLE

Part I : Separation of Magnesium and Zinc :

1.	Given solution of [Mg^{2+} + Zn^{2+}] diluted to	100 ml
2.	Amount of given dil.solution used for ion exchange separation	10 ml

3.	Effluent solution diluted to	250 ml
4.	Diluted effluent solution used for estimation	25ml

Part II : Estimation of Magnesium

Observations :

1. Sol^n in burette :	-0.01M EDTA solution
2. Sol^n in conical flask :	-25 ml diluted effluent sol^n of Mg (neutralize this solution by adding 2 N NaOH + ammonia buffer
3.Indicator :	3/4T.T.)
4.End point :	-Pinch of solid Eriochrome black -T Wine red to sky blue

Observation Table :

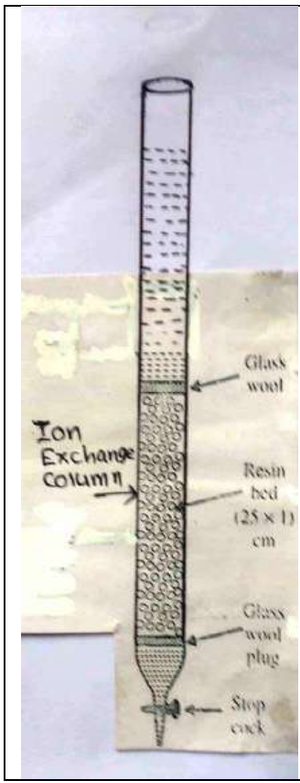
Burette level	Burette Reading in ml			C.B.R.
	I	II	III	
Final level				X=…….ml
Initial level				
Difference				

Part III : Estimation of Zinc

Observations :

1. Sol^n in burette :	-0.01M EDTA solution
2. Sol^n in conical flask :	-25 ml diluted effluent sol^n of Zn (neutralize this solution by adding 2 N NaOH + ammonia buffer 3/4
3.Indicator :	T.T.) -Pinch of solid Eriochrome black -T
4.End point :	-Wine red to sky blue

Observation Table : As previous, C.B.R.=Y=……ml



Reactions :

1) $Mg^{2+} + 2Cl^- \longrightarrow MgCl_2$

2) $Zn^{2+} + 4Cl^- \longrightarrow ZnCl_4^{2-}$

3) $2[(Res.NMe_3^+)Cl^-]_{(s)} + ZnCl_4^{2-}{}_{(sol)} + MgCl_{2\ (sol)} \longrightarrow [(Res.NMe_3^+)_2\ ZnCl_4^{2-}]_{(s)} + 2Cl^-_{(sol)} + MgCl_{2\ (sol)}$

4) $MgCl_2 + Na_2H_2EDTA \longrightarrow Na_2MgEDTA + 2HCl$

5) $[(Res.NMe_3^+)_2\ ZnCl_4^{2-}] + 2HNO_3 \longrightarrow [(Res.NMe_3^+)Cl^-] + Zn(NO_3)_2 + 2HCl$

6) $Zn(NO_3)_2 + Na_2H_2EDTA \longrightarrow Na_2[ZnEDTA] + 2\ HNO_3$

Calculations :

Part II: Estimation of Magnesium	**Part III : Estimation of zinc**
From the above equation we get	From the above equation we get
$Na_2H_2EDTA = Mg^{2+}$	$Na_2H_2EDTA = Zn^{2+}$
1000 ml 1M EDTA = 1 mole of Mg^{2+}	1000 ml 1M EDTA = 1 mole of Zn^{2+}

i.e. 1000 ml 1 M EDTA = 24.31g Mg^{2+} Now 1 ml 0.01 M EDTA = 0.0002431 g Mg^{2+} ∴ X ml 0.01 M EDTA = X x 0.000243 g Mg^{2+} i.e. A = ……. g Mg^{2+} Thus Amount of Mg^{2+} in 25 ml effluent sol^{n} =A g Amount of Mg^{2+} in250 ml effluent sol^{n} = Ax10 g i.e. B = …….g Here , Amount of Mg^{2+} in 10 ml dil. given sol^{n} = B g Amount of Mg^{2+} in100 ml dil.given sol^{n} = B x 10 g i.e. C = ……..g	i.e. 1000 ml 1M EDTA = 65.38 g Zn^{2+} Now, 1 ml of 0.01 M EDTA = 0.0006538 g Zn^{2+} ∴Y ml of 0.01 M EDTA = 0.0006538 x X g of Zn^{2+} i.e. D = ……..g Zn^{2+} Thus Amount of Zn^{2+} in 25 ml effluent sol^{n} = D g Amount of Zn^{2+} in 250 ml effluent sol^{n} = D x 10 g i.e. E = …….g Here, Amount of Zn^{2+} in10 ml diluted given sol^{n} = E g Amount of Zn^{2+} in100 ml diluted given sol^{n}= E x10 g i.e. F = …… g

Results :

1.	25 ml diluted effluent sol^{n} containing Mg^{2+} ions required 0.01 M EDTA sol^{n}	X	=……ml
2.	25 ml of diluted effluent sol^{n} containing Zn^{2+} ions required 0.01 M EDTA sol^{n}	Y	=……ml
3.	Quantity of Mg^{2+} in the given sol^{n}	= C =……g	= ……..x 10^{-3} kg
4	Quantity of Zn^{2+} in the given sol^{n}	= F =……g	= ……. x 10^{-3} kg

19. ESTIMATION OF SODIUM BY ION EXCHANGE METHOD

AIM: To determine the amount of sodium present in the given sample of common salt, using cation exchange resin followed by acid-base titration.

CHEMICALS: **1.** Strongly acidic cation exchange resin Amberlite IR- 120 provided in ion exchange column in H form.

2. Stock solution of common salt in 100 cm^3 measuring flask.

3. 0.05N NaOH solution. **4.** Phenolphthalein indicator **5.** Distilled Water.

APPARATUS: 1.Ion exchange column,2. Burette, 3. Pipettes(10 and 25 cm^3) 4. Conical Flask, 5. Funnel, 6. Beakers, 7. 100 and 250 cm^3, standard measuring flasks etc.

Theory : When common salt solution free from other cations, is passed through a strongly acidic cation exchange resin(in hydrogen form), an equivalent amount of H^+ ions are displaced by sodium ions from the given solution. The displaced H^+ ion solution can be titrated against standard solution of strong base which gives the amount of sodium present.

POCEDURE:

Part –A: Separation of sodium ion using cation exchanger :-

1. Dilute the given stock solution of common salt with distilled water up to 100 mL mark in measuring flask. 2. Wash the ion exchange column with 100 mL distilled

water to remove the acid present in column if any. 4. Drain the column and adjust the liquid level 1 cm above the resin bed. 5. Adjust the clean 250 cm^3 standard measuring flask to collect the effluent from column. 6. Pipette out 10 cm^3 of diluted salt solution and transfer it quantitatively to the top of the column. 7.Adjust the flow rate of the effluent at 4-5 mL per min and collect the effluent in 250 cm^3 standard measuring flask. 8. Now wash the column with 100 cm^3 distilled water maintaining the same flow rate and collect the washing (Effluent) in the same measuring flask. 9. Dilute the effluent up to 250 cm^3 mark with distilled water.

Part- B: Estimation of Sodium:-

1. Fill the burette with 0.05 N NaOH by taking usual precautions. 2. Pipette out 25 cm^3 of diluted effluent in a conical flask, add to it 2-3 drops of Phenolphthalein indicator and titrate it against 0.05N NaOH till colour changes colourless to pink. 3. Find out CBR and say it is 'X' cm^3

OSERVATIONS:

Part –A: Separation of sodium ion using cation exchanger :-

1.	Given salt solution diluted to	100 ml
2.	Amount of given diluted solution used for ion exchange separation	10 ml
3.	Effluent solution diluted to	250 ml
4.	Diluted effluent solution used for estimation	25ml

Part- B: Estimation of Sodium:-

1. Solution in burette - 0.05N NaOH
2. Solution in conical flask - 25 cm^3 diluted effluent solution.
3. Indicator - Phenolphthalein
4. End point Colourless to pink

Observation Table: As per previous experiments

CBR = X =……..cm^3

REACTIONS:

$$NaCl + Res^-H^+ \longrightarrow Res^-Na^+ + H^+Cl^-$$

$$H^+Cl^- + NaOH \longrightarrow NaCl + H_2O$$

CALCULATIONS:

❖ We have CBR = X =……..cm^3

25 cm^3 of dil. effluent solution = X cm^3 of 0.05 N NaOH solution.

Therefore, 250 cm^3 dil. effluent solution = X x 10 cm^3 of 0.05 N NaOH Sol^n

i.e. 10 cm^3 of diluted salt solution = X x 10 cm^3 of 0.05 N NaOH solution

Therefore, 100 cm^3 of dil. salt solution = X x 10 x 10 cm^3 of 0.05 N NaOH sol^n

= X x 100 cm^3 of 0.05 N NaOH sol^n

A) Amount of Sodium in the given salt solution:

Now,

1 cm^3 of 0.05N NaOH solution = 0.00115 g of sodium

Therefore,100 cm^3 of 0.05N NaOH solution = 0.00115 x 100 x X g of Sodium.

i.e. A = …….. g of sodium.

B) Amount of Sodium Chloride in the given salt solution :

Now,

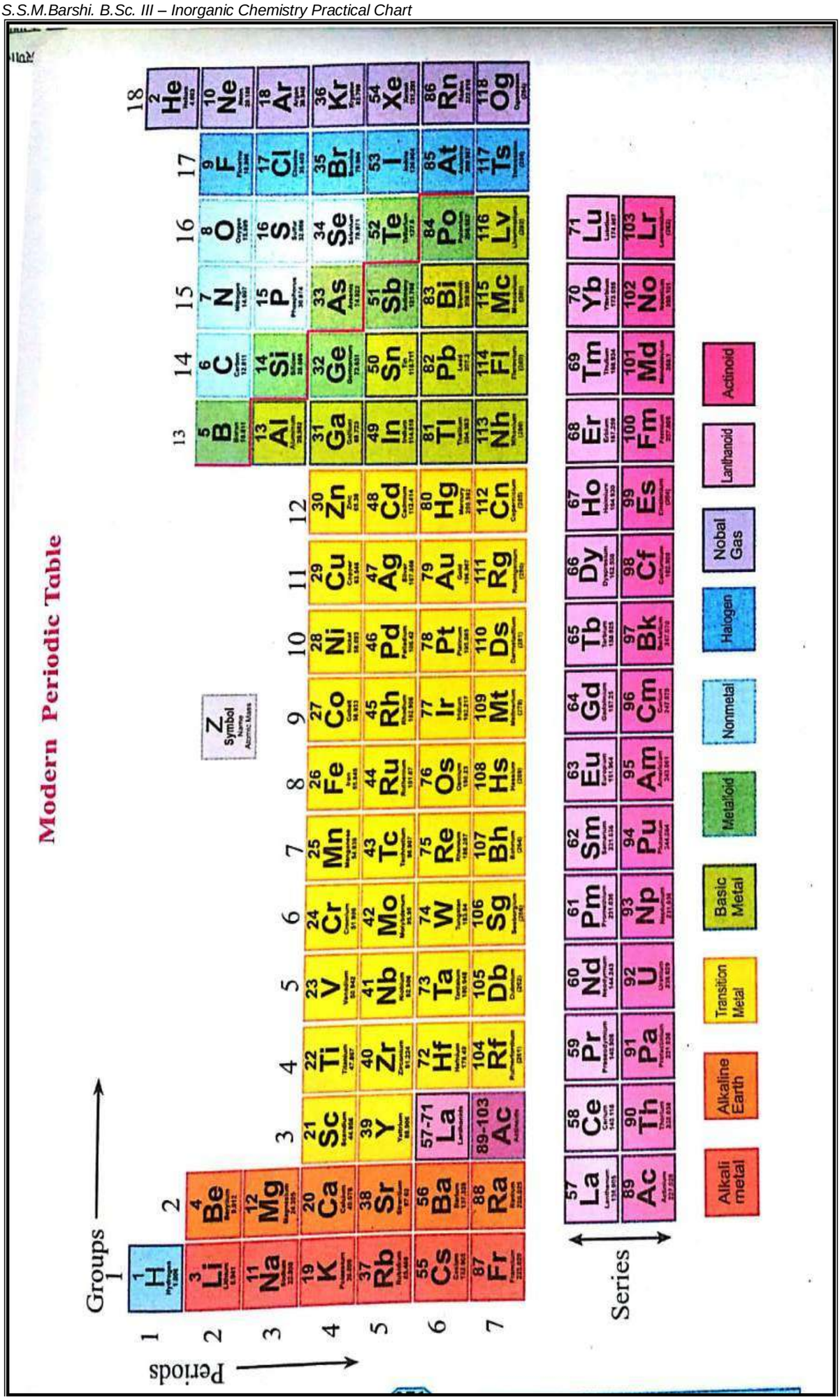
1 cm^3 of 0.05N NaOH solution = 0.002921 g of Sodium chloride

Therefore,100 cm^3 of 0.05N NaOH solution = 0.002921 x A g of NaCl.

i.e. B = …….g of Sodium chloride

RESULTS :

1.	25 cm^3 of diluted effluent solution required(X) cm^3 of 0.05N NaOH solution.	X cm^3 =	…… cm^3
2.	Quantity of Sodium in the given salt sample solution	A g = A x 10^{-3} kg =	……g …… x 10^{-3} kg
3.	Quantity of Sodium chloride in given sample solution	B g = B x 10^{-3} =	……g …….x 10^{-3} kg


Modern Periodic Table
Groups
Periods
Series
Z
Symbol
Name
Atomic Mass
Alkali metal
Alkaline Earth
Transition Metal
Basic Metal
Metalloid
Nonmetal
Halogen
Nobal Gas
Lanthanoid
Actinoid

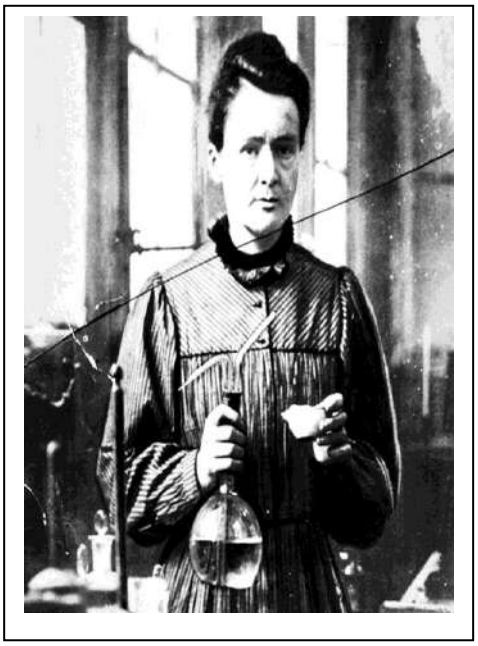
SHRI SHIVAJI MAHAVIDYALAYA, BARSHI

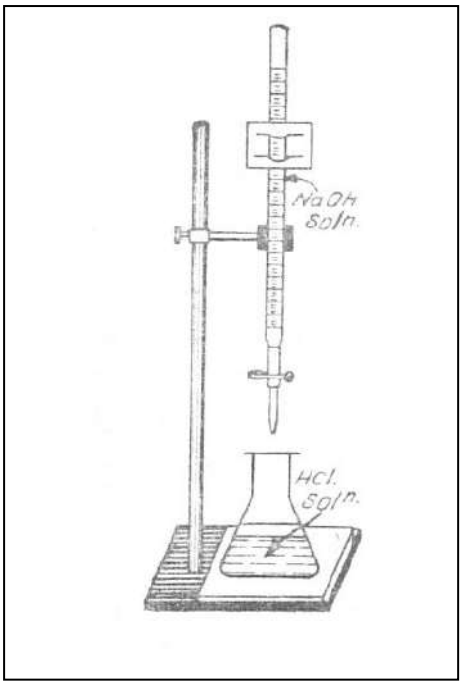
DEPARTMENT of CHEMISTRY

B. Sc. I Practical Chart

Name of Student**:** …….. Roll No**:** ……

INDEX





CONTENTS

	iv) Spot Test No. 4 Radical L=……. Radical M= ……..				
11	**Chromatography :** You are given solution containing two basic radicals in a container marked K bearing your table number i) Separate two cations by using paper chromatography method and ii) Identify the cations and calculate R_f values.				
	i) Chromatography –I: Radicals ……&……				
	ii) Chromatography –II: Radicals ……&……				
	Inorganic Preparations				
12	**Preparation of ferrous ammonium sulfate** To prepare ferrous ammonium sulphate (F.A.S.) from ferrous sulphate and ammonium sulphate.				
	C) ORGANIC CHEMISTRY				
	Organic Quantitative Analysis				
13	**Estimation of Aniline** To estimate the amount of aniline in the given solution.				
14	**Estimation of Amide** To estimate the amount of acetamide in the given solution.				
15	**Estimation of Aspirin** To estimate the amount of aspirin in the given solution.				
	Organic Preparation				
16	**Preparation of Benzoic Acid** To prepare benzoic acid from benzamide				
	Organic Qualitative Analysis (Organic Spotting)				
17	**Identification of organic compounds**				
	Org. Comp. No.	**Name of Organic Compound**	**Structural Formula**		
	1				
	2				
	3				
	4				
	5				

A) PHYSICAL CHEMISTRY

1. & 2. Chemical Kinetics No 1 / 2

Aim **:** To investigate hydrolysis of given methyl acetate in presence of 0.5 N HCl/H_2SO_4

Chemicals **:** 0.1 N NaOH, 0.5 N HCl / H_2SO_4, methyl acetate, ice and phenolphthalein

Apparatus **:** Burette, 5ml pipette, stoppered bottles, conical flask, etc.

Procedure **:** Perform the experiment as follows.

1. Take the following solutions in two separate (clean and dry) stoppered bottles.
   Bottle No. 1 – 5 ml methyl acetate.
   Bottle No. 2 – 100 ml 0.5 N HCl / H_2SO_4
   Keep these two bottles in water bath to attain the same temperature
2. Mean while fill the burette with 0.1 N NaOH and take about 100 ml ice cold water or 2-3 ice pieces and two drops of phenolphthalein indicator in conical flask .
3. Transfer the solution from bottle No.2 to bottle No.1 completely and shake well. Note the time of mixing and immediately pipette out 5 ml reaction mixture in conical flask containing ice and indicator and titrate it with 0.1 N NaOH solution till colour changes from colourless to pink. Note this reading as T_0 .
4. Again fill the burette with 0.1 N NaOH solution. Take ice and indicator in conical flask.
5. In this way, titrate 5 ml of the reaction mixture after every 10 minutes from the start of the reaction. These readings are called T_t. Ask for T_∞ reading.
6. Calculate the values of k by using formula and plot the graph of log $\frac{a}{a-x}$ $Vs\ time\ (t)$. Calculate the value of k by using slope of the graph.

Reaction :

$$CH_3COOCH_3 + H_2O \xrightarrow{HCl\ /\ H_2SO_4} CH_3COOH + CH_3OH$$

Observations :

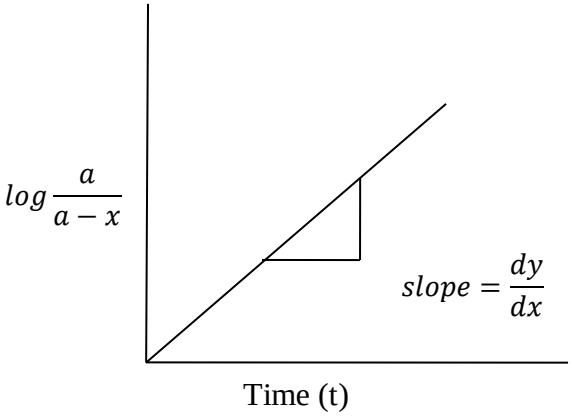
1. In burette **:** 0.1 N NaOH
2. In conical flask **:** Ice + indicator + 5 ml reaction mixture
3. Indicator **:** Phenolphthalein
4. End Point **:** Colourless to pink

Observation table:

Given: T_∞ = ……. ml a = T_∞ - T_o = ---------- ml

Time in minutes t	Titration Reading T_t ml	x = T_t - To	a – x	$\frac{a}{a-x}$	$log\frac{a}{a-x}$	$k = \frac{2.303}{t} log\frac{a}{a-x}$ min^{-1}
0	T_0 =	0.0		1.000	0.0000	----------
10	T_{10} =					
20	T_{20} =					
30	T_{30} =					
40	T_{40} =					
50	T_{50} =					
					Mean k =	min^{-1}

Calculations: Show all details of all calculations. Detail calculation means use of formulae, substitution of values in formulae and calculations by calculator / log table.



k by calculation:

$$\text{Velocity constant } k = \frac{2.303}{t} \log\frac{a}{a-x}$$

Where,

t = time in minutes

a = initial concentration of reactant

a – x = amount of reactant remained at time t

T_∞ = titration reading at infinite time

k by graph:

k = 2.303 × slope

Results:

1. Mean k by calculation = ---------- min^{-1}
2. k by graph = ---------- min^{-1}

Conclusion: Values of 'k' obtained by using first order equation are fairly constant and therefore reaction is pseudo-unimolecular.

3. Chemical Kinetics No-3

Aim : To investigate the reaction between $K_2S_2O_8$ and KI solutions with equal concentrations of the reactants.

Chemical : 0.1 N $K_2S_2O_8$, 0.1 N KI, 0.002 N $Na_2S_2O_3$ solution, ice, starch indicator etc.

Apparatus : Burette, 5ml pipette, stoppered bottles, conical flask etc.

Procedure : Perform the experiment as follows

1. Take the following solutions in two separate (clean and dry) bottles.
   Bottle No. 1 - 20 ml 0.1 N $K_2S_2O_8$ + 20 ml distilled water
   Bottle No.2 - 20 ml 0.1 N KI + 20 ml distilled water
   Keep these two bottles in a water bath to attain the same temperature.
2. Fill the burette with 0.002 N $Na_2S_2O_3$ solution.
3. Add the solution from bottle No. 2 to 1 completely. Note the time of mixing. The solution is stirred and placed in a water bath throughout the experiment. It is shaken occasionally. (Do not take zero time reading).
4. At intervals of 10, 15, 20, 25, 30 and 40 minutes from the mixing time, pipette out 10 ml reaction mixture in the conical flask containing ice pieces and starch indicator. Titrate the reaction mixture against 0.002 N $Na_2S_2O_3$ solution. The end point will be blue to colourless.
5. Calculate the values of k using the formula for a bimolecular reaction.
6. Calculate the values of ' a ' and ' b '.
7. Plot the graph of 1/ (a- x) against time (t). Calculate the value of k from the graph.

Observations :

1. In Burette : 0.002 N $Na_2S_2O_3$
2. In Conical flask: Ice + Indicator + 10 ml reaction mixture
3. Indicator : Starch (8 to 10 drops)
4. End point : Blue to colourless

Calculation of the initial concentration (a and b)

Normality of $K_2S_2O_8$ in mixture:

$K_2S_2O_8$ (original) ***Vs*** $K_2S_2O_8$ (in reaction mix.)

$N_1V_1 = N_2V_2$

$0.1 \times 20 = N_2 \times 80$

$$N_2 = \frac{0.1 \times 20}{80}$$

$N_2 = 0.025$ N

Initial concentration of $K_2S_2O_8$ (a) in terms 0.002 N $Na_2S_2O_3$ solution:

$K_2S_2O_8$ (in reaction mix.) ***Vs*** 0.002N $Na_2S_2O_3$ sol^n

$N_2V_2 = N_3V_3$

$0.025 \times 10 = 0.002 \times V_3$

$$V_3 = \frac{0.025 \times 10}{0.002}$$

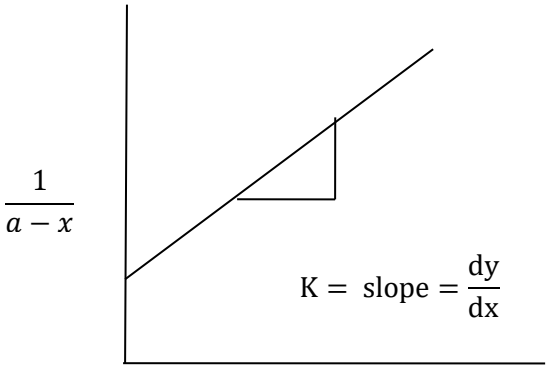
$V_3 = 125$

$\therefore$ a = V_3 = 125 ml

As the normality and volume of KI using is same as that of $K_2S_2O_8$ hence the initial concentration of KI (b) in terms of 0.002 N $Na_2S_2O_3$ is 125 ml, therefore a = b = 125 ml

Observation table:

a = 125 ml $Na_2S_2O_3$			b = 125 ml $Na_2S_2O_3$	
Time in min 't'	Titration reading 'x' ml	a – x	$\frac{1}{a-x}$	$k = \frac{x}{t\,a\,(a-x)}$ lit. mol^{-1} min^{-1}
10				
15				
20				
25				
30				
40				
			Mean k =	lit. mol^{-1} min^{-1}



Results:

1. Mean k by calculation = …… lit. mol^{-1} min^{-1}
2. Mean k by graph = ……lit. mol^{-1} min^{-1}

Conclusion: The graph $1/(a - x)$ against time (t) is a straight line which shows that the reaction is a bimolecular.

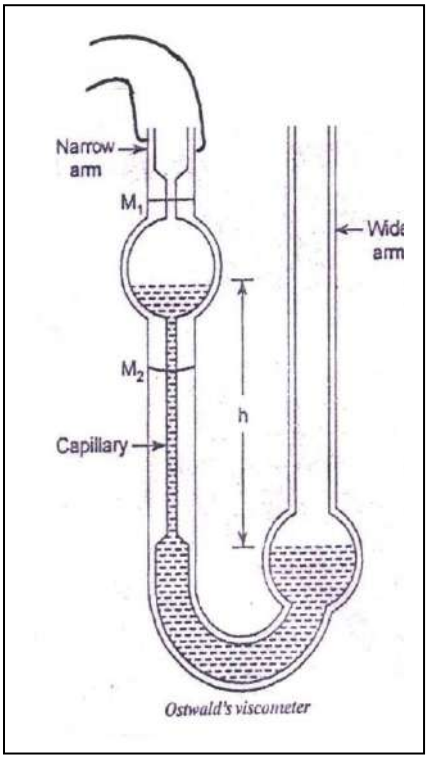
4. Viscosity

Aim : To determine the viscosity of given liquids A and B

Chemicals : Drying liquid:-acetone, pure liquid: – A and B, distilled water etc.

Apparatus : Ostwald's viscometer, dryer, stop watch.

Procedure :



1. Viscometer is washed with acetone and dries it by passing current of air using dryer. Attach piece of rubber tube to the narrow arm of viscometer and clamp it in a perfectly vertical position.
2. Introduce, required volume of liquid 'A' into the viscometer, Suck the liquid 'A' by means of rubber tube till it raises little above the upper mark.
3. Allow the liquid to flow through the capillary tube into the lower bulb. Start the stop watch when liquid level just passes the upper mark and stop it when just reaches the lower mark. Note down the time in seconds. i.e. time required for the flow of liquid from upper to lower mark.
4. Repeat the measurement of time of flow three times and take the mean reading as the time of flow.
5. Remove liquid A from viscometer, rinse it with acetone and dry it.
6. Rinse viscometer with liquid B and introduce the same volume of liquid B and determine the time of flow as explained above.
7. Repeat this procedure for distilled water. Ask for the densities of liquids A,B and viscosity of distilled water.

Observation table:

Liquid	Time of flow (t) seconds				Density (d)	Viscosity (η)
	I	II	III	Mean (t)		
A						
B						
Distilled water					1.00	8.91

Calculation : Calculate the viscosity of the liquid by using formula.

$$\frac{\eta_1}{\eta_2} = \frac{d_1 \times t_1}{d_2 \times t_2}$$

$$\therefore \eta_2 = \frac{\eta_1 \times d_2 \times t_2}{d_1 \times t_1}$$

Where: -
η_1 = Viscosity of distilled water (8.91)
η_2 = Viscosity of liquid A or B.
d_1 =Density of distilled water (1.00)
d_2 = Density of liquid A or B.
t_1 = Time of flow for distilled water
t_2 = Time of flow for liquid A or B.

Result :

1. Viscosity of liquid (A) = …….. millipoise
2. Viscosity of liquid (B) = …….. millipoise

5. Equivalent weight of Mg

Aim : To determine the equivalent weight of given magnesium metal by hydrogen displacement method using eudiometer.

Apparatus : Eudiometer, hydrometer jar, crucible, water bath etc.

Chemicals : Two Mg metal pieces, concentrated HCl etc.

Procedure :

1. Clean the magnesium metal pieces with sand paper and weigh it accurately. (Weight should be about 0.030 to 0.050 g).
2. Take about 15 ml concentrated HCl in graduated eudiometer and then fill it completely with water.
3. Invert the eudiometer in water bath by closing the mouth of eudiometer with right hand thumb.
4. Now roll the weighed metal piece and insert carefully in the mouth of eudiometer. Magnesium reacts with HCl and hydrogen gas begins to evolve and gets collected at the upper part of eudiometer by displacing diluted acid. When reaction is over wait for some time to allow gas in the eudiometer to attain room temperature.
5. Insert a crucible below the mouth of eudiometer and introduce it in hydrometer jar (tall cylinder filled with water).
6. Equalize the levels of water in eudiometer and hydrometer by raising or lowering the eudiometer. This will make the pressure of moist hydrogen gas in eudiometer equal to atmospheric pressure. Now record the volume of gas i .e. $\mathbf{V_1}$ ml at room temperature.
7. By using same procedure record the volume of gas collected for another magnesium piece.

Observations :

P_0 = Atmospheric pressure = 760 mm,

T_1 = 273 + R.T. = ……...K

V_0 = Volume of H_2 at NTP

T_0 = Absolute temperature = 273 K

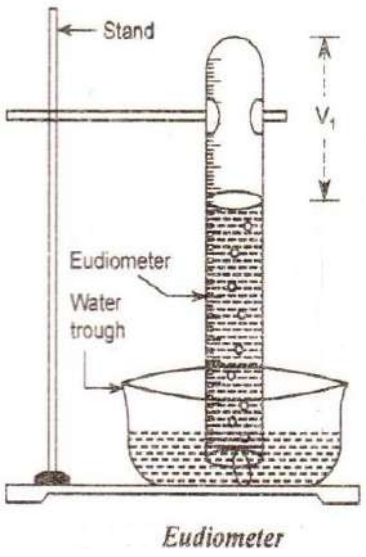
V_1 = Volume of H_2 collected at room temperature and atmospheric pressure.

P_1= Pressure of dry gas = barometric pressure – aqueous tension

P_1 = (P – p) = ……mm

Given :

1. Barometric pressure (P) = …….. mm
2. Aqueous tension (p) = ……..…… mm
3. Room temperature (T_1) = ……^{0}C + 273 = -------- K

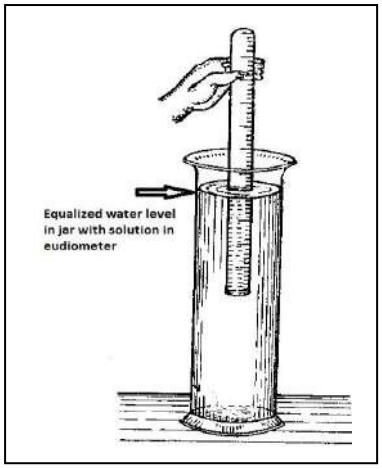


Eudiometer

Observation table:

Sr. No	Wt of the Metal (W gm)	Volume of H_2 evolved at R.T. (V_1) ml	Volume of H_2 evolved at N.T.P (Vo) ml	Volume for 0.1 gm of metal $= \frac{V_0 \times 0.1}{W}$	Equivalent Weight of metal
1					
2					
Mean equivalent weight of metal =					

Reaction : $Mg + 2\,HCl \longrightarrow MgCl_2 + H_2\uparrow$



Calculations : Give calculations in detail

1. **Volume of gas reduced to NTP:**

$$\frac{P_0\ V_0}{T_0} = \frac{P_1\ V_1}{T_1}$$

$$V_0 = \frac{P_1\ V_1\ T_0}{T_1\ P_0} = ----\ ml$$

Where,

$P_0 = 760$ mm

$T_0 = 273$ K

$P_1 = P - p =$ ------ mm

2. Volume for 0.1 gm of metal $= \frac{V_0 \times 0.1}{W}$

3. **Equivalent weight of metal which liberates 11,200 ml of pure and dry H_2 at NTP.**

∵ V_0 ml is volume of H_2 at NTP displaced by W gm metal

∴ 11,200 ml of H_2 at NTP will be displaced by the metal = Equivalent weight

$$\text{Equivalent weight} = \frac{11200 \times W}{V_0}$$

Results: Mean equivalent weight of magnesium metal = ---------

6. Heat of ionization

Aim : To determine the heat of ionization (ΔHi) of weak acid

Apparatus : Beakers, $\frac{1}{10}^{th}$ thermometer, measuring cylinder, polythene bottle etc.

Chemicals : Z N strong acid (HCl), Z N strong base (NaOH), Z N weak acid (CH_3COOH).

Procedure : Perform the experiment in two parts:

Part A: Heat of neutralization of strong acid (HCl) by strong base (NaOH) i.e. ΔH_1:

1. Take 100 ml of given strong base (NaOH) in a polythene bottle and record its temperature ($t_1\ ^0C$)
2. Take 100 ml given strong acid (HCl) in a beaker and record its temperature ($t_2{}^0C$). Mean of the two temperatures is taken as initial temperature (t^0C). Then add the acid carefully to base in polythene bottle, stir the mixture well and record the maximum steady temperature ($t_3\ ^0C$).

Part B: Heat of neutralization of weak acid (CH_3COOH) by strong base (NaOH) i.e. ΔH_2:

1. Take 100 ml of given strong base (NaOH) in a polythene bottle and record its temperature ($t_1\ ^0C$)
2. Take 100 ml given weak acid (CH_3COOH) in a beaker and record its temperature ($t_2\ ^0C$). Mean of the two temperatures is taken as initial temperature ($t\ ^0C$). Then add the acid carefully to base in polythene bottle, stir the mixture well and record the maximum steady temperature ($t_3\ ^0C$).

Observations:

Part A: Heat of neutralization of strong acid (HCl) by strong base (NaOH) i.e. ΔH_1:

Vol. of acid + base	Temp of base ($t_1\ ^0C$)	Temp of acid ($t_2\ ^0C$)	Initial temp ($t\ ^0C$) $t = \frac{t_1 + t_2}{2}$	Temp of mixture ($t_3\ ^0C$).	Rise in temp $\Delta t = t_3 - t$	Heat of neutralization **ΔH_1** cal
200 ml						

Part B: Heat of neutralization of weak acid (CH_3COOH) by strong base (NaOH) i.e. ΔH_2:

Vol. of acid + base	Temp of base ($t_1\ ^0C$)	Temp of acid ($t_2\ ^0C$)	Initial temp ($t\ ^0C$) $t = \frac{t_1 + t_2}{2}$	Temp of mixture ($t_3\ ^0C$)	Rise in temp $\Delta t = t_3 - t$	Heat of neutralization **ΔH_2** cal
200 ml						

Given **:** 1. Water equivalent of polythene bottle (W) = 0.0 cal.
2. Normality of acid or base (Z) = ----------N

Calculations :

a) Heat evolved (Q) & heat of neutralization (ΔH_1) for Part A:

Heat evolved (Q) = (200 + W) Δt = ---------- cal.

$$\text{Heat of neutralization } (\Delta H_1) = \frac{10 \times Q}{Z} = -----\text{cal.}$$

b) Heat evolved (Q) & heat of neutralization (ΔH_2) for Part B:

Heat evolved (Q) = (200 + W) Δt = ---------- cal.

$$\text{Heat of neutralization } (\Delta H_2) = \frac{10 \times Q}{Z} = -----\text{cal.}$$

c) Heat of ionization (ΔHi) of weak acid:

$\Delta Hi = \Delta H_2 - \Delta H_1$ = ---------- cal.

= ---------- joule (1 cal = 4.184 joules)

Result **:**

1. ΔH_1 = - (----------) cal.
2. ΔH_2 = - (----------) cal.
3. ΔHi = + (----------) cal.
   = + (----------) joules.

B) INORGANIC CHEMISTRY

INORGANIC QUANTITATIVE ANALYSIS

7. Volumetric Estimation-I: $H_2C_2O_4$ versus NaOH

Aim : To prepare standard solution of oxalic acid and determine the strength of sodium hydroxide (NaOH) solution in terms of normality & kg/dm^3.

Apparatus : Volumetric flask (250 ml), beaker , blass rod , funnel , watch glass ,burette , pipette , conical flask.

Chemicals : 1.Oxalic acid 2. water 3. NaOH solution 4. phenolphthalein indicator

Procedure :

Part A: Determination of equivalent weight of oxalic acid and preparation of standard solution

Molecular weight of oxalic acid is 126 and its basicity is 2

$$\therefore \text{ Eq. wt. of oxalic acid } = \frac{\text{Molecular Weight}}{\text{Basicity}} = \frac{126}{2} = 63$$

∴ Equivalent weight of oxalic acid is 63, now for preparing

1000 ml ---- 1 N oxalic acid solution ≅ 63 g of oxalic acid

1000 ml ---- 0.1 N oxalic acid solution ≅ 6. 3 g of oxalic acid

250 ml ---- 0.1 N oxalic acid solution ≅ $\frac{250\times0.1\times63}{1000} = 1.575$ g

Weigh accurately 1.575 g of oxalic acid on watch glass, dissolve it in 50 to 75 ml of distilled water in a beaker by stirring with glass rod and transfer the solution to 250 ml volumetric flask. Rinse the beaker and glass rod 4 -5 times with distilled water and transfer the washing to the flask. Dilute the solution up to mark with distilled water and shake well.

Part B: Determination of normality and strength of NaOH

Rinse and fill the burette with sodium hydroxide solution up to zero mark .Pipette out 25 ml of oxalic acid solution in a conical flask and add 2-3 drops of phenolphthalein indicator. Add NaOH solution from the burette with constant shaking. Go on adding NaOH solution until a faint permanent pink colour is obtained and note the burette reading, repeat the titration for two more readings.

Observations:

Part A

1. Wt. of empty watch glass (W_1) = ……. g
2. Wt. of oxalic acid taken (W_2) = 1.575 g
3. Wt. of watch glass + oxalic acid = W= (W_1+ W_2) = ……g

Part B

1. In burette : 0.1 N NaOH solution
2. By pipette : 25 ml oxalic acid solution
3. Indicator : phenolphthalein
4: End point : colourless to faint permanent pink

Reaction: $H_2C_2O_4 + 2\ NaOH \longrightarrow Na_2C_2O_4 + 2H_2O$

Observation table:

Burette level	Burette reading in ml			C.B.R
	I	II	III	
Final level				X =……. ml
Initial level	0.0	0.0	0.0	
Difference				

Calculations : C.B.R. (X) = ……ml

a) Normality of given NaOH **b) Strengh of given NaOH**

Oxalic acid Vs NaOH

$N_1V_1 = N_2 V_2$

$0.1 \times 25 = N_2 \times$ 'X'

$N_2 = \frac{0.1 \times 25}{X}$

Strength of given NaOH = Normality x equivalent weight of NaOH

= N x 40 =Z = …… g/dm^3

∴ Strength of NaOH = Z x 10^{-3} kg/dm^3

RESULTS : 1. Normality of given NaOH solution = …….. N.

2. Strength of NaOH (Z) = ……. g/dm^3

3. Strength of NaOH (Z x 10^{-3}) = …….. x 10^{-3} kg/dm^3

8. Volumetric Analysis-II: $KMnO_4$ versus $H_2C_2O_4$

Aim **:** To prepare standard solution of oxalic acid and determine the strength potassium permanganate ($KMnO_4$) solution in terms of normality & kg/dm^3.

Chemicals: Given $KMnO_4$ solution, oxalic acid crystalline, dilute H_2SO_4, distilled water.

Procedure : Part A: Determination of equivalent weight of oxalic acid and preparation of standard solution

Molecular weight of oxalic acid is 126 and its basicity is 2

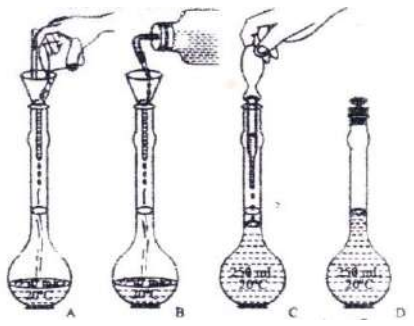
$\therefore$ Eq. wt. of oxalic acid $= \frac{\text{Molecular weight}}{\text{Basicity}} = \frac{126}{2} = 63$

∴ Equivalent weight of oxalic acid is 63, now for preparing

1000 ml ---- 1 N oxalic acid solution ≅ 63 g of oxalic acid

1000 ml ---- 0.1 N oxalic acid solution ≅ 6. 3 g of oxalic acid

250 ml ---- 0.1 N oxalic acid solution ≅ $\frac{250 \times 0.1 \times 63}{1000} = 1.575$ g



Weigh accurately 1.575 g of oxalic acid on watch glass, dissolve it in 50 to 75 ml of distilled water in a beaker by stirring with glass rod and transfer the solution to 250 ml volumetric flask. Rinse

the beaker and glass rod 4-5 times with distilled water and transfer the washing to the flask. Dilute the solution up to mark with distilled water and shake well.

Part B: Determination of normality and strength of $KMnO_4$

Rinse and fill the burette with potassium permanganate solution up to zero mark ,pipette out 25 ml of oxalic acid solution in a conical flask and add to it one test tube of dilute sulphuric acid. Heat this content to 60 to 70 ^{0}C on a boiling water bath . Add $KMnO_4$ solution from the burette with constant shaking. Go on adding $KMnO_4$ solution until a permanent faint pink colour is obtained and note the burette reading. Repeat the titration for two more readings.

Observations:

Part A

1. Wt. of oxalic acid taken (W) = 1.575 g

Part B

1. In burette : 0.1 N $KMnO_4$ solution
2. By pipette : 25 ml oxalic acid solution
3. Indicator : $KMnO_4$ itself.
4: End point : colourless to permanent faint pink

Reaction: $2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow$

Observation table:

Burette level	**Burette reading in ml**			**C.B.R**
	I	**II**	**III**	
Final level				X =…..ml
Initial level	0.0	0.0	0.0	
Difference				

Calculations : C.B.R. (X) = ……ml

a) Normality of given $KMnO_4$

Oxalic acid Vs $KMnO_4$

$N_1V_1 = N_2 V_2$

$0.1 \times 25 = N_2 \times$ 'X'

$N_2 = \frac{0.1 \times 25}{X}$

b) Strength of given $KMnO_4$

Strength of given $KMnO_4$ = Normality x equivalent weight of $KMNO_4$

= N x 31.6 = …… g/dm^3

∴ Strength of $KMnO_4$ = Z x 10^{-3} kg/dm^3

Results:

1. Normality of given $KMnO_4$ solution = …….. N.
2. Strength of $KMnO_4$ (Z) = ……. g/dm^3
3. Strength of $KMnO_4$ (Z x 10^{-3}) = …….. x 10^{-3} kg/dm^3

9. Volumetric Analysis-III: $K_2Cr_2O_7$ versus $[Fe(SO_4)(NH_4)_2SO_4).6H_2O]$

Aim : To prepare standard solution potassium dichromate ($K_2Cr_2O_7$) and determine the strength ferrous ammonium sulphate (F.A.S.) solution solution in terms of normality & kg/dm^3.

Chemicals: Crystalline $K_2Cr_2O_7$, F.A.S .solution, phosphoric acid-sulphuric acid mix, diphenyl amine, dilute H_2SO_4

Procedure:

Part A: Determination of equivalent weight of $K_2Cr_2O_7$ & Preparation of 0.1 N $K_2Cr_2O_7$ sol^n:

In presence of acid $K_2Cr_2O_7$ supplies oxygen according to following equation .

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

i.e . $K_2Cr_2O_7 \equiv 3(O) \equiv 6H$

$$\therefore \text{Eq. Wt of } K_2Cr_2O_7 = \frac{\text{mole. wt. of } K_2Cr_2O_7}{6} = \frac{294.2}{6} = 49.03$$

Equivalent weight of $K_2Cr_2O_7$ in acidic medium is 49.03, now for preparing $K_2Cr_2O_7$ solution

1000 ml --- 1N $K_2Cr_2O_7$ Solution = 49.03 g of $K_2Cr_2O_7$

1000 ml --- 0.1N $K_2Cr_2O_7$ Solution = 4.903 g of $K_2Cr_2O_7$

250ml --- 0.1 N $K_2Cr_2O_7$ Solution = ?

$$= \frac{250\times0.1\times4.903}{1000\times0.1} = 1.226\ g \quad K_2Cr_2O_7$$

Weigh accurately 1.226g of $K_2Cr_2O_7$ crystals on a watch glass. Dissolve it in about 50to 75 ml of distilled water in a beaker and transfer the solution to 250 ml measuring flask .Rinse the beaker and glass rod 4 to 5 times with distilled water and transfer the washing to the flask .Dilute this solution to up to the mark with distilled water and shake well .

Part B: Determination of normality and strength of F.A.S. $[Fe(SO_4)(NH_4)_2SO_4).6H_2O]$

Pipette out 25 ml of F.A.S. solution in conical flask, add to it 25 ml **phosphoric acid-sulphuric acid solution** (masking reagent) Add 100 ml of distilled water and then add 3 drops of diphenylamine indicator .Run the dichromate solution in to the flask, go in adding $K_2Cr_2O_7$ solution until the green colour changes to grey green, then add dichromate drop wise until the first tinge of purple or violet blue colour appears. Repeat the procedure for 3 times and find out C.B.R.

Observations:

Part A

Wt of $K_2Cr_2O_7$ solid (W_2) = 1.226 g

Part B

1. In burette = 0.1N $K_2Cr_2O_7$ solution
2. By pipette = 25 ml F.A.S. (Given)
3. Indicator = diphenyl amine (3 Drops)
4. End point = green to violet blue

Reactions :

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$[FeSO_4(NH_4)_2SO_4] + H_2SO_4 + (O) \longrightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + H_2O] \times 3$$

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4.(NH_4)_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe(SO_4)_3 + 6(NH_4)_2SO_4 + 7H_2$$

Observation table:

Burette level	Burette reading in ml			C.B.R
	I	II	III	
Final level				X=……. ml
Initial level	0.0	0.0	0.0	
Difference				

Calculations: C.B.R. (X) = …… ml

a) Normality of given F.A.S. solution

F.A.S. Vs $K_2Cr_2O_7$

$N_1V_1 = N_2V_2$

$\therefore N_1 = \frac{0.1 \times X}{25}$

b) Strength of F.A.S.

= Normality x Eq. Wt of F.A.S

= N_1 x 392 = Z g/dm^3

$\therefore$ Strength of $KMnO_4$ = Z x 10^{-3} kg/dm^3

Results: 1. Normality of F.A.S. solution = …….. N

2. Strength of F.A.S. (Z) = …… g/ dm^3

3. Strength of F.A.S (Z x 10^{-3}) = …… x 10^{-3} kg/dm^3

INOGANIC QUALITATIVE ANALYSIS

10. Spot Tests of Basic Radicals

Aim: To detect basic radicals from solutions given in two containers marked as L and M. Give the complete report of the test performed.

Note: i) Carry out Confirmatory test. if first test of particular radical is positive.

ii) If first test of particular radical is negative then try for next radical (Show the final tests to the examiner and take his signature on the final test).

iii) The reagent of each test is written above it.

Sr. No.	REAGENT	OBSERVATION	INFERENCE
1.	RUBEANIC ACID On drop of original solution (O.S.) on a filter paper + One drop of the reagent and expose it to ammonia	a) Blue colour. b) Brown colour. c) Olive green colour.	Ni^{++} present Co^{++} present. Cu $^{++}$present.
	C.T. For Ni $^{++}$ DIMETHYL GLYOXIME One drop of O.S .on filter paper + One drop of the reagent and expose it to ammonia	Rose red colour	Ni $^{++}$confirmed

	C.T. For CO $^{++}$ ∝ –NITROSO β – NAPTHOL One drop of O.S. on filter paper + One drop of reagent **C.T. For Cu $^{++}$** CUPRON : (α – Benzoin oxime) One drop of O.S. on a filter paper + One drop of the reagent and expose it to ammonia .	Red brown colour Green colour	Co $^{++}$confirmed Cu $^{++}$confirmed
2.	AMMONIUM SULPHOCYANIDE One drop of O.S. on spot plate + One drop of reagent POTASSIUM FERROCYNIDE **C.T. For Fe^{+++}** One drop of O.S. on filter paper + One drop of reagent	Blood red colour Deep blue colour	Fe $^{+++}$present Fe $^{+++}$confirmed
3	ALIZARIN One drop of O.S. on filter paper , dry it + One drop of reagent and expose to ammonia **C.T. For Al^{+++}** ALUMINON 2 drops of O.S. on spot plate or in small T.T.+ 1 drop of dil HCl + 4 drops of ammonium acetate + 3 drops of reagent, wait for 5 minutes + excess of ammonical ammonium carbonate	Red colour Red colour \ ppt	Al $^{+++}$present Al $^{+++}$confirmed
4.	P NITROBENZENE AZORESORCINOL (MAGNESON –I) Four drops of O. S in a spot plate + One drop of dilute HCl + One drop reagent + Several drops of NaOH. **C.T. For Mg $^{++}$** TITAN YELLOW : One drop of O. S in a spot plate + One drop of reagent + 4 drop of NaOH	Blue ppt . or blue colour Red ppt or colour	Mg $^{++}$present Mg $^{++}$confirmed
5.	DILUTE H_2SO_4 : Two drop of O. S in a fusion or small test tube + One drop of reagent **C.T. For Pb $^{++}$** GALLOCYNINE : One drop of O. S in a spot plate + $NaHCO_3$ solution + One drop of reagent	White ppt. or turbidity Blue or bluish violet ppt or colour	Pb $^{++}$present Pb $^{++}$confirmed

6.	<u>AMMONIUM MERCURY THIOCYANATE</u> One drop of O. S in a spot plate + one drop of cobalt nitrate + 3-4 drops of ammonium mercuric thiocynate and rub with glass rod **C.T. For Zn^{++}** <u>POTASSIUM FERRICYNIDE AND DIPHENYLAMINE :</u> One drop of O. S in a spot plate + One drop of potassium ferricyanide + One drop of diphenylamine in glacial acetic acid	Immediate blue ppt or violet colour Green ppt	Zn^{++}present Zn^{++}confirmed

Result:

Container	Radical Detected
L	
M	

11. Chromatography

Aim **:** You are given solution containing two basic radicals in a container marked 'K' bearing your table number. (i) Separate the two cations by using paper chromatography method and (ii) identify the cations and calculate R_f values. give report of the test performed.

Apparatus: Strips of Whatman paper, cylinder with lid, sharp pencil, glass dropper, sprayer, infrared lamp.

Chemicals: Solvent (mixture of acetone, conc. HCl and distilled water in the ratio 90:5:5), mixture given for separation, Rubeanic acid , dimethyl glyoxime, ammonia.

Procedure:

1. Pour about 20 to 25 ml solvent in cylinder and close it with lid.
2. Take Whatman paper strip and draw lightly a reference line on it by pencil at 3 cm from one end and make a mark at the centre of reference line .
3. Apply drop of given mixture on the centre of reference line, Allow to dry the applied solution by dryer or Infra red lamp . Repeat same procedure for 2 to 3 times.
4. Dip the strip in the cylinder taking care that the reference line must be well above the solvent level as
5. shown in fig 8(a) .
6. Allow the solution to run for 45 minutes (distance about 10 to 15 cm from reference line).
7. Remove the strip from cylinder and dry it. Mark a solvent front with a dotted line by pencil.
8. Detect the radicals by applying the spraying reagent.
9. Paste your chromatogram on your answer book/ journal.
10. Show the distance travelled by radicals and solvent front as shown in fig. 8 (b).
11. Calculate the rate of flow (R_f) values for the detected radicals.

Observation table:

Spraying Reagent	Colour developed	Radicals
Rubeanic acid and expose to ammonia.	i . Blue	Ni^{++} present
	ii .Yellow orange or yellowish brown	Co^{++} present
	iii. Olive green	Cu^{++} present

Calculations:-

1. by first radical (ℓ_1) = …… cm
2. Distance travelled by second radical (ℓ_2) = …… cm
3. Distance travelled by solvent front (L) = ……..... cm

$$\mathbf{R_f\ value} = \frac{\textbf{Distance travelled by solute radical}}{\textbf{Distance travelled by solvent front}}$$

1. $\mathbf{R_f}$ **value for first radical** $= \frac{\ell_1}{L}$ =……
2. $\mathbf{R_f}$ **value for second radical** $= \frac{\ell_2}{L}$ =……..

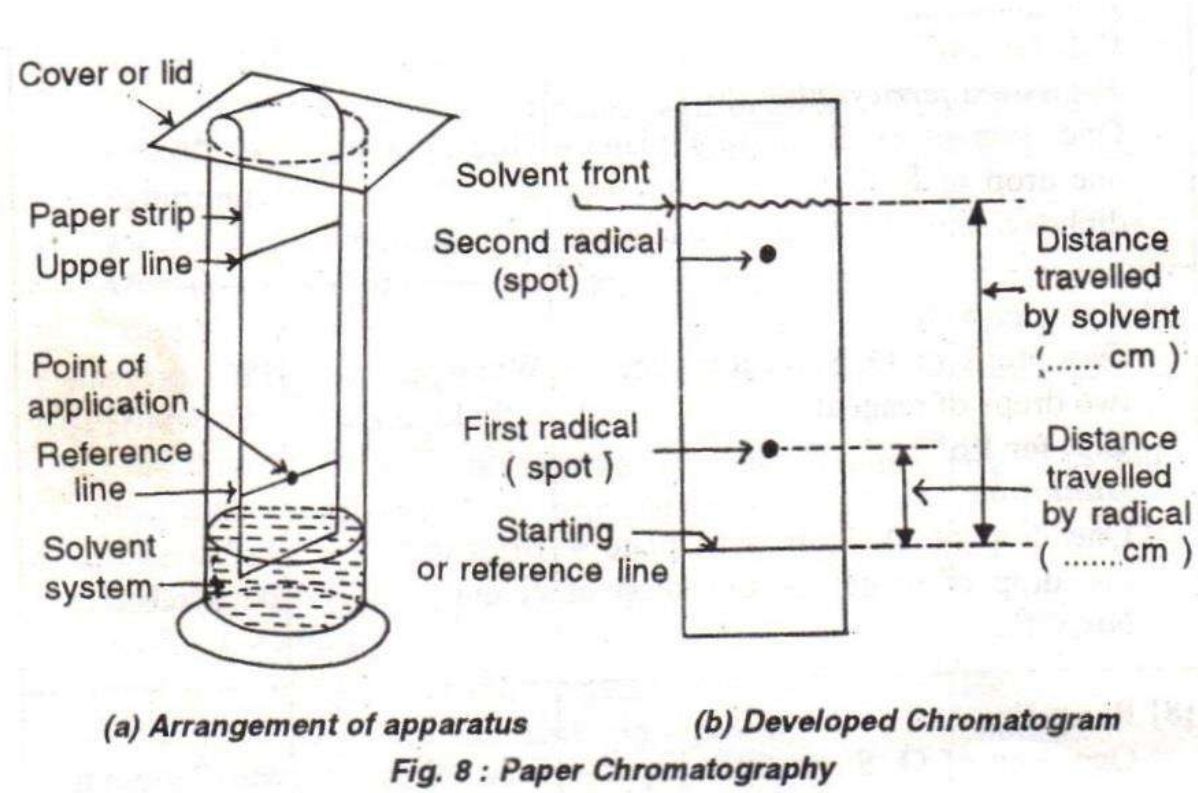


Fig. 8 : Paper Chromatography

Results:

Sr. No.	Name of radical detected	R_f value
1.	First radical …….	…….
2.	Second radical ……	…….

Inorganic Preparations

12. Preparation of Ferrous Ammonium Sulphate (Mohr's Salt)

Aim : To prepare ferrous ammonium sulphate (F.A.S.) from ferrous sulphate and ammonium sulphate.

Chemicals : Ferrous sulphate ($FeSO_4.7H_2O$), ammonium sulpahte $(NH_4)_2\ SO_4$, ethanol etc.

Apparatus : 250 ml Beakers, glass rod, measuring cylinder etc.

Procedure :

1. Weigh 10 g of Ferrous Sulphate and transfer in to beaker. Add in it about 60ml distilled water and 5 ml dil. H_2SO_4, dissolve by boiling the solution.
2. Add to it 5g of ammonium sulphate with constant stirring.
3. Add a bright iron nail to maintain iron content.
4. Boil the solution till the crystallization points is just reached (avoid formation of crystal masses).
5. Cool and add about 10 ml ethyl alcohol. Faint green coloured crystals of F.A.S. are obtained.
6. Now filter the product on Buchner funnel and wash the product with little alcohol. Dry and weigh the product.

Reaction:

$$FeSO_4 + (NH_4)_2\ SO_4 + 6H_2O \longrightarrow FeSO_4\ (NH_4)_2SO_4 \cdot 6H_2O$$

Simple salt Simple salt Double salt (F.A.S.)

Calculations:

a) Theoretical yield	**b) Percent % yield**
From chemical reaction we get $FeSO_4.7H_2O = FeSO_4(NH_4)_2SO_4.6H_2O$ 278 392 278 g Ferrous sulphate = 392 g F.A.S $\therefore$ 10 g Ferrous sulphate = $\frac{10 \times 392}{278}$ g F.A.S. = 14 g $\therefore$ Theoretical yield of product (A) = 14 g	Weight of the product = X = ……g $\therefore$ 14 g product F.A.S = 100 % yield $\therefore$ X g product F.A.S = $\frac{X \times 100}{14}$ % i.e. B = ……%

Results:

1.	Colour of the product F.A.S.	Faint green	
2	Weight of the product F.A.S.	X = …… g	=…….. x 10^{-3}Kg
3.	Theoretical yield of product	A =……. g	=…….. x 10^{-3}Kg
4.	Practical % yield of product	B =……. %	

C) ORGANIC CHEMISTRY

13. Estimation of Aniline

Aim **:** To estimate the amount of aniline in the given solution.

Apparatus **:** Burette, 10 ml pipette, conical flask, stoppered bottles etc.

Chemicals **:** 0.1 N $Na_2S_2O_3$ solution, brominating solution (0.1 N approx.), aniline solution, 10 % KI solution, starch indicator, concentrated HCl.

Principle **:** Aniline reacts with bromine which is liberated from the brominating solution to give tri- bromo derivative. From the amount of bromine (in terms of iodine (I_2) used) aniline can be estimated.

Part I: Blank titration

Procedure **:** Pipette out 10 ml of brominating solution in the conical flask. Add to it 25 ml distilled water, 10-15 ml about 1 test tube of KI solution and 3 ml conc. HCl. Titrate the liberated iodine against the sodium thiosulphate solution using starch as an indicator (10 drops). End point is blue to colourless. Note the burette readings. Repeat the procedure for two more readings and determine constant burette reading as 'X' ml.

Observations :

1. In burette **:** 0.1 N $Na_2S_2O_3$ solution
2. In conical flask **:** 10 ml brominating solution + 25 ml distilled water + 1 test tube 10 % KI + 3 ml conc . HCl
3. Indicator **:** Starch (10 drops) or 1 ml
4. End point **:** Blue to colourless

Observation table:

Burette level	**Burette reading in ml**			**C.B.R.**
	I	**II**	**III**	
Final level				
Initial level	0.0	0.0	0.0	X =……ml
Difference				

Part II: Back titration

Procedure **:** Pipette out 10 ml of brominating solution in a stoppered bottle. Add to it 10 ml aniline solution by pipette, 25 ml distilled water and 3 ml conc. HCl solution. Wait for 10 minutes. Then add about 10-15 ml (about 1 test tube) of 10 % KI solution. Titrate the liberated iodine against 0.1 N sodium thiosulphate solution using starch as an indicator. End point is blue to colourless. Note the burette reading. Repeat the procedure for two more readings and determine the constant burette reading as 'Y' ml.

Observations :

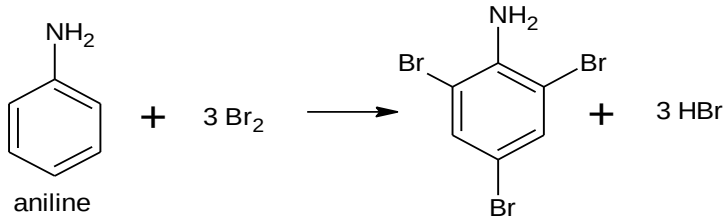
1. In burette **:** 0.1 N $Na_2S_2O_3$ solution
2. In conical flask **:** 10 ml brominating solution + 10 ml aniline solution + 25 ml distilled water + 3 ml conc. HCl (wait for 10 min.) + 1 T.T. 10 % KI
3. Indicator **:** Starch (10 drops) or 1 ml
4. End point **:** Blue to colourless

Observation table:

Burette level	Burette reading in ml			C.B.R.
	I	II	III	
Final level				Y=……..ml
Initial level	0.0	0.0	0.0	
Difference				

Reactions :

$$KBrO_3 + 5\,KBr + 6\,HCl \longrightarrow 6\,KCl + 3\,Br_2 + 3\,H_2O$$



aniline

2,4,6 tribromo aniline

$$Br_2 + 2\,KI \longrightarrow 2\,KBr + I_2$$

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2\,NaI$$

Calculations :

C.B.R of the back titration = Y ml = ---------- ml

C.B.R of the blank titration = X ml = ---------- ml

X – Y = V = …….. – …….... = ……. ml

V = ……ml is the amount of brominating solution in terms of sodium thiosulphate solution consumed by 10 ml of aniline solution

From the above chemical reactions

1 mole of aniline ≡ 3 mole of Br_2 ≡ mole of I_2 ≡ 6 mole of $Na_2S_2O_3$

Thus

6 mole of $Na_2S_2O_3$ ≡ 1 mole of aniline

∴ 6000 ml 1 N $Na_2S_2O_3$ ≡ 93 g of aniline

60,000 ml 0.1 $Na_2S_2O_3$ ≡ 93 g of aniline

∴ V ml 0.1 N $Na_2S_2O_3$ = $\frac{V \times 93}{60{,}000}$ g of aniline in 10 ml solution

i.e. Z = …….. g of aniline in 10 ml solution

Strength of aniline in g / L

10 ml of aniline solution ≡ Z g of aniline

∴ 1000 ml aniline solution ≡ $\frac{Z \times 1000}{10}$ g of aniline

= Z × 100 g of aniline

i.e. Strength of aniline = Z × 100 = …… g / L

Strength of aniline in kg / dm^3

Strength of aniline in given solution = Z × 100 g / L

Now strength of aniline in kg/dm^3 = $\frac{Z \times 100}{1000}$

∴ Strength of aniline in kg / dm^3 = Z × 0.1

=…… kg / dm^3

Results:

1. Strength of aniline	= Z × 100	= …… g / L
	= Z × 0.1	=…… kg / dm^3

14. Estimation of Amide

Aim : To estimate the amount of acetamide in the given solution.

Apparatus: 250 ml volumetric flask, conical flask, round bottom flask, burette, pipette (25 ml), sand bath, glass funnel etc.

Chemicals : Given acetamide solution, 1N KOH solution, 0.1 N HCl solution, phenolphthalein etc.

Part I: Back titration

Procedure : To the given solution of acetamide in a round bottom flask, add 25 ml of 1 N KOH solution. Cover the flask with funnel. Heat the flask on sand bath till ammonia gas is evolved completely. (Test with moist turmeric paper i.e. it should not turn brown or red). Cool this solution and transfer the solution to the 250 ml volumetric flask carefully by using funnel. Wash the round bottom flask 2-3 times with distilled water and transfer it to the volumetric flask and dilute the solution up to the mark with distilled water. Shake well the solution. Pipette out 25 ml of this diluted solution in conical flask and titrate against 0.1 N HCl solution using phenolphthalein indicator. End point is pink to colourless. Take three readings and find out C.B.R. Note this C.B.R. as 'Y' ml.

Observation :

1. In burette : 0.1 N HCl
2. In conical flask : 25 ml diluted reaction mixture by pipette
3. Indicator : Phenolphthalein (2-3 drops)
4. End point : Pink to colourless

Observation table:

Burette level	**Burette reading in ml**			**C.B.R.**
	I	II	III	
Final level				Y=……ml
Initial level	0.0	0.0	0.0	
Difference				

Part II: Blank titration

Procedure : Pipette out 25 ml of 1 N KOH in to the 250 ml volumetric flask and dilute up to the mark with distilled water. Shake well. Titrate 25 ml of this diluted solution against 0.1N HCl solution by using phenolphthalein indicator. End point is pink to colourless. Take three readings and find out C.B.R and note this C.B.R as 'X' ml

Observation :

1. In burette : 0.1 N HCl
2. In conical flask : 25 ml diluted KOH solution by pipette
3. Indicator : Phenolphthalein (2-3 drops)
4. End point : Pink to colourless

Observation table:

Burette level	**Burette reading in ml**			**C.B.R.**
	I	**II**	**III**	
Final level				X =….. ml
Initial level	0.0	0.0	0.0	
Difference				

Reactions:

$$CH_3CONH_2 + KOH \longrightarrow CH_3COOK + NH_3\uparrow$$

$$KOH + HCl \xrightarrow{\text{Neutralisation}} KCl + H_2O$$

Calculation :

C.B.R of the back titration = Y ml = ---------- ml

C.B.R of the blank titration = X ml = ---------- ml

X – Y = V = …….. – …….... = ……. ml

∴ V ml = -------- ml is the amount of KOH solution consumed during hydrolysis of acetamide in terms of 0.1N HCl solution

KOH solution consumed in terms of 0.1 N HCl for hydrolysis of acetamide in the given solution

= V X 10 ml

=……ml

From the above chemical reactions ,

1 mole of KOH solution ≡ 1 mole of acetamide (CH_3CONH_2)

∴ 1000 ml of 1N KOH solution ≡ 59 g of acetamide.

∴ 1000 ml of 1N HCl solution ≡ 59 g of acetamide

∴ 1000 ml of 0.1N HCl solution ≡ 5.9 g of acetamide

i. e. 10,000 ml of 0.1N HCl solution ≡ 59 g of acetamide

∴ V ml of 0.1N HCl solution = $\frac{V \times 59}{10{,}000}$ g of acetamide

i. e. A = …… g of acetamide in 25 ml of diluted solution.

∴ Amount of acetamide present in 250 ml solution = A × 10 g

i.e. B =……. g of acetamide.

Results :

1. KOH solution consumed in terms of 0.1 N HCl solution for hydrolysis of acetamide in the given solution	= V × 10 ml	= ---------- ml
2. Quantity of acetamide in the given solution	= B g	=…….g
	= B × 10^{-3} kg	= ……10^{-3} kg

15. Estimation of Aspirin

Aim : To determine the amount of aspirin (Acetyl salicylic acid) from aspirin tablet.

Apparatus: Burette , pipette , conical flask , 250 cc volumetric flask , beakers , watch glass , weight box etc .

Chemicals: Asprin tablet, 0.1 N oxalic acid solution, approximate 0.1 N NaOH solution, Phenolphthalein

Procedure:

A) Preparation of standard oxalic acid solution and standardization of NaOH solution :

I. Preparation of standard oxalic acid solution: Weigh accurately, 1.575 g of oxalic acid on a watch glass and transfer it in a beaker, dissolve it in minimum amount of distilled water and transfer this solution to 250 ml volumetric flask. Dilute the contents up to the mark with distilled water and shake well.

II. Standardization of NaOH solution :

1. Clean all the glasswares and rinse with disitilled water .
2. Fill the burette with 0.1 N (approx) NaOH solution .
3. Pipette out 25 ml exactly 0.1 N oxalic acid solution in conical flask .
4. Add phenolphthalein indicator.
5. Titrate with NaOH solution till colour changes from colorless to faint pink colour. Repeat the procedure for two more readings and determine CBR as ‘a’ml.
6. Calculate the normality of NaOH solution . Let the normality be ‘ X’ N .

B) Estimation of Aspirin:

1. Weigh the given aspirin tablet accurately on a clean watch glass.
2. Transfer it to 250 ml beaker and dissolve in minimum amount of ethyl alcohol (20 ml) .
3. Transfer this solution to 100 ml volumetric flask and dilute up to mark with distilled water.
4. Titrate 25 ml of this diluted solution against X N NaOH solution using phenolphthalein as an indicator till colour changes from colorless to faint pink colour. Repeat the procedure for two more readings and determine CBR as ‘b’ml.

Observations and observations table:

A. Preparation of standard oxalic acid solution and standardization of NaOH solution :

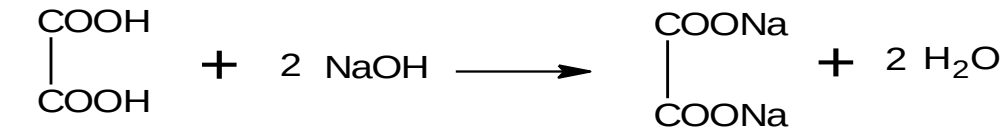
Observations I :

1. In burette : NaOH solution
2. By pipette : 25 ml 0.1N oxalic acid solution
3. Indicator : Phenolphthalein
4. End point : Colourless to pink

Observation Table :

Burette Level	Burette Reading in ml			C.B.R
	I	II	III	
Final level				a =…..ml
Initial level				
Difference				

Reaction:



oxalic acid — sodium salt of oxalic acid

Calculations :

Oxalic acid NaOH

$N_1V_1 = N_2V_2$

$0.1 \times 25 = N_2 \times a$

$N_2 = \frac{0.1 \times 25}{a}$ N

i.e. $N_2 = X =$ --------- N

Thus exact normality of NaOH = X = …….. N

B. Estimation of aspirin :

Observations II :

Weight of aspirin tablet (W) = ---------- g

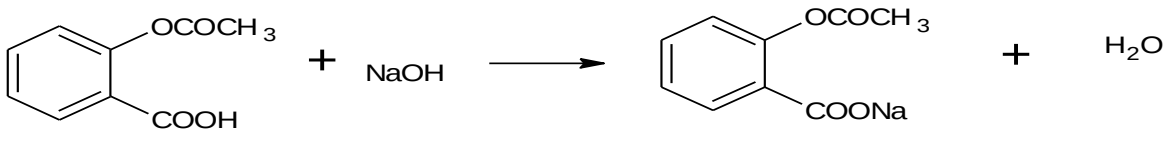
Observations III :

1. In Burette : X N NaOH Solution
2. By pipette : 25 cc diluted aspirin tablet solution
3. Indicator : Phenolphthalein
4. End point : : Colourless to pink

Observation table :

Burette Level	Burette reading in ml			C.B.R
	I	II	III	
Final Level				b=…..ml
Initial Level				
Difference				

Reaction :



Aspirin — Sodium salt of Aspirin

Calculations :

1. Amount of aspirin in given tablet

From chemical reaction we know

1 mole of NaOH solution ≡ 1 mole of aspirin ≡ 180 g of aspirin

i.e.1000 ml of 1 N NaOH solution ≡ 180 g of aspirin

∴ b ml of X N NaOH solution ≡ $\frac{b \times X \times 180}{1{,}000 \times 1}$ g of aspirin

i.e Y ≡ ---- g of aspirin

Now 25 ml of diluted aspirin tablet solution contain = Y =…….. g of aspirin

∴ 100 ml of aspirin tablet solution contain = Y x 4 = …… g of aspirin

i.e . Z = ……..g of aspirin

2. Percentage of aspirin in given tablet :

Weight of aspirin tablet = W = …… g

∴ W g of aspirin tablet = Z g aspirin

∴ 100g of aspirin tablet = $\frac{100 \times Z}{W}$ % aspirin

i.e. A = ---------- % aspirin

i.e. Percentage of aspirin in given tablet = ----------% aspirin

Results:

1. Exact normality of NaOH solution	= X N	= …….. N
2. Amount of aspirin in the given tablet	= Z g = Z x 10^{-3} kg	= …….. g = …….. x 10^{-3} kg
3. Percentage of aspirin in given tablet	= A %	= …….. %

16. Preparation of Benzoic acid

Aim **:** To prepare benzoic acid from benzamide.

Chemicals **:** Benzamide, 10 % NaOH, conc. HCl etc.

Apparatus **:** Conical flask, funnel , beakers etc.

Procedure **:**

1. Take X gm (3 / 4 / 5 gm) of benzamide and 50 ml of 10% NaOH solution in a conical flask
2. Add about 20 ml distilled water and 2 to 3 porcelain pieces to it.
3. Cover the conical flask with stem cut funnel and heat the flask on sand bath for 30 minutes. During boiling ammonia gas is evolved. (Completion of reaction is tested by turmeric paper.)

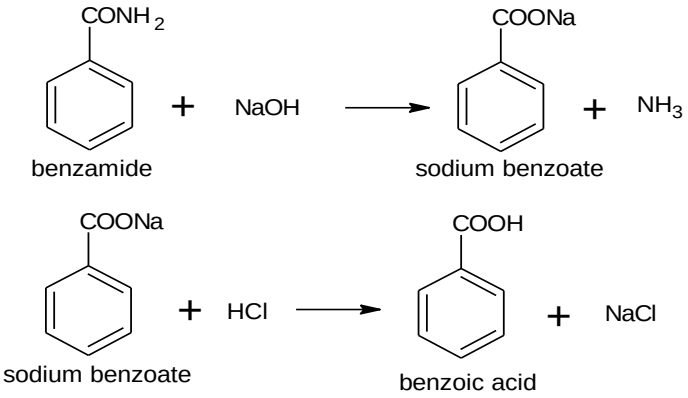
4. Now cool the solution in ice cold water and add conc. HCl until the mixture becomes strong acidic. Benzoic acid separates immediately.
5. Filter the product using Buckner funnel and wash with cold water.
6. Dry and weigh the product. Suppose the weight of the crude product is Z g
7. Recrystallize the product from hot water.

Recrystallization: Take about 2 g of product in clean and dry test tube. Add ¾th test tube distilled water to it and 2 porcelain pieces. Then boil the solution carefully. Filter this solution through cotton plug in another test tube. Cool the filtrate in ice bath to get crystals of benzoic acid. Again filter this solution through filter paper and dry the recrystallized product.

Observations :

1. Weight of benzamide (X) = ……g
2. 10 % NaOH = …… ml

Reactions :



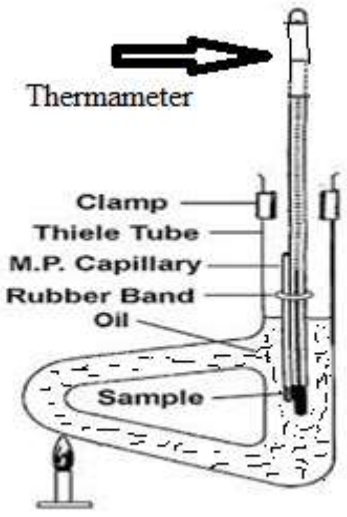
Result:

1. Weight of the crude product	= Z g	=……g
	= Z x 10^{-3} kg	=……. x 10^{-3} kg

17. Identification of Organic Compounds (Organic Spotting)

A systematic identification of an organic compound involves the following steps.

I. Determination of physical constant
II. Preliminary tests
III. Detection of elements
IV. Determination of group
V. Identification of organic compound by comparing physical constant and taking confirmatory test.
VI. Summary



Organic Compound No. -------

I) Physical Constant:

M.P. / B.P. of given organic compound = ---------- ^0C

II) Preliminary Tests:

Sr. No.	Test	Observation	Inference
1.	State	Solid	Benzoic acid , Oxalic acid, Cinnamic acid, Glucose , Resorcinol, β-Naphthol, p-Toludine, m-Dinitrobenzene, Thiourea etc. may be present
		Liquid	Acetone , Aniline , Chloroform, Chlorobenzene may be present
2.	Colour	a) White solid	Benzoic acid, Oxalic acid, Cinnamic acid, Glucose, Thiourea etc. may be present .
		b) Colourless liquid	Acetone, Chloroform, Chlorobenzene Ethyl acetate may be present
		c) Brown or pink liquid	Aniline may be present
		d) Brown or pink solid	Resorcinol or β-Naphthol may be present
		e) Yellow or orange solid	m- Dinitrobenzene and p-Toludine may be present
3.	Odour	a) Fruty smell	Ethyl acetate may be present
		b) Pleasant	Acetone may be present
		c) Sweet smell	Chloroform, Chlorobenzene etc
		d) Phenolic	Resorcinol, β-Naphthol may be present
		e) Fishy smell	Aniline may be present
		f) No particular odour	Benzoic acid, Cinnamic acid, Salicylic acid, Glucose, Thiourea, m-Dinitrobenzene etc. may be present
4.	**Solubility Test :** i) Solid compound + water	a) Soluble in cold water	Resorcinol, Oxalic acid may be present
		b) Soluble in hot water	Benzoic acid, Cinnamic acid may be present
		c) Insoluble	β- Naphthol, p-Toludine, m-Sinitrobenzene may be present
	ii) Liquid compound + water	a) Miscible with water	Acetone may be present
		b) Immiscible with water	Aniline, Chloroform, Chlorobenzene etc
		c) Immiscible and lighter then water	Ethyl acetate may be present
5.	Organic compound + $NaHCO_3$ solution	Effervescence of CO_2	Benzoic acid , Salicylic acid , Cinnamic acid may be present
6.	Organic compound + NaOH, Shake well	Soluble	Resorcinol or β- Naphthol may be present

7.	Organic compound + dilute $KMnO_4$	a) Decolourization of $KMnO_4$ solution b) No decolourization of $KMnO_4$ solution	Unsaturated compound is present Saturated compound is present
8.	Organic compound + dil. HCl	Soluble	p-Toludine, Aniline may be present
9.	**Burning Test** Heat small amount of substance on piece of glass rod or Cu foil	a) Burns with sooty flame b) Burns with non-sooty flame c) Green flame for liquid compound d) Green flame for solid compound	Aromatic compound is present Aliphatic compound is present Halogen containing compound is present Thiourea is present

III) Detection of elements (Lassiagne's test or sodium fusion test):

Take 3/4 test tube distilled water in clean evaporating dish. Take a dry piece of freshly cut sodium (Na) metal in a three fusion tubes. Heat the fusion tube gently to melt the Na metal. Add little amount of organic compound to it. (For liquid compound use 5 ml pipette for dropping the liquid in fusion tube) Initially heat slowly and then strongly until fusion tube is red hot. Drop red hot fusion tube in evaporating dish containing distilled water. Repeat the procedure for remaining fusion tube.

Boil contents of evaporating dish for some time (till half of the distilled water in evaporating dish evaporates) and filter through ordinary filter paper in a large test tube. This filtrate is known as sodium extract. Use this filtrate / extract for detection of elements.

Sr. No.	Test	Observation	Inference
1.	**Test for nitrogen :** 1 ml filtrate + 1 ml freshly prepared saturated $FeSO_4$ solution + 1 drop of NaOH boil well, cool well & add excess of conc. HCl	Blue ppt or green coloration	Nitrogen (N) is present
	$Na + C + N \longrightarrow NaCN$ $2\,NaCN + FeSO_4 \longrightarrow Fe(CN)_2 + Na_2SO_4$ $Fe(CN)_2 + 4\,NaCN \longrightarrow Na_4[Fe(CN)_6]$ $3\,Na_4[Fe(CN)_6] + 4\,FeCl_3 \xrightarrow{HCl} Fe_4[Fe(CN)_6]_3 \downarrow + 12\,NaCl$ Ferric ferrocyanide (prussian blue)		
2.	**Test for sulphur** 2 ml filtrate +1 drop of NaOH + few drops of sodium nitropruside solution	Purple or violet colouration	Sulphur (S) is present
	$2\,Na + S \longrightarrow Na_2S$ Sodium sulphide $Na_2S + Na_2[Fe(CN)_5\,NO] \longrightarrow Na_4[Fe(CN)_5NOS]$ Sodium nitroprusside — purple colour		
3.	**Test for halogens :** 2-3 ml filtrate +1-2 ml dil HNO_3, boil , cool + $AgNO_3$ solution	White or yellow ppt	Halogen (X) present

	$Na + X \longrightarrow \underset{\text{Sodium halide}}{NaX}$ (X = Cl, Br or I) $NaX + AgNO_3 \longrightarrow \underset{\text{Silver halide}}{AgX\downarrow} + NaNO_3$

∴ **Given organic compound contains element C, H, and (O) and ……**

IV) Determination of group:

Group I – Compounds containing element C, H and (O):

Sr. No.	Test	Observation	Inference
	A) Acid (-COOH)		
1.	Organic compound + Saturated $NaHCO_3$ solution	Effervescence of CO_2	Benzoic acid Oxalic acid Cinnamic acid May be present
	$R\,COOH + NaHCO_3 \longrightarrow R\,COONa + CO_2\uparrow$		
2.	Organic compound + water	Completely soluble & acidic to litmus	Oxalic acid is present
Preparation of neutral solution: 1 g organic compound + 10 ml NH_4OH solution in a beaker ⟶ boil off ammonia gas (Test with turmeric paper it should not turn brown) ⟶ Neutral solution			
3.	Neutral solution + $FeCl_3$ solution	Buff coloured or reddish Brown ppt	Benzoic acid or Cinnamic acid is present
4.	Neutral solution + $CaCl_2$ solution	a) White precipitate insoluble in acetic acid b) No white precipitate	Cinnamic acid is present Benzoic acid is present
	B) Phenols (Ar-OH)		
1.	Organic compound + water, shake well, compound dissolves + $FeCl_3$ solution by pipette	Violet coloration	Resorcinol is present
2.	Organic compound + water, shake well, compound remains insoluble + boil + $FeCl_3$ solution	Green coloration immediately changing to white.	β -Naphthol is present
	$6\,Ar\,OH + FeCl_3 \longrightarrow \underset{\substack{\text{complex ion}\\\text{(violet colour)}}}{[(ArO)_6Fe]^{3-}} + 3\,HCl + 3\,H^+$		
	C) Neutral		
1.	**Ester:** Liquid organic compound + NaOH + $FeCl_3$	Red colour	Ethyl acetate is present

2.	**Carbohydrate:** Organic compound + Fehling's solution and warm	Red ppt.	Glucose is present
3.	**Ketones :** Organic compound + few drops of sodium nitroprusside solution + 2 drops of NaOH	Blood red coloration	Acetone is present

Group II: Compounds containing the elements C, H, (O) and N:

Sr. No.	Test	Observation	Inference
1.	**Amines ($-NH_2$):** Use two test tubes for this test. **In first test tube** : Organic compound + 3 times conc. HCl, cool and dilute with water + few drops of $NaNO_2$ (sodium nitrite) solution, cool well in ice cold water. **In second test tube** : β – Naphthol + NaOH, shake well, cool well in ice cold water. Add solution of first test tube into second test tube.	Orange dye stuff	Aniline, p-Toludine is present
	Colour of organic compound	a) Yellowish brown coloured liquid. b) Brownish solid	Aniline is present p-Toludine is present
	$Ar\,NH_2 + NaNO_2 + HCl \xrightarrow[\text{low temparature}]{\text{diazotization}} Ar\,N{=}N.Cl + NaCl + 2\,H_2O$ diazonium salt $Ar\,N{=}N.Cl$ + beta naphthol in NaOH $\xrightarrow[\text{low temparature}]{\text{coupling}}$ orange dye + HCl diazonium salt		
2.	**Nitrohydrocarbon:** Compound + 2 ml alcohol + 2 drops $CaCl_2$ solution + small quantity of Zn dust, boil for few minutes and filter into Tollen's reagent.	Grey or black ppt	m- Dinitrobenzene present
(Tollen's reagent**:** 2 ml $AgNO_3$ solution + drop of NaOH + add NH_4OH till ppt. dissolves.)			

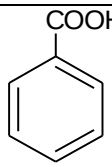
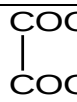
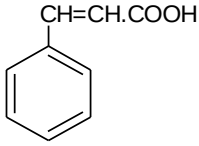
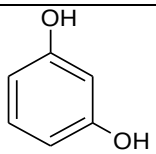
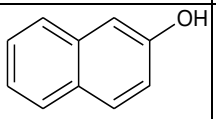
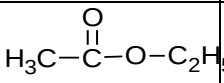
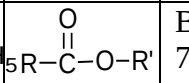
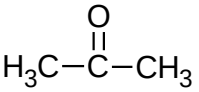
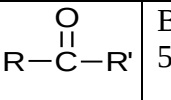
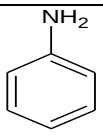
Group III: Compounds containing the elements C, H, (O), N and S:

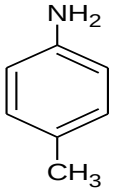
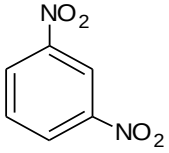
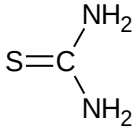
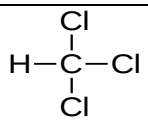
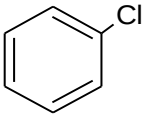
1.	Heat organic compound in test tube till it melts + cool + water + aqueous $FeCl_3$	Red coloration	Thiourea present

Group IV: Compounds containing C, H, (O) and Halogen**:**

1.	1 ml sodium extract + $CHCl_3$ + Cl_2 water in excess shake well	Colourless chloroform layer	Chlorobenzene , Chloroform present

V) Identification of the organic compound by comparing their practical / observed M.P. / B.P. with the theoretical M.P. / B.P.

Sr. No.	Name of organic compound	Molecular formula	Structural formula	Functional group	M.P. / B.P.	Confirmatory test
Group I: Compound containing elements C, H, (O).						
A) Acids						
1.	Benzoic acid	$C_7H_6O_2$		-COOH	M.P. 122^0C	Organic compound +2 ml ethyl alcohol + 4 to 5 drops of conc. H_2SO_4 + 2 porcelain pieces and heat **:-** Sweet smell of ester.
2.	Oxalic acid	$C_2H_2O_4$		-COOH	M.P. 101^0C	Organic compound + $CaCl_2$ Sol^n **:-** White ppt.
3.	Cinnamic acid	$C_9H_8O_2$		> C=C< -COOH	M.P. 133^0C	Organic compound + drop of dil. H_2SO_4 + 1ml dil. $KMnO_4$ solution & warm **:-** decolourization of $KMnO_4$
B) Phenols						
4.	Resorcinol	$C_6H_4(OH)_2$		Ar-OH	M.P. 110^0C	Organic compound + NaOH shake well + few drops chloroform and boil **: -** Brilliant red colour.
5.	β – Napthol	$C_{10}H_7OH$		Ar-OH	M.P. 122^0C	Organic compound + chloroform ($CHCl_3$) + copper fillings + NaOH solution and heat **: -** blue colouration.
C) Neutrals						
6.	**a. Ester :** Ethyl acetate	$C_4H_8O_2$			B.P. 78 ^0C	Liquid organic compd . + water + 2 drops of phenolphalein + very dilute NaOH, drop by drop till pink colour is developed & warm **:-** pink colour disappears .
7.	**b. Ketone:** Acetone	C_3H_6O			B.P. 56 ^0C	Organic compound + m-Dinitrobenzene + excess of NaOH solution, shake well **:-** Violet coloration which fades slowly.
Group II: Compounds containing elements C, H, (O) and N.						
A) Amines						
8.	Aniline	$C_6H_5NH_2$		Ar-NH_2	B.P. 184 ^0C	Organic compd. +KOH + $CHCl_3$ + few pieces of porcelain + heat **:-** dirty smell of carbylamines.

9.	p-Toludine	$CH_3(C_6H_4)NH_2$		$Ar\text{-}NH_2$	M.P. 44 ^{0}C	Compound + Water + concentrated HCl + shake well + few drops of $FeCl_3$ **:-** Red orange colour
B) Nitrohydrocarbon:						
10	m-Dinitro benzene	$C_6H_4(NO_2)$		$-NO_2$	M.P. 90^{0}C	Organic compound + 1-2 ml acetone + few drops of NaOH **:-** Deep violet coloration
Group III : Compounds containing elements C,H,(O), N and S:						
11.	Thiourea	$C\ H_4N_2S$		>C=S	M.P. 180^{0}C	Compound + acetic acid warm + aqueous[$K_4Fe\ (CN)_6$] → Green colour changes to blue on standing .
Group IV : Compound containing C,H,(O) and X :						
12.	Chloroform	$CHCl_3$		-Cl	B.P. 61^{0}C	Compound + β-Naphthol in NaOH + Copper filling → Heat →Blue colour changes to brown on standing.
13.	Chlorobenzene	C_6H_5Cl		-Cl	B.P. 132^{0}C	It is colourless liquid and aromatic in nature

Confirmatory test of …….

Test	Observation	Inference

VI) Summary:

1. Appearance **:** Solid / Liquid.
2. Nature **:** Aliphatic / Aromatic
3. Elements **:** C, H, (O) and ……
4. Functional group **:**
5. Physical constant **:** a) Theoretical M.P. / B .P. = …… ^{0}C

b) Practical M.P. / B .P. = …… ^{0}C

On the basis of confirmatory test and summary, the given organic compound is ……

Result:

Name of organic compound	Molecular formula	Structural formula

-----xxx----------xxx------

Theory Examination Pattern (CBCS)

Semester I	Semester II
University Examination Chemistry Paper No. I = 40 marks Chemistry Paper No. II = 40 marks	**University Examination** Chemistry Paper No. III = 40 marks Chemistry Paper No. IV = 40 marks
Internal examination (Unit Test / Home Assignment) for <u>each Paper</u> = 10 marks	

Annual Practical Examination Pattern (CBCS)

Total Marks = 100

- **Internal practical examination** (As per schedule given by college) **= 20 marks**
- **University practical examination** (Annual as per schedule given by university) **= 80 marks**

University practical examination paper is of 80 marks. Duration of this examination is one day. There will be two practicals. Distribution of 80 marks university practical examination is as follows:

Section	Marks
Two experiments from Physical / Inorganic / Organic / Analytical sections	70
Oral	05
Journal	05
Total	**80**

Journal Writing Format:

			Date:
			Title of Experiment
	Reactions:		Aim:
	Figures:		Chemicals:
	Observations:		Apparatus:
	Observation Table:		Procedure:
	Calculations:		Results:

Punyashlok Ahilyadevi Holkar Solapur University, Solapur
Dnyanteerth Nagar, Kegaon, Solapur - Pune National Highway, Solapur- 413255, Maharashtra (India)

Printed on: 17/08/2022

Blank Mark List For

College Name: Shri Shivaji Mahavidyalaya, Barshi(SMB)

Bachelor of Science (Hons)-I

B.Sc(Hons)Regular - CBCS Pattern 2019 - B.Sc(Hons)-I Sem-II

Paper Code : 19201204-Botany Practical

			Practical CA (Min Mark: 0 Max Mark: 20)		
Sr No	Seat No	Name	PRN No	Marks	Total
1	002157	ADSUL ATHRAV SHRIRAM	202101082003635		17
2	002158	AGALAVE RHISHIKESH HEMANT	202101082005738		18
3	002161	ANDHARE RATUJA RAVINDRA	202101082003895		19
4	002164	Atkare Sanika Sanjaykumar	202101082004020		17
5	002165	ATKARE TEJASVI KRISHNAT	202101082003681		15
6	002169	BAGWAN MASIRA ARIF	202101082003887		19
7	002170	BAHIREWAR AISHWARY TUKARAM	202101082004072		15
8	002172	BARATE MADHURI RAMESHWAR	202101082005739		16
9	002173	BARBOLE PRANALI DATTATRAYA	202101082003657		20
10	002174	BARBOLE ROHINI SANJAY	202101082003679		20
11	002176	BHATLAVANDE URMILA TANAJI	202101082003816		19
12	002180	BHOITE PRAGATI ASHOK	202101082004775		14
13	002181	BHOLE VAISHNAVI RAJKUMAR	202101082004920		12
14	002183	BHOSALE VAISHNAVI RAJENDRA	202101082005393		13
15	002184	bhosale vaishanavi vishwanath	202101082004016		15
16	002185	BHOSALE VAISHNAVI DILIPKUMAR	202101082003643		17
17	002186	BHUITE SAKSHI RAMCHANDRA	202101082005384		18
18	002188	CHAUDHARI SHANKAR ASHOK	202101082003650		19
19	002189	CHAVAN SAUDAGAR UTTAM	202101082003660		20
20	002190	CHAVAN SHIVANAND DHARMARAJ	202101082004032		AB
21	002192	CHAVAN VAISHNAVI ABHIMAN	202101082013193		11
22	002193	Chobe Pooja Hanumant	202101082003883		11
23	002197	DESHMUKH GEETANJALI SUBRAO	202101082004719		18
24	002199	DEVKATE SAKSHI HANUMANT	202101082003820		10
25	002201	DHERE SANKET RAMHARI	202101082005375		18
26	002202	DOIFODE SHWETA SAMBHAJI	202101082003673		14
27	002204	FAPAL PRIYANKA PANDURANG	202101082003675		18
28	002206	GADKAR SARIKA SHANKAR	202101082003678		20
29	002208	GAIKWAD AJAY BALASAHEB	202101082004768		13
30	002209	GAIKWAD ANKITA ANNASAHEB	202101082004868		17
31	002210	GAIKWAD GAURI PRASHANT	202101082005339		12
32	002211	GAIKWAD SANIKA NITIN	202101082004770		19
33	002212	GALANDE ANUSHKA SURYAKANT	202101082004922		15
34	002213	GALANDE ANKITA KAMALAKAR	202101082004924		10
35	002214	GAVALI PRATHAMESH RAJENDRA	202101082004083		07
36	002215	GAVASANE VAISHNAVI RAMESH	202101082003758		15
37	002216	GAVHANE PRANITA NEMINATH	202101082003722		17
38	002218	GAWARE RUTIKA BHARAT	202101082005740		16
39	002219	Ghaytidak Vijay Maruti	202101082004025		19
40	002220	GHAYTIDAK VRUSHALI APPASAHEB	202101082003720		19
41	002224	GHOLAP VIJAY SHIVAJI	202101082003763		15
42	002225	GHOLAVE VAISHNAVI WALCHAND	202101082004030		17
43	002226	GHYTIDAK SHWETA NARSINH	202101082003762		20
44	002232	GURAV GORAKH JAGANNATH	202101082003215		9
45	002233	HANDE SAKSHI SANTOSH	202101082003868		10
46	002234	JADHAV ADITI ABHAY	202101082003697		14
47	002235	JADHAV ATHARV BANDU	202101082004893		9
48	002236	JADHAV DEVAYANI VINAYAK	202101082003658		17
49	002237	JADHAV DNYANESHWARI CHANDRAKANT	202101082003686		13

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Dr. Parthraj R. Kshirsagar

Head
Department of Botany
Shri Shivaji Mahavidyalaya
Barshi, Dist-Solapur

Blank Mark List For

College Name: Shri Shivaji Mahavidyalaya, Barshi(SMB)

Bachelor of Science (Hons)-I

B.Sc(Hons)Regular - CBCS Pattern 2019 - B.Sc(Hons)-I Sem-II

Paper Code : 19201204-Botany Practical

			Practical CA (Min Mark: 0 Max Mark: 20)		
Sr No	Seat No	Name	PRN No	Marks	Total
50	002239	JADHAV ROHIT PRASHANT	202101082003826		12
51	002242	JADHAV VAISHNAVI NANDKUMAR	202101082003718		9
52	002243	JAGTAP MIRABAI VAJINATH	202101082003886		11
53	002245	JAGTAP SHRUTI SATISH	202101082003829		18
54	002246	JAGTAP VIJAYA SAHADEV	202101082003708		15
55	002248	JAVHERI DAKSHA VISHAL	202101082004028		11
56	002251	KADAM SAMARTH BHAUSAHEB	202101082003693		17
57	002252	KADAM TANAYA ARJUN	202101082004885		16
58	002253	KALE RUTUJA SHASHIKANT	202101082003772		14
59	002259	KAPASE ISHWARI GANESH	202101082003828		11
60	002260	KAPSE SAKSHI RAKESH	202101082003659		11
61	002262	KARKAR OMRAJE SANTOSH	202101082004778		13
62	002263	KASABE SAGAR RAJENDRA	202101082003209		10
63	002267	KATMORE VAISHNAVI BAPU	202101082004891		11
64	002270	KAZI FIZA MUDASSSIR	202101082003818		16
65	002271	KHADBADE NIKITA SHANKAR	202101082003667		19
66	002272	KHAIRE AISHWARYA BHAUSAHEB	202101082005386		12
67	002275	KHANDAGALE ABHISHEK BALASAHEB	202101082004879		8
68	002276	KHARADE RADHA SHIVHARI	202101082004718		8
69	002279	KHODSE BALIKA BAPURAO	202101082005251		14
70	002280	KHUNE PRITI KIRAN	202101082005742		12
71	002281	KOLEKAR SHRADDHA SHASHIKANT	202101082003709		18
72	002283	KOLTE KESHAV ANIL	202101082005381		16
73	002284	KONDHARE SWAPNALI SHANTILAL	202101082005247		12
74	002285	KONDHARE ASHWINI BALASAHEB	202101082003646		15
75	002287	KOTWAL ALFIYA PASHA	202101082005243		14
76	002288	KSHIRSAGAR AMOL DATTATRAYA	202101082003956		10
77	002289	KULKARNI SOURABH SURESH	202101082003831		14
78	002292	LAD TEJA VIVEK	202101082005302		16
79	002293	LANDAGE FATEMABI AYYUB	202101082003759		15
80	002294	Landage Rohan Chaitanya	202101082003960		12
81	002296	LANDAGE SNEHA BALASAHEB	202101082003760		16
82	002298	LOKARE KIRTI SURESH	202101082003690		18
83	002299	LOMATE RAJNANDINI RAJENDRA	202101082003682		19
84	002301	MACHALE GAYATRI VITTHAL	202101082003648		18
85	002304	MALI OM SACHIN	202101082003661		13
86	002305	MALI VAISHNAVI VIJAYKUMAR	202101082005745		20
87	002306	MANE AISHWARYA SUBHASH	202101082003753		18
88	002307	MANE ANJALI KASHINATH	202101082003684		14
89	002308	MANE JANVHI RAHUL	202101082005065		18
90	002309	MANJARE RUTUJA KRUPACHRYA	202101082003994		18
91	002310	MANJARE SHWETA RAVINDRA	202101082005073		15
92	002314	MATE DATTATRYA ARJUN	202101082005017		11
93	002315	MISAL SANSKRUTI CHANDRAKANT	202101082005744		12
94	002317	MOHITE PRIYANKA SANTOSH	202101082003665		08
95	002318	MOKHANDE GANESH RAMKRUSHNA	202101082004774		13
96	002319	MOMALE SHRADDHA KAKASAHEB	202101082003866		10
97	002321	MORE ANKITA RAMKRUSHNA	202101082004882		18
98	002323	MUJAWAR SAHIL HASAN	202101082003821		09
99	002325	MULANI ANJUM SHARIF	202101082003664		18
100	002327	MULUK PRATIKSHA VILAS	202101082005336		18
101	002329	NAGAME RUPESH BABASAHEB	202101082004041		9
102	002330	NAGMODE DNYANESHWARI VIKAS	202101082003832		13
103	002331	NAGTILAK ASHWINI VIJAY	202101082004007		15

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Dr. Prathraj Kshirsagar

Head
Department of Botany
Shri Shivaji Mahavidyalaya
Barshi Dist-Solapur

Blank Mark List For

College Name : Shri Shivaji Mahavidyalaya, Barshi(0809)

Bachelor of Science (Botany)

B.Sc(Hons)Regular - CBCS Pattern 2019 - B.Sc(Hons) I Sem II

Paper Code : 19201208 Botany Practical

Sr.No.	Seat No.	Name	Practical I A (Min Mark : 8 Max Mark : 20)		
			Pdfx No	Marks	Total
104	002535	NALAWADE VAISHNAVI SANJAY	20210108200380		16
105	002536	NARUTE MAYURI BAPURAO	20210108200373		11
106	002540	PALKE VAISHNAVI MAHESH	20210108200433		15
107	002542	PANSE VAISHNAVI VAMAN	20210108200377		17
108	002543	PARASE DIGVIJAY CHANDRAKANT	20210108200516		15
109	002544	PATHAN SUJAN SALIM	20210108200373		12
110	002547	PATIL ANKITA AVINASH	20210108200488		10
111	002549	PATIL SANSHI ASHOK	20210108200574		12
112	002551	PATIL SHWETA VILAS	20210108200364		16
113	002552	PATIL YASHRAJ DATTATRAY	20210108200471		11
114	002553	PAUL TUSHAR SHRIRAM	20210108200487		11
115	002554	PAWAR AKSHATA DATTATRYA	20210108200370		16
116	002556	PAWAR PRAGATI SANTOSH	20210108200388		14
117	002557	PAWAR RANVEER RAJESH	20210108200368		17
118	002560	PAWAR SHRADDHA SANTOSH	20210108200381		13
119	002562	PISAL SIDDHANT SIDDHESHWAR	20210108201309		15
120	002563	RALIT UTKARSH UDHAS	20210108200374		12-
121	002565	RALIT NEHA SANJAY	20210108200381		16
122	002566	RALIT SANJYOTI SHRIKANT	20210108200407		20
123	002568	SALUNKE SWAPNALI NARAYAN	20210108200392		17
124	002569	SAPKAL DIGVIJAY KAKASAHEB	20210108200389		18
125	002570	Sarwade Samarth Sagar	20210108200386		17
126	002571	SATPUTE GANESH RAVSAHEB	20210108200403		12
127	002573	SHAIKH SANIYA NAYUM	20210108200375		13
128	002574	SHAIKH SALAMA AMJAD	20210108200524		20
129	002575	SHAIKH AMAN HUSSAIN	20210108200364		15
130	002576	SHAIKH BUSHRA MANSOOR	20210108200374		17
131	002578	SHELAKE AISHWARYA BHASKAR	20210108200371		20
132	002579	SHELAKE DIPALI SATISH	20210108200507		13
133	002580	SHELAKE DIPALI SHANKAR	20210108200406		12
134	002583	SHINDE SALONI RAMESH	20210108200524		16
135	002584	SHINDE ASMITA AMAR	20210108200367		16
136	002586	SHINDE SANDIP VAIJINATH	20210108200367		16
137	002587	SHINDE SHWETA SANJAY	20210108200539		14
138	002588	SHINDE VAISHNAVI APPASAHEB	20210108200408		19
139	002595	TAMBOLI SOHEL MOHAMMAD	20210108200376		18
140	002596	TELANGE KIRTI ANKUSH	20210108200369		13
141	002597	THAKARE MAYURI GANESH	20210108200321		19
142	002599	THONGE ANUJA ASHRAB	20210108200389		18
143	002600	THORE SHAKSHI AVIRATH	20210108200398		19
144	002601	TIKATE VAISHNAVI RAMKRUSHNA	20210108200501		12-
145	002602	TORADMAL SHWETA DASHARATH	20210108200540		11
146	002605	VILE VIPUL SHRIRAM	20210108200387		17
147	002606	WAGH PRAJAKTA SANJAY	20210108200539		17
148	002608	WAGHMARE AMRUTA RATNADIP	20210108200394		19
149	002609	WAGHMARE ANIRUDH RUSHIKANTH	20210108200372		13
150	002611	WAGHMARE DHANASHRI MOHAN	20210108200367		17
151	002613	WAGHMARE PRATIKSHA SAHEBRAO	20210108200372		08
152	002614	WAGHMARE ROHIT PANDURANG	20210108201310		17
153	002617	YADAV ANURAG RAJENDRA	20210108200365		17
154	002618	YADAV NIRANJAN NANDKUMAR	20210108200365		16
155	002619	YADAV PRITI UMESH	20210108200371		17
156	002620	YAMPURE AKASH NAGNATH	20210108200322		14

Total No of Students Found In This Paper : 156

Report Generated By: Mahadik S.D. on 17/08/2022 01:39:02pm

Dr. Pashtraj R. Kshirsagar

Head
Department of Botany
Shri Shivaji Mahavidyalaya
Barshi, Dist-Solapur

Punyashlok Ahilyadevi Holkar Solapur University, Solapur

Jeyantieerth Nagar, Kegaon, Solapur - Pune National Highway, Solapur- 413255, Maharashtra (India)

Printed on: 17/08/2022

Blank Mark List For

College Name: Shit Shivaji Mahavidyalaya, Barshi(SMB)

Bachelor of Science (Hons) I

B.Sc(Hons)Regular - CBCS Pattern 2019 - B.Sc(Hons)-I Sem-II

Paper Code: 18201204 Botany Practical

			Practical UA (Min Mark: 0 Max Mark: 80)		
Sr.No	Seat No	Name	PRN No	Marks	Total
1	002157	ADOLE ATHRAV SHRIRAM	2021010820036 35	64	
2	002158	AGALAVE RUSHIKESH TUKARAM	2021010820057 38	75	
3	002181	ANGHARE RATNA RAVINDRA	2021010820038 95	70	
4	002184	Atkare Sarika Sanjay Kumar	2021010820040 20	70	
5	002185	ATKARE TEJASVI KRISHNAT	2021010820036 81	72	
6	002189	BAGWAN MASIRA ARIF	2021010820038 87	72	
7	002170	BAPANEWAR AISHWARY TUKARAM	2021010820040 72	66	
8	002172	BARATE MADHURI RAMESHWAR	2021010820057 39	73	
9	002173	BARBOLE PRANALI DATTATRAYA	2021010820036 57	72	
10	002174	BARBOLE ROHINI SANJAY	2021010820036 79	78	
11	002175	BHATLAVANDE URMILA TANAJI	2021010820038 16	76	
12	002180	BHANTE PRAGATI ASHOK	2021010820047 75	71	
13	002181	BHANE VAISHNAVI RAJKUMAR	2021010820049 20	66	
14	002183	BHAUSALE VAISHNAVI RAJENDRA	2021010820053 93	69	
15	002184	Bhosale vaishanavi vishwanath	2021010820040 16	73	
16	002185	BHAUSALE VAISHNAVI DILIPKUMAR	2021010820036 43	64	
17	002186	BHUTE SAKSHI RAMCHANDRA	2021010820053 84	74	
18	002188	CHAUDHARI SHANKAR ASHOK	2021010820036 50	69	
19	002189	CHAVAN SAUDAGAR UTTAM	2021010820036 60	68	
20	002190	CHAVAN SHIVANAND DHARMARAJ	2021010820040 32	AB	AB
21	002192	CHAVAN VAISHNAVI ABHIMAN	2021010820131 93	67	
22	002193	Chobe Pooja Hanumant	2021010820038 83	63	
23	002197	DESHMUKH GEETANJALI SUDHAO	2021010820047 19	65	
24	002199	DEVKATE SAKSHI HANUMANT	2021010820038 20	41	
25	002201	DHERE SANKET RAMHARI	2021010820053 75	72	
26	002202	DHOLDE SHWETA SAMBHAJI	2021010820036 73	71	
27	002204	GAPAL PRIYANKA PANDURANG	2021010820036 75	71	
28	002206	GAIKAR SARIKA SHANKAR	2021010820036 78	74	
29	002208	GAIKWAD AJAY BALASAHEB	2021010820047 68	67	
30	002209	GAIKWAD ANKITA ANNASAHEB	2021010820048 68	65	
31	002210	GAIKWAD GAURI PRASHANT	2021010820053 39	68	
32	002211	GAIKWAD SANIKA NITIN	2021010820047 70	69	
33	002212	GALANDE ANUSHKA SURYAKANT	2021010820049 22	66	
34	002213	GALANDE ANKITA KAMALAKAR	2021010820049 24	59	
35	002214	GAVALI PRATHAMESH RAJENDRA	2021010820040 83	31	
36	002215	GAVHANE VAISHNAVI RAMESH	2021010820037 58	70	
37	002219	GAVHANE PRANITA NENINATH	2021010820037 22	72	
38	002218	GAVHANE RUTIKA BHARAT	2021010820056 40	58	
39	002219	Ghadyalak Vijay Manik	2021010820040 25	67	
40	002220	GHATTIYAK VRUSHALI APPASAHEB	2021010820037 20	67	
41	002224	GHOLAP VIJAY SHIVAJI	2021010820037 63	40	
42	002225	GHOLAVE VAISHNAVI BALCHAND	2021010820040 30	67	
43	002226	GHOTIYAK SHWETA NARSINH	2021010820037 62	72	
44	002232	GURAV GORAKH JAGANNATH	2021010820032 15	66	
45	002233	HANGE SAKSHI SANTOSH	2021010820038 68	67	
46	002234	JADHAV ADITI AMRAY	2021010820036 97	69	
47	002235	JADHAV ATHARV RANDEV	2021010820048 93	67	
48	002236	JADHAV DEVYANI VINAYAK	2021010820036 58	79	
49	002237	JADHAV DNYANESHWARI CHANDRAKANT	2021010820036 88	72	

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Dr. Pathraj R. Kshirsagar

Head
Department of Botany
Shri Shivaji Mahavidyalaya
Barshi, Dist-Solapur

Blank Mark List For					
College Name: Shri Shivaji Mahavidyalaya, Barshi(SMB)					
Bachelor of Science (Hons)-I					
B.Sc(Hons)Regular - CBCS Pattern 2019 - B.Sc(Hons)-I Sem-II					
Paper Code : 19201204-Botany Practical			Practical UA (Min Mark: 0 Max Mark: 80)		
Sr.No	Seat No	Name	PRN No	Marks	Total
50	002239	JADHAV ROHIT PRASHANT	202101082003826	70	
51	002242	JADHAV VAISHNAVI NANDKUMAR	202101082003718	70	
52	002243	JAGTAP MIRABAI VAJINATH	202101082003886	72	
53	002245	JAGTAP SHRUTI SATISH	202101082003829	68	
54	002246	JAGTAP VIJAYA SAHADEV	202101082003708	71	
55	002248	JAVHERI DAKSHA VISHAL	202101082004028	67	
56	002251	KADAM SAMARTH BHAUSAHEB	202101082003693	78	
57	002252	KADAM TANAYA ARJUN	202101082004885	77	
58	002253	KALE RUTUJA SHASHIKANT	202101082003772	76	
59	002259	KAPASE ISHWARI GANESH	202101082003828	77	
60	002260	KAPSE SAKSHI RAKESH	202101082003659	69	
61	002262	KARKAR OMRAJE SANTOSH	202101082004778	59	
62	002263	KASABE SAGAR RAJENDRA	202101082003209	68	
63	002267	KATMORE VAISHNAVI BAPU	202101082004891	65	
64	002270	KAZI FIZA MUDASSSIR	202101082003818	69	
65	002271	KHADBADE NIKITA SHANKAR	202101082003667	75	
66	002272	KHAIRE AISHWARYA BHAUSAHEB	202101082005386	69	
67	002275	KHANDAGALE ABHISHEK BALASAHEB	202101082004879	69	
68	002276	KHARADE RADHA SHIVHARI	202101082004718	70	
69	002279	KHODSE BALIKA BAPURAO	202101082005251	63	
70	002280	KHUNE PRITI KIRAN	202101082005742	70	
71	002281	KOLEKAR SHRADDHA SHASHIKANT	202101082003709	78	
72	002283	KOLTE KESHAV ANIL	202101082005381	74	
73	002284	KONDHARE SWAPNALI SHANTILAL	202101082005247	71	
74	002285	KONDHARE ASHWINI BALASAHEB	202101082003646	76	
75	002287	KOTWAL ALFIYA PASHA	202101082005243	66	
76	002288	KSHIRSAGAR AMOL DATTATRAYA	202101082003956	71	
77	002289	KULKARNI SOURABH SURESH	202101082003831	67	
78	002292	LAD TEJA VIVEK	202101082005302	72	
79	002293	LANDAGE FATEMABI AYYUB	202101082003759	71	
80	002294	Landage Rohan Chaitanya	202101082003960	60	
81	002296	LANDAGE SNEHA BALASAHEB	202101082003760	69	
82	002298	LOKARE KIRTI SURESH	202101082003690	73	
83	002299	LOMATE RAJNANDINI RAJENDRA	202101082003682	75	
84	002301	MACHALE GAYATRI VITTHAL	202101082003648	75	
85	002304	MALI OM SACHIN	202101082003661	61	
86	002305	MALI VAISHNAVI VIJAYKUMAR	202101082005745	75	
87	002306	MANE AISHWARYA SUBHASH	202101082003753	76	
88	002307	MANE ANJALI KASHINATH	202101082003684	67	
89	002308	MANE JANVHI RAHUL	202101082005065	75	
90	002309	MANJARE RUTUJA KRUPACHRYA	202101082003994	72	
91	002310	MANJARE SHWETA RAVINDRA	202101082005073	67	
92	002314	MATE DATTATRYA ARJUN	202101082005017	49	
93	002315	MISAL SANSKRUTI CHANDRAKANT	202101082005744	65	
94	002317	MOHITE PRIYANKA SANTOSH	202101082003665	33	
95	002318	MOKHANDE GANESH RAMKRUSHNA	202101082004774	67	
96	002319	MOMALE SHRADDHA KAKASAHEB	202101082003866	69	
97	002321	MORE ANKITA RAMKRUSHNA	202101082004882	72	
98	002323	MUJAWAR SAHIL HASAN	202101082003821	46	
99	002325	MULANI ANJUM SHARIF	202101082003664	68	
100	002327	MULUK PRATIKSHA VILAS	202101082005336	77	
101	002329	NAGAME RUPESH BABASAHEB	202101082004041	59	
102	002330	NAGMODE DNYANESHWARI VIKAS	202101082003832	64	
103	002331	NAGTILAK ASHWINI VIJAY	202101082004007	70	

Report Generated By: Mahadik S.D. on 17/08/2022 01.39.02pm

Shri Shivaji Mahavidyalaya, Barshi
DEPARTMENT OF BOTANY

Date: 17/06/2022

To,
The Principal,
Shri Shivaji Mahavidyalaya, Barshi
Dist: Solapur

Subject: Permission for Botanical excursion of B. Sc. I and II

Respected Madam,

With respect to above subject, Department of Botany is going to organize one day botanical excursion at Ramling hills, on 20/06/2022. As a part of syllabus of Punyashlok Ahilyadevi Holkar Solapur University, Solapur, botanical excursion report is compulsory in practical examination. Following faculty members will conduct this tour. Please sanction duty leave as well as TA and DA of these faculty members.

Sr. No.	Name of the Faculty
1	Mr. Patil P. A.
2	Mr. Bhise D. S.
3	Dr. Gaikwad S. P.
4.	Dr. Kshirsagar P. R.
5.	Dr. Gawali M. T.
6.	Ms Ganje P. B.
7.	Mr. Karanjakar S. N.

So, I hereby request to grant the permission for this excursion.

Thanking You,

Yours Faithfully,

विभाग प्रमुख
वनस्पतिशास्त्र विभाग
श्री शिवाजी महाविद्यालय बार्शी,
बार्शी, जि. सोलापूर

Allowed
Revd
18/06/2022

Shri Shivaji Mahavidyalaya, Barshi
Department of Botany
Botanical Excursion
Date 20/06/2022

Sr. No.	Name	Class
1.	Anbhule Ankita Atul	B. Sc. II
2.	Andhare Sakshi Bhausaheb	B. Sc. II
3.	Bagal Pratiksha Nagnath	B. Sc. II
4.	Bagale Ritu Laxman	B. Sc. II
5.	Barangule Aishwarya Vijay	B. Sc. II
6.	Chaudhari Rohan Govind	B. Sc. II
7.	Deshmukh Manoj Tukaram	B. Sc. II
8.	Dhanake Durga Maruti	B. Sc. II
9.	Doiphode Minal Maruti	B. Sc. II
10.	Doke Vijay Abasaheb	B. Sc. II
11.	Dupare Rohit Mahavir	B. Sc. II
12.	Gadade Pravin Bajrang	B. Sc. II
13.	Gaikwad Amruta Somnath	B. Sc. II
14.	Gaikwad Dnyaneshwar Nanasaheb	B. Sc. II
15.	Gaikwad Gouri Dhanaji	B. Sc. II
16.	Ghemad Ranjit Tulsidas	B. Sc. II
17.	Gholap Prajakta Subhash	B. Sc. II
18.	Gudekar Gayatri Vijay	B. Sc. II
19.	Hore Pranjali Hanumant	B. Sc. II
20.	Humbe Madhuri Bapu	B. Sc. II
21.	Jadhav Vaishnavi Vishwanath	B. Sc. II
22.	Jamdade Vaishnavi Nitin	B. Sc. II
23.	Kadam Pradnya Naganath	B. Sc. II
24.	Kadam Pratiksha Kamlakar	B. Sc. II
25.	Kadam Priti Somnath	B. Sc. II
26.	Kadam Rutuja Ramhari	B. Sc. II
27.	Kashid Rohit Rajkumar	B. Sc. II
28.	Kawale Aditya Ankush	B. Sc. II
29.	Kazi Shaista Tamij	B. Sc. II
30.	Khatal Manisha Mohan	B. Sc. II
31.	Kore Shivshankar Surykant	B. Sc. II
32.	Landage Rohini Chaitanya	B. Sc. II
33.	Limkar Saurabh Saudagar	B. Sc. II
34.	Maske Vaishnavi Dattatray	B. Sc. II
35.	Misal Shraddha Nandkumar	B. Sc. II
36.	More Chaitali Baliram	B. Sc. II
37.	Mulani Afrin Akbar	B. Sc. II
38.	Muthal Nikita Navanath	B. Sc. II
39.	Naiknaware Shweta Umesh	B. Sc. II

Chemica

Name :- Deshmukh Bharati Suryakant.

class :- B.sc. III

Roll no :- 3012

scientist :- Alfred Werner.

Shri. Shivaji Mahavidyalay, Barshi...

Biography

Alfred Werner

Born :- 12 December 1866
Mulhouse, Haut-Rhin
Alsace, France.

Died :- 15 November 1919
(aged 52)
Zurich, Switzerland.

Nationality :- Swiss

Alma mater :- University of Zurich. ETH Zurich

known for :- Configuration of transition metal complexes

Awards :- Nobel Prize for chemistry (1913)

Scientific career

Fields :- Inorganic chemistry.

Institutions :- University of Zurich

Doctoral :- Arthur Rudolf

Advisor :- Hantzsch, Marcellin
Berthelot.

Research

Coordination Chemistry

In 1893, Werner was the first to propose correct structures for coordination compounds containing complex ions, in which a central transition metal atom is surrounded by neutral or anionic ligands.

For example, it was known that cobalt forms a "complex" hexammine cobalt (III) chloride, with formula $CoCl_3 \cdot 6NH_3$, but the nature of the association indicated by the dot was mysterious. Werner proposed the structure $[Co(NH_3)_6]Cl_3$, With the Co^{3+} ion surrounded by six NH_3 at the vertices of an octahedron. The three Cl^- are dissociated as free ions. Which Werner confirmed by measuring the conductivity of the compound in aqueous solution, and also by chloride anion analysis using precipitation with Silver nitrate. Later, magnetic susceptibility analysis was also used to confirm Werner's proposal for the chemical nature of $CoCl_3 \cdot 6NH_3$.

For complexes with more than one type of ligand, Werner succeded in explaining the no. of isomers observed. For e.g., he explained the existence of two tetrammine isomers, "$Co(NH_3)_4Cl_2$", one green and one purple. Werner proposed that these are two geometric isomers of formula $[Co(NH_3)_4Cl_2]Cl$, with one Cl^- ion dissociated as confirmed by conductivity measurments. The Co atom is surrounded by four NH_3 and two Cl^- ligands at the vertices of an octahedron.

Werner also prepared complexes with optical isomers, and in 1914 he reported the first synthetic chiral compound lacking C, known as hexol with Formula $[Co(C_6NH_3)_4(OH)_2]Br_6$

Nature of Valence

Before, Werner, Chemists defined the valance of an element as the number of its bonds without distingui-sh different types of bond, However, in complexes, such as $[Co(NH_3)_6]Cl_3$ for example, Werner considered that the Co-Cl bonds corresponds to a "primary" value of 3 at long distance, While the Co-NH_3 bonds which correspond-to a "Secondary" or weaker valance of 6 at shorter distance. This secondory valence of 6 he referred to as the co-ordination number which he defined as the no. of molecules directly linked to the central metal atom. In other complexes he found coordination number of 4 or 8.

on these views, and other similar views, in 1904, Richard Abegg formulated what is now known as Abbegg's rule which states that the difference between the maxi-mum positive and negative valence of an electron is frequently eight. This rule was used later in 1916 when Gilbert N. Lewis formulated the 'octet rule" in his Cubical atom theory.

In modern terminology, Werner's primary valence cor-rsponds to the oxidation state, and his secondory valence is called coordination number. The Co-Cl bonds are now classed as ionic, and each Co-N bond is a coordinate coralent bond between the Lewis acid Co^{3+} and the Lewis base NH_3.

* chemica *

Name :- chikane Komal Sharad

class :- B.Sc. III

Roll no :- 3009

Takashi Nagai

Nagai Takashi, 3 February 1908, Matsue - 1 May 1951, Nagasaki) was a physician specializing in radiology, a convert to Roman Catholicism, & a survior of the atomic bombing of Nagasaki. His subsequent life of prayer & seevice earned him the affe-ctionate title "Saint of Urakami" & has subsequently been honoured with the title of seevent of God.

Takashi Nagai

In mourning for his wife (1946)

Born	3 February 1908 Matsue City, Shimane Prefecture
Died	1 May 1951 (aged 43) Nagasaki, Japan
Nationality	Japan
Scientific career	
Fields	Radiology

- Contents :
- Life :-
  - Early years.
- Life in Nagasaki.
- Conversion to catholicism
- Sino-Japanese War
- World War II
- Relief Activities
- Postwar years
- Thought :
  - Use of Nuclear Power
- Works :
  - Bibilography :
    - Writing
    - Translation
    - Editing & Writing
- Media :

★ Life :-

★ Early Yeaes:

Takashi Nagai was born in 1908 on 3 Feb. His father, Noboru Nagai, was trained in Western medicine; his paternal grandfather, Fumitaka Nagai, was a practitioner of traditional herbal medicine. His mother, Tsune, was the descendant of an old family of Samurai. In Japanese, Takashi means "nobility".

Nagai was raised in the rural area of Mitoya & raised in according to the teachings of Confucius & the Shinto religion. In 1920, he commenced his secondary studies at Matsue High School boarding at his cousin's home, not far from Matsue. Occidental sciences & the materialistic spirit were dominant among his profesor & came to hear the story of the teaching of Christ from this teacher.

Life in Nagasaki :-

In April 1928, he joined the Nagasaki Medical College. The reason he chose the college is unclear, as he did not explain it clearly to his parents, siblings, friends or classmates; nor did he write anything about it.

During his studies he embarked upon the spiritual journey that would eventually lead him from atheism to Catholicism. The college was located 500meters from Urakami Cathedral, but Nagai had faith only in Science, humanity & Japan.

Chemica

NAME - Sarde Madhuri Ramdas

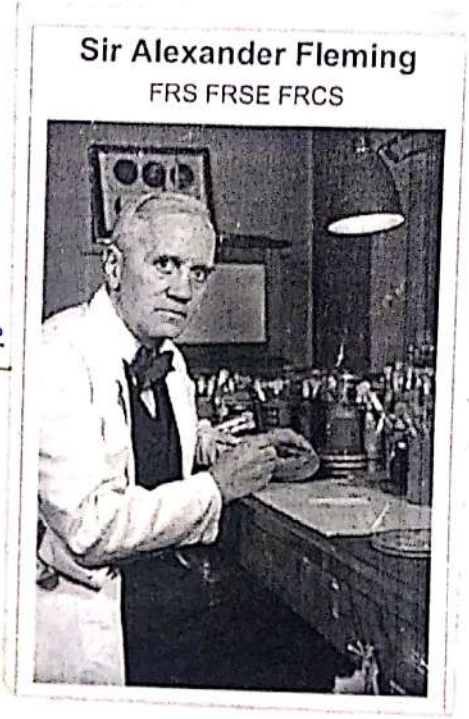
class - B.Sc - III

Roll No - 3067

Scientist - Alexander Fleming

Shri Shivaji Mahavidyalaya
Barshi

Sir Alexander Fleming



Born - 6 August 1881
Darvel, East Ayrshire
Scotland

Died - 11 March 1955
(aged 73)
London, England

Citizenship - British

Alma mater - Royal Polytechnic Institution - St Mary's Hospital Medical School - Imperical college London

Known for - Discovery of Penicillin [1]

Awards - FRS (1943)
Nobel Prize (1945) [2]
FRSE
FRCS [Eng]
Knight Bachelor (1944)

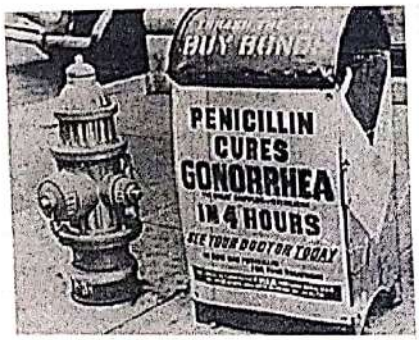
Scientific career

Fields - Bacteriology, immunology

Research

Work before Penicillin

During world war, I Fleming witnessed the death of many soldiers from sepis resulting from infected wounds. Antiseptics, which were used at the time to treat infected wounds, often worsened the injuries. [9] In an article he submitted for the identical medical journal The Lancet during world war I, Fleming described an ingenious experiment, which he was able to conduct as a result of his own glass blowing skills, in which he explained why antiseptics were killing more soldiers than infection itself during world war I. Antiseptics worked well on the surface, but deep wounds tended to shelter anaerobic bacteria from the antiseptic agent, & antiseptics seemed to remove beneficial agents produced that protected the Patients in these cases at least as well as they removed bacteria, & did nothing to remove the bacteria that were out of reach. [10] Sir Almroth Wright strongly supported Fleming's findings, but despite this, most army Physicians over the course of the war continued to use antiseptics even in cases where this worsened the condition of the Patients. [7]



Miracle cure.

Antibiotics

Fleming's accidental discovery & isolation of Penicillin in September 1928 marks the start of modern antibiotics. Before that, several scientists had published or pointed out that mould or Penicillium Sp. were able to inhibit bacterial growth, & even to cure bacterial infections in animals. Ernest Duchesne in 1897 in his thesis "contribution to the study of vital competition in micro-organisms: antagonism between moulds & microbes", [21] or also Clodomiro Picado Twight whose work at Institut Pasteur in 1923 on the inhibiting action of fungi of the "Penicillin SP" genre in the growth of staphylococci drew little interest from the direction of the Institut at the time. Fleming was the first to push these studies further by isolating the penicillin, & by being motivated enough to promote his discovery at a larger scale. Fleming also discovered very early that bacteria developed antibiotic resistance whenever too little penicillin was used or when it was used for too short a period. Almroth coright had predicted antibiotic resistance even before it was noticed during experiments. Flemings cautioned about the use of penicillin in his many speeches around the world. on 26 June 1945, he made the following cautionary Statements. "— the microbes are educated to resist Penicillin & a host of penicillin-fast organism is bred out—