CHEMISTRY TEACHERS FORUM OF
KARNATAK UNIVERSITY AFFILIATED COLLEGES

CHEM-FORUM
2017-18

COMPREHENSIVE B.Sc. CHEMISTRY
LABORATORY MANUAL

As per
Karnatak University Syllabus
2012-2013

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CHEMISTRY TEACHERS FORUM OF KARNATAK UNIVERSITY AFFILIATED COLLEGES

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CHEMISTRY TEACHERS' FORUM OF KARNATAK UNIVERSITY AFFILIATED COLLEGES

CHEM – FORUM: 2017-18

B.SC. CHEMISTRY COMPREHENSIVE LABORATORY MANUAL

EDITORIAL BOARD

Name of the Book : **Comprehensive B.Sc. Chemistry Laboratory Manual**

No. of pages in the book : 300

No. of copies printed : 150

Purpose of printing : To have common procedure to conduct B.Sc. Chemistry Practicals without any ambiguity.

Publication Coordinator: President and Secretary, Chem – Forum: 2017-18.

Publication Convener : Dr. K. S. Katagi, Karnatak Science College, Dharwad

Published by Chem – Forum : 2017-18 in association with Karnatak Science College, Dharwad
On 22nd June, 2018.

Books are distributed by Karnatak College, Dharwad to all the affiliated Colleges of Karnatak University 02 copies each as a service to the society with a motto of ***"Quality Education for All"*** *on the occasion of its centenary celebration.*

Foreword

Chemistry is an experimental science. The fundamental basis for Chemistry is experiments. It is through experiments that we study and explain certain phenomenon. Chemistry lives by experiments by combining or separating, by analysis or synthesizing different substances.

Experiments carried out in many undergraduate chemistry courses have not been upgraded for quite some time, while the situation has changed a lot in the leading universities in the country. The quality of experiments carried out in many B.Sc. Chemistry laboratories also remains substandard and rudimentary due to the outcome of lack of proper instruction material and infrastructure.

A Laboratory Manual prepared by Chemistry Teachers' Forum is enormously important for Chemistry education. This manual is an attempt to bring in tested, modern and interesting Chemistry experiments especially to undergraduate students of Karnatak University and to all Chemistry students in general. Eighty carefully described experiments show that you do not need a lot of sophisticated material or advanced equipment to teach students, especially undergraduates, how to do interesting experiments in chemistry.

For the future, it is important to teach students how to do Modern Chemistry. This laboratory manual is a valuable contribution to Undergraduate Chemistry Education. Each experiment has been carefully elaborated; materials, equipments and even time required for performing the experiments have also been listed. You will find a theoretical description of the background of the materials employed and practical uses of the different substances produced. This will be interesting especially for younger students who aim to become future experts in Chemistry.

Working with this laboratory manual will be a great pleasure and success for every teacher who wants to motivate interested and talented students.

Vidyanand Revankar
Chairman
Board of Studies in Chemistry
Karnatak University, Dharwad

PREFACE

The idea of writing this manual came when we realized that our B.Sc. Course with Chemistry as Optional Subject do not have a common and comprehensive Laboratory Manual for all the experiments prescribed in the syllabi. In this direction all the Chemistry Teachers met in 24th Annual Convention of **Chem – Forum** held on the 25th May, 2018 and thereby resolved unanimously to prepare a single, properly structured book which guides us how to conduct the practicals for under graduate students effectively with a special emphasis to unambiguous. This book is therefore an attempt to provide a single manual for the purpose.

The most important feature of this Manual is that it not only focuses on the procedure for all the experiments but also provides the instructions to the examiners along with questions to conduct the practical examinations. It can act as a lighthouse for examiners. It also narrates the list of specific and commonly using reagents and methods of preparation with calculations.

Foremost on behalf of Chem – Forum, I wish to express my indebtedness to Prof. Pramod B. Gai, Hon'ble Vice Chancellor, Karnatak University, Dharwad who inspired us to prepare this manuscript for the benefits of the students, teachers and lab-staff. I am grateful to Prof. Pancharatna K, Dean, Faculty of Science, and Prof. V. K Revankar, Chairman, P.G. Department of Chemistry for their constant motivation and guidance.

I am deeply thankful to Dr (Smt) M. S. Salunke and Dr. H.Y. Merwade, Chairpersons of Inorganic and Organic Sections respectively and their team along with the team members of Physical Section for preparing this manual in a record time of just 15 days.

I also owe my special thanks to all the members of Chem – Forum for their splendid contributions to provide draft for this manual. This Manual would not have seen the light of day without the combined efforts of the Team of Chemistry Faculty of Karnatak University' affiliated Colleges especially the efforts of Dr. S.B. Sajjanar for coordinating with all the committees and contribution towards the development of this manual.

I sincerely thank Dr. C.F. Mulimani, Principal, Karnatak Science College, Dharwad for encouraging with financial support on the occasion of centenary celebration of Karnatak College. I am deeply thankful to Dr. K. S. Katagi for his untiring zeal in designing the front cover and also getting the print copies on time.

I would like to thank the entire Chemistry Faculty of Karnatak Science College and J.S.S. College, Dharwad for their active participation in finalizing the draft. Hope, all the Chemistry faculty members of affiliated colleges of Karnatak University ensures a culture of good lab practices, examinations and lab safety in the colleges.

Written in a very simple and lucid language, the students, teachers and lab-staff should find this book extremely handy and useful. A complete e-version of this manual is available on the Karnatak Science College website (www.kscd.ac.in) and Karnatak University website too (www.kud.ac.in).

Though all the efforts have been made to make this book error free, yet some errors might have crept inadvertently. Suggestions from the readers for the improvement of the book are most welcome.

Finally, I hope that this book will be helpful not only to the Karnatak University's under graduate students but also for students of other Universities where many of these experiments are included in their syllabi.

Dr. Suresh M Tuwar : President, Chem – Forum: 2017-18.

Contents

KARNATAK UNIVERSITY
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NAAC Accredited
DHARWAD
"University with Potential for Excellence"
"ಉತ್ಕೃಷ್ಟ ಸಾಮರ್ಥ್ಯ ಹೊಂದಿರುವ ವಿಶ್ವವಿದ್ಯಾಲಯ"

ಪ್ರೊ. ಪ್ರಮೋದ ಭೀ. ಗಾಯಿ
ಕುಲಪತಿಗಳು ಎಂ.ಎಸ್‌ಸಿ., ಪಿಎಚ್.ಡಿ.,

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DATE : 22-06-2018

MESSAGE

It is my pleasure to write about the Comprehensive Laboratory Manual for the entire B.Sc Course with Chemistry as optional subject of Karnatak University. I congratulate all the members of the Editorial Board for translating this unique idea into action which is first of its kind in the annals of Karnatak University. This is a model worthy of emulation by all the Science departments which will go a long way in introducing Uniformity in teaching conducting and evaluation of practicals.

I must say that the Karnatak College which is celebrating its centenary year has lived up to its reputation in its selfless service to provide quality education for all the undergraduate students.

I congratulate all the concerned teachers of the Chem- Forum- a vibrant body of chemistry teachers for their concern and care towards the student community. I hope to see such Comprehensive/Educative manuals in other subjects which will serve the purpose of Quality Education to all especially in the remote areas of Karnatak University Jurisdiction.

I once again congratulate all the teachers from different Colleges and wish them all success.

Prof. Pramod B. Gai

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B.Sc I Sem
Inorganic Chemistry Experiments

Total No of hours/week : 4Hrs Total No of hours/week : 4Hrs
Total No. of Hours : 54 Hrs Total No. of Hours : 54 Hrs

Distribution of Marks:	
Accuracy	
Standardization titration	10 marks
Main titration	16 marks
Technique & Presentation	04 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

Deduction of Marks for accuracy:

Standardization titration:	**Main titration**:
± 0.2 cc -10 marks	± 0.2 cc -16 marks
± 0.4 cc- 08 marks	± 0.4 cc- 14 marks
± 0.6 cc- 06marks	± 0.6 cc- 12 marks
± 0.8cc- 04 marks	± 0.7 cc- 10 marks
Above ± 0.8 cc- zero marks	± 0.8 cc- 08 marks
	± 0.9 cc- 06 marks
	above ± 1.0 – zero mark

1. CALIBRATION OF GLASSWARE AND WEIGHTS USING CHEMICAL BALANCE

Aim: To calibrate the glass wares (pipette, burette and volumetric flask) and weights (both grams and milligrams) and to use the chemical balance.

Apparatus: Pipette, burette, conical flasks, volumetric flask, funnel, beaker etc.

Calibration of glass wares: In laboratory work a measuring flask, pipette or burettes are used in volumetric experiments. For accurate knowledge of the volume of liquids taken by these, the glassware needs calibration.

Pipettes: Two types of pipettes, volumetric and graduated (or measuring) are available. A volumetric pipette is meant for highly accurate work whereas a graduated pipette is not very accurate. A volumetric pipette is an accurate volume conveyor and unless it is properly used, the volumes discharged by it will not be constant. 10 cc, 25 cc & 50 cc pipettes will have the respective delivery times of 20 seconds, 30 seconds & 35 seconds; if the pipette is forced to discharge too rapidly, the discharged volume will vary.

A pipette is always held vertical and the liquid is allowed to drain itself out. When the liquid is allowed to drain out, the last drop would not be forced out either by blowing or by warming the bulb; however, the jet end is brought in contact with the sides of the beaker or conical flask is as to enable any last drop portion which may freely flow out. This procedure ensures that always the same constant volume is drained out of the pipette.

Calibration of pipettes (10 cc & 25 cc)

1. 10 cc and 25 cc pipettes are rinsed with chromic acid mixture and dipped in it for overnight.
2. Both of them are washed thoroughly and then rinsed with distilled water.
3. Distilled water is sucked up in to 10 cc pipette up to the mark and it is then released in to a previously weighed clean & dry conical flask.
4. The time required for the release of last drop from the pipette is noted down.
5. The mass of the flask along with water is determined.
6. The volume of the water is found with the help of mass and density formula.

$$\text{Volume} = \frac{\text{Mass}}{\text{Density}}$$

7. The same procedure is repeated for 25 cc pipette also.

Observations

1. Mass of empty and dry conical flask = M_1 = ---g
2. Mass of conical flask + water M_2 = ---g
3. Mass of water = $M_2 - M_1$ = ---g

Calibration of burette

1. Burette is filled with chromic acid mixture and kept it overnight and thoroughly washed with water.
2. Burette is finally filled with distilled water. Water is run out until the meniscus is exactly on the zero mark and out flow is then stopped.
3. 5 cc of water from the burette (i.e. from 0-5) released in to a previously weighed clean & dry conical flask.
4. The mass of conical flask with water is determined and mass of 5cc water is calculated.
5. Again 5 cc of water from the burette from 5-10 is released in to the same conical flask and weighed it again and mass of 5cc water is calculated.
6. This process is repeated up to 50 cc.
7. The density of water at room temperature is noted down.
8. The volume of water is calculated by the formula, $\text{Volume} = \frac{\text{Mass}}{\text{Density}}$
9. The volume correction is noted down.

Mass of 1 litre of water & volume of 1 g of water at various temperatures

Temp (° C)	Density (g/cc)	Mass	Volume of 1 g of water	Temp (° C)	Density (g/cc)	Mass	Volume of 1 g of water
20	0.9982	997.18	1.0028	24	0.9975	996.38	1.0036
21	0.9979	997.00	1.0030	25	0.9970	996.17	1.0038
22	0.9977	996.80	1.0032	26	0.9967	995.93	1.0041
23	0.9975	996.60	1.0034	27	0.9965	995.69	1.0043

Observations

1. Laboratory temperature = ---° C
2. Density of water at laboratory temperature = ………….g/cc

Burette levels	Mass of water	Volume of water	Correction
0-5			
5-10			
10-15			
15-20			
20-25			
25-30			
30-35			
35-40			
40-45			
45-50			

Calibration of measuring flasks (250 cc & 100 cc) relative to a burette

1. 250 cc Measuring flask is filled with chromic acid mixture and kept it overnight and thoroughly washed with water and dried.
2. The measuring flask is filled with distilled water that is added from the calibrated burette.
3. How much water is required to fill up to the mark is noted down.
4. From the mass of the water volume is calculated and hence the volume of standard flask is calibrated. Or

1. 100 cc Measuring flask is filled with chromic acid mixture and kept it overnight and thoroughly washed with water and dried.
2. 100 cc Measuring flask is weighed and the mass is noted down.
3. The measuring flask is filled with distilled water up to the mark and the adhered drops of water are removed by absorbing with filter paper.
4. Measuring flask containing distilled water is weighed again and the mass is noted down.
5. Using the mass of water filled up in the measuring flask volume is calculated.

Observations

1. Mass of empty and dry measuring flask = M_1 = ---g
2. Mass of measuring flask + water M_2 = ---g
3. Mass of water = M_2- M_1 = ---g
4. Laboratory temperature = --- ^{o}C
5. Density of water at laboratory temperature = ….g/cc

Calibration of weights

Weights should be calibrated in order to detect any inaccuracies in weights themselves. This is especially so in the case of fractional weights sold as separate unit. The weights used to be weighed for weighing on a chemical balance should be calibrated. i.e. their irregularities should be checked and corrected by comparing against the weights of standard weight box.

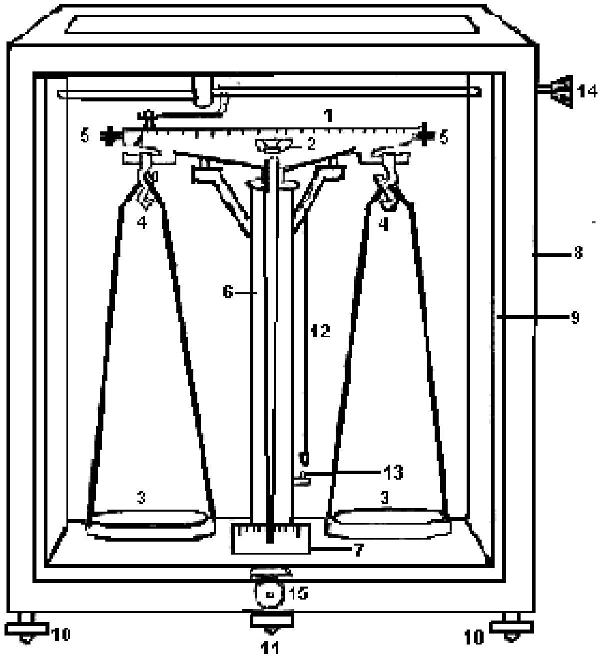
1. For calibration work the balance is first adjusted to zero resting point and the weights to be calibrated are compared one by one against the corresponding weights of standard weight box.
2. The gram weights are usually provided with screw top. Their irregularities can be corrected by unscrewing the top and altering the fine sand or minute lead shots in the cavity.
3. For milligrams it is convenient to select only the good once and reject those showing variations. However heavier milligrams can be reduced in weights by rubbing against emery paper or cutting with a pair of scissors.

Fractional weights	Corrected weight
500 mg	
200 mg	
200 mg	
100 mg	
50 mg	
20 mg	
20 mg	
10 mg	

Use of Chemical balance

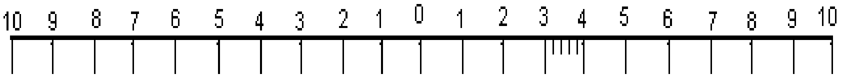
A chemical balance is a device used to measure the mass of an object(substance) accurately by comparing its weight with that of the standard weights from a weight box.

FIGURE: It consists of following parts**.**

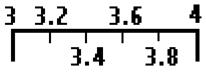


1. Beam of balance
2. Fulcrum of agate knife edge
3. Balance pans
4. Stirrup with double hooks
5. Adjustment screws
6. Pointer
7. Pointer scale
8. Balance case
9. Front opening for the case
10. Levelling screws
11. Pointed peg
12. Plumb line
13. Index
14. Handle for rider movement
15. Knob

Main scale division



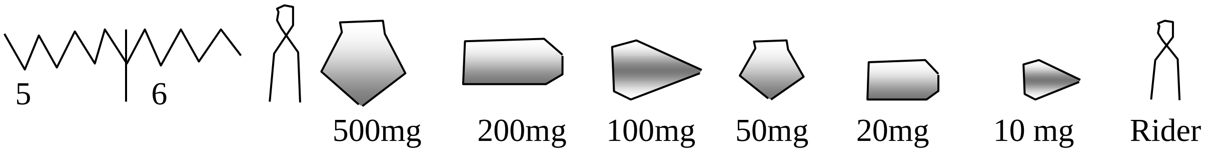
Subscale division



Weight box: Two weight boxes are used 1. Gram weight box. 2.Fractoinal weight box.

Gram weight box consists brass weights coated with nichrome of 1, 2, 2, 5, 10, 20, 20, 50 & 100 grams

Fractional weight box consists 10, 20,20 50,100,200, 200, 500 milli grams & a rider of 10mg



Use of rider: The weights less than 10 mg(0.0010 g) can be avoided conveniently by the use of rider. The beam of balance is divided in to ten divisions on either side and each division is subdivided in to 5. When the rider is placed on 1st subdivision next to 0, it weighs 0.0002 g, 2nd 0.0004, 3rd 0.0006 etc. When it is place on main division 1, it weighs 0.0010 g. similarly if placed on 2nd, 3rd etc it weighs 0.002, 0.003 g respectively and if placed on 10th main division then the weight is 0.0100 g.

Procedure (B): I. Adjustment of Balance

1. The leveling screws at the base are adjusted so that the plumb point suspended behind the pillar is exactly above the index below. This indicates that the balance is perfectly horizontal.

2. The knob is turned gently so that the beam is raised and the pointer oscillates on the pointer scale. If the pointer oscillates equally on both sides of the pointer scale, the balance is said to be adjusted. Otherwise, knob is turned back and the adjustment screw nuts are turned suitably. The movement of the pointer is again checked for its oscillation equally on either side of the pointer scale.

II.To find the mass of an object:

1. The object to be weighed is placed on the left side pan when the balance is at rest. The weights are placed on the right side pan in the descending order using forceps. Every time after placing the weight, the knob is turned gently to see whether the pointer moves equally on either sides of the pointer scale. The least weight that is used in weighing is 10 mg. When the last 10 mg weight is less than the actual weight of the substance, the rider is placed on any of the main scale or sub scale division on the beam so that the pointer moves equally on either side of the pointer scale. This completes the weighing of the given object.

2. The weights placed on the pan as well as the weight due to the rider together form the weight of the object. If the rider is placed to the right part of the beam, its weight is added to the weight in the pan. If the rider is placed on the left pan its weight is deducted from the weight in the pan. This gives the accurate weight of the object.

Note:

1. The knob should be turned only gently. Otherwise the stirrups as well as beams will be dislocated due to sudden jerk.
2. Weights from the weight box as well as milligram weights should be transferred using forceps only.
3. Hot articles should not be weighed as they spoil the pans.
4. The doors should be closed when the oscillation of the pointer is to be observed.
5. The balance beam should be brought to rest while transferring the objects or weights.
6. Chemical substances should be weighed using weighing bottles only.

OBSERVATION:

Gram	**Rider weight 10 mg** ↔			
5 1	0 0 5 1	0 0 0 0 2 1	0 0 0 0 0 0 5	6
6	6	3	5	6

RESULT: The accurate mass of the object (watch glass) is **6.6356**g

Use of Digital or Electronic Balance

1. Place the electronic balance on a flat & stable surface table
2. Press the “ON” button and wait for the balance to show zeroes on the digital screen.
3. Use a container (like weighing bottle, watch glass or beaker) for the object (never place directly on the pan.
4. Note down the weight of the container and press “TARE” or “ZERO” button to automatically deduct the weight of the container from the future calculations. The digital display will show zero again, indicating that the container’s mass is stored in the balance memory.
5. Carefully add the substance to the container. Ideally this is done with the container still on the platform, but it may be removed if necessary.
6. Place the container with the substance back on the balance platform if necessary and record the mass as indicated by the digital display.

2. DETERMINATION OF HYDROCHLORIC ACID

Aim: To Prepare the standard oxalic acid solution, standardization of sodium hydroxide solution and to determine the hydrochloric acid in the given solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Oxalic acid, hydrochloric acid, sodium hydroxide, phenolphthalein indicator, etc.

Theory: Though Oxalic acid ($H_2C_2O_4.2H_2O$. Mol.wt = 126) is not a primary standard (satisfactory secondary standard) but due to the stability, non-hygroscopic & solubility. It is a dibasic acid with the gram equivalent mass (E) as 63. The amount of oxalic acid crystals to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of oxalic acid is used to standardize the secondary standard solutions like sodium hydroxide, potassium permanganate etc.

The given sodium hydroxide solution is standardized by titrating against the standard solution of oxalic acid using phenolphthalein as an indicator till the colour changes from colourless to pale pink.

$$2NaOH + H_2C_2O_4 \longrightarrow Na_2C_2O_4 + 2H_2O$$

The amount of hydrochloric acid present in the given solution is determined by titrating against the standard solution of sodium hydroxide using phenolphthalein as an indicator till the colour changes from colourless to pale pink.

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

Procedure

A. Preparation of standard (0.05 N) oxalic acid solution:

Accurately weighed (0.7875 g) oxalic acid is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of sodium hydroxide solution:

The burette is washed with water, rinsed with the given sodium hydroxide solution and filled with the same solution up to the zero mark (Avoid air bubbles). The pipette is washed with distilled water and rinsed with oxalic acid solution. The given conical flask is washed with distilled water.

Exactly 25 cc of oxalic acid solution is pipetted out into the clean conical flask. Two drops of phenolphthalein indicator is added and the solution is titrated against oxalic acid solution till the colour changes from colourless to pale pink. The burette reading is noted. The titration is repeated to get concordant values and determined exact normality of sodium hydroxide solution.

C. Determination of Hydrochloric acid:

The hydrochloric acid solution supplied in the 250 cc volumetric flask is diluted up to the mark with distilled water. It is shaken well for uniform concentration or *Sample solution of HCl may be directly supplied*. 25 cc of this solution is pipetted out into a clean conical flask and 2 to 3 drops of phenolphthalein indicator is added and titrated against sodium hydroxide solution till the colour changes from colourless to pale pink. The titration is repeated to get concordant values. Using appropriate formula Hydrochloric acid in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05N) oxalic acid solution

The amount of oxalic acid required = $w = \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 63 \times 250}}{\mathbf{1000}} = 0.7875$ **g**

Mass of empty watch glass : m_1 = ... g
Mass of watch glass + oxalic acid : m_2 = ... g
Mass of oxalic acid : $(m_2 - m_1)$ = ... g

If the value of mass of oxalic acid weighed differs from 0.7875 g then, normality of oxalic acid solution is calculated by

$\therefore$ Normality of oxalic acid solution $= \frac{\text{Mass of oxalic acid x 4}}{\text{Eq.mass of oxalic acid}}$

$= \frac{(m_2 - m_1) \text{ x } 4}{63} = \ldots\ldots\ldots\ldots\ldots.\text{N}$

B. Standardization of Sodium hydroxide:

Solution taken in the burette : NaOH solution

Solution taken in the conical flask : 25 cc oxalic acid solution

Indicator used : Phenolphthalein

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading(A)	Final reading(B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of NaOH V_1 = volume of NaOH (CBR)

N_2 = Normality of oxalic acid and V_2 = volume of oxalic acid(25cc)

Normality of NaOH , $N_1 = \frac{N_2 \ V_2}{V_1}$ = ... N

C. Determination of the amount of hydrochloric acid:

Solution taken in the burette : NaOH solution

Solution taken in the conical flask : 25 cc HCl solution

Indicator used : Phenolphthalein

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of HCl V_1 = Volume of HCl =25 cc

N_2 = Normality of NaOH V_2 = Volume of NaOH(CBR)

Therefore, Normality of HCl, $N_1 = \frac{N_2 \ V_2}{V_1}$ = ... N

We know that equivalent mass of HCl = 36.5

Amount of HCl present in dm^3 of solution (X) = Normality of HCl x Eq.mass of HCl
=.......... g

Amount of HCl present in 250 cc of solution = $\frac{X}{4}$
=.......... g

Result:

1.	Normality of oxalic acid solution	.............N
2.	Normality of Sodium hydroxide solution	.............N
3.	Normality Hydrochloric acid solution	.............N
4.	Amount of Hydrochloric acid present in 250 cc of solution	.............g

3. DETERMINATION OF SODIUM HYDROXIDE

Aim: To prepare of standard sodium carbonate solution, standardize hydrochloric acid solution and to determine sodium hydroxide in the given solution.

Apparatus : Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Sodium carbonate, hydrochloric acid, sodium hydroxide, phenolphthalein indicator methyl red indicator etc.

Theory : Sodium carbonate (Mol.wt = 106) is referred as the primary standard in the titrimetric analysis with a gram equivalent mass (E) as 53.It produces weakly basic solution on dissolution in water. The amount of sodium carbonate to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of sodium carbonate is used to standardize the secondary standard solutions like hydrochloric acid.

The given hydrochloric acid solution is standardized by titrating against the standard solution of sodium carbonate using methyl red as an indicator till the colour changes from yellow to red.

$$Na_2CO_3 + 2HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

The amount of sodium hydroxide present in the given solution is determined by titrating against the standard solution of hydrochloric acid using phenolphthalein as an indicator till the colour changes from pink to colourless.

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

Procedure:

A. Preparation of standard (0.05 N) Sodium carbonate solution:

Accurately weighed (0.6625 g) sodium carbonate is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of hydrochloric acid solution:

The burette is washed with water, rinsed with the given hydrochloric acid solution and filled with the same solution up to the zero mark (Avoid air bubbles). The pipette is washed with distilled water and rinsed with sodium carbonate solution. The given conical flask is washed with distilled water.

Exactly 25 cc of sodium carbonate solution is pipetted out into the clean conical flask. Two drops of methyl red indicator is added and the solution is titrated against sodium carbonate solution till the colour changes from yellow to red. The burette reading is noted. The titration is repeated to get concordant values and exact normality of hydrochloric acid solution is calculated.

C. Determination of the amount of sodium hydroxide:

The sodium hydroxide solution supplied in the 250 cc volumetric flask is diluted up to the mark with distilled water. It is shaken well for uniform concentration or *sample solution of NaOH may be directly supplied*. 25 cc of this solution is pipetted out into a clean conical flask and 2 to 3 drops of phenolphthalein indicator is added and titrated against of hydrochloric acid solution till the colour changes from pink to colourless. The titration is repeated to get concordant values. Using appropriate formula sodium hydroxide in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05N) sodium carbonate solution

The amount of sodium carbonate required $= \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 53 \times 250}}{\mathbf{1000}} = 0.6625\ \mathbf{g}$

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + sodium carbonate : m_2 = ……..g

Mass of sodium carbonate : $(m_2 - m_1)$ = --------- g

If the value of mass of sodium carbonate weighed differs from 0.6625 g then, normality of sodium carbonate solution is calculated by

∴ Normality of sodium carbonate solution = $\frac{\text{Mass of sod. carbonate x 4}}{\text{Eq.mass of sod.carbonate}}$

$$= \frac{(m_2 - m_1) \times 4}{53} = \dots\dots.\text{N}$$

B. Standardization of hydrochloric acid:

Solution taken in the burette : HCl solution

Solution taken in the conical flask : 25 cc of sodium carbonate

Indicator used : Methyl red

Colour change at the end point : Yellow to red

Tabulations

Trial No	Burette readings (cc)		Volume of HCl added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of HCl V_1 = Volume of HCl (CBR)

N_2 = Normality of sodium carbonate and V_2 = Volume of sodium carbonate

Therefore, Normality of HCl, $N_1 = \frac{N_2\ V_2}{V_1}$ = ... N

C. Determination of Sodium hydroxide:

Solution taken in the burette : HCl solution

Solution taken in the conical flask : 25 cc of NaOH solution

Indicator used : Phenolphthalein

Colour change at the end point : Pink to colourless

Tabulations

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of NaOH V_1 = Volume of NaOH (25)

N_2 = Normality of HCl and V_2 = Volume of HCl (CBR)

Therefore, Normality of NaOH, $N_1 = \frac{N_2\ V_2}{V_1}$ = ... N

We know that equivalent mass of NaOH = 40

Amount of NaOH present in dm^3 of solution (X) = Normality of NaOH x Eq.mass of NaOH

=......... g

Amount of NaOH present in 250 cc of solution = $\frac{X}{4}$

=......... g

Result:

1.	Normality of sodium carbonate solution	............N
2.	Normality of hydrochloric acid solution	............N
3.	Normality of sodium hydroxide solution	............N
4.	Amount of sodium hydroxide present in 250 cc of solution	............g

4. DETERMINATION OF SODIUM CARBONATE AND SODIUM HYDROXIDE FROM THE MIXTURE

Aim: To prepare the standard sodium carbonate solution, standardize hydrochloric acid solution and to determine sodium carbonate and sodium hydroxide from their mixture of the solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Sodium carbonate, hydrochloric acid, phenolphthalein indicator and methyl orange, etc.

Theory: Sodium carbonate (Mol.wt. =106) is referred as the primary standard in the titrimetric analysis with the gram equivalent mass (E) of 53 and forms weakly basic solution in water. The amount of sodium carbonate to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of sodium carbonate is used to standardize the secondary standard solutions like hydrochloric acid.

The given hydrochloric acid solution is standardized by titrating against the standard solution of sodium carbonate using phenolphthalein as an indicator till the colour changes from pink to colourless.

$$Na_2CO_3 + 2HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

The sodium carbonate and sodium hydroxide present in the given solution is determined by titrating against the standard solution of hydrochloric acid by the selective use of indicators. The sodium hydroxide undergoes complete neutralisation with HCl to form NaCl, where as sodium carbonate reacts with HCl to form first $NaHCO_3$ and then NaCl. The phenolphthalein is added as first indicator which decolorizes its pink colour in alkaline medium when NaOH and half Na_2CO_3 are neutralised (Na_2CO_3 turns to $NaHCO_3$). The $NaHCO_3$ is slightly acidic in nature (pH =4); hence phenolphthalein is not a suitable indicator for its determination. Therefore, methyl orange is used as the second indicator which shows the complete neutralisation ($NaHCO_3$ turns to NaCl) of solution of NaOH and Na_2CO_3 mixture

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

$$Na_2CO_3 + HCl \longrightarrow NaCl + NaHCO_3$$

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

Procedure:

A. Preparation of standard (0.1 N) Sodium carbonate solution:

Accurately weighed (1.325g) sodium carbonate is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250 cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. **Standardization of hydrochloric acid solution**:

The burette is washed with water, rinsed with the given hydrochloric acid solution and filled with the same solution up to the zero mark (Avoid air bubbles). The pipette is washed with distilled water and rinsed with sodium carbonate solution. The given conical flask is washed with distilled water. Exactly 25 cc of sodium carbonate solution is pipetted out into the clean conical flask. Two drops of phenolphthalein indicator is added and the solution is titrated against sodium carbonate solution till the colour changes from pink to colourless. The burette reading is noted. The titration is repeated to get concordant values and determined exact strengtg(normality) of hydrochloric acid solution.

C. **Determination of sodium carbonate and sodium hydroxide from their mixture of the solution**:

The mixture of sodium hydroxide and sodium carbonate solution supplied in the 250 cc volumetric flask is diluted up to the mark with distilled water or *ready mixture of the solution may be directly supplied*. 25 cc of this solution is pipetted out into a clean conical flask and 2 to 3 drops of phenolphthalein indicator is added and titrated against hydrochloric acid solution till the colour changes from pink to colourless. The burette reading is noted as V_1. The titration is further continued by adding 2-3 drops of methyl orange indicator till the colour changes from yellow to red. The burette reading is noted as V_2. The above procedure repeated to get concordant values.

Using appropriate formula sodium carbonate and sodium hydroxide from the given mixture is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.1N) sodium carbonate solution

The amount of sodium carbonate required = $= \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.1 \times 53 \times 250}}{\mathbf{1000}} = 1.325$ **g**

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + sodium carbonate : m_2 = ……..g

Mass of sodium carbonate : $(m_2 - m_1)$ = --------- g

If the value of mass of sodium carbonate weighed differs from 1.325 g then, normality of sodium carbonate solution is calculated by

∴ Normality of sodium carbonate solution $= \frac{\text{Mass of sod. carbonate x 4}}{\text{Eq.mass of sod.carbonate}}$

$= \frac{(m_2 - m_1) \times 4}{53} =$ .... N

B. Standardization of hydrochloric acid:

Solution taken in the burette : HCl solution
Solution taken in the conical flask : 25 cc of sodium carbonate
Indicator used : Methyl red
Colour change at the end point : Yellow to orange red

Tabulations

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 =Normality of HCl V_1 = Volume of HCl (CBR)

N_2= Normality of sodium carbonate and V_2 = volume of sodium carbonate

Therefore, Normality of HCl, $N_1 = \frac{N_2\ V_2}{V_1}$ = ... N

C. Determination of sodium carbonate and sodium hydroxide from their mixture of the solution:

Solution taken in the burette : HCl solution

Solution taken in the conical flask : 25 cc of mixture of NaOH and Na_2CO_3 solution

Indicator used : Phenolphthalein for first stage & methyl orange for second stage

Colour change at the end point : For first stage Pink to colourless for second stage yellow to red

Tabulations **I stage (with Phenolphthalein)** $NaOH + HCl \xrightarrow{\quad} NaCl + H_2O$

$$Na_2CO_3 + HCl \xrightarrow{\quad} NaCl + NaH\,CO_3$$

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			CBR = V_1 cc
2	0.0			
3	0.0			

II stage (with Methyl red) $NaHCO_3 + HCl \xrightarrow{\quad} NaCl + H_2O + CO_2\uparrow$

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	Final Level of I stage			CBR = V_2 cc
2	,,			
3	,,			

Concordant burette reading for V_1 =.......cc & for V_2 = ...cc

Calculations :

Determination of amount of Na_2CO_3

V_1: Volume of HCl used for complete neutralization of NaOH and half neutralization of Na_2CO_3(conversion of Na_2CO_3 to $NaHCO_3$)

V_2 : Volume of HCl used for half neutralization of Na_2CO_3

∴ Volume of HCl required for half neutralization of Na_2CO_3 $= V_2 - V_1 =$ ….c.c.

∴ Volume of HCl required for complete neutralization of Na_2CO_3 $2\,(V_2 - V_1) =$ ..c.c.

$$N_1 = \frac{N_2\ V_2}{V_1} = N_{\text{Sod carbonate}} = \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{V_{\text{Sod.carbonate}}} = \frac{N_{\text{HCl}} \times 2\,(V_2 - V_1)}{25} = ..\mathbf{N}$$

∴Grams per litre of sodium carbonate$= N_{Na_2CO_3} \times \textbf{equivalent mass of } \mathbf{Na_2CO_3 = N \times 53 = X}$

∴Amount of Na_2CO_3 present in the given 250 cc solution $= \frac{X}{4} = ..\mathbf{g} = \mathbf{a}$

Determination of amount of NaOH

∴ Volume of HCl required for complete neutralization of NaOH = total volume of HCl – volume of HCl required for neutralization of Na_2CO_3 = $V_2 - 2\,(V_2 - V_1)$ =…cc

Equation used $N_1 = \frac{N_2\ V_2}{V_1}$

$$N_{\text{NaOH}} = \frac{N_{\text{HCl}} \times V_{\text{HCl}}}{V_{\text{NaOH}}} = \frac{N_{\text{HCl}} \times V_2 - 2\,(V_2 - V_1)}{V_{\text{NaOH}}} = ..\mathbf{N}$$

∴ Grams per litre of NaOH$= N_{\text{NaOH}} \times$ equivalent mass of NaOH $= \mathbf{N \times 40 = Y}$

∴ Amount of NaOH present in the given 250 cc solution $= \frac{Y}{4} =..$ g = b

Percentage of Na_2CO_3 = $\frac{a}{a+b} \times 100 = \cdots$ %

Percentage of NaOH = $\frac{b}{a+b} \times 100 = \cdots$ %

Result:

1.	Normality of sodium carbonate solution	............N
2.	Normality of hydrochloric acid solution	............N
3.	Amount of NaOH present in 250 cc of the mixture	............g
4.	Amount of Na_2CO_3 present in 250 cc of the mixture	............g
5.	Percentage (%) of NaOH in the mixture	..........%
6.	Percentage (%) of Na_2CO_3 in the mixture	..........%

Note: NaOH & Na_2CO_3 solutions are prepared separately and added separately using two burettes in to the same volumetric flask to distribute to students.

5. DETERMINATION OF SODIUM CARBONATE AND SODIUM BICARBONATE FROM THEIR MIXTURE

Aim: To prepare the standard sodium carbonate solution, standardize hydrochloric acid solution and to determine sodium carbonate and sodium bicarbonate from their mixture of the solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Sodium carbonate, hydrochloric acid, sodium bicarbonate, phenolphthalein and methyl orange etc.

Theory: Sodium carbonate (Mol.wt = 106) is referred as the primary standard in the titrimetric analysis with the gram equivalent mass (E) of 53 and its aqueous solution is weakly basic in nature. The amount of sodium carbonate to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of sodium carbonate is used to standardize the secondary standard solutions like hydrochloric acid.

The given hydrochloric acid solution is standardized by titrating against the standard solution of sodium carbonate using phenolphthalein as an indicator till the colour changes from pink to colourless.

$$Na_2CO_3 + 2HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

The sodium carbonate and sodium bicarbonate present in the given solution is determined by titrating against the standard solution of hydrochloric acid by the selective use of indicators. The sodium carbonate alone present in the given mixture reacts first with HCl to form $NaHCO_3$. The phenolphthalein is added as an indicator to know the end point of the titration which turns its colour from pink to colourless. As the $NaHCO_3$ is slightly acidic in nature, phenolphthalein is not a suitable indicator at this stage. The sodium bicarbonate formed from sodium carbonate and sodium bicarbonate present in the given mixture neutralises with next addition of HCl to form NaCl. During this stage of titration, methyl orange is selected as suitable indicator, to know the end point of titration which turns its colour from yellow to red.

$$Na_2CO_3 + HCl \longrightarrow NaCl + NaHCO_3$$

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

Procedure

A. Preparation of standard (0.1 N) Sodium carbonate solution:

Accurately weighed (1.325 g) sodium carbonate is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250 cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of hydrochloric acid solution:

The burette is washed with water, rinsed with the given hydrochloric acid solution and filled with the same solution up to the zero mark (Avoid air bubbles). The pipette is washed with distilled water and rinsed with sodium carbonate solution. The given conical flask is washed with distilled water.

Exactly 25 cc of sodium carbonate solution is pipetted out into the clean conical flask. Two drops of phenolphthalein indicator is added and the solution is titrated against sodium carbonate solution till the colour changes from pink to colourless. The burette reading is noted. The titration is repeated to get concordant values and these are used to calculate exact strength or normality of hydrochloric acid solution.

C. Determination of sodium carbonate and sodium bicarbonate from their mixture:

The mixture of sodium carbonate and sodium bicarbonate solution supplied in the 250 cc volumetric flask is diluted up to the mark using distilled water or *ready mixture of the solution may be directly supplied*. 25 cc of this solution is pipetted out into a clean conical flask and 2 to 3 drops of phenolphthalein indicator is added and titrated against the standard hydrochloric acid solution till the colour changes from pink to colourless. The burette reading is noted as V_1. The titration is further continued by adding 2-3 drops of methyl orange indicator till the colour changes from yellow to red. The burette reading is noted as V_2. The above procedure repeated to get concordant values.

Using the appropriate formula amounts sodium carbonate and sodium bicarbonate in the given mixture is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.1 N) sodium carbonate solution

The amount of sodium carbonate required $= \frac{\mathrm{N} \times \mathrm{E} \times \mathrm{V}}{1000} = \frac{0.1 \times 53 \times 250}{1000} = 1.325\ \mathrm{g}$

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + sodium carbonate : m_2 = ……..g

Mass of sodium carbonate : $(m_2 - m_1)$ = --------- g

If the value of mass of sodium carbonate weighed differs from 1.325 g then, normality of sodium carbonate solution is calculated by

∴ Normality of sodium carbonate solution = $\frac{\text{Mass of sod. carbonate} \times 4}{\text{Eq.mass of sod.carbonate}}$

$$= \frac{(m_2 - m_1) \times 4}{53} = \ldots\ldots \mathrm{N}$$

B. Standardisation of hydrochloric acid:

Solution taken in the burette : HCl solution

Solution pipetted out in conical flask : Sodium carbonate

Indicator used : Methyl orange

Colour change at the end point : Yellow to orange red

Tabulations

Trial No	Burette readings (cc)		Volume of HCl added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of HCl V_1 = Volume of HCl (CBR)

N_2 = Normality of sodium carbonate and V_2 = Volume of sodium carbonate

Therefore, Normality of HCl, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

C. Determination of sodium carbonate and sodium bicarbonate from their mixture:

Solution taken in the burette : HCl solution

Solution pipetted out in conical flask : Mixture of Na_2CO_3 and $NaHCO_3$ solution

Indicator used : Phenolphthalein for first stage & methyl orange for second stage

Colour change at the end point : For first stage Pink to colourless for second stage yellow to red

Tabulations **I stage (with Phenolphthalein)** $Na_2CO_3 + 2HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			CBR = V_1 cc
2	0.0			
3	0.0			

II stage (with Methyl red) $Na_2CO_3 + HCl \longrightarrow NaCl + NaHCO_3$

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

Trial No	Burette readings (cc)		Volume of HCl added (B – A) (cc)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	Final Level of I stage			CBR = V_2 cc
2	,,			
3	,,			

Concordant burette reading for V_1 =.......cc & for V_2 = ...cc

Calculations

Determination of amount of Na_2CO_3

V_1: Volume of HCl used for half neutralization of Na_2CO_3.

V_2 :Volume of excess of HCl used for remaining half neutralization of Na_2CO_3 **and** complete neutralization of $NaHCO_3$.

∴ Volume of HCl required for half neutralization of Na_2CO_3 = V_1 =… c.c.

∴ Volume of HCl required for complete neutralization of Na_2CO_3 = 2 × V_1 = …c.c.

Concentration or strength of sodium carbonate

$$\mathrm{N}_1 = \frac{\mathrm{N_2\ V_2}}{\mathrm{V_1}} = \mathrm{N}_{\textbf{Sod carbonate}} = \frac{\mathrm{N_{HCl} \times V_{HCl}}}{\mathrm{V_{Sod.carbonate}}} = \frac{\mathrm{N_{HCl} \times 2V_1}}{\mathrm{V_{Sod.carbonate}}} \ldots = \mathbf{N}.$$

We know that, equivalent mass of Na_2CO_3 = 53

∴ Grams per litre of sodium carbonate

$$= \mathrm{N}_{\textbf{Sod carbonate}} \times \text{equivalent mass of Sod carbonate} = \mathbf{N \times 53 = X}$$

∴ Amount of Na_2CO_3 present in the given 250 cc solution = $\frac{\mathbf{X}}{\mathbf{4}} = = \cdots \mathbf{g} \ \ = \mathbf{a}$

Determination of amount of $NaHCO_3$

∴ Volume of HCl required for complete neutralization of $NaHCO_3$

= total volume of HCl – volume of HCl required for neutralization of Na_2CO_3

= V_2 - 2 × V_1 =…. c.c.

Concentration or strength of sodium bicarbonate

$$\mathrm{N}_1 = \frac{\mathrm{N_2\ V_2}}{\mathrm{V_1}} = \mathrm{N}_{\mathbf{NaHCO_3}} = \frac{\mathrm{N_{HCl} \times V_{HCl}}}{\mathrm{V_{NaHCO_3}}} = \frac{\mathrm{N_{HCl} \times \ V_2 - 2V_1}}{\mathrm{V_{NaHCO_3}}} \ldots = \mathbf{N}$$

We know that, equivalent mass of $NaHCO_3$ = 84

∴ Grams per litre of $NaHCO_3$= $\mathrm{N_{NaHCO_3}} \times \text{equivalent mass of } \mathrm{NaHCO_3} = \mathbf{N \times 84 = Y}$

∴ Amount of $NaHCO_3$ present in the given 250 cc solution = $\frac{\mathbf{Y}}{\mathbf{4}} = \cdots \ \mathbf{g} \ \ = \mathbf{b}$

Percentage of Na_2CO_3 = $\frac{\mathbf{a}}{\mathbf{a+b}} \times 100 = \cdots \%$: Percentage of $NaHCO_3$ = $\frac{\mathbf{b}}{\mathbf{a+b}} \times 100 = \cdots \%$

Result:

1.	Normality of sodium carbonate solution	.........N
2.	Normality of hydrochloric acid solution	........N
3.	Amount of Na_2CO_3 present in 250 cc of the mixture	.........g
4.	Amount of $NaHCO_3$ present in 250 cc of the mixture	........g
5.	Percentage (%) of Na_2CO_3 in the mixture	… ….%
6.	Percentage (%) of $NaHCO_3$ in the mixture	…….%

Note: Na_2CO_3 & $NaHCO_3$ solutions are prepared separately and added separately using two burettes in to the same volumetric flask distribute to students..

6. DETERMINATION OF OXALIC ACID AND SULPHURIC ACID FROM THEIR MIXTURE

Aim: To prepare the standard oxalic acid solution, standardize NaOH and $KMnO_4$ solutions and to determine oxalic acid and sulphuric acid from their mixture of the solution

Apparatus : Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Sodium hydroxide, oxalic acid, potassium permanganate, phenolphthalein, Sulphuric acid.

Theory: Though Oxalic acid ($H_2C_2O_4.2H_2O$. Mol.wt = 126) is not a primary standard (satisfactory secondary standard), but due to the stability, non-hygroscopic nature & solubility. It is a dibasic acid with the gram equivalent mass (E) as 63. The amount of oxalic acid crystals to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of oxalic acid is used to standardise the secondary standard solutions like sodium hydroxide, potassium permanganate etc.

The given sodium hydroxide solution is standardized by titrating against the standard solution of oxalic acid using phenolphthalein as an indicator till the colour changes from colourless to pale pink.

The titration of oxalic acid versus potassium permanganate is an example of redox titration in which oxalic acid gets oxidised to carbon dioxide and potassium permanganate gets reduced to manganese sulphate. This reaction is carried out in acidic medium by adding 2N sulphuric acid and titrated against $KMnO_4$ in hot condition (60-70°C). In this titration $KMnO_4$ acts as self indicator changes colour from colourless to pale pink near the end point.

The given mixture of the solution contains both oxalic acid and sulphuric acid. Oxalic acid present in the mixture can be estimated by titrating a known volume of the mixture against standard $KMnO_4$ solution. The following reactions take place.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$[\ H_2C_2O_4 + [O] \longrightarrow H_2O + 2CO_2\uparrow] \times 5$$

$$\overline{2KMnO_4 + 3H_2SO_4 + 5\,H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow}$$

The total acid present in the mixture can be estimated by titrating a known volume of the mixture against standard NaOH solution using phenolphthalein indicator. The following reactions take place.

$$H_2C_2O_4 + 2NaOH \longrightarrow Na_2C_2O_4 + 2H_2O$$

$$H_2SO_4 + 2NaOH \longrightarrow Na_2SO_4 + 2H_2O$$

Procedure:

A. Preparation of standard (0.05 N) oxalic acid solution:

Accurately weighed (0.7875 g) oxalic acid is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of sodium hydroxide solution:

The burette is washed with water, rinsed with the given sodium hydroxide solution and filled with the same solution up to the zero mark (Avoid air bubbles). The pipette is washed with distilled water and rinsed with oxalic acid solution. The given conical flask is washed with distilled water.

Exactly 25 cc of oxalic acid solution is pipetted out into the clean conical flask. Two drops of phenolphthalein indicator is added and the solution is titrated against oxalic acid solution till the colour changes from colourless to pale pink. The burette reading is noted. The titration is repeated to get concordant values. Using these readings the exact normality of sodium hydroxide is calculated.

C. Standardization Potassium permanganate solution:

Exactly 25 cc of oxalic acid solution is pipetted out into a clean conical flask. $1^1/_2$ test tube of dilute sulphuric acid (2N) is added and heated nearly to boiling. It is then titrated against potassium permanganate solution taken in the burette. The end point is reached when permanent pale pink colour is obtained. The titration is repeated to get concordant values and the exact normality of Potassium permanganate solution is determined by using concordant reading.

D. Determination of oxalic acid from the mixture of oxalic acid and sulphuric acid solution.

The acid mixture supplied in the 250 cc standard flask is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution of the mixture of oxalic acid and sulphuric acid solution may be directly supplied*. 25 cc of the acid mixture is pipetted out into a clean conical flask. $1^1/_2$ test tube of dilute H_2SO_4 is added and the solution is heated to (60-70°C) just short of boiling. The hot solution is titrated against standard $KMnO_4$ solution till permanent pink colour is obtained. The titration is repeated to get concordant values and the amount of oxalic acid in the given mixture is calculated.

E. Determination of sulphuric acid from the mixture of oxalic acid and sulphuric acid solution.

25 cc of the acid mixture is pipetted out into a clean conical flask and 2 drops of phenolphthalein indicator is added, the solution is titrated against standard NaOH solution till permanent pale pink colour is obtained. The titration is repeated to get concordant values. Using the appropriate formula amounts of oxalic acid and sulphuric acid in the given mixture is determined.

Observation and calculation:

A. preparation of 250 cc of standard (0.05 N) oxalic acid solution

The amount of oxalic acid required $= \frac{\text{NEV}}{1000}$

$$= \frac{0.05\times 63 \times 250}{1000} = 0.7875 \text{ g}$$

Mass of empty watch glass $m_1 = \ldots\ldots..$g

Mass of watch glass + ***oxalic acid*** : m_2 = ……..g

Mass of ***oxalic acid*** : $(m_2 - m_1)$ = --------- g

If the value of mass of oxalic acid weighed differs from 0.7875 g then, normality of oxalic acid solution is calculated by

Therefore Normality of oxalic acid solution $= \frac{\text{Mass of oxalic acid x 4}}{\text{Eq.mass of oxalic acid}}$

$$= \frac{(m_2 - m_1) \times 4}{63} = \ldots\ldots.\mathrm{N}$$

B. Standardization of Sodium hydroxide:

Solution taken in the burette : NaOH solution

Solution pipetted out in a conical flask : 25 cc Oxalic acid

Indicator used : Phenolphthalein

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1V_1 = N_2V_2$

N_1 =Normality of NaOH V_1 = volume of NaOH (CBR)

N_2= Normality of oxalic acid and V_2 = volume of oxalic acid (25)

Therefore, Normality of NaOH, $N_1 = \frac{N_2\ V_2}{V_1}$ =.......... N

C. Standardization of Potassium permanganate solution:

Solution taken in the burette : $KMnO_4$ solution

Solution taken in the conical flask : 25cc oxalic acid+ $1^1/_2$ test tube 2N sulphuric acid

Indicator used : $KMnO_4$ solution itself

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of $KMnO_4$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of $KMnO_4$ V_1 = Volume of $KMnO_4$ (CBR)

N_2 = Normality of oxalic acid and V_2 = Volume of oxalic acid(25)

Therefore, Normality of $KMnO_4$, $N_1 = \frac{N_2 V_2}{V_1}$ =.......... N

D. Determination of oxalic acid from the mixture of oxalic acid and sulphuric acid solution:

Solution taken in the burette : $KMnO_4$ solution

Solution taken in the conical flask : 25 cc of mixture of oxalic acid and sulphuric acid solution + + $1^1/_2$ test tube 2N sulphuric acid and heated to 70 – 80 ^oC.

Indicator used : $KMnO_4$ solution itself

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volum of $KMnO_4$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations:

Determination of oxalic acid from the mixture of oxalic acid and sulphuric acid solution

Let ‘**a**’ be the normality of $H_2C_2O_4$ and ‘**b**’ the normality of H_2SO_4

∴ Total normality of the mixture = (a+b)

Equation used $N_1 V_1 = N_2 V_2$

N_1 =Normality of oxalic acid from the mixture of oxalic acid and sulphuric acid solution= (a+b)

V_1 = volume of oxalic acid from the mixture of oxalic acid and sulphuric acid solution = 25 cc

N_2 = Normality of $KMnO_4$ and V_2 = Volume of $KMnO_4$

Normality of oxalic acid from the mixture of oxalic acid and sulphuric acid solution:

$$N_1 = \frac{N_2\ V_2}{V_1} = N_1 = \frac{N_2 \times CBR}{25} =$$

= ------N = a

We know that, equivalent mass of oxalic acid = 63.0

Amount of oxalic acid from the mixture of oxalic acid and sulphuric acid solution present in dm^3 of solution (X) = Normality of oxalic acid x Eq.mass of oxalic acid (63) = ... g

Amount of oxalic acid from the mixture of oxalic acid and sulphuric acid solution present in 250 cc of solution = $\frac{X}{4}$ =...... g = ‘R’

E. Determination of sulphuric acid from the mixture of the solution.

Solution taken in the burette : NaOH solution

Solution taken in conical flask : 25 cc of mixture of oxalic acid and sulphuric acid**.**

Indicator used : Phenolphthalein

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations :

Determination of sulphuric acid from the mixture of the solution

Equation used $N_1 V_1 = N_2 V_2$

N_1 = total Normality of mixture of oxalic acid and sulphuric acid

V_1 = Volume of mixture of oxalic acid and sulphuric acid (CBR),

N_2 = Normality of NaOH and V_2 = Volume of NaOH

Total Normality of mixture of oxalic acid and sulphuric acid, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N= ***(a+b)***

Total Normality of sulphuric acid from the mixture of oxalic acid and sulphuric acid = (**a+b)**

∴ Normality of sulphuric acid = b = (**a + b) - a**

We know that, equivalent mass of Sulphuric acid = 49.0

Amount of sulphuric acid from the mixture of oxalic acid and sulphuric acid solution present in dm^3 of solution (Y) = (**a + b) - a** x Eq. Mass of sulphuric acid(49) =.......... g

Amount of sulphuric acid from the mixture of oxalic acid and sulphuric acid solution present in 250 cc of solution $= \frac{Y}{4}$ = ... g = ‘S’

Percentage of Oxalic acid = $\frac{R}{R+S} \times 100 =$... %

Percentage of Sulphuric acid = $\frac{S}{R+S} \times 100 =$...%

: **Result:**

1.	Normality of oxalic acid solution	......N
2.	Normality of sodium hydroxide solution	.......N
3.	Normality of potassium permanganate solution	......N
4.	Amount of oxalic acid from the mixture of oxalic acid and sulphuric acid solution	.....g
5.	Amount of sulphuric acid from the mixture of oxalic acid and sulphuric acid solution	......g
6.	Percentage of Oxalic acid	...%
7.	Percentage of Sulphuric acid	%

7. DETERMINATION OF MOHR'S SALT

Aim: To prepare the standard solution of oxalic acid, standardize potassium permanganate solution and to determine Mohr's salt (Ferrous Ammonium Sulphate, $FeSO_4.(NH_4)_2SO_4.6H_2O$) solution.

Apparatus : Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals : Oxalic acid, potassium permanganate, Mohr's salt and sulphuric acid.

Theory : Oxalic acid ($H_2C_2O_4.2H_2O$. Mol.wt = 126) is referred as satisfactory secondary standard because of its stability, non-hygroscopic nature & solubility in the titrimetric analysis. It is a dibasic acid with the gram equivalent mass (E) as 63. The amount of oxalic acid crystals to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of oxalic acid is used to standardise the secondary standard solutions like sodium hydroxide, potassium permanganate etc.

Potassium permanganate and oxalic acid are oxidizing and reducing agents respectively. The titration of oxalic acid versus $KMnO_4$ is an example of redox titration in which oxalic acid gets oxidised to carbon dioxide and $KMnO_4$ gets reduced to manganese sulphate. This reaction is carried out in acidic medium by adding 2N sulphuric acid and titrated against $KMnO_4$ in hot condition (60-80° C). In this titration $KMnO_4$ acts as self indicator which changes colourless to light pink at the end point.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$[\ H_2C_2O_4 + [O] \longrightarrow H_2O + 2CO_2\uparrow] \times 5$$

$$\overline{2KMnO_4 + 3H_2SO_4 + 5\ H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow}$$

Mohr's salt (FAS) is a reducing agent. To determine the amount of Mohr's salt ($FeSO_4.(NH_4)_2SO_4.6H_2O$) present in the given solution, the solution is diluted to 250 cc in the standard flask and a known volume of this solution is titrated against $KMnO_4$ solution. $KMnO_4$ solution oxidizes ferrous sulphate to ferric sulphate.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$[\ 2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O\ ] \times 5$$

$$\overline{2KMnO_4 + 8H_2SO_4 + 10FeSO_4 \longrightarrow K_2SO_4 + 5\ Fe_2(SO_4)_3 + 2MnSO_4 + 8H_2O}$$

Procedure:

A. Preparation of standard (0.05 N) oxalic acid solution:

Accurately weighed (0.7875 g) oxalic acid is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization Potassium permanganate solution: Exactly 25 cc of oxalic acid solution is pipetted out into a clean conical flask. $1^1/_2$ test tube of dilute sulphuric acid (2N) is added and heated nearly to boiling. It is then titrated against potassium permanganate solution taken in the burette. The end point is reached when permanent pale pink colour is obtained. The titration is repeated to get concordant values and is used to calculate exact normality of potassium permanganate.

C. Determination of Mohr's salt: The Mohr's salt solution supplied in the standard flask is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution of Mohr's salt may be directly supplied*. Exactly 25 cc of this solution is pipetted out into a clean conical flask. $1^1/_2$ test tube of dilute sulphuric acid is added and the solution is titrated against potassium permanganate solution taken in the burette. The end point is reached when permanent pale pink colour is obtained. The titration is repeated to get concordant values.

Using the appropriate formula, amount Mohr's salt present in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05N) oxalic acid solution

The amount of oxalic acid required = w $= \frac{\mathrm{N \times E \times V}}{1000} = \frac{0.05 \times 63 \times 250}{1000} = 0.7875$ g

Mass of empty watch glass : m_1 = ... g

Mass of watch glass + oxalic acid : m_2 = ... g

Mass of oxalic acid : $(m_2 - m_1)$ = ... g

If the value of mass of oxalic acid weighed differs from 0.7875 g then, normality of oxalic acid solution is calculated by

$$\therefore \text{Normality of oxalic acid solution} = \frac{\text{Mass of oxalic acid} \times 4}{\text{Eq.mass of oxalic acid}}$$

$$= \frac{(m_2 - m_1) \times 4}{63} = \ldots\ldots\ldots\ldots\ldots.\text{N}$$

B. Standardization of potassium permanganate solution:

Solution taken in the burette : $KMnO_4$ solution

Solution taken in the conical flask : 25 cc oxalic acid + $1^1/_2$ test tube of 2N H_2SO_4

Indicator used : $KMnO_4$ solution itself

Colour change at the end point : Colourless to pale pink

Trial No	Burette readings (cc)		Volume of $KMnO_4$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1 V_1 = N_2 V_2$

N_1 =Normality of $KMnO_4$ V_1 = volume of $KMnO_4$ (CBR)

N_2 = Normality of oxalic acid and V_2 = volume of oxalic acid

Therefore, Normality of $KMnO_4$, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

C. Determination of Mohr's salt:

Solution taken in the burette : $KMnO_4$ solution

Solution taken in the conical flask :25cc Mohr's salt solution + $1^1/_2$ test tube of 2N H2SO4

Indicator used : $KMnO_4$ solution itself

Colour change at the end point : Colourless to pale pink

Tabulations

Trial No	Burette readings (cc)		Volume of $KMnO_4$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of Mohr's salt V_1 = Volume of Mohr's salt

N_2 = Normality of $KMnO_4$ and V_2 = Volume of $KMnO_4$

Therefore, Normality of Mohr's salt solution, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

We know that, equivalent mass of Mohr's salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$]= 392.1

Amount of Mohr's salt present in dm^3 of solution (X)

= Normality of Mohr's salt x Eq.mass of Mohr's salt(392.1)

=.......... g

Amount of Mohr's salt present in 250 cc of solution $= \frac{X}{4}$=.......... g

Result:

1.	Normality of oxalic acid solution	.............N
2.	Normality of $KMnO_4$ solution	.............N
3.	Normality of Mohr's salt solution	.............N
4.	Amount of Mohr's salt present in 250 cc of solution	.............g

8. DETERMINATION OF Fe^{2+} ION

Aim: To prepare the standard Mohr salt solution, standardize potassium dichromate solution and to determine Fe^{2+} ion in the given solution.

Apparatus : Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Mohr's salt,potassium dichromate , sulphuric acid, diphenyl amine indicator etc.

Theory: Mohr's salt or Ferrous ammonium sulphate-FAS [$FeSO_4.(NH_4)_2SO_4.6H_2O$, *Mol.wt* = 392.1] is a reducing agent. Its molecular mass is equal to its gram equivalent mass (E) as 392.1. The amount of Mohr's salt to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of Mohr's salt is used in this experiment to standardize potassium dichromate solution.

The titration of Mohr's salt v/s potassium dichromate is an example of redox titration in which Fe^{2+} ions of the Mohr's salt gets oxidised to Fe^{3+} ions and potassium dichromate gets reduced to chromic sulphate. This reaction is carried out in acidic medium by adding 1 test tube sulphuric acid - phosphoric acid mixture and diphenylamine indicator. The added phosphoric acid lowers the oxidation potential of ferric–ferrous ion system by forming a complex $[Fe(HPO_4)]^+$ with ferric ions this gives the sharp colour change of the indicator at the end point.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$[2FeSO_4(NH_4)_2SO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3(NH_4)_2SO_4 + H_2O] \times 3$$

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

Procedure:

A. Preparation of standard (0.05N) Mohr's salt solution:

Accurately weighed (4.901g) Mohr's salt is transferred to a beaker and is made in to paste with 5 cc of conc. sulphuric acid and further it is dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of potassium dichromate solution:

Exactly 25 cc of Mohr's salt solution is pipetted out into a clean conical flask. 1 test tube of sulphuric acid-phosphoric acid mixture and 7-8 drops of diphenylamine indicator are added and immediately titrated against the potassium dichromate solution till the colour changes from pale green to deep violet.

The titration is repeated to get concordant values. Exact normality of potassium dichromate is calculated by using concordant readings.

C. Determination of Fe^{2+} ions present in the given solution:

The Fe^{2+} ions present in the given solution is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution containing* Fe^{2+} ions *may be directly supplied*. Exactly 25 cc of this solution is pipetted out into a clean conical flask. 1 test tube of sulphuric acid-phosphoric acid mixture is added and the solution is titrated against potassium dichromate solution using diphenyl amine as an indicator till the colour changes from pale green to deep violet. The titration is repeated to get concordant values. Using appropriate formula the amount of Fe^{2+} ions present in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05N) Mohr's salt solution

The amount of Mohr's salt required = w = $\frac{N \times E \times V}{1000} = \frac{0.05 \times 392.1 \times 250}{1000} = 4.901$ **g**

Mass of empty watch glass : m_1 = ….…… g

Mass of watch glass + Mohr's salt : m_2 = ….…… g

Mass of Mohr's salt : $(m_2 - m_1)$ = ….…… g

If the value of mass of* Mohr's salt *weighed differs from 4.901 g then, normality of* Mohr's salt *solution is calculated by

$$\therefore \text{Normality of Mohr's salt solution} = \frac{\text{Mass of Mohrs salt} \times 4}{\text{Eq.mass of Mohrs salt}}$$

$$= \frac{(m_2 - m_1) \times 4}{392.1} = \ldots\ldots.\text{N}$$

B. Standardisation of potassium dichromate solution:

Solution taken in the burette : $K_2Cr_2O_7$ solution

Solution taken in the conical flask : 25 cc containing Fe^{2+} ions + 1 test tube of H_2SO_4-H_3PO_4 mixture

Indicator used : Diphenyl amine

Colour change at the end point : Pale green to deep violet

Tabulations

Trial No	Burette readings (cc)		Volume of $K_2Cr_2O_7$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of $K_2Cr_2O_7$ V_1 = Volume of $K_2Cr_2O_7$ (CBR)

N_2 = Normality of Mohr's salt and V_2 = Volume of Mohr's salt

Therefore, Normality of $K_2Cr_2O_7$, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

C. Determination of Fe^{2+} ions present in the given solution:

Solution taken in the burette : $K_2Cr_2O_7$ solution

Solution taken in the conical flask :25cc Mohr'ssalt+1 test tube of H_2SO_4-H_3PO_4 mixture

Indicator used : Diphenyl amine

Colour change at the end point : Pale green to deep violet

Tabulations

Trial No	Burette readings (cc)		Volume of $K_2Cr_2O_7$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations

: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of Fe^{2+} ions, V_1 = Volume of Fe^{2+} ions,

N_2 = Normality of $K_2Cr_2O_7$ and V_2 = Volume of $K_2Cr_2O_7$

Therefore, Normality of Fe^{2+} ions, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

We know that, equivalent mass of Fe^{2+} ions = 55.85

Amount of Fe^{2+} ions present in dm^3 of solution (X)

= Normality of Fe^{2+} ions x Eq.mass of Fe^{2+} ions(*55.85)*

=........ g

Amount of Fe^{2+} ions present in 250 cc of solution = $\frac{X}{4}$

=.......... g

Result:

1.	Normality of Mohr's salt solution	.............N
2.	Normality of potassium dichromate solution	.............N
3.	Normality of Fe^{2+} ions solution	.............N
4.	Amount of Fe^{2+} ions present in 250 cc of solution	.............g

9. DETERMINATION OF FERROUS AND FERRIC IONS IN A GIVEN MIXTURE

Aim: To determine the ferrous and ferric ions in a given mixture of the solution using standard potassium dichromate solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Potassium dichromate, hydrochloric acid, stannous chloride, mercuric chloride, diphenyl amine indicator etc.

Theory: Potassium dichromate (Mol.wt = 294.18) is referred as the primary standard and strong oxidising agent in acidic medium in the titrimetric analysis. its equivalent mass (E) i.e Eqt.wt = Mol.wt/6=49.03. The amount of potassium dichromate solution to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The ferrous iron in the solution is determined by titrating a known volume of the solution against standard $K_2Cr_2O_7$ solution in presence of 2N H_2SO_4 using diphenyl amine indicator.

$$FeCl_3 + SnCl_2 \longrightarrow FeCl_2 + SnCl_4$$

$$2HgCl_2 + SnCl_2 \longrightarrow \underset{\text{white silky ppt.}}{Hg_2Cl_2\downarrow} + SnCl_4$$

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$[2FeCl_2 + 2HCl + [O] \longrightarrow 2\,FeCl_3 + H_2O] \times 3$$

$$K_2Cr_2O_7 + 6FeCl_2 + 6HCl + Fe^{2+} + 7H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + Fe_2(SO_4)_3 + 6FeCl_3 + 7H_2O$$

To determine the ferric iron, it is first reduced to the ferrous state by using stannous chloride in presence of hydrochloric acid in hot condition. The excess of stannous chloride is removed by adding mercuric chloride. The total iron (Fe^{+2} & Fe^{+3}) in the solution is determined by titrating against the standard solution of $K_2Cr_2O_7$ in acidic medium by using sulphuric acid(2N), phosphoric acid and diphenyl amine indicator till the colour changes from pale green to violet. *[The $SnCl_2$ is added in drop wise to convert all ferric ions in to ferrous ions. The excess $SnCl_2$ added is removed by adding $HgCl_2$ in one lot thereby a silky white precipitate of mercurous chloride is obtained. If no silky white precipitate is obtained it indicates the insufficient addition of $SnCl_2$. If black precipitate is obtained it indicates the conversion of $HgCl_2$ to Hg, which interferes in further reaction. Hence, in the above two cases, the solution should be rejected]*.

Procedure:

A. Preparation of standard (0.05 N) potassium dichromate solution:

Accurately weighed (0.613 g) potassium dichromate is transferred to a beaker and dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Determination of Fe^{2+} ions present in the given solution:

The ferrous and ferric ion present in the given solution is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution containing the ferrous and ferric ion may be directly supplied*. Exactly 25 cc of this solution is pipetted out into a clean conical flask. $1^1/_2$ test tube of dilute sulphuric acid (2N) and $^1/_2$ test tube of phosphoric acid is added and the solution is titrated against potassium dichromate solution using diphenyl amine as an indicator till the colour changes from pale green to deep violet. The titration is repeated to get concordant values and calculated the amount of Fe^{2+} ions present in the given solution**.**

C. Determination of total iron (Fe^{2+} and Fe^{3+} ions) present in the given solution:

Exactly 25 cc of the ferrous and ferric ion solution is pipetted out into a clean conical flask. 5 cc of conc. HCl is added and the solution is heated to boiling so that the solution turns to yellow colour. Then $SnCl_2$ is added in drop wise from a burette with shaking till the solution becomes colourless. Two more drops of $SnCl_2$ are added to make sure that the reduction is complete. The solution is cooled under tap water to room temperature. Half test tube of mercuric chloride solution is added in one lot to get a silky white precipitate (If no silky white precipitate obtained or a black precipitate appears, then the solution should be rejected). $1^1/_2$ test tube of sulphuric acid (2N), $^1/_2$ test of phosphoric acid and 5-7 drops of diphenyl amine indicator are added, and the solution is titrated against standard $K_2Cr_2O_7$ solution till the colour changes from pale green to dark violet. The titration is repeated to get concordant values. Using appropriate formula the amount of Fe^{2+} and Fe^{3+} ions in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05 N) potassium dichromate solution

The amount of potassium dichromate required $= \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 49 \times 250}}{\mathbf{1000}} = 0.613$ **g**

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + potassium dichromate : m_2 = ……..g

Mass of potassium dichromate : $(m_2 - m_1)$ = --------- g

If the value of mass of* potassium dichromate *weighed differs from 0.613 g then, normality of* potassium dichromate *solution is calculated by

$$\therefore \text{Normality of potassium dichromate solution} = \frac{\text{Mass of potassium dichromate} \times 4}{\text{Eq.mass of potassium dichromate}}$$

$$= \frac{(m_2 - m_1) \times 4}{49.03} = \ldots\ldots \text{N}$$

B. Determination of Fe^{2+} ions present in the given solution:

Solution taken in the burette : $K_2Cr_2O_7$ solution

Solution taken in the conical flask : 25 cc of ferrous and ferric ion + $1^1/_2$ test tube of 2N sulphuric acid+ $^1/_2$ test tube of phosphoric acid

Indicator used : Diphenyl amine

Colour change at the end point : Pale green to deep violet

Tabulations

Trial No	Burette readings (cc)		Volume of $K_2Cr_2O_7$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of Fe^{2+} ions V_1 = Volume of Fe^{2+} and Fe^{3+} ion,

N_2 = Normality of $K_2Cr_2O_7$ and V_2 = Volume of $K_2Cr_2O_7$

Therefore, Normality of Fe^{2+} ions, $\mathbf{N_1} = \frac{N_2 V_2}{V_1}$ = ... N

We know that, equivalent mass of Fe^{2+} *ions= 55.85*

Amount of Fe^{2+} ions present in dm^3 of solution (X)

= Normality of Fe^{2+} ions x Eq.mass(55.85) of Fe^{2+} ions =.......... g

Amount of Fe^{2+} ions present in 250 cc of solution $= \frac{X}{4}$ =.......... g = ‘***a***’

C. Determination of total iron (Fe^{2+} and Fe^{3+} ions) present in the given solution:

Solution taken in the burette : $K_2Cr_2O_7$ solution

Solution taken in the conical flask : 25 cc of ferrous and ferric ion + $SnCl_2$ + $HgCl_2$ + $1^1/_2$ test tube of 2N sulphuric acid+ $^1/_2$ test tube of phosphoric acid

Indicator used : Diphenyl amine

Colour change at the end point : Pale green to deep violet

Tabulations

Trial No	Burette readings (cc)		Volume of $K_2Cr_2O_7$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations :

Equation used $N_1 V_1 = N_2 V_2$

N_1 =Normality of total iorn (Fe^{2+} and Fe^{3+} ions)

V_1 = Volume of total iorn (Fe^{2+} and Fe^{3+} ions)

N_2 = Normality of $K_2Cr_2O_7$ and

V_2 = Volume of $K_2Cr_2O_7$

Normality of total iron (Fe^{2+} and Fe^{3+} ions), $\mathbf{N_1 = \frac{N_2\ V_2}{V_1}}$ = ------N

We know that, equivalent mass of Fe^{2+} *ions = 55.85*

Amount of total iron (Fe^{2+} and Fe^{3+} ions) present in dm^3 of solution (X)

= Normality of total iron (Fe^{2+} and Fe^{3+} ions) x Eq.mass of total iron (Fe^{2+} and Fe^{3+} ions)

=.......... g

Amount of total iron (Fe^{2+} and Fe^{3+} ions) present in 250 cc of solution $= \frac{X}{4}$ =........ g = ***'b'***

Amount of Fe^{3+} ions present in 250 cc of solution = **b - a =...........g**

Result:

1.	Normality of K_2Cr_2O solution	.........N
2.	Amount of Fe^{2+} ions present in 250 cc of solution	........g
3.	Amount of total ions (Fe^{2+} and Fe^{3+}ions) present in 250 cc of solution	.........g
4.	Amount of Fe^{3+} ions present in 250 cc of solution	........g

10. DETERMINATION OF IODINE

Aim: To prepare the standard potassium dichromate solution, standardize of sodium thiosulphate solution and to determine iodine in the given solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Sodium thiosulphate solution, potassium dichromate, sulphuric acid, potassium iodide solution, starch etc.

Theory: Potassium dichromate (Mol.wt = 294.18) is referred as the primary standard and strong oxidising agent in presence of dilute sulphuric acid in the titrimetric analysis. Its molecular mass is six times as that of its equivalent mass (E) i.e Eq.wt = Mol.wt/6=49.03. The amount of potassium dichromate solution to be weighed to prepare any volume (V) and any concentration (N) is calculated by using the equation NEV/1000. The standard solution of potassium dichromate is used in this experiment to standardize sodium thiosulphate solution.

The standardization of sodium thiosulphate is carried out by taking sodium thiosulphate in the burette and potassium dichromate, sulphuric acid and KI solution in the conical flask using starch as an indicator. The iodine liberated during the reaction between $K_2Cr_2O_7$ and KI reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate. Hence, it is an example of redox reaction. This titration is called iodometric titration wherein oxidising agent i.e. $K_2Cr_2O_7$ liberates iodine from KI solution and the liberated iodine is titrated against $Na_2S_2O_3$. It is an indirect titration of iodine. There is another type of titration called iodimetric titration where in directly iodine solution is involved.

$$K_2Cr_2O_7 + 7H_2SO_4 + 6KI \longrightarrow 4K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O + 3I_2$$

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

$$\text{Hence, } K_2Cr_2O_7 = 3I_2 = 6Na_2S_2O_3$$

Procedure:

A. Preparation of standard (0.05 N) potassium dichromate solution:

Accurately weighed (0.613 g) potassium dichromate is transferred to a beaker and dissolved by adding distilled water. The solution is carefully transferred (along with washings) to a 250cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization sodium thiosulphate solution:

Exactly 25 cc of potassium dichromate solution is pipetted out into a clean conical flask. To this 10 cc of dilute sulphuric acid (2N) and 10 cc of KI solution are added. The liberated iodine formed during the reaction is titrated against the sodium thiosulphate solution till the colour changes to pale yellow. 1 cc of starch indicator is added so that the whole solution becomes blue colour. The titration is continued by adding sodium thiosulphate solution in dropwise till the colour changes from blue to colourless*[blue colour is not completely discharged because of the presence of $Cr_2(SO_4)_3$]*. The titration is repeated to get concordant values and exact concentration or normality of sodium thiosulphate is calculated.

C. Determination of iodine ions present in the given solution:

The given iodine present in the volumetric flask is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution containing iodine may be directly supplied*. Exactly 25 cc of this solution is pipetted out into a clean conical flask and titrated against the sodium thiosulphate solution till the colour changes to pale yellow. 1 cc of starch indicator is added so that the whole solution becomes blue colour. The titration is continued by adding sodium thiosulphate solution in dropwise till the colour changes from blue to colourless. The titration is repeated to get concordant values. Using appropriate formula the amount of iodine present in the given solution is determined.

Observations and Calculation

A. Preparation of 250 cc of standard (0.05N) potassium dichromate solution

The amount of potassium dichromate required $= \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 49 \times 250}}{\mathbf{1000}} = 0.613$ **g**

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + potassium dichromate : m_2 = ……..g

Mass of potassium dichromate : $(m_2 - m_1)$ = --------- g

If the value of mass of* potassium dichromate *weighed differs from 0.613 g then, normality of* potassium dichromate *solution is calculated by

∴ Normality of potassium dichromat**e** solution $= \frac{Mass\ of\ potassium\ dichromate \times 4}{Eq.mass\ of\ potassium\ dichromate}$

$$= \frac{(m_2 - m_1) \times 4}{49.03} = \ldots\ldots.\text{N}$$

B. Standardisation of sodium thiosulphate solution:

Solution taken in the burette : $Na_2S_2O_3$ solution

Solution taken in the conical flask : 25 cc $K_2Cr_2O_7$ + 10 cc of 2N H_2SO_4 + 10 cc of 10% KI solution

Indicator used : Starch (To be added after getting pale yellow colour to the reaction mixture)

Colour change at the end point : Blue to colourless

Tabulations

Trial No	Burette readings (cc)		Volume of $Na_2S_2O_3$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of $Na_2S_2O_3$ V_1 = Volume of $Na_2S_2O_3$ (CBR)

N_2 = Normality of $K_2Cr_2O_7$ and V_2 = Volume of $K_2Cr_2O_7$

Therefore, Normality of $Na_2S_2O_3$, $N_1 = \frac{N_2\ V_2}{V_1}$ = ... N

C. Determination of iodine present in the given solution:

Solution taken in the burette : $Na_2S_2O_3$ solution

Solution taken in the conical flask : 25 cc Iodine solution

Indicator used : Starch (To be added after getting pale yellow colour to the reaction mixture)

Colour change at the end point : Blue to colourless

Tabulations

Trial No	Burette readings (cc)		Volume of $Na_2S_2O_3$ added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality iodine present in the given solution V_1 = Volume of iodine taken

N_2 = Normality of $K_2Cr_2O_7$ and V_2 = Volume of $K_2Cr_2O_7$

Therefore, Normality of iodine, $N_1 = \frac{N_2\ V_2}{V_1}$ = ... N

We know that, equivalent mass of iodine = 127

Amount of iodine present in the given dm^3 of solution (X)

= Normality of iodine x Eq.mass of iodine(127)

=.......... g

Amount of iodine present in 100 cc of solution = $\frac{X}{10}$ =.......... g

Result:

1.	Normality of potassium dichromate solution	.............N
2.	Normality of sodium thiosulphate solution	.............N
3.	Normality of iodine present in the given solution	.............N
4.	Amount of iodine present in the 100 cc of solution	.............g

11. DETERMINATION OF ZINC

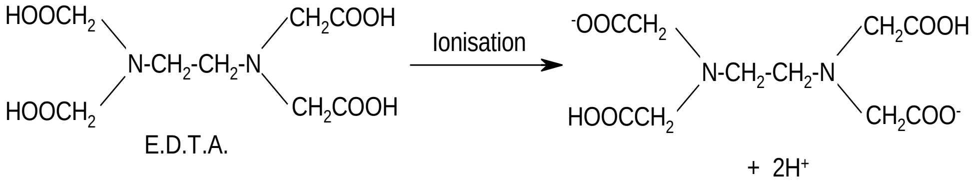
Aim: To prepare the standard zinc sulphate solution, standardize of EDTA solution and to determine Zn^{2+} in the given solution.

Apparatus: Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Ammonium chloride, ammonium hydroxide, EDTA, zinc sulphate, Eriochrome black-T indicator etc,

Theory: $ZnSO_4.7H_2O$ (Mol.wt =M= 287.54),though not a primary standard, but quiet stable, easily water soluble compound used for standardisation of EDTA in the complexometric titration. The amount of $ZnSO_4.7H_2O$ to be weighed to prepare any volume (V) and any concentration (m) is calculated by using the equation (m)MV/1000. The standard solution of $ZnSO_4$ is used in this experiment to standardize di- sodium salt of EDTA.

EDTA (Ethylene diamine tetraacetic acid) is a reagent which forms a complex compound with metal ions such as Zn^{2+}, Ca^{2+}, Mg^{2+} etc.



The EDTA is used in the form of its disodium salt (Na_2-EDTA.). It acts as hexa-dentate ligand during the formation of metal complex. The ionization of the metal complex depends on the p^H of the solution. It is kept nearly 10 by adding a suitable buffer. In this titration, the pH sensitive *Erichrome black-T* (EBT) metal ion indicator is used. The solution is initially wine red due to the formation of less stable metal - indicator complex (*M-EBT*). As titration proceeds, the added EDTA snatches all the metal ions from the wine red coloured M-EBT complex and forms more stable colourless metal - EDTA complex *(M-EDTA*) leaving behind blue colour due to EBT anions. Therefore, at the end point the solution turns wine red to blue.

The titration of $ZnSO_4$ solution v/s EDTA is an example of complexometric titration in which Zn^{2+} forms a complex with EDTA. This reaction is carried out at the pH around 10 using the suitable buffer solution. Eriochrome black-T is used as an indicator which turns wine red to blue at the end point.

$$Na_2H_2EDTA + Zn^{2+} \longrightarrow ZnH_2EDTA(complex) + 2\,Na^+$$

Procedure:

A. Preparation of standard (0.05 M) zinc sulphate solution:

Accurately weighed (1.4377 g) $ZnSO_4.7H_2O$ is transferred to a beaker and dissolved by adding distilled water. The solution is carefully transferred (along with washings) to the 100 cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

(Method of Preparation of pH =10 buffer solution : It is prepared by taking 17.5 g of NH_4Cl *and 150 cc of 17 N* NH_4OH *solution and further diluting with distilled water up to the mark in 250 cc volumetric flask)*

B. Standardization of di-sodium salt of EDTA solution:

Exactly 25 cc of zinc sulphate solution is pipetted out into a clean conical flask. To this 5 cc of buffer solution and 4 drops of Eriochrome black–T indicator are added. It is titrated against EDTA solution till the colour changes from wine red to blue. The titration is repeated to get concordant values.

C. Determination of zinc ions present in the given solution:

The zinc ions present in the given solution is diluted up to the mark by adding distilled water. It is shaken well for uniform concentration *or ready solution containing* zinc ions *may be directly supplied*. Exactly 25 cc of zinc ions solution is pipetted out into a clean conical flask. To this 5 cc of buffer solution and 4 drops of Eriochrome black–T indicator are added. It is titrated against EDTA solution till the colour changes from wine red to blue. The titration is repeated to get concordant values. Using appropriate formula amount of zinc ions present in the given solution is determined.

Observations and Calculation

A. preparation of 100 cc of standard (0.05M) zinc sulphate solution:

The amount of zinc sulphate required = $\frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 287.54 \times 100}}{\mathbf{1000}} = 1.4377$ **g**

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + zinc sulphate : m_2 = ……..g

Mass of zinc sulphate : $(m_2 - m_1)$ = --------- g

If the value of mass of* zinc sulphate *weighed differs from 1.4377 g then, molarity of* zinc sulphate *solution is calculated by

∴ Molarity of zinc sulphate solution $= \frac{\text{Mass of } ZnSO_4.7H_2O \times 4}{\text{Mol.mass of } ZnSO_4.7H_2O}$

$$= \frac{(m_2 - m_1) \times 4}{287.54} = \ldots\ldots.\text{N}$$

B. Standardization of di-sodium salt of EDTA solution:

Solution taken in the burette : Di-sodium salt of EDTA solution

Solution taken in the conical flask : 25 cc of zinc sulphate solution + 5 cc of buffer.

Indicator used : Eriochrome black-T

Colour change at the end point : wine red to blue

Tabulations

Trial No.	Burette readings (cc)		Volume of EDTA added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations :Equation used $M_1 V_1 = M_2 V_2$

M_1 = Molarity of EDTA V_1 = Volume of EDTA (CBR)

M_2 = Molarity of $ZnSO_4.7H_2O$ and V_2 = Volume of $ZnSO_4.7H_2O$ (25)

Molarity of EDTA , $M_1 = \frac{M_2\ V_2}{V_1}$ = ------M

C. Determination of zinc ions present in the given solution:

Solution taken in the burette : Di-sodium salt of EDTA solution

Solution taken in the conical flask : 25 cc of zinc ion solution + 5 cc of buffer.

Indicator used : Eriochrome black-T

Colour change at the end point : wine red to blue

Tabulations

Trial No	Burette readings (cc)		Volume of EDTA added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $M_1 V_1 = M_2 V_2$

M_1= Molarity of Zn^{2+} V_1 = Volume of Zn^{2+}

M_2 =Molarity of EDTA and V_2 = Volume of EDTA (CBR),

Molarity of Zn^{2+}, $M_1 = \frac{M_2\ V_2}{V_1}$ = ------M

We know that, atomic mass of Zn^{2+} *= 65.38*

Amount of Zn^{2+} present in the given dm^3 of solution (X)

= Molarity of Zn^{2+} x Mol.mass of Zn^{2+} =.......... g

Amount of Zn^{2+} present in 250 cc of solution = $\frac{X}{4}$ =.......... g

Result:

1.	Molarity of zinc sulphate solution	.............M
2.	Molarity of EDTA solution	.............M
3.	Molarity of zinc ions present in the given solution	.... M
4.	Amount of zinc ions present in the 250 cc of solution	.............g

12. DETERMINATION OF HARDNESS OF WATER

Aim: To determine the temporary, permanent and total hardness of water using standard EDTA method.

Apparatus : Burette, pipette, conical flask, volumetric flask, funnel, beaker etc.

Chemicals : Di-sodium salt of EDTA, Eriochrome black-T indicator, Buffer solution etc.

Theory : The hardness of water is due to the presence of dissolved salts mainly Ca^{+2} and Mg^{+2}. Hard water doesn't give lather easily with soap, as the hardness causing ions react with soap to form insoluble precipitate. There are three types of hardness of water viz. Temporary hardness, Permanent hardness and Total hardness. *Temporary hardness* is due to bicarbonates of calcium and magnesium, which can be removed by physical treatment that is by simple boiling. On boiling, the bicarbonates are converted into insoluble carbonates of calcium and magnesium. *Permanent hardness* is due to the presence of chloride and sulphates of calcium and magnesium, which are removed only by chemical treatment. *Total hardness* is the sum of temporary and permanent hardness. The hardness is expressed in terms of ppm (parts per million) or mg / litre of calcium carbonate.

Hardness of water is determined by titrating a known volume of water sample with disodium salt of EDTA, using Eriochrome Black-T as indicator in the presence of buffer of pH = 10. When a few drops of the indicator is added, a wine red coloured complex is formed. This reveals that the indicator forms a wine red coloured complex with calcium and magnesium ions. When the solution is titrated against EDTA (Ethylene Diamine Tetra Acetic acid) which is a strong complexing agent, reacts quantitatively with the metal (calcium, magnesium present in the water sample) ions and forms M-EDTA (stable complex). Near the end point when all the metal ions are exhausted from M-EBT complex which are snatched away by EDTA, the indicator molecules are set free in the solution to give blue colour. Therefore, the end point of the titration is observed through colour change from wine red to blue.

Procedure:

A. Preparation of standard (0.05 M) zinc sulphate solution:

Accurately weighed (1.4377 g) $ZnSO_4.7H_2O$ is transferred to a beaker and dissolved by adding distilled water. The solution is carefully transferred (along with washings) to the 100 cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get homogeneous solution.

B. Standardization of di-sodium salt of EDTA solution:

Exactly 25 cc of **)** zinc sulphate solution is pipetted out into a clean conical flask. To this 5 cc of buffer (pH-10) solution and 4 drops of Eriochrome black–T indicator are added. It is titrated against EDTA solution till the colour changes from wine red to blue. The titration is repeated to get concordant values. Using appropriate formula exact concentration of EDTA is determined.

C. Determination of total hardness of water:

Exactly 50 cc of hard water solution is pipetted out into a clean conical flask. 5 cc of buffer (pH-10) solution and 2 drops of Eriochrome black-T indicator are added. The solution is titrated against EDTA solution till the colour changes from the wine red to blue. The titration is repeated to get concordant values. Using suitable formula total hardness of given sample of water is determined.

D. Determination of permanent hardness of water:

Exactly 250 cc of hard water is transferred a 500 cc beaker using a standard volumetric flask. It is boiled for 50 minutes and cooled. The magnesium and calcium carbonates precipitated are filtered off and the filtered is collected in a 250 cc standard flask. The volume is then made up to the mark with distilled water and shaken well for uniform concentration. Exactly 50 cc of this water is pipetted out into a clean conical flask. 1cc of buffer solution and 2-3 drops of Eriochrome black-T indicator are added. The solution is titrated against EDTA solution till the colour changes from the wine red to blue. The titration is repeated to get concordant values and permanent hardness of water sample is determined.

E. Determination of temporary hardness of water:

The temporary hardness is calculated by deducting the permanent hardness from the total hardness.

Observations and Calculation

A. preparation of 100 cc of standard (0.05M) zinc sulphate solution:

The amount of $ZnSO_4.7H_2O$ required = $\frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 287.54 \times 100}}{\mathbf{1000}} = 1.4377$ **g**

Mass of empty watch glass : m_1 = ……..g

Mass of watch glass + zinc sulphate : m_2 = ……..g

Mass of zinc sulphate : $(m_2 - m_1)$ = --------- g

If the value of mass of* zinc sulphate *weighed differs from 1.4377 g then, molarity of* zinc sulphate *solution is calculated by

∴ Molarity of **zinc sulphate** solution = $\frac{\text{Mass of } ZnSO_4.7H_2O \times 4}{\text{molecular mass of } ZnSO_4.7H_2O}$

$$= \frac{(m_2 - m_1) \times 4}{287.54} = \dots\dots\text{N}$$

B. Standardization of di-sodium salt of EDTA solution:

Solution taken in the burette : Di-sodium salt of EDTA solution

Solution taken in the conical flask : 25 cc of zinc sulphate solution + 5 cc of buffer.

Indicator used : Eriochrome black-T

Colour change at the end point : wine red to blue

Tabulations

Trial No	Burette readings (cc)		Volume of EDTA added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations : Equation used $M_1 V_1 = M_2 V_2$

M_1 = Molarity of EDTA, V_1 = Volume of EDTA (CBR)

M_2 = Molarity of $MgSO_4.7H_2O$ and V_2 = Volume of $ZnSO_4.7H_2O$

Molarity of EDTA, $\mathbf{M_1 = \frac{M_2 \times V_2}{V_1}}$ = ------M

C. Determination of total hardness of water:

Solution taken in the burette : Di-sodium salt of EDTA solution

Solution taken in the conical flask : 50 cc of hard water sample + 5 cc of buffer.

Indicator used : Eriochrome black-T

Colour change at the end point : wine red to blue

Trial No	Burette readings (cc)		Volume of EDTA added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial (A)	Final (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations :

Equation used $M_1 V_1 = M_2 V_2$

M_1= Molarity of hard water V_1 = Volume of of hard water(25)

M_2 = Molarity of EDTA V_2 = Volume of EDTA (CBR)

Molarity of of hard water : $\mathbf{M_1 = \frac{M_2 \times V_2}{V_1}}$ = ------M

We know that Mol.mass of $CaCO_3$ = 100

Total hardness of water in terms of mass of $CaCO_3$ in 1000 g or dm^3 *(1000 cc)* of water

= Molarity of hard water X Molecular mass of $CaCO_3$(100). = ...g

Total hardness of water in terms of mass of $CaCO_3$ in million gram of water

= Molarity of hard water Molecular mass of $CaCO_3$ X 10^6 / 1000

= ....ppm or mg/ dm^3 = ***a***

D. Determination of permanent hardness of water:

Solution taken in the burette : Di-sodium salt of EDTA solution

Solution taken in the conical flask : 50 cc of hard water after removal of temporary hardness + 5 cc of buffer.

Indicator used : Eriochrome black-T

Colour change at the end point : wine red to blue

Tabulations

Trial No	Burette readings (cc)		Volume of EDTA added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations:

Equation used $M_1 V_1 = M_2 V_2$

M_1= Molarity of hard water V_1 = Volume of of hard water

M_2 =Molarity of EDTA and V_2 = Volume of EDTA (CBR)

Molarity of of hard water, $\mathbf{M_1 = \frac{M_2 \times V_2}{V_1}}$ = ------M

We know that Mol.mass of $CaCO_3$ = 100

Permanent hardness of water in terms of mass of $CaCO_3$ in 1000 g or dm^3 *(1000 cc)* of water

= Molarity of hard water X Molecular mass of $CaCO_3$ = ...g

Permanent hardness of water in terms of mass of $CaCO_3$ in million gram of water

= Molarity of hard water Molecular mass of $CaCO_3$(100) X 10^6 / 1000

= ....ppm or mg/ dm^3 = ***b***

D. Determination of temporary hardness of water:

Temporary hardness of water = Total hardness of water - Permanent hardness of water

a- b = ...ppm or mg/ dm^3

Result:

1.	Molarity of zinc sulphate solution	.............M
2.	Molarity of EDTA solution	.............M
3.	Total hardness of water	.............ppm
4.	Permanent hardness of water	.............ppm
5.	Temporary hardness of water	.............ppm

Reference Books:

1. A Text Book of Quantitative Inorganic Analysis (3rd edition) - A.I. Vogel.
2. Fundamentals of Analytical Chemistry (8th edition)- Skoog, West,Holler & Crouch.
3. Analytical Chemistry(6th Edition)- Gary Christian
4. A Textbook of Systematic Practical Chemistry-Dr.R.S.Hamsagar
5. A hand book of Analytical Chemistry – Subhash & Satish & Lal.

B.Sc II Sem
Organic Chemistry Experiments

Total No of hours/week : 4Hrs Total No of hours/week : 4Hrs
Total No. of Hours : 54 Hrs Total No. of Hours : 54 Hrs

Exp No.	**CONTENTS**	Page No.
1.	Explanation regarding crystallization, fractional crystallization, sublimation, fractional distillation, distillation under reduced pressure and steam distillation.(Students should write in the journal regarding the above)	48
2.	Recrystallisation and determination of melting point of crystallized solid. Mixed melting point determination and its importance may be mentioned.	55
3.	Preparation of acetanilide from aniline	58
4.	Preparation of phthalimide from phthalic anhydride	60
5.	Preparation of *p*-nitroacetanilide from acetanilide	62
6.	Preparation of aspirin from salicylic acid	64
7.	Preparation of $1,1^{I}$-bis-2-naphthol from 2-naphthol	66
8.	Estimation of Phenol by bromination method	68
9.	Estimation of Aniline by bromination method	71
10.	Estimation of Acetamide	74
11.	Estimation of Carboxylic acids	77
12.	Estimation of Aspirin	80

Note:

In a batch of ten students, for six students preparation experiments and for four students estimations may be given in the practical examination. Two different preparations and two different estimations may be distributed to the students. Selection of experiments may be done by the students based on the picking up of chits. Viva questions may be asked on any of the experiments prescribed in the practical syllabus. During practical examination chart is not allowed, wherever necessary simple procedure may be given.

1. PURIFICATION OF ORGANIC COMPOUNDS

Organic compounds never occur singly at a source but are found associated with many other compounds. Quite often, these compounds have similar properties. An organic compound synthesized in the laboratory is contaminated with other substances produced in side reactions of the process. The purification of organic compounds is therefore important and is the first step before the elucidation of the molecular structure. Following are the commonly employed methods of purification.

I.A. METHODS OF PURIFICATION OF SOLID COMPOUNDS

1. CRYSTALLISATION:

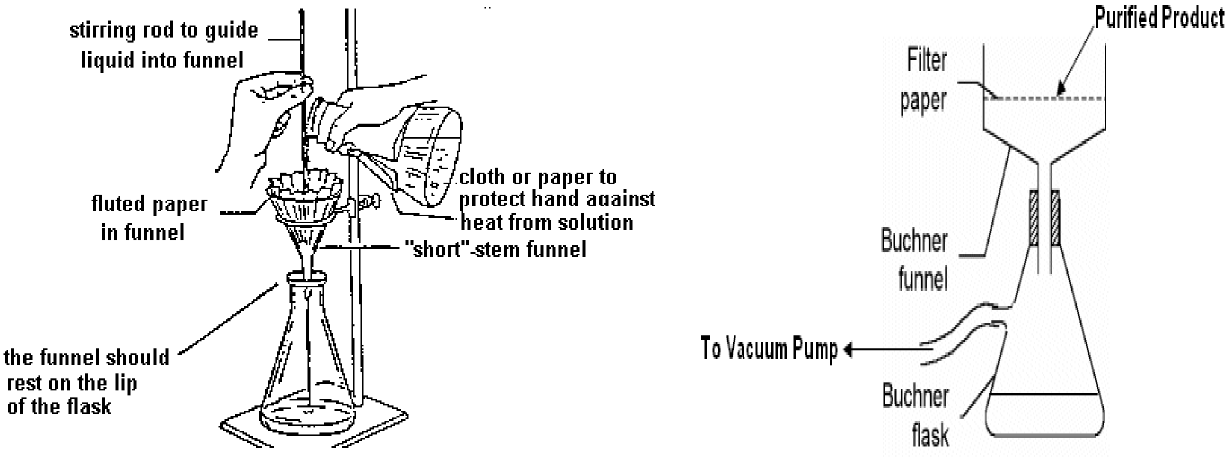
An impure compound is converted into its crystals this process is known as crystallisation. *The differences in the solubility of the compound and impurities in a solvent is the concept used in this method*. If the compound is highly soluble in one solvent and is sparingly soluble in another solvent then the compound is recrystalised using mixture of respective solvents. Also, the solvent chosen is such that the solubility of the compound is much less at room temperature and relatively more as the temperature increases. A saturated solution of the substance is prepared at a high temperature and filtered while hot. A fluted filter paper is used for rapid filtration. On cooling the filtrate, the pure compound crystalises out from the solution and highly soluble impurities remain in the solution.

If the solid is associated with coloured impurities then the colour is removed by adsorption technique using activated charcoal treatment. The size of crystals depends on the manner of cooling. Very gradual cooling gives big crystals but not very pure, whereas quick cooling gives tiny pure crystals.

- If the solute is less soluble than the impurities, it crystallizes first leaving behind the impurities in the solution. The most commonly used solvents are: Water (2) Rectified spirit (3) Absolute ethanol (4) Acetic acid (5) Acetone (6) Methyl alcohol (7) Chloroform (8) Benzene (9) Carbon tetrachloride (10) Ethyl acetate.

The process of crystallization comprises the following steps: (1) Selection of solvent (2) Preparation of solution of solute (compound) in the chosen solvent. (3) Decolourisation of solution by using activated charcoal (4) Filtration of solution to remove suspended solids (5) Crystallisation. (6) Washing the crystals (7) Drying.

Procedure: The impure organic compound is dissolved in a solvent in which it is sparingly soluble at room temperature and relatively soluble at high temperature to get nearly saturated solution. The hot solution is filtered to separate insoluble impurities and is allowed to cool. Pure compound crystallizes out from the solution and highly soluble impurities remain in the solution. If the compound is highly soluble in one solvent and sparingly soluble in another solvent then the compound is recrystallised using mixture of these solvents. If the compound is associated with colored impurities then the colour is removed by adsorption using activated charcoal.



Rapid filtration of a hot solution using fluted filter paper

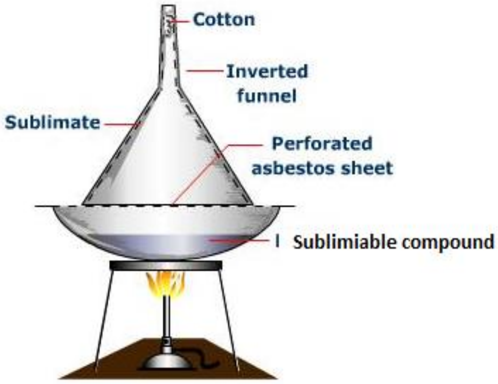
Fig. Filteration Technique

(a) FRACTIONAL CRYSTALLISATION :

This method is useful when two or more components of a mixture need to be purified and all the components are soluble in a particular solvent. The whole process is actually a series of crystallisations (*viz. Separation of sugar and salt).* The difference in the solubilities of components in the solvent is made use of in this process. The mixture of compound X and compound Y is dissolved in the solvent at a higher temperature and then cooled. On cooling the crystals of the less soluble component say X, separates out. The solution is filtered to obtain the crystals of X contaminated with the crystals of Y. These crystals are further subjected to the above process and eventually the crystals of pure X are obtained. The filtrate after the removal of the less soluble component X is concentrated by boiling. On cooling this filtrate, crystals of Y separates out. These are again contaminated with the component X which can be removed by repeated crystallisation. Thus, separation and purification of the components is possible. ***Example***: Mixture of benzoic acid and naphthalene can be separated from hot water in which benzoic acid is soluble while naphthalene is insoluble.

(b) SUBLIMATION:

Sublimation is a process where a solid directly changes to vapour on heating. Particularly some substances whose vapour pressure becomes equal to the atmospheric pressure much before their respective melting points, are capable of undergoing sublimation (*viz. Naphthalene, benzoic acid, camphor, anthracene etc*.,) This process is very useful for the separation and purification of solids which sublime on heating from non-volatile solids **(Fig.1)**.



Procedure: The impure solid is taken on a china dish and covered with a perforated asbestos sheet or paper. An inverted funnel is placed over it. The end of the stem is plugged with cotton or paper and the sides are kept cooled. The dish is heated gently on a sand bath the crystals sublime and condense on the inner sides of the funnel. The non volatile impurities are left behind.

Fig. 1. Sublimation

1.B METHODS OF PURIFICATION FOR LIQUIDS

DISTILLATION:

Distillation is a method applied to separate constituents of liquid mixture which are differing in their boiling points [Benzene (80^0C), Aniline (184^0C), Chloroform (61^0C), Benzaldehyde (184 ^{0}C)]. *The normal boiling point of a liquid is that temperature at which its vapour pressure is equal to the atmospheric pressure*. All the organic liquids after heating are converted into vapours based on their fixed boiling point. In this process the liquid is allowed to boil, the vapours are condensed and collected.

(a) SIMPLE DISTILLATION: The simple distillation is a process applied for volatile

liquids which boil without decomposition at atmospheric pressure and contain non volatile impurities. Simple distillation can be applied in cases where the two liquids differ in their boiling points by 40^0C or more. (*Viz. Mixture of* $C_6H_6 + C_6H_5NH_2$, $CHCl_3 + C_6H_5NH_2$, $C_4H_{10}O + C_6H_5CH_3$)

The apparatus consists (*Fig.3*) of a distillation flask (*a round bottom flask with a side tube*) connected to a condenser. A clean receiver is kept at the end of the condenser. A cork carrying a thermometer is fitted in the flask in such a way that the bulb of the thermometer is near the side tube of the distilling flask. Depending upon the boiling point of the liquid, an air condenser (*for liquids with boiling point greater than 130* ^0C) or a Liebig’s condenser (*water cooled condenser*) is used. Further, heating may be direct on a wire gauze or on a sand bath. A few glass beads or porcelain bits are added to the liquid to prevent bumping of the liquid during heating.

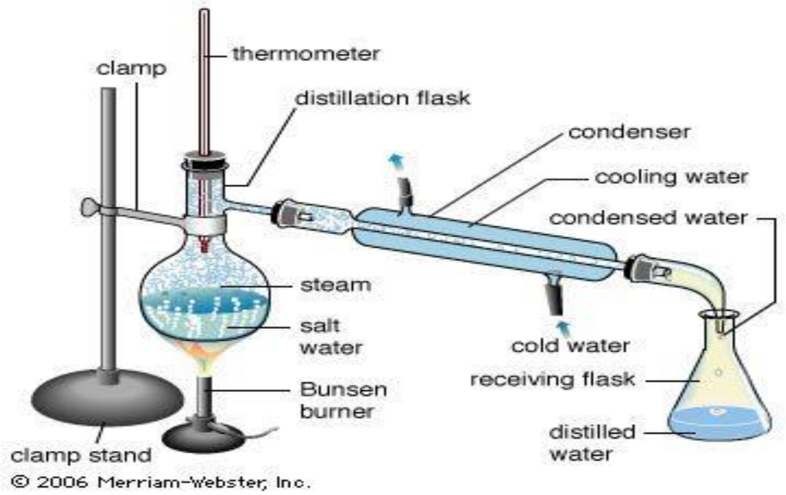


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Fig.3 Simple distillation

The flask and the condenser are clamped then heated therby the liquid boils and distillation starts. The vapours of pure liquid pass through the side tube, condense and are collected in the receiver. The impurities are left behind in the distillation flask.

(b) FRACTIONAL DISTILLATION:

In this process the distillate is collected in fractions under different temperatures which is known as fractional distillation. In cases where the boiling points of the components are fairly close (*Viz.* $[C_6H_6 + C_6H_5CH_3]$, $[CH_3COCH_3 + CH_3OH]$ *etc.)* a fractionating column is used (*Fig.4*). Different types of fractionating columns are known but all of them work on the same principle.

Fig: ***Fractional distillation***

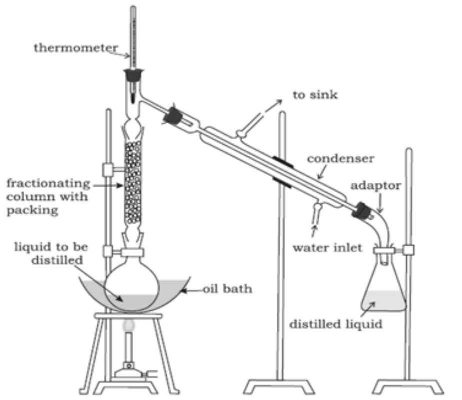


Fig. 3. *Fractional distillation. The vapours of lower boiling fraction reach the top of the column first followed by vapours of higher boiling fractions.*

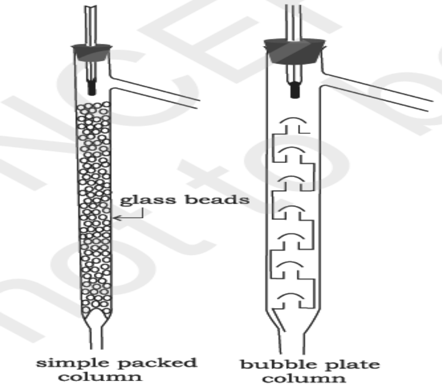


Fig. 4. Fractionating

The fractionating column is fitted to the distilling flask at its mouth. A condenser is connected at the top end of the column; a thermometer is also fixed in the open end of the column. The apparatus is set as in the case of simple distillation. On heating the liquid mixture, the vapours of both the components rise up through the fractionating column but the less volatile component condenses after moving some height.

As it flows down, it meets the upcoming vapour mixture and due to heat exchange, the vapours of the more volatile component continue to move up while the vapours of the less volatile component condense and flow down. Thus the vapours of the more volatile component reach the upper end of the column, condense in the condenser and are collected in the receiver. The less volatile component is left behind in the flask. The process may be repeated to get very pure liquids. Thus two or more liquids from a mixture can be easily separated using a fractionating column.

(c) DISTILLATION UNDER REDUCED PRESSURE:

High boiling liquids as well as liquids which decompose at or below their normal boiling points cannot be purified by distillation at ordinary atmospheric pressure. These are generally distilled under reduced pressure or at vacuum when the liquid boils at a lower temperature. A special apparatus is used for this purpose (*Fig.5*).

During this process the atmospheric pressure is reduced whereby the liquid will boil earlier than its normal boiling point (*Viz. Distillation for cane juice and glycerol in sugar and soap industry respectively)* then the safe separation of compound is achieved. A double necked flask called Claisen's flask is employed. A cork carrying a thermometer is fitted into the smaller of the two necks. Through the main neck, a long capillary tube dipped into the liquid is introduced.

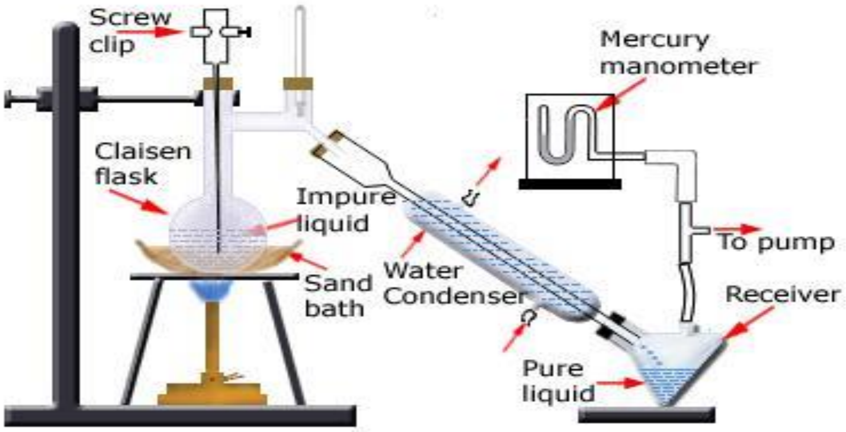


Fig.5. Distillation under reduced pressure

The upper end of the capillary tube is closed by means of a clip. This prevents bumping of the liquid, more pronounced in distillation under reduced pressure. A condenser is joined to the side tube which in turn is connected to a filtration flask. The filtration flask serves as a receiver and its side tube is connected to a vacuum pump through a manometer. The distillation carried out after reducing the pressure. The liquid boils at temperatures much below its normal boiling point and any decomposition of the compound is prevented. *Ex*ample manufacture of cane sugar and glycerol.

(d) STEAM DISTILLATION :

In steam distillation, the liquid boils when the sum of vapour pressure due to the organic liquid (p_1) and that due to water (p_2) becomes equal to the atmospheric pressure (p) that is given as $p = p_1 + p_2$. , since the p_1 is lower than p, the organic liquid vaporises at lower temperature than its boiling point.

The mixture of immiscible liquids boils when the sum of the vapour pressure of components becomes equal to the external pressure. In this process, the organic liquid is distilled along with steam so that the mixture boils at a temperature below 100^0C (*since boiling point of pure water is 100* ^{0}*C*). Therefore, this type of distillation is essentially a co-distillation with water which is carried out when a solid or liquid immiscible with water and is steam volatile containing non volatile impurities. In steam distillation, the vapours coming out at every stage is a mixture of steam and organic liquid so that the temperature at which the mixture boils is lower than the boiling point of either of the components. (*Viz., Purification of aniline, nitrobenzene, Chlorobenzene.o-and p- Nitrophenol can be separated by steam distillation. During steam distillation, o-nitrophenol is carried over by steam while p- nitrophenol remains in the flask).*

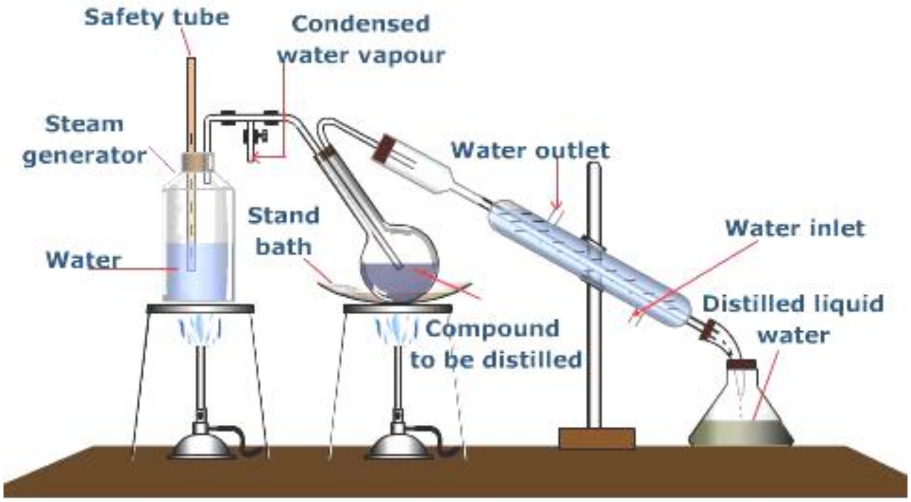


Fig.6. Steam distillation

A steam generator is connected to a round bottom flask containing the organic liquid (*Fig.6*). The flask is clamped in a slanting position and connected to a condenser then to a receiver. (*The slanting position of the flask prevents the liquid from splashing into the condenser*). The steam enters the flask and the liquid begins to boil. Vapours of organic liquid and steam pass over, get condensed and collect in the receiver. To avoid too much condensation of steam in the flask, the flask is gently heated till the distillation is over. Further,the mixture of organic liquid and water collected in the receiver is then separated using a separating funnel and finally dried.

2. RECRYSTALLISATION AND DETERMINATION OF MELTING POINT AND MIXED MELTING POINT OF THE CRYSTALLISED SOLID

The crude products obtained after most of the preparations of organic compounds are contaminated may be with un-reacted starting materials and substances from side-reactions. These can often be eliminated by a simple process known as re-crystallisation.

Re-crystallization is a rapid and convenient way of purifying a solid organic compound. These crystals are the purest form of the substance having definite crystalline structure and shape (*provided the appropriate conditions are maintained*). The product to be purified is dissolved in the hot appropriate solvent. As the solvent cools, the solution becomes saturated with respect to the substance which then crystallizes and the impurities remain in the solution. If the compound is highly soluble in one solvent and is sparingly soluble in another solvent then the compound is re-crystallized using mixture of those respective solvents.

a) MELTING POINT: The temperature at which a solid melts is known as the melting point (MP) of that substance. The melting point is a physical property of a solid, this helps to identify a substance. In practice, a solid usually melts over a range of temperatures rather than at one specific temperature. For this reason it is more useful to speak of a melting point range. If the compound melts over a very narrow range, it can usually be assumed that the compound is relatively pure. Conversely, compounds that melt over a wide range are assumed to be relatively impure.

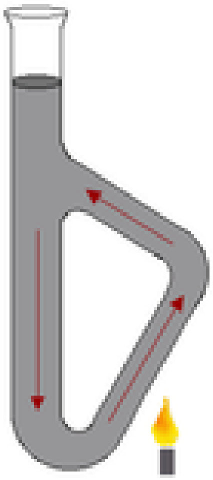


Fig.7a. Thele's tube

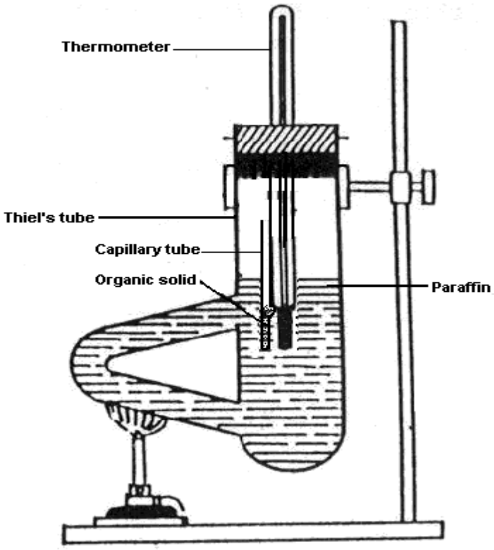


Fig.7b . Melting point determination

An experimental setup is depicted hereunder with **Fig.7b.** A Thiele's tube shown in ***Fig.7a*** is a glass instrument. This is filled with paraffin oil. The Thiele's tube may also be used to determine the boiling point of liquid sample.

The bent shape of the Thiele tube allows for formation of convection currents in the oil when it is heated. These currents maintain a fairly uniform temperature distribution throughout the oil in the tube. The side arm of the tube is designed to generate these convection currents and thus transfer the heat from the flame evenly and rapidly throughout the heating oil.

Take a capillary tube close at its one end by heating on a flame. Now, ground the given organic compound (*whose melting point is to be determined*) into a fine powder on a porcelain tile using spatula. Fill small amount of the compound into the capillary tube. Tie the capillary tube (*containing the substance*) parallel to the thermometer bulb using a thread. The whole assembly is introduced in to a Thele's tube containing liquid paraffin in such a way that the bulb of the thermometer and capillary tube are parallel to the upper side arm of the Thele's tube. Heat the lower side arm of the Thele's tube slowly with a wavy flame using a micro burner. During heating observe the temperature carefully so also the physical state of the solid. When compound in the capillary tube starts to melt, the temperature slowly rises to a particular point the solid suddenly melts completely. This is considered as the melting point of the given organic compound.

b) MIXED MELTING POINT:

The phenomenon of melting point depression can be applied to the identification of unknown pure substances. In the majority of the cases the presence of a foreign substance will lower the melting point of a pure organic compound.

This hint is utilized in the so called **mixed melting point** of pure organic compound. For example, if you measure the melting point of a sample at 160°C, you will find from the MP table that this is the same melting point for several different reference compounds. The substance can be identified by determining its mixed melting point wherein, the sample is mixed one-by-one with small amounts of the references and the mixed melting point is determined in each case. Whenever the melting point of the sample is depressed by mixing a small amount of a reference with it, the two substances cannot be identical. If the melting point of the mixture does not drop it indicates that the reference substance which was added is identical to the sample (*i.e. the sample has been identified*).

As an illustration, take pure benzoic acid *(MP = 122* ^{0}*C)* and 2-naphthol *(MP = 123* ^{0}*C)* ***or*** Acetanilide (MP = 114 ^{0}C) and Antipyrin (MP = 113 ^{0}C). Approximately equal mass (*50 mg : 50 mg*) of the two compounds either combinations mentioned above are placed on a clean porcelain tile. These are mixed thoroughly using a spatula. The capillary tube is filled with requisite amount of the mixture. Place it in the melting point apparatus alongside the capillary tubes filled with each of the two components. The considerable depression of the melting point behavior of the mixture and pure substances will clearly show the melting point lower than that of the pure substances.

Similarly, the determination of mixed melting point of of urea and succinic acid mixture is as follows. *Mixtures of urea: succinic acid in 1:3 and 3:1 ratios (by mass) have been prepared.Record the melting point range of the 1:3 ratio sample and then record the melting point range of the 3:1 ratio sample. Qualitatively, note the melting behaviour of these mixed samples as compared to those of the pure samples*.

3. PREPARATION OF ACETANILIDE FROM ANILINE

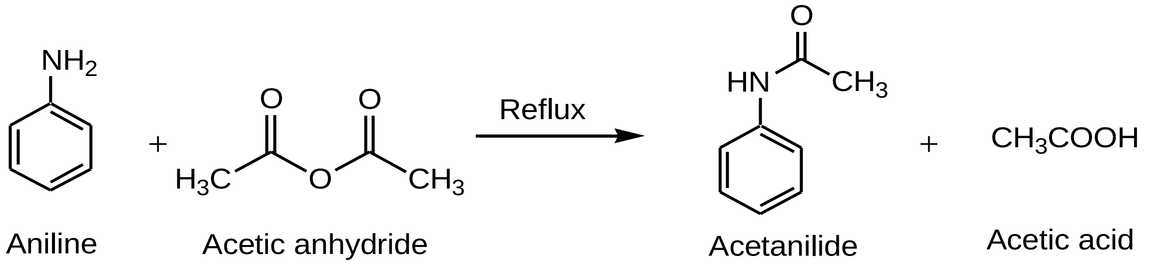
Aim : To prepare acetanilide from aniline.

Chemicals required: Aniline, acetic anhydride or acetyl chloride, glacial acetic acid, zinc dust etc.
Apparatus required: 100 or 250 cc R.B. Flask, water condenser, Buchner funnel etc.

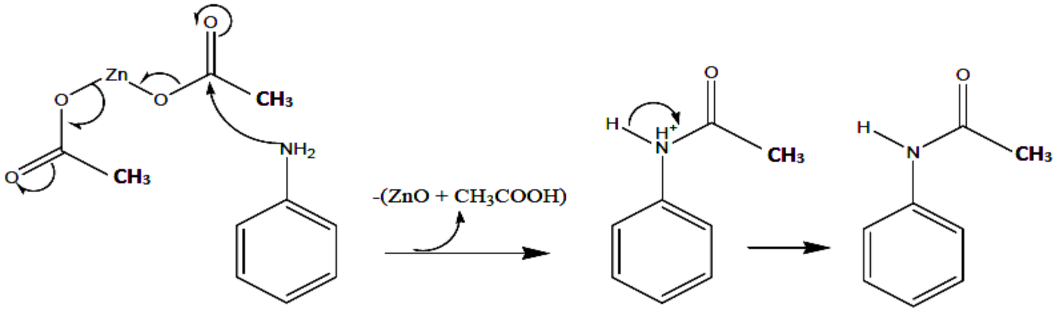
Principle:

The replacement of the hydrogen atom from the compounds containing –OH, $-NH_2$, -NH- groups by acetyl ($-COCH_3$) group is called acetylation. Acetyl chloride or acetic anhydride is used as acetylating agent. Acetylation of aniline can be readily achieved on reacting with acetic anhydride / acetyl chloride. Acetyl chloride, though most reactive, is very difficult to handle, because it is corrosive and has tendency to get hydrolysed by moisture. Acetic anhydride and acetic acid with zinc dust are most commonly employed for the reaction.

In presence of zinc dust acetic acid can be used as acetylating agent to form acetanilide. *The Zn dust is used as a catalyst and reducing agent which prevents oxidation of aniline.*



Mechanism:



Procedure:

Take 5cc of aniline, 10-12cc of glacial acetic acid and 0.5g of zinc dust in a 100cc round bottom flask. Shake the reaction mixture thoroughly and then reflux it gently for about 45 minutes using water condenser. The reaction mixture is then carefully poured into 250 cc beaker containing 100 cc of ice cold water. Acetanilide is separated slowly. After 15 min., the acetanilide is collected by filtration using Buchner funnel and washed the product with cold water. Dry the product by pressing between the filter papers and weigh to record the yield. Use a small amount for re-crystallization with hot water (If necessary, charcoal may be used for decolourisation).

Uses: Acetanilide is used as one of the precursor in the synthesis of penicillin and other pharmaceuticals. It has also applications in the intermediation in rubber accelerator synthesis, dye intermediate synthesis, and also in the synthesis of camphor.

CALCULATION OF PERCENTAGE YIELD OF THE PRODUCT

$$\%\ \text{Yield} = \frac{\text{Experimental Yiled}}{\text{Theoretical Yield}} \times 100$$

Result:

S.No.	Particulars	
1.	Name of the product obtained	
2.	Molecular and structural formula	
3.	Mass of the product (g)	
4.	% Yield of the product obtained.	
5.	Solvent used for re-crystallization	
6.	Colour and nature of the re-crystallized product	
7.	Melting point **(a) Experimental** **(b) Literature**	 **-------- ^{0}C** **114-115 ^{0}C**

4.PREPARATION OF PHTHALIMIDE FROM PHTHALIC ANHYDRIDE

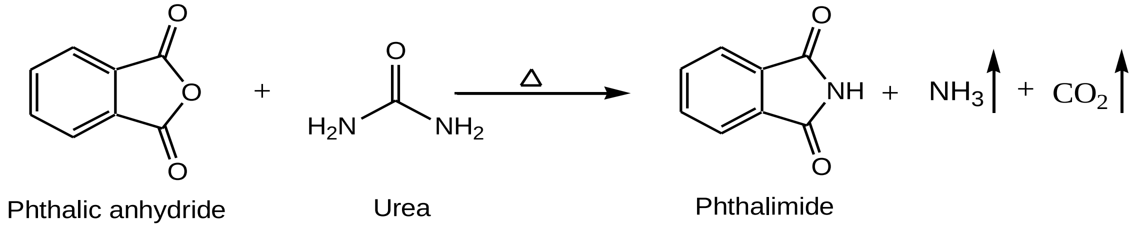
Aim: To Prepare Phthalimide from phthalic anhydride.

Chemicals : Phthalic anhydride, urea and rectified spirit or ether.

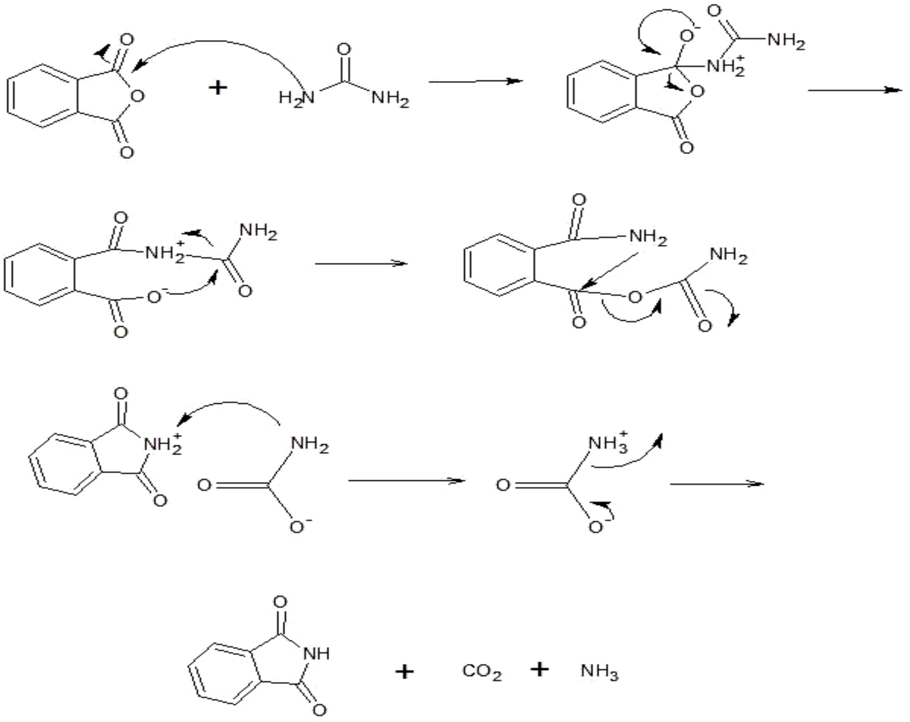
Apparatus required: 250cc conical flask or R. B. Flask, beaker, glass rod, Buchner funnel etc.

Principle:

Phthalic anhydride reacts with urea and undergoes condensation reaction to form phthalimide with the elimination of ammonia and carbon dioxide.



Mechanism:



Procedure:

4 g of phthalic anhydride and 2 g of urea is mixed thoroughly in a pestle mortar. The mixture is introduced into a 250 cc conical flask. The flask is heated on a sand bath. The reaction begins with frothing of the mass and the temperature is raised to about 160°C. Heating was stopped when the frothing subsides; cold water added to the spongy solid mass. The content was filtered in a Buchner funnel with suction. Product is washed with water. The product dried by pressing it with the filter paper and mass of the product noted.

A minimum quantity of the product is used for re-crystallisation using rectified spirit or ether as the solvent. The re-crystallized product is dried and used for the determination of its melting point.

CALCULATION OF PERCENTAGE YIELD OF THE PRODUCT

$$\% \text{ Yield } = \frac{\text{Experimental Yiled}}{\text{Theoretical Yield}} \times 100$$

Uses: Phthalimide is used in plastics, in chemical synthesis, etc.,

Result:

S.No.	Particulars	
1.	Name of the product obtained	
2.	Molecular and structural formula	
3.	Mass of the product (g)	
4.	% Yield of the product obtained.	
5.	Solvent used for re-crystallization	
6.	Colour and nature of the re-crystallized product	
7.	Melting point **(c) Experimental** **(d) Literature**	 **-------- ^{0}C** **233 - 234 ^{0}C**

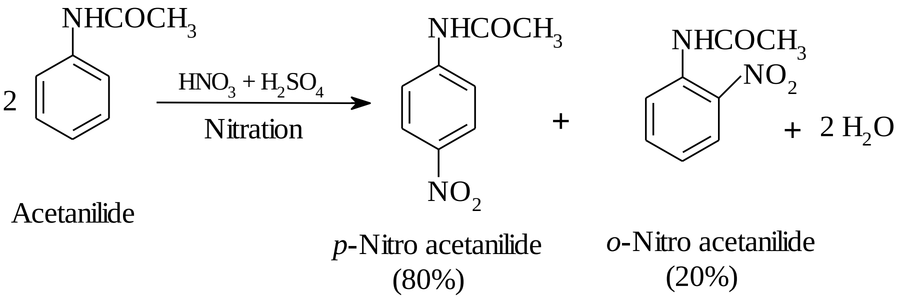
5. PREPARATION OF *p*-NITRO ACETANILIDE FROM ACETANILIDE

Aim : To prepare *p*-nitroacetanilide from acetanilide

Chemicals: Acetanilide, glacial acetic acid, conc. H_2SO_4 and conc. HNO_3, alcohol etc.

Apparatus required: 250 ml Beaker, Glass rod, Ice bath, Wash bottle, Filter paper, Buchner funnel and Thiele's apparatus for Melting point determination.

Principle: *p*-nitroacetanilide is prepared by nitration of acetanilide with nitrating mixture (mixture of conc.HNO_3 and conc.H_2SO_4) in cold condition. Anilide (-$NHCOCH_3$) group is ortho, para directing group there fore during nitration it also gives small amount of ortho nitroacetanilide.

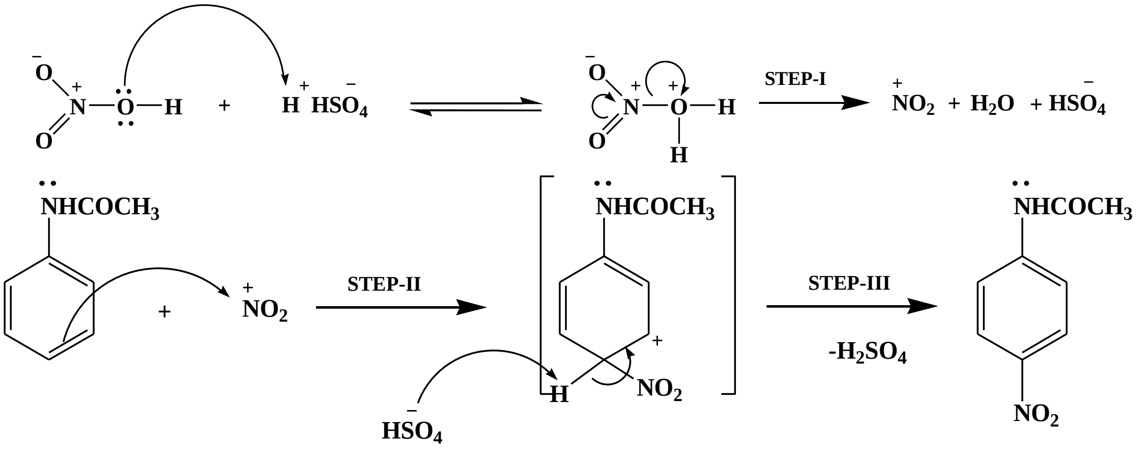


Mechanism:

Step-I: Generation of electrophile

Step-II: Electrphilic attack

Step-II: Regaining the aromaticity by deprotonation



Procedure:

5 g of acetanilide is taken in a 100 cc conical flask and is dissolved by adding 5 cc of glacial acetic acid.10 cc of conc. sulphuric acid is added carefully and the reaction mixture is cooled in an ice bath. 6 cc of conc. nitric acid is added to the reaction mixture drop wise with constant shaking *(so that the temperature does not rise above* $10^{\circ}C$*)*. After the addition of acid, the reaction mixture is shaken for about 30 minutes. The reaction mixture is poured into a beaker containing ice water. The content is filtered off and then product is washed with cold water. The product dried by pressing it with the filter paper and mass of the product noted.

A minimum quantity of the product is used for recrystallisation using alcohol as the solvent. The recrystallized product is dried and used for the determination of its melting point.

Uses: p-Nitroacetanilide is used as an intermediate in the production of some dyes.

CALCULATION OF PERCENTAGE YIELD OF THE PRODUCT

$$\%\ Yield = \frac{Experimental\ Yiled}{Theoretical\ Yield} \times 100$$

Result:

S.No.	Particulars	
1.	Name of the product obtained	
2.	Molecular and structural formula	
3.	Mass of the product (g)	
4.	% Yield of the product obtained.	
5.	Solvent used for re-crystallization	
6.	Colour and nature of the re-crystallized product	
7.	Melting point	
	(a) Experimental	**-------- ^{0}C**
	(b) Literature	**214-215 ^{0}C**

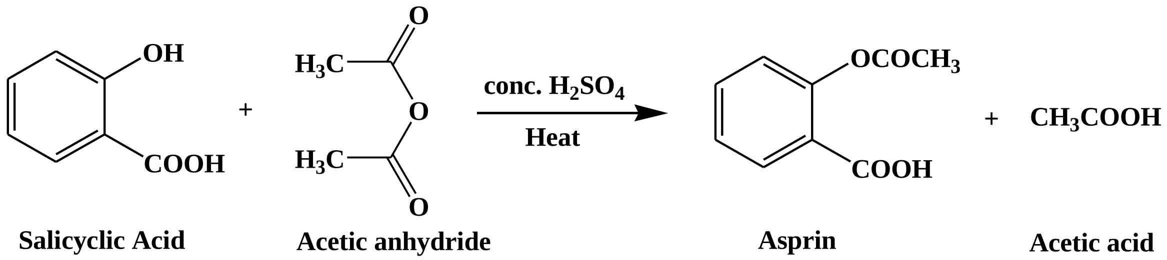
6 . PREPARATION OF ASPIRIN FROM SALICYLIC ACID

Aim : To prepare aspirin from salicylic acid.

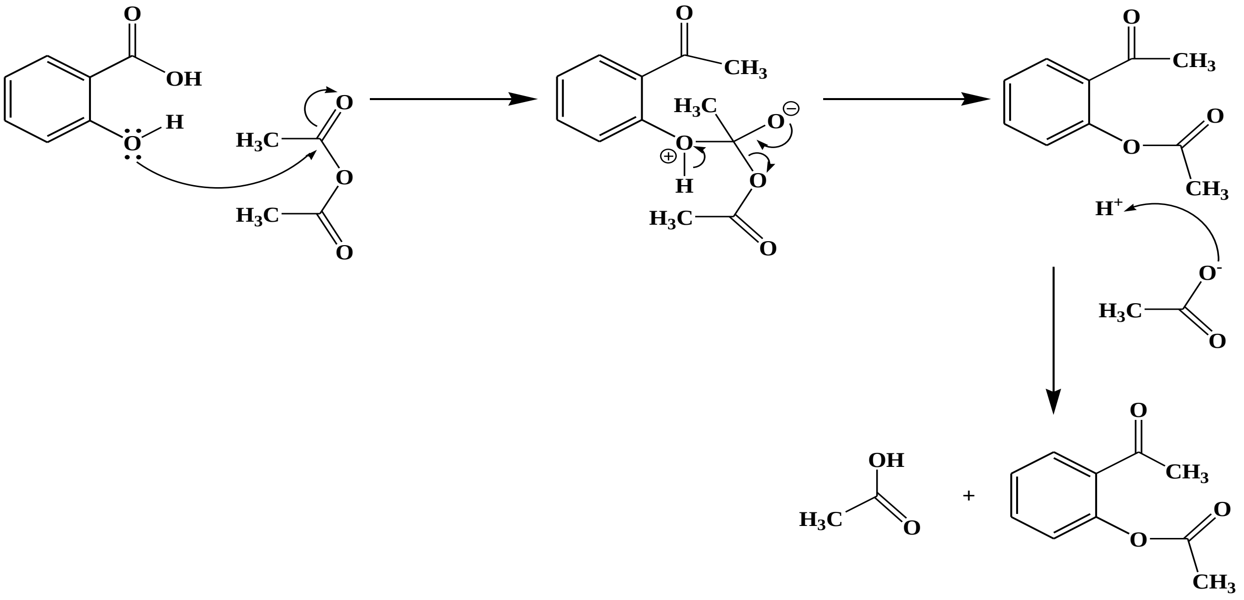
Chemicals: Salicylic acid, acetic anhydride, $FeCl_3$, H_2SO_4, ethanol, etc.

Apparatus required: 250 cc conical flask, Glass rod, Funnel etc.

Aspirin is also known as acetylsalicylic acid. This is an important drug worldwide and conventionally is prepared by an acid-catalyzed process. Preparative method uses acetic anhydride in the reaction instead of acetic acid. An acid catalyst, like sulfuric acid or phosphoric acid, is used to speed up the process.



Mechanism:



Procedure:

4.0 g of salicylic acid is taken in a 250 cc conical flask, 6 cc of acetic anhydride and 5 drops of concentrated sulfuric acid are added to the flask. Then contents are swirled gently and the flask is placed in a boiling water bath for 20 minutes along with stirring using a glass rod (*the entire solid must completely be dissolved*). The flask is removed from the boiling water bath and allowed to cool to the room temperature. (*Formation of crystals during cooling is avoided !*). The reaction mixture poured slowly into a 250 cc beaker containing about 50 cc of ice water, mixed thoroughly and placed the beaker in ice water bath; it is kept undisturbed until crystals grown. The contents filtered in a Buchner funnel with suction. The product obtained is washed with about 20 cc of ice water followed by 10 cc of cold ethanol. The product dried by pressing it with the filter paper and mass of the product noted.

A minimum quantity of the product dissolved by adding ethanol in a 100 cc conical flask, warm it on water bath then the contents filtered. The product obtained is dried. The re-crystallized product used to determine its melting point.

Uses: Aspirin is often used as analgesic, antipyretic, anti-inflammatory agent. Aspirin is also used at low doses to prevent heart attacks, strokes, and blood clot formation in people at high risk of developing blood clots. It has also been established that low doses of aspirin may be given immediately after a heart attack to reduce the risk of another heart attack or of the death of cardiac tissue. Aspirin is one of the most widely used medicine in the world, with an estimated 40,000 tonnes of it being consumed each year.

Result:

S.No.	Particulars	
1.	Name of the product obtained	
2.	Molecular and structural formula	
3.	Mass of the product (g)	
4.	% Yield of the product obtained.	
5.	Solvent used for re-crystallization	
6.	Colour and nature of the re-crystallized product	
7.	Melting point a) **Experimental** b) **Literature**	 **-------- ^{0}C** **134 - 135 ^{0}C**

7. PREPARATION OF 1,1'- *bis*-2-NAPHTHOL FROM 2-NAPHTHOL

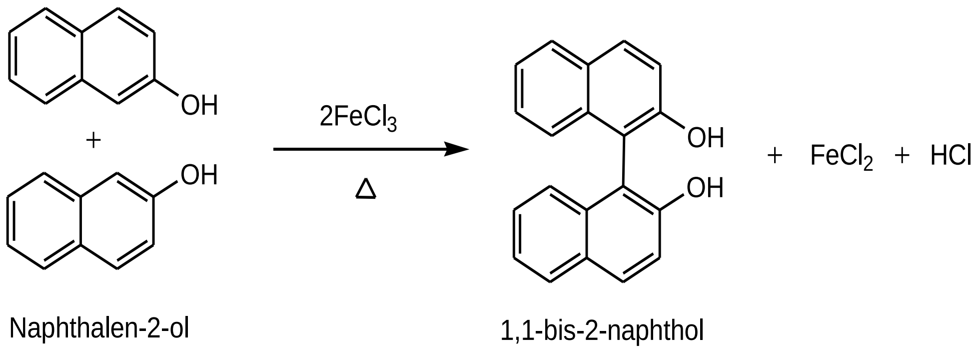
Aim : To Prepare 1,1-bis-2-naphthol from 2-naphthol.

Chemicals: 2-Naphthol, Toluene and $FeCl_3$ solution etc.

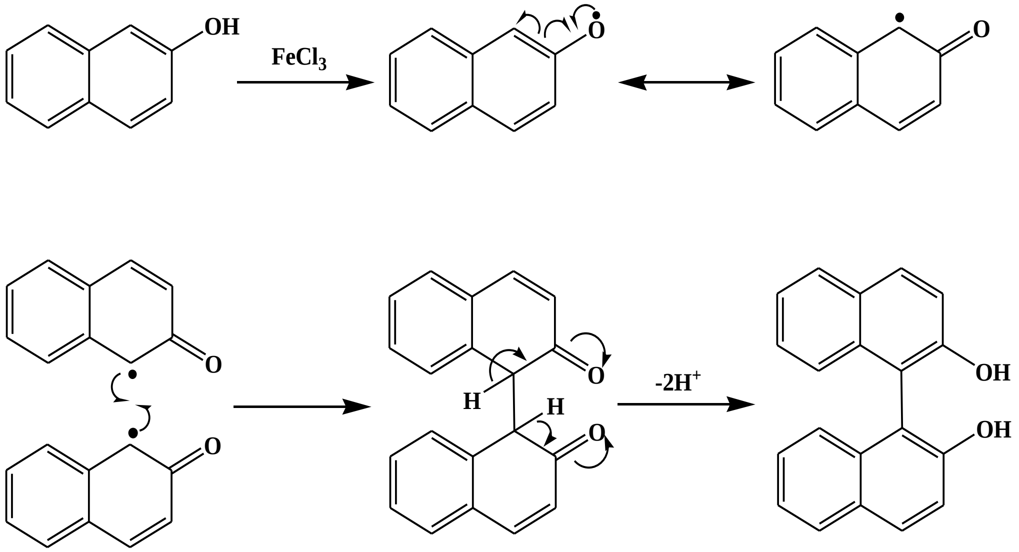
Apparatus required: 250 cc beaker, watch glass rod, funnel, etc.

Principle:

The 2-Naphthol readily undergoes oxidation with iron (III) chloride by a single electron transfer process. This leads to the formation of mesomerically stabilized radical species which then dimerises. It is called oxidative coupling reaction. The 1,1`-bis-2 naphthol forms coloured complex with iron.



Mechanism:



Procedure:

1.5 g of 2'-naphthol is taken in a 250 cc conical flask and 20 cc of water is added to it. The contents are subjected to boiling. To the boiling reaction mixture, 3.0 g of ferric chloride in 10-15 cc of water is added slowly with vigorous stirring till the oily drops of 2-naphthol disappear. The 1,1'-bis-2-naphthol separated out, further the heating is continued for another 5-10 minutes. Filter the hot suspension. The product obtained is washed with hot water. The product dried by pressing it with the filter paper and mass of the product noted.

A minimum quantity of the product is used for recrystallisation using toluene as the solvent. The recrystallized product is dried and used for the determination of its melting point.

Uses: 1,1'-bis-2-naphthol (BINOL) is used as a ligand for transition-metal catalysed asymmetric synthesis.

Result:

S.No.	**Particulars**	
1.	Name of the product obtained	
2.	Molecular and structural formula	
3.	Mass of the product (g)	
4.	% Yield of the product obtained.	
5.	Solvent used for re-crystallization	
6.	Colour and nature of the re-crystallized product	
7.	Melting point a) **Experimental** c) **Literature**	 **-------- ^{0}C** **218 – 219 ^{0}C**

8. ESTIMATION OF PHENOL BY BROMINATION METHOD

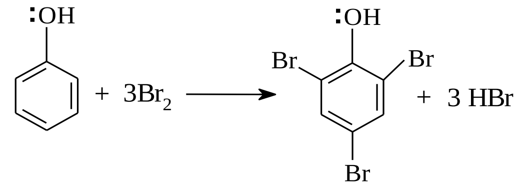
Aim: To estimate the amount of phenol in the given solution.

Chemicals: Phenol solution (provided in volumetric flask), 0.1 N $Na_2S_2O_3$ solution, brominating mixture, 10% KI solution, starch solution, conc. HCl etc.,

Apparatus required: Burette, Pipette , Iodine flasks, conical flask etc.

Principle: On adding a known volume of brominating mixture to the phenol solution, tribromophenol is precipitated. The excess of bromine that liberates an equivalent amount of iodine on addition of potassium iodide is determined by titrating the reaction mixture with standard solution of sodium thiosulphate using starch as an indicator. Thus, knowing the amount of bromine solution consumed then the quantity of phenol in the given solution is calculated.

$$5KBr + KBrO_3 + 6HCl \longrightarrow 6KCl + 3Br_2 + 3H_2O \quad \text{..................................(1)}$$



..................................(2)

Phenol

2,4,6 - Tribromophenol

$$2KI + Br_2 \longrightarrow 2KBr + I_2$$

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

Sodium tetrathionate

Procedure:

A. Blank titration:

Pipette out 10 cc of brominating mixture (*KBr* + *KBrO₃*) is taken in an iodine flask, 20 cc of distilled water, 5 cc concentrated HCl and 5 cc of 10% KI solution is added. The liberated iodine is titrated against 0.1N $Na_2S_2O_3$ using starch as an indicator. The burette reading is noted as **'P'** cc**.**

B. Main titration:

The given solution of phenol diluted using distilled water up to the mark in a 100 cc volumetric flask. Pipette out *10* cc of this diluted solution is taken in an iodine flask. 20 cc of distilled water, 3 cc of conc. HCl is added. Add 10 cc of brominating mixture. The reaction flask is stopperd / corked. The reaction flask allowed standing for 10 minutes in the dark. Then 3 cc of 10% KI solution is added. The liberated iodine is titrated against 0.1N $Na_2S_2O_3$ using starch as an indicator. The burette reading is noted as **'Q'** cc.

Observations:

A. Blank Titration:

Solution taken in the burette : 0.1N $Na_2S_2O_3$solution

Solution taken in the conical flask : 10 cc brominating mixture + 20 cc of water 3 cc HCl + 3 cc of 10 % KI

Indicator used : Starch solution

Colour change : Pale yellow → Blue → Colourless

Trial No.	Burette readings in cc		Volume of $Na_2S_2O_3$ added (B – A) cc	Concordant burette reading = P cc
	Initial (A)	Final (B)		
1.				
2.				
3.				

Main titration.

Solution taken in the burette : 0.1N $Na_2S_2O_3$solution

Solution taken in the conical flask : 10 cc phenol solution + 20 cc of distilled water + 3 cc HCl + 10 cc brominating solution + 3 cc of 10 % KI

Indicator used : Starch solution

Colour change : Pale yellow → Blue → Colourless

Trial No.	Burette readings in cc		Volume of $Na_2S_2O_3$ added (B – A) cc	Concordant burette reading = Q cc
	Initial (A)	Final (B)		
1.				
2.				

Calculation:

From the equation:

1 mole of phenol = 3 moles of Br_2 = 6 equivalents of bromine = 6 moles of iodine = 60,000 cc of 0.1N $Na_2S_2O_3 . 5H_2O$

∴ 1 cc of 0.1N $Na_2S_2O_3$ = $\frac{94}{60,000}$ = 0.001567 g of phenol.

Where, the molecular mass of phenol = 94

a) 0.1N $Na_2S_2O_3$ required for 25 cc of brominating mixture in the ***Blank titration*** = **P** = ……cc

b) 0.1N $Na_2S_2O_3$ required for the excess of brominating mixture added in the ***Main titration*** = **Q** = ……cc

c) The brominating solution in terms of 0.1N $Na_2S_2O_3$ solution= **P-Q** = **S** = …… cc

d) Volume of phenol in the 10 cc of diluted phenol solution = **S** x 0.001567 g

e) Amount of phenol present in the given solution = 10 x **S** x 0.001567 g = **T** g /100 cc

Result: The amount of phenol present in 1000 cc of the given sample solution = **T** x 10 = ………g/dm^3

9. ESTIMATION OF ANILINE BY BROMINATION METHOD

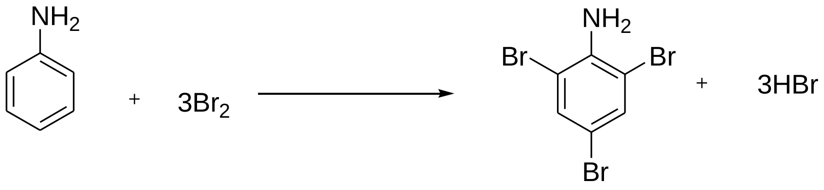
Aim: To estimate volumetrically the amount of aniline in the given solution.

Chemicals: Anilne solution (provided in volumetric flask), 0.1 N $Na_2S_2O_3$ solution, brominating mixture, 10% KI solution , starch solution, conc.HCl etc.,

Apparatus required: Burette (50ml), Pipette (25ml), two iodine flasks (250ml) and one 250 ml conical flask.

Principle: On adding a known volume of brominating solution to the aniline solution, tribromo aniline is precipitated. The excess of bromine that liberates an equivalent amount of iodine on addition of potassium iodide is determined by titrating the reaction mixture with standard solution of sodium thiosulphate using starch as an indicator. Thus, knowing the amount of bromine solution consumed then the quantity of aniline in the given solution is calculated.

$$KBrO_3 + 5KBr + 6HCl \longrightarrow 6KCl + 3H_2O - 3Br_2$$



$$Br_2 + 2KI \longrightarrow 2KBr - I_2$$

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

Procedure:

A. Blank titration:

Pipette out *10* cc of brominating mixture (*KBr + KBrO*$_3$) is taken in an iodine flask, 20 cc of distilled water, 3 cc concentrated HCl and 3 cc of 10% KI solution is added. The liberated iodine is titrated against 0.1N $Na_2S_2O_3$ using starch as an indicator. The burette reading is noted as **'P'** cc**.**

B. Main titration:

The given solution of aniline diluted using distilled water up to the mark in a 100 cc volumetric flask. Pipette out *10* cc of this diluted solution is taken in an iodine flask. 20 cc of distilled water, 3 cc of conc. HCl is added. Add 10 cc of brominating solution. The reaction flask is stoppered / corked. The reaction flask is allowed to stand for 10 minutes in the dark.

Then 3 cc of 10% KI solution is added. The liberated iodine is titrated against 0.1N $Na_2S_2O_3$ using starch as an indicator. The burette reading is noted as **'Q'** cc.

Observation:

A. Blank Titration:

Solution taken in the burette : 0.1N $Na_2S_2O_3$ solution

Solution taken in the conical flask : 10 cc brominating mixture + 20 cc of water 5 cc HCl + 3 cc of 10 % KI

Indicator used : Starch solution

Colour change : Pale yellow → Blue → Colourless

Trial No.	Burette readings in cc		Volume of $Na_2S_2O_3$ added (B – A) cc	Concordant burette reading = P cc
	Initial (A)	Final (B)		
1.				
2.				
3.				

B . Main titration:

Solution taken in the burette : 0.1N $Na_2S_2O_3$ solution

Solution taken in the conical flask : 10 cc aniline solution + 20 cc of distilled water + 3 cc HCl + 10 cc brominating mixture + 3 cc of 10 % KI

Indicator used : Starch solution

Colour change : Pale yellow → Blue → Colourless

Trial No.	Burette readings in cc		Volume of $Na_2S_2O_3$ added (B – A) cc	Concordant burette reading = Q cc
	Initial (A)	Final (B)		
1.				
2.				

Calculation:

From the above equation,

1 mole of phenol = 3 moles of Br_2 = 6 equivalents of bromine= 6 moles of iodine

= 60,000 cc of 0.1N $Na_2S_2O_3 . 5H_2O$

∴ 1 cc of 0.1N $Na_2S_2O_3$ = 93 / 60,000 = 0.00155 g of aniline

Where, the molecular mass of aniline is 93.

a) Volume of 0.1N $Na_2S_2O_3$ required for 25 cc of brominating mixture

Blank titration = **P** = ……cc

b) Volume of 0.1N $Na_2S_2O_3$ required for the un-reacted brominating mixture

Main titration = **Q** = ……cc

c) The brominating mixture used interms of 0.1N $Na_2S_2O_3$ solution = **P – Q = R** = …… cc

d) Volume of aniline in the 10 cc of diluted aniline solution = **R** x 0.00155 g

e) Amount of aniline present in the given solution = 10 x **R** x 0.00155 g

= **S =………**g / 250 cc

Result: The amount of aniline present in 1000 cc of the given sample solution = **S** x 10

= ………g/dm^3

10. ESTIMATION OF ACETAMIDE BY HYDROLYSIS METHOD

Aim: To estimate the amount of acetamide present in the given solution by hydrolysis method.

Chemicals Required: 1N KOH solution, amide solution (Given), 0.1N H_2SO_4 and phenolphthalein indicator.

Apparatus Required: Measuring flask, R.B. flask, air condenser, Burette, pipette etc.

Principle:

The acetamide is hydrolysed completely by boiling it with an excess of KOH (*Approx. 1N*) solution. Ammonia is expelled by boiling and excess of alkali is added which is determined by titrating the hydrolysed mixture against standard solution of sulphuric acid. By knowing the amount of alkali consumed for hydrolysis, the quantity of acetamide in the given solution can be calculated.

$$\underset{\text{Acetamide}}{CH_3CONH_2} + KOH \longrightarrow \underset{\text{Potassium actate}}{CH_3COOK} + NH_3\uparrow \quad ....(1)$$

$$\underset{\text{Potassium acetate}}{CH_3COOK} + H_2SO_4 \longrightarrow \underset{\text{Acetic acid}}{CH_3COOH} + K_2SO_4 \quad ....(2)$$

$$KOH + H_2SO_4 \longrightarrow K_2SO_4 + H_2O \quad ....(3)$$

Procedure:

A. Blank Titration:

25 cc of alcoholic KOH solution is taken (*using pipette*) in a 250 cc measuring flask, is diluted up- to the mark using distilled water. After shaking the diluted solution a homogeneous solution is obtained. 25 cc of this diluted solution pipette out into a 250 cc conical flask and 2-3 drops of phenolphthalein indicator is added. This is titrated against standard solution of 0.1N H_2SO_4 till the pink colour disappears. The burette reading is noted as **'P'** cc.

B. Main Titration:

Given acetamide solution is taken in an iodine flask, 25 cc (*using a pipette*) of alcoholic KOH solution and 1-2 pieces of boiling chips is added. It is refluxed on sand bath for 90 minutes by connecting the air condenser. After the hydrolysis is over, the reaction mixture is cooled then washed the condenser with a little amount of distilled water and collected the washings in to the flask. The condenser was disconnected. The flask is heated for another 10-15 minutes (*Till there is no evolution of ammonia which is confirmed by testing with a red litmus paper*) using a funnel over the mouth of the flask and cooled. The contents transferred into a 250 cc measuring flask. The iodine flask is washed 2-3 times using distilled water and the washings transferred into the measuring flask then diluted up to the mark using distilled water. then shake well to get uniform solution.

Pipette out 25 cc of this diluted solution in a 250 cc conical flask and add 2-3 drops of phenolphthalein as an indicator then titrate against standard solution of 0.1N H_2SO_4 till the pink colour disappears. Note down the burette as **'Q'** cc.

Observation:

A. Blank Titration:

Solution taken in the burette : 0.1N H_2SO_4 solution

Solution taken in the conical flask : 25 cc alc.KOH solution

Indicator used : Phenolphthalein

Colour change : Pink → Colourless

Trial No.	Burette readings in cc		Volume of H_2SO_4 added (B – A) cc	Concordant burette reading = P cc
	Initial (A)	Final (B)		
1.				
2.				
3.				

Main titration:

Solution taken in the burette : 0.1N H_2SO_4solution

Solution taken in the conical flask : 25 cc diluted Acetamide solution

Indicator used : Phenolphthalein

Colour change : Pink → Colourless

Trial No.	**Burette readings in cc**		**Volume of H_2SO_4 added (B – A) cc**	**Concordant burette reading = Q cc**
	Initial (A)	**Final (B)**		
1.				
2.				
3.				

CALCULATION:

From the above equation,

1 mole of acetamide (*Mol. mass = 59 g*) ≡ 1 mole of KOH ≡ 1000 cc of 1N KOH.

∴ 1 cc of 0.1 N KOH = 59 / 10,000 = 0.0059 g of acetamide

a) Volume of 0.1N H_2SO_4 required for 25 cc of diluted KOH solution from the ***Blank titration*** = **P** = ……cc

b) Volume of 0.1N H_2SO_4 required for the un-reacted KOH solution from the ***Main titration*** = **Q** = ……cc

c) Volume of the KOH solution used in terms of 0.1N H_2SO_4 solution= **P- Q** = **R** = …cc

d) Volume of acetamide in the 25 cc of diluted acetamide solution = **R** x 0.0059 g

(*Since 1 cc of 0.1 N KOH ≡ 1 cc of 0.1 N H_2SO_4 ≡ 0.0059 g of acetamide*)

e) Amount of acetamide present in the given solution = 10 x **R** x 0.0059 g

= **S =………**g / 250 cc

Result: The amount of acetamide present in 1000 cc of the given sample solution = **S** x 4

= …g / dm^3

11. ESTIMATION OF CARBOXYLIC ACID

AIM: To estimate the amount of carboxylic acid present in the given solution.

Chemicals: Oxalic acid, NaOH, carboxylic acid solution and phenolphthalein indicator. **Apparatus: V**olumetric flask, conical flask, Beaker, glass rod, watch glass etc.

Principle:

Organic compounds which contained the carboxylic group that show appreciable acidity. Whether the group is aliphatic or aromatic, saturated or unsaturated, substituted or un-substituted, the properties of carboxylic group are essentially the same. As their name implies carboxylic acids are acidic. Therefore, these acids react with bases like NaOH and $NaHCO_3$ to give metal carboxylate salts, RCO_2^- . The carboxylic acids with more than 6 carbons are only slightly soluble in water, but alkali metal salts of carboxylic acids are generally quite water soluble because they are ionic in nature.

In fact the determination of amountcarboxylic acids is achieved by titrating with the standard solution of a base. The strength of the NaOH is determined using standard solution of oxalic acid. Using the known strength of NaOH the amount of carboxylic acid is determined.

$$\underset{\textit{Insoluble in } H_2O}{RCOOH + NaOH} \longrightarrow \underset{\textit{Soluble in } H_2O}{RCOONa + H_2O}$$

Procedure:

A. Preparation of standard 0.1 N oxalic acid solution:

Accurately weighed (1.575 g) oxalic acid is transferred to a beaker and is dissolved by adding distilled water. The solution is carefully transferred (*along with washings*) to a 250 cc volumetric flask and further diluted up to the mark using distilled water. It is shaken well to get a homogeneous solution.

B. Blank titration: **Standardization of sodium hydroxide solution:**

The burette is washed with water, rinsed with the given sodium hydroxide solution and filled with the same solution. The pipette and conical flask are rinsed with distilled water.

Exactly 25 cc of oxalic acid solution is pipette out into a 250 cc conical flask. Add 2-3 drops of phenolphthalein as an indicator. The solution is titrated against NaOH solution till the colour changes from colourless to pink. The burette reading is noted. The titration is repeated to get concordant values.

C. Main titration: **Estimation of carboxylic acid**

Make up the given carboxylic acid solution into 250 cc volumetric flask using distilled water and shake well to make the solution homogenous. Pipette out 25 cc of diluted solution in- to a clean 250 cc conical flask then add 2-3 drops of phenolphthalein as an indicator. Titrate the reaction mixture against standard NaOH solution till pink colour is obtained. Note down the burette reading. Repeat the titration for concordant reading.

Observation:

A. Preparation of 250 cc of standard 0.1N oxalic acid solution

The amount of oxalic acid required = NEV/1000 = 0.1x 63 x 250/1000 = 1.575 g

Mass of empty watch glass : m_1 = ... g

Mass of watch glass + oxalic acid : m_2 = ... g

Mass of oxalic acid : $(m_2 - m_1)$ = ... g

$$\therefore \text{Normality of oxalic acid solution} = \frac{\text{Mass of oxalic acid x 4}}{\text{Eq.mass of oxalic acid}}$$

$$= \frac{(\text{m2} - \text{m1})\text{ x 4}}{63} = \dots\dots\dots\dots\dots.\text{N}$$

B. Blank Titration: Standardization of sodium hydroxide

Solution taken in the burette : NaOH solution

Solution taken in the conical flask : 25 cc oxalic acid solution

Indicator used : Phenolphthalein

Colour change at the end point : Colourless → Pale pink

Trial No.	**Burette readings in cc**		**Volume of NaOH added (B – A) cc**	**Concordant burette reading (CBR) cc**
	Initial (A)	**Final (B)**		
1.				
2.				
3.				

Calculations: Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of NaOH V_1 = Volume of NaOH (CBR)

N_2 = Normality of oxalic acid and V_2 = Volume of oxalic acid

Normality of NaOH , $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

C. Main Titration: Determination of the amount of carboxylic acid

Solution taken in the burette : NaOH solution

Solution taken in the conical flask : 25 cc carboxylic acid solution

Indicator used : Phenolphthalein

Colour change at the end point : Colourless → Pale pink

Trial No.	Burette readings in cc		Volume of NaOH added (B – A) cc	Concordant burette reading (CBR) cc
	Initial (A)	Final (B)		
1.				
2.				
3.				

Equation used $N_1 V_1 = N_2 V_2$

N_1 = Normality of carboxylic acid V_1 = Volume of carboxylic acid

N_2 = Normality of NaOH V_2 = Volume of NaOH

Therefore, Normality of carboxylic acid, $N_1 = \frac{N_2 V_2}{V_1}$ = ... N

We know that equivalent mass of carboxylic acid *= 60.0*

Amount of carboxylic acid present in dm^3 of solution (**X**)

= Normality of carboxylic acid x Eq. mass of carboxylic acid = **X** g =.......... g / 1000 cc

∴ Amount of Carboxylic acid present in 250 cc of solution = $\frac{X}{4}$ =........ g / 250 cc

Result: The amount of carboxylic acid present in 1000 cc of the given sample solution

=…g /dm^3

12. ESTIMATION OF ASPIRIN

Aim: To estimate the amount of Aspirin.

Appartus: Burette, Pipette, Conical flak, Measuring flask and conical flask etc.

***Chemicals*:** 0.2M NaOH , 0.05M H_2SO_4 , Phenolphthalein etc

Principle:

Aspirin is an acetylsalicylic acid, has pharmacological significance. This was first synthesized in 1893 by Felix Hofmann, a chemist for the German firm of Bayer. It is a weak acid that also undergoes slow hydrolysis; i.e., each aspirin molecule reacts with two hydroxide ions. A known amount of standard sodium hydroxide solution is used in excess to hydrolyse the known mass of aspirin. The unreacted sodium hydroxide which remains in the reaction mixture after hydrolysis is then titrated with standard acid. The amount of aspirin is calculated (*from the equation*) by knowing the amount of alkali consumed during the hydrolysis.

$$\underset{\text{Acetyl salicylic acid}}{CH_3COOC_6H_4COOH} + 2NaOH \rightarrow \underset{\text{Sodium acetate}}{CH_3COONa} + \underset{\text{Sodium salicylate}}{HOC_6H_4COONa} + H_2O$$

Procedure:

A. Blank Titration:

Pipette out 25 cc of 0.2M NaOH solution into a conical flask and add 2-3 drops of phenolphthalein as an indicator then titrate against standard solution of 0.2 M HCl till the pink colour disappears (*reverse titration*). Note down the burette reading as **'P'** cc.

B. Main Titration:

Take aspirin tablet / aspirin solution, 20 cc of ethanol and 20 cc of distilled water, into a 250 cc Erlenmeyer flask (*Iodine flask*). Add 25 cc (*using a pipette*) of 0.2M NaOH solution to the reaction mixture and 1-2 pieces of boiling chips. Set the reaction mixture for the reflux (*by connecting the air condenser*) on a water bath for about 20 minutes, swirl the flask occasionally. After the hydrolysis is over disconnect the condenser, cool the reaction mixture to room temperature.

The reaction mixture is titrated against standard solution of 0.2M HCl using phenolphthalein as an indicator till the pink colour disappears (*reverse titration*). Note down the burette reading as **'Q'** cc.

Observations:

A. Blank Titration:

Solution taken in the burette : 0.2M solution HCl

Solution taken in the conical flask : 25 cc NaOH solution

Indicator used : Phenolphthalein

Colour change : Pink → Colourless

Trial No.	**Burette readings in cc**		**Volume of HCl added (B – A)cc**	**Concordant burette reading = P cc**
	Initial (A)	**Final (B)**		
1.				
2.				
3.				

B. Main titration.

Solution taken in the burette : 0.2 M HCl solution

Solution taken in the conical flask : Entire reaction mixture (*after hydrolysis*)

Indicator used : Phenolphthalein

Colour change : Pink → Colourless

Trial No.	**Burette readings in cc**		**Volume of HCl added (B – A)cc**	**Concordant burette reading = Q cc**
	Initial (A)	**Final (B)**		
1.				
2.				
3.				

Calculation:

a. Volume of 0.2 M HCl required for 25 cc of NaOH solution from the ***Blank titration*** = **P** = ……cc

Volume of 0.2 M HCl required for the un-reacted NaOH solution from ***Main titration*** = **Q** = ….cc

b. Volume of NaOH solution used in terms of 0.2M HCl solution = (**P- Q**) = **R** cc

We know from the reaction that,

2 moles of HCl ≡ 2 moles of NaOH ≡ 1 mole of $CH_3COOC_6H_4COOH$ (*aspirin*)

≡ 180 g aspirin

Where , the mol. mass of aspirin =180

∴ 1 mole of HCl ≡ 1 mole of NaOH ≡ 1/2 mole of $CH_3COOC_6H_4COOH$ (*Aspirin*)

≡ 180/2 g Aspirin = 90 g Aspirin

∴ 1000 cc of 1M HCl ≡ 90 g Aspirin

∴ 1 cc of 1M HCl ≡ 0.090 g Aspirin

∴ **R** cc of **Y** M HCl ≡ 0.090 x **R** x **Y** = **S** =…..g Aspirin

= **S** X1000 = ***T*** mg Aspirin

*Where **Y** = Exact Molarity of HCl used in the titration*

∴ The amount of Aspirin present per tablet = ***T*** / Number of tablets used in the experiment

= ….mg / Tablet

Result: The amount of aspirin present per tablet =……..mg.

B.Sc II SEMESTER PRACTICAL EXAMINATION

SCHEME OF EVALUATION			
PREPARATION OF AN ORGANIC COMPOUND		**ESTIMATION OF AN ORGANIC COMPOUND**	
Yield	**16**	**Blank titration**	**08**
Nature of crystals	**04**	**Main titration**	**12**
Technique and presentation	**04**	**Technique and presentation**	**05**
Purity and M.P	**04 + 02**	**Calculation**	**05**
Journal	**05**	**Journal**	**05**
Viva-voce	**05**	**Viva-Voce**	**05**
TOTAL	**40**	**TOTAL**	**40**

AWARD OF MARKS BASED ON ACCURACY					
Yield in %	**Max. Marks**	**Blank Titration (cc)**	**Max. Marks**	**Main Titration (cc)**	**Max. Marks**
< 10%	**16**	**< ± 0.2**	**08**	**± 0.2**	**12**
11-15 %	**14**	**±0.4**	**06**	**±0.4**	**10**
16-20 %	**12**	**±0.6**	**04**	**±0.6**	**08**
21-25 %	**10**	**±0.8**	**02**	**±0.7**	**06**
26-30 %	**08**	**> ±0.8**	**ZERO**	**±0.8**	**04**
> 30 %	**ZERO**			**±0.9**	**02**
				> ±0.9	**ZERO**

Note:

In case of determination of Carboxylic acid just to satisfy the guidelines of distribution of marks mentioned in scheme of evaluation; in this experiment the Standardization of sodium hydroxide solution shall be treated as ***Blank titration*** and then the marks should be awarded accordingly.

B.Sc III Semester

EXPERIMENTS IN PHYSICAL CHEMISTRY

Total No of Hours/Week	: 04 Hours	Practical	:40 Marks
Total No of Hours	: 52 Hours	IA	:10 Marks

Expt. No	Experiments	Page No.
1	Explanation regarding errors, types of errors, accuracy, precision, significant figures and standard deviation (students should write in the journal regarding the above).	85
2	Determination of the velocity constant and effect of concentration on velocity constant of second order reaction KI + $K_2S_2O_8$ (a = b).	89
3	Determination of surface tension and parachor of benzene series or alcohol series.	91
4	Determination of viscosity of toluene and carbon tetrachloride by Ostwald's Viscometer method.	93
5	Determination of enthalpy of ionization of acetic acid by calorimetric method.	95
6	Determination of degree of dissociation of KCl by Landsberger's method.	98
7	To study the effect of acid strength on hydrolysis of methyl acetate using HCl and H_2SO_4.	100
8	To study the adsorption of acetic acid on animal charcoal.	102
9	Determination of surface tension and parachor of toluene, xylene and n-hexane and calculate the atomic parachor of Carbon and Hydrogen.	104
10	Determination of viscosity of binary liquid mixtures of Toluene & carbon tetrachloride and to calculate the percentage composition of the unknown mixture.	106
11	Study of the distribution of benzoic acid between water and toluene.	108
12	Determination of heat of solution of KNO_3 by solubility method.	110

Scheme of Marking:

Accuracy	= 18
Proper Technique and Presentation	= 03
Calculation (Calculation + Graph)	= 09 (5+4)
Viva voce	= 05
Journal	= 05
Total	**= 40 Marks**

NB: 1. scientific calculators are not allowed.

2. Use A4 size graph sheets.

Expt.No.I: Explanation regarding errors, types of errors, accuracy, precision, significant figures and standard deviation

1. Errors, Accuracy, Precision, Significant figures and Standard deviation.

ERRORS: It is defined as the numerical difference between a measured value and the absolute or true value of an analytical determination. The absolute or true value of a quantity is however never known. All that we can use only an accepted value. The value for any quantity is 'accepted' when the uncertainty in this value is less than the uncertainty in some other quantity with which the given quantity is to be compared. It is very rare that two methods may yield identical accepted values.

The error in a measured quantity is represented either as absolute error or relative error.

Absolute error (E): The absolute error in a measurement is expressed as:

$E = x_i - x_t$ where, x_i is the measured value and x_t is the true or accepted value for a given measurement.

Relative error (E_r): The relative error in a measurement is expressed as:

$$E \quad \frac{x_i - x_t}{x_t}$$

where, x_i is the measured value and x_t is the true or accepted value for a given measurement.

Relative error is generally expressed as percentage of error.

$$E_r \quad \frac{x_i - x_t}{x_t} \; X$$

Types of errors:

A) Systematic error or Determinant error: These are errors, which can be avoided, or whose magnitude can be determined. ***Systematic errors are three types,* they are:**

i) Operational and Personal error, ii) Instrumental and Reagent error and iii) Method error.

i) Operational and personal error: The errors are mostly physical in nature and occur when sound analytical technique is not followed.

Example: a) Incomplete drying of analytical sample before weighing.

b) Mechanical loss of materials during sample dissolution from effervesces or from bumping.

c) Incorrect technique involving transfer of solution.

Personal errors may arise from the inability of an individual to make certain observations accurately:

a) Some persons are unable to judge colour changes sharply in visual titrations.

b) The personal decisions include the estimation of a value between two scale divisions of a burette or a meter.

ii) Instrumental and Reagent error:-These errors arise from the faulty construction of balances, the use of uncelebrated or improperly calibrated weights, graduated glassware and other instruments. The attack of reagents upon glassware, porcelain, etc., resulting in the introduction of foreign materials and the use of reagents containing impurities.

iii) Method errors: These are the most serious errors because often they can be difficult to detect.

Examples:- a) A pH meter that has been wrongly standardized,

b) Faulty detector response in chromatographic and spectrographic methods,

c) Errors in classical analysis include solubility of precipitates and the decomposition or volatilization on ignition of weighing forms in gravimetry,

d) Errors may arise in titrimetry if there are differences between the observed end point and the stoichiometric equivalence point of a reaction.

B) Random error or Indeterminate error:- Indeterminate or random errors arises from uncertainties which are inevitably associated with every physical or chemical measurements. These are random or accidental errors whose sources, though many, cannot be positively identified.

Example:- In gravimetric analysis of aluminium, aluminium oxide shows high weight due to the presence of water vapour and can be easily controlled, whereas the analyst has no control over the erratic or random type of errors.

ACCURACY: It is the agreement of a particular value to the true value or most probable value of the result.

PRECISION:- It refers to the closeness of various measurements for the same quantity.

* Accuracy expresses the correctness of a measurement.

* Precision expresses the reproducibility of a measurement.

Precision always accompanies accuracy, but a high degree of precision does not imply accuracy.

Example:- If the true value for a result is 2.00 g and a student ‘A’ takes two measurements and reports the results as 1.95 g and 1.93 g. These values are precise as they are close to each other but are not accurate.

Another student repeats the experiment and obtains 1.94g and 2.05g as the results for two measurements. These observations are neither precise nor accurate.

When a third student repeats these measurements and reports 2.01g and 1.99 g as the results. These values are both precise and accurate.

SIGNIFICANT FIGURES:- Every experiment has some amount of uncertainty associated with it. However one would always like the results to be precise and accurate. The significant figures in a numerical expression are defined as **“all those digits whose values are known with certainty with one additional digit whose value is uncertain”.** The uncertainty in the experimental or the calculated values is indicated by mentioning the number of significant figures.

Examples:1) If the mass of a substance, determined by using a balance which is capable of measuring accurately to the nearest milligram is reported as **2.03765** gram, then only the first four figures are meaningful. In actual measurement, the last digit known with certainty is 7. The digit 6 is uncertain and indicates only that the mass is more than **2.037** but less than **2.038.** The last digit 5 is meaningless and superfluous. Thus by definition **2.03765** has only five significant figures, of which four figures are certain and one figure is uncertain.

2) The position of decimal is of no relevance in determining the number of significant. For example, 0.2341, 2.341, 23.41, 234.1 & 2341 all have **four significant figures**. A **zero** is not a significant figure when used for locating a decimal but becomes significant when used at the end of the expression. Thus **0.0304 has three significant figures** where as the expression **0.03040 has four Significant figures**.

3) In the quantities1.2680 g and 1.0062 g the zero is significant, but in the quantity 0.0025 kg the **zero is not significant figures, they serve only to locate the decimal point** and can be omitted by proper choice of units, i.e. 2.5 g. the first two numbers contain five significant figures, but 0.0025 contains only two significant figures.

Rounding Off the numerical expression

When the answer to a calculation contains too many significant figures, it must be rounded off. There are 10 digits that can occur in the last decimal place in a calculation. One way of rounding off involves ***underestimating*** the answer for five of these digits (0, 1, 2, 3, and 4) and *overestimating* the answer for the other five (5, 6, 7, 8, and 9).

This approach to rounding off is summarized as follows:

i) If the digit is smaller than 5, drop this digit and leave the remaining number unchanged. Thus, 1.684 becomes 1.68.

ii) If the digit is 5 or larger, drop this digit and add 1 to the preceding digit. Thus, 1.247 becomes 1.25.

iii) Rounding off the following numbers:

a) 75.8437 for **three** significant figures: 75.8437 ⟶ 75.8

b) 9.857862 for **four** significant figures: 9.857862 ⟶ 9.858

c) 0.236578 for **five** significant figures: 0.236578 ⟶ 0.23658

STANDARD DEVIATION: The term standard deviation is commonly used in statistics as a measure of precision. This quantity is obtained by the summation of the squares of the individual deviations from the mean, dividing the sum by n-1, where n is the number of measurements and then taking the square root.

If we consider a series of n observations arranged in ascending order of magnitude,

$$x_1, x_2, x_3, \ldots, x_{n-1}, x_n$$

the arithmetic mean is given by

$$\bar{x} = \frac{x_1, x_2, x_3, \ldots, x_{n-1}, x_n}{n}$$

The spread of the values is measured most efficiently by the standard deviations, defined by:

$$s = \sqrt{\frac{(x_1-\bar{x})^2 + (x_2-\bar{x})^2 + \ldots + (x_n-\bar{x})^2}{n-}}$$

In this equation the denominator is (n-1) rather than n when the number of values is small.

The equation may also be written as:

$$s = \sqrt{\frac{\sum(x-\bar{x})^2}{n-}}$$

Expt.No.2 SECOND ORDER REACTION

Aim: To determine the velocity constant and study the effect of concentration on velocity constant of second order reaction : KI + $K_2S_2O_8$, when a = b.

Chemicals: 0.05N $K_2S_2O_8$, 0.05N KI, 0.002N $Na_2S_2O_3$, starch etc.,

Apparatus: Burette, pipette, reagent bottles, conical flask, stop watch etc.,

Theory: When KI is added to $K_2S_2O_8$, the iodine is liberated. Progress of the reaction is studied by titrating a known volume of reaction mixture against std. $Na_2S_2O_3$ solution at a regular interval. The reaction is proceeding through a second order; therefore its velocity constant, k is calculated by using the integrated rate equation; $\boldsymbol{k} = \frac{1}{t}\,\frac{x}{a(a-x)}$ When a=b

Where 'a' and 'b' are initial concentration of KI and $K_2S_2O_8$ respectively

't' is time in minute,

'x' is concentration of KI and $K_2S_2O_8$ reacted at time 't'.

Velocity constant 'k' is independent on concentration of reactants at a given temperature. It can be proved by studying the reaction at two different concentrations of reactants.

Reactions: $2KI + K_2S_2O_8 \longrightarrow 2K_2SO_4 + I_2$

$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$ (Sodium tetrathionate)

Procedure: Set-I: a=b: 50 cc of 0.05N KI + 50 cc of 0.05N $K_2S_2O_8$.

1 Pipette out 50 cc of $K_2S_2O_8$ and 50 cc of KI in two separate clean and dry conical flasks. Stopper both the flasks and keep them in water bath at room temperature.
2 When the content attains room temperature, mix KI to $K_2S_2O_8$ solution and immediately start the stopwatch. Cork the flask tightly and keep it in a water bath.
3 At 5th minute, pipette out 10 cc of the reaction mixture into a conical flask containing few pieces of ice cubes and titrate rapidly the liberated iodine against 0.002N $Na_2S_2O_3$ using starch indicator. Record the burette reading when the colour changes from blue to colour less.
4 Repeat the same titration for 10, 15, 20, 25 and 30 minutes.
5 Calculate 'a' in terms of volume, then calculate 1/ (a-x) and record up to **4th decimal**.
6 Calculate the second order velocity constant, '***k***' using a suitable equation.
7 Plot a graph of 1/(a-x) against time, which gives a straight line. Slope of the straight line is '***k***'.
8 To study the effect of concentration on velocity constant, repeat the entire experiment with the following concentrations of KI and $K_2S_2O_8$.

Set –II : 25 cc 0.05N KI + 25 cc dist. water & 25 cc 0.05N $K_2S_2O_8$ + 25 cc dist. water.

* Calculate '***k***' value and compare this with the value of '***k***' for set-I.
Note: At a constant temperature, velocity constant *k* is independent upon concentration of reactants provided other conditions are constant.

Observations:

Time in minute	Titre readings 'x' in cc	a-x	1 /a-x	$k = \frac{1}{t}\frac{x}{a(a-x)}$ dm^3 mol^{-1} min^{-1}
5				
10				
15				
20				
25				
30				

Calculations:

1. **Calculation of 'a' in terms of volume.**

Set-I: When 50 cc of 0.05N KI and 50 cc 0.05N $K_2S_2O_8$ solutions are mixed, the operating normality of $K_2S_2O_8$ or KI, in the mixture becomes 0.05/2 = 0.025.

When 10 cc of the reaction mixture is taken and titrated against 0.002N $Na_2S_2O_3$ then,

From $N_1 V_1 = N_2V_2$ equation

$10 \times 0.025 = V \times 0.002$

$$V = \frac{10 \times 0.025}{0.002} = 125 \text{ cc} \quad \text{, Hence, a = 125 cc}$$

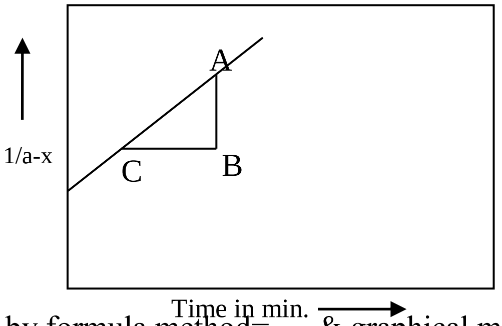
Set-II: 25cc 0.05N KI + 25cc dist. water & 25cc 0.05N $K_2S_2O_8$ + 25cc dist. water.

In this case the operating normality of $K_2S_2O_8$ or KI, in the reaction mixture becomes 0.05/4 = 0.0125.

$$V = \frac{10 \times 0.0125}{0.002} = 62.5\text{cc} \quad \text{, Hence, a= 62.5cc}$$

2. **Calculation of velocity constant (*k*):** $k = \frac{1}{t}\frac{x}{a(a-x)}$ dm^3 mol^{-1} min^{-1}

Nature of the graph:



Slope =

Results: i) Value of ***k*** for set I by formula method=.,…& graphical method =……dm^3 mol^{-1} min^{-1}

ii) Value of ***k*** for set II by formula method=… & graphical method =…… dm^3 mol^{-1} min^{-1}

Conclusion: k for both the sets i.e., for two different concentrations are found to be same. Hence, velocity constant of a reaction is independent on concentration

Expt.No.3 SURFACE TENSION

Aim: Determination of Surface tension of liquids benzene, toluene and o-xylene, by drop number method and calculate the Parachor of these liquid series.

Chemicals: Liquids: A (Benzene), B (Toluene) and C (o-xylene).

Apparatus: Trauts Stalagmometer with pinchcock, beaker, etc.,

Theory: Surface tension is one of the physical properties of the liquid. It is defined as the force in dynes acting on a surface at right angles to any line of unit length. This can be determined by drop number method using Stalagmometer. The surface tension can be calculated according to the following expression:

$$\gamma_L = \frac{n_w \times d_L}{n_L \times d_w} \times \gamma_w$$

Where γ_w = surface tension of water (72 dyne/cm)

γ_L = surface tension of liquid

n_w = no. drops of water, n_L = no.of drops for liquid,

d_w = density of water, d_L = density of liquid.

The magnitude of surface tension is used to calculate the parachor of liquid series, which are additive and constitutive properties. The parachor is calculated using the following equation.

$$P = \frac{M \gamma^{1/4}}{d}$$

Where, P = parachor, d = density

γ = surface tension of liquid & M= Mol.Wt. of liquid.

When the parachor are calculated for the liquids of given homologous series, parachor values of difference in the atoms / group can be calculated.

Procedure:

1. Clean the Stalagmometer by ether / acetone. Dry it thoroughly by blowing hot air from rubber air blower and attach a clean and dry rubber with a screw clip in the middle to the upper end of Stalagmometer.
2. Clamp the apparatus exactly in a vertical position, lower end should be slightly inside the edge of the beaker to avoid disturbance while falling the drops by air.
3. Loosen the screw clip and suck the liquid taken in a small beaker, so that it is well above the upper round mark (care must taken to see the liquid does not enter the rubber tube). Close the tubes with the screw clips.
4. Adjust the flow of liquid using the screw clip, so that the number of drops falling from the flat end is between 20-25 drops per minute. Do not disturb this adjustment.
5. Suck the liquid again little above the upper mark and count the number of drops when the liquid flows from the upper round mark to the lower mark.

6. Repeat the same procedure for at least three times and calculate the average number of drops for the liquid.
7. Rinse the stalagmometer with ether / acetone and blow the air to dry before using for new liquid.
8. Take similar readings for other liquids under study and finally for distilled water.
9. Calculate the surface tension and parachor of each liquid by using the suitable formula and finally the parachor of CH_2..

Observations:-Tabulation

Liquids	No. of drops of liquids	Mean(n)	Density g/cc	Surface tension(dyne/cm)	Molar. mass	Parachor (P)
Benzene (A)	i ii iii		0.8784		78	**P_A =**
Toluene (B)	i ii iii		0.8660		92	**P_B=**
Xylene **(C)**	i ii iii		0.8811		106	**P_C =**
Water	i ii iii		1.00		18	

Calculation :

1) Surface tension of liquid : $\gamma_L = \frac{n_w \text{ x } d_L}{n_L \text{ x } d_w} \text{ x } \gamma_w$

2) Calculation of Parachor: $P = \frac{M\,\gamma^{1/4}}{d}$

3) Parachor of $-CH_2$: $P_B - P_A = P_C - P_B$

Results:

Surface Tension of (dyne/cm)	Benzene	Toluene	Xylene
Parachor of $-CH_2$:			

Note: 1. Theoretical value of Parachor for CH_2: 39
2. Alcohol series like, n-propanol, n- butanol, n- pentanol can also be used to calculate the Parachor of CH_2.

Expt.No.4 VISCOSITY

Aim: To determine the coefficient of viscosity of toluene and carbon tetrachloride by drop number method using Ostwald's Viscometer.

Chemicals: Pure toluene and carbon tetrachloride.

Apparatus: Ostwald's Viscometer, beaker, specific gravity bottle, weight box, etc.,

Theory: Viscosity is a resistance exerted by a liquid against the displacement of its own molecules. It is expressed in terms of coefficient of viscosity (η). It is defined as 'the force acting on unit area to maintain unit difference of velocity between two parallel layers of liquid 1 cm apart'. It can be calculated using Poiseuille's equation when η of other liquid is known.

$$\eta_L = \frac{\eta_w \text{ x } t_L d_L}{t_w\, d_w} \text{ -----}$$

Where 't_L' is time of flow of liquid,
't_w' is time of flow of water
'd_L' is density of liquid & 'd_w' is density of water

Experimentally, this can be determined with the help of Ostwald's viscometer by taking water as a reference liquid having 'η_w' , the viscosity coefficient 0.0089 poise.

Procedure:

1. Clean the Viscometer with acetone or ether and dry it thoroughly by blowing hot air from rubber air blower.
2. Clamp the Viscometer in a perfectly vertical position.
3. Attach a clean piece of rubber tube to the narrow arm of the viscometer.
4. Take 10 cc of the toluene with the help of a pipette into the wider arm of viscometer.
5. Suck the liquid by a rubber tube into the capillary side bulb up to a little above the upper mark. Allow the liquid to flow down through the capillary and at the same time record carefully the time required for the liquid to flow from upper mark to the lower mark on the capillary arm using stopwatch. Take at least three readings with each liquid and find the mean time in second (t_L).
6. Rinse the Viscometer with ether / acetone and blow the air to dry before using for new liquid.
7. Repeat the same procedure for carbon tetrachloride and lastly find the time of flow for water (t_w).
8. Determine the density of toluene and carbon tetrachloride using a specific gravity bottle. Calculate the viscosity coefficient of each liquid using the suitable formula.

Observations

1) Determination of Densities:

1. Weight of empty specific gravity bottle = w_1 = -----g
2. Weight of specific gravity bottle + toluene = w_2 = -----g
3. Weight of specific gravity bottle + carbon tetrachloride = w_3 = -----g
4. Weight of specific gravity bottle + water = w_4 = -----g
5. Weight of toluene = $w_2 - w_1$ = -----g
6. Weight of carbon tetrachloride = $w_3 - w_1$ = -----g
7. Weight of water = $w_4 - w_1$ = -----g

$$\text{Density of toluene} = \frac{\text{Weight of Toluene}}{\text{Weight of water}} = \frac{(w_2 - w_1)}{(w_4 - w_1)} = \text{------g/cc}$$

$$\text{Density of carbon tetrachloride} = \frac{\text{Weight of carbon tetrachloride } (w_3 - w_1)}{\text{Weight of water } (w_4 - w_1)} = \text{------g/cc}$$

2) Determination of coefficient of viscosity (η):

Liquids	Density (d) g/cc	Time of flow in second (t)	Mean(t)	$\eta_L = \frac{\eta_w\ x}{t_w\, d_w}$ ---- poise
Toluene		i ii iii		
Carbon tetrachloride		i ii iii		
Water		i ii iii		$\eta_w = 0.0089$

Calculation: Coefficient of viscosity

$$\eta_L = \frac{\eta_w\ x}{t_w\, d_w} \text{ ---- poise}$$

Results. 1) Coefficient of Viscosity of Toluene = ------poise

2) Coefficient of Viscosity of Carbon tetrachloride = ------poise

Note:

1. Theoretical Coefficient of Viscosity of Toluene = 0.00526 poise
2. Theoretical Coefficient of Viscosity of Carbon tetrachloride = 0.0097poise

Expt.No.5 CALORIMETRY

Aim: To determine the enthalpy of ionization of acetic acid by calorimetric method.

Chemicals: Conc.H_2SO_4, 0.5N CH_3COOH and 0.5N NaOH soln., phenolphthalein indicator.

Apparatus: Thermos flask, 1/10th ^0C thermometer, magnifying lens, measuring cylinder, burette, pipette etc.

Theory: When an acid is added to a base, heat is liberated. The quantity of heat liberated is expressed in terms of enthalpy of neutralization in kJ/eqv. It is defined as a change in enthalpy when one gram equivalent mass of acid is completely neutralized by one gram equivalent mass of base.

It is determined using calorimeter. In the determination of quantity of heat liberated in calorimeter, there will be a loss of heat due to the absorption of heat by the thermometer, wall of the calorimeter, stirrer, water etc. This loss of heat is called water equivalent of calorimeter. It can be measured by measuring the quantity of heat liberated ‘Q’ for a given concentration of H_2SO_4.

However, for a weak acid or weak base, the enthalpy of neutralization is always less than that of neutralization of strong acid by strong base i.e., -57.32 kJ. This is because of the fact that these acids or bases do not undergo complete ionization in solution. Some of the heat is consumed in ionizing theses acids and bases. The heat required for ionizing the weak acid or the weak base is known as the enthalpy of ionization.

It can be calculated from the enthalpy of neutralization of weak acid from strong base. The neutralization of a weak acid is equal to the combination of $H^+_{(aq)}$ and $OH^-_{(aq)}$ to form unionized water and heat accompanying in the ionization of a weak acid. The neutralization of CH_3COOH with NaOH can be represented as:

i) $CH_3COOH_{(aq)} + OH^-_{(aq)} \longrightarrow CH_3COO^-_{(aq)} + H_2O_{(l)}$: ΔH_1 = ---- kJ.

ii) $H^+_{(aq)} + OH^-_{(aq)} \longrightarrow H_2O_{(l)}$: $\Delta H_2 = -57.32$ kJ

and ionization of weak acid

iii) $CH_3COOH_{(aq)} \longrightarrow CH_3COO^-_{(aq)} + H^+_{(aq)}$: ΔH_3 = ---- kJ

Where, ΔH_1 = enthalpy of neutralization of weak acid by strong base,
ΔH_2 = enthalpy of neutralization and ΔH_3 = enthalpy of ionization.
On adding Eqs. (ii) & (iii), we get
$CH_3COOH_{(aq)} + OH^-_{(aq)} \longrightarrow CH_3COO^-_{(aq)} + H_2O_{(l)}$
$\Delta H_1 = -57.32 + \Delta H_3$ or $\Delta H_3 = \Delta H_1 + 57.32$ = ------kJ mol^{-1}

Procedure

I) Determination of water equivalent of calorimeter: *w* (Calorimetric constant).

1. Take 98 cc distilled water in a thermos flask provided with a stirrer and a thermometer and kept at a room temperature.

2. Start stopwatch and temperature is noted for every minute up to 5 minute (T_1 ^{0}C). Add 2cc of conc. H_2SO_4 at 5.5 minute, stirred well and note down the temperature at 6th min.
3. Record the temperature continuously at the interval of one minute up to 15th minute (T_2 ^{0}C).
4. Plot the graph of temperature v/s time and calculate the difference in temperature (ΔT_w).
5. Titrate the content of H_2SO_4solution in the calorimeter by pipetting out 10cc to 100 cc conical flask and titrate against 0.5N NaOH solution using phenolphthalein indicator.
6. Calculate the normality and find out the amount of heat liberated (Q) by the calculated normality of H_2SO_4 solution using standard graph.
7. water equivalent of calorimeter (*W*) can be calculated using the formula: $W = Q/\Delta T_w$

II) Determination of enthalpy of neutralization.

1. Take 50 cc of 0.5N CH_3COOH in a thermos flask and 50cc of 0.5N NaOH in another flask/beaker.
2. Note down the temperature of acid (T_3 ^{0}C) for 05 minute as shown table 2.
3. Now, note down the temperature of base (T_4 ^{0}C) separately up to 5 minutes with slow stirring and mix the base to the acid in the thermos flask at 5.5 minutes.
4. Note the temperature at 6th minute and continue to record the temperature for every minute with stirring up to 15th minute (T_5 ^{0}C).
5. Plot the graph of temperature v/s time and calculate ΔT_N.

Observations

Table-1: Determination of water equivalent of calorimeter or Calorimetric constant (***W***).

98 cc H_2O + 2 cc H_2SO_4 (mix at 5.5 min.)

Time (min)	1	2	3	4	5	5.5	6	7	8	9	10	11	12	13	14	15
Temp	← T_1 (water) →					**Mixing**	← T_2 (Solution) →									
(^{0}C)						-----										

Determination of normality of H_2SO_4 solution:

10 cc of the above H_2SO_4 solution is pipette out in a conical flask and titrated against 0.5 N NaOH using phenolphthalein indicator.

$$(N_1V_1)_{H_2SO_4} = (N_2V_2)_{NaOH}$$

$$N_1 = \frac{0.5 \times V_2}{10} = \text{-------}N$$

Find out the 'Q' from the standard graph for corresponding normality = ---- kJ.

Calculations of water equivalent of calorimeter = $Q/\Delta T_w$ = ------k J/^{0}C.

2) Table-2: Determination of enthalpy of neutralization:-

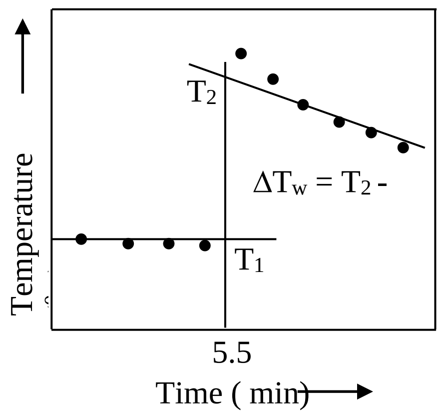
50 cc 0.5 N CH_3COOH + 50 cc 0.5 N NaOH (mix at 5.5 min.)

Time (min)	1	2	3	4	5	5.5	6	7	8	9	10	11	12	13	14	15
Temp(^{0}C)	← T_3 & T_4 →					**mix**	← T_5 (Solution) →									
Acid (T_3)																
Base (T_4)																

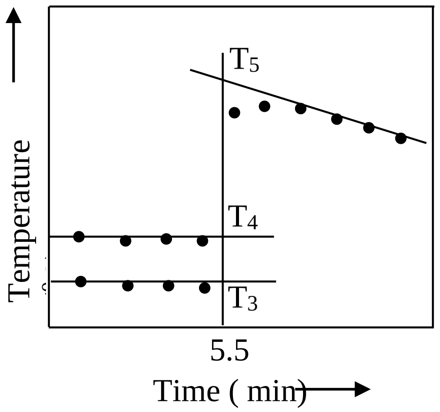
Determination of ΔT_w & ΔT_N from the plot of temperature against time,

Nature of the graph as shown below.

Graph-1.Determination of



Graph-2.Determination of



$$\Delta T_N = T_5 - \frac{T_3 + T_4}{2}$$

Enthalpy of neutralizatio n of CH_3COOH (ΔH_1):

$$= -\left[\frac{1000 \text{ x } W \text{ x } \Delta T_N}{(\text{Normality x Volume})_{\text{acid or base}}}\right]$$

$$= - \frac{1000 \text{ x } W \text{ x } \Delta T_N}{0.5 \text{ x } 50}$$

= ----- kJ

Enthalpy of neutralization of CH_3COOH: (ΔH_1) = -57.32 + Enthalpy of ionization of CH_3COOH: (ΔH_3)

$\Delta H_1 = -57.32+\Delta H_3$

$\Delta H_3 = \Delta H_1+57.32$

= ---- kJ mol^{-1}

Result: Enthalpy of ionization of CH_3COOH = ------kJ mol^{-1}

Note: 1. Theoretical value of Enthalpy of neutralization of CH_3COOH = - 55.43 kJ mol^{-1}

2. Theoretical value of Enthalpy of ionization of CH_3COOH = +1.89-kJ mol^{-1}

Expt.No.6 EBULLIOSCOPY

Aim: To determine the degree of dissociation of KCl by Landsberger's method.

Chemical: Solid KCl.

Apparatus: Landsberger's apparatus, thermometer (1/10th ^{o}C), steam generator, etc.

Theory: When a soluble substance is added to a solvent, its boiling point increases. It is one of the colligative properties. Hence, increase in boiling point is directly proportional to its concentration. This elevation of boiling point (ΔT) is related to its molecular mass (M) as:

$$M_E = \frac{1000 \text{ x } k_b \text{ x w}}{W \Delta T}$$

Where, M_E = Mol. wt. of solute

w = weight of solute

W = weight of solution = volume of solution

ΔT = rise in boiling point.

If the solute is an electrolyte, its molecular mass is determined as above. It will not be the same as its molecular mass, but it may be lower than its molecular mass. It can be equated with the following relation.

$$M_E = \frac{1000 \text{ x } k_b \text{ x w}}{W \Delta T} (1+\alpha)$$

i.e $(1+\alpha) = M_T/ M_E$

$$\alpha = [M_T/M_E] - 1$$

Where, α is degree of dissociation,
M_T is theoretical molecular mass of KCl (74.5)

Procedure:

1. Pipette out 15 cc of water in a graduated tube of Landsberger's apparatus and connect it to the steam generator.
2. Pass the steam into the graduated tube of Landsberger's apparatus till the water drops come out of the outer jacket regularly and temperature remains constant. Now, disconnect the graduated tube from the generator. Note down the temperature immediately. This gives the boiling point of water T_w. Repeat the determination of B.P. of water for another two times with fresh water till to get concordant B.P.
3. Weigh exactly 0.5 g KCl and dissolve in 15 cc of water in a beaker. Then transfer the solution into a clean graduated tube. Pass the steam and find the boiling point of solution as above and record it as T_s.
4. Take out the graduated tube, cool under tap water to attain room temperature and note down the volume of solution W.
5. Repeat the same procedure with 1.0 and 1.5 g of KCl in 15 cc of water separately and note down their respective boiling points and volumes.
6. Calculate the molecular weight of the solute using a suitable formula.

Observations:

Boiling point of water, T_1= -----------, T_2……… & T_3……….^{0}C

Therefore, concordant B.P. of water = T_w =……. ^{0}C

Ebullioscopic constant of water (k_b) = 0.525

Table.

Sl.No.	Wt. of KCl 'w'g	B.P. of soln. T_s ^{0}C	Rise in temp. $\Delta T = T_s - T_w$ (^{0}C)	Volume of soln.= Wt. of soln= W g	$M = \frac{1000 \times k_b \times w}{W \Delta T}$
1	0.5				
2	1.0				
3	1.5				

$$\frac{\text{Theoretical molecular weight } (M_T)}{\text{Experimental molecular weight } (M_E)} = 1+\alpha$$

$$\alpha = \frac{M_T}{M_E} - 1$$

Results: 1. Molecular weight of given electrolyte = i ------ii--------iii--------

2. Degree of dissociation = i ------ii--------iii--------

Conclusion: 1. As concentration increases degree of dissociation decreases.

2. As concentration increases Molecular mass also slightly increases.

Note: KCl is an electrolyte and it gives two particles, K^+ & Cl^-. Therefore, molecular weight of KCl is almost half of its theoretical molecular weight.

Expt.No.7 FIRST ORDER REACTION

Aim: To study the effect of acid strength on hydrolysis of methyl acetate using HCl and H_2SO_4.

Chemicals: Methyl acetate, 0.5N HCl, 0.5N H_2SO_4, 0.1N NaOH, phenolphthalein indicator.

Apparatus: Burette, pipette, conical flask, water bath, funnel etc.,

Theory: $CH_3COOCH_3 + H_2O \xrightarrow{H^+} CH_3COOH + CH_3OH$

The hydrolysis of methyl acetate is a first order reaction and its rate of reaction is proportional to first power of methyl acetate. Water is in large excess, therefore there in no appreciable change in its concentration. Hence, it will not appear in rate equation. Progress of the reaction is monitored by measuring increases in conc. of CH_3COOH by titrating against standard NaOH solution. The first order velocity constant can be calculated using integrated rate equation;

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

Where, ‘a’ is initial concentration and

‘a-x’ is concentration at time ‘t’.

The rate of hydrolysis of methyl acetate is accelerated by mineral acid, H^+. Hence, rate is directly proportional to the concentration of H^+. Existence of free H^+ is depending on dissociation of acids. Dissociation of HCl is greater than that of H_2SO_4. This can be proved kinetically.

Procedure: **Set-I:_ 5 cc methyl acetate + 100 cc 0.5N HCl**

1. In a clean and dry conical flask, take 5 cc methyl acetate and in another flask 100 cc 0.5N HCl solution. Keep them in water bath to attain room temperature for 5 min.
2. Fill 0.1N NaOH solution in a cleaned and rinsed burette. Take 2-3 ice pieces and 1or 2 drops phenolphthalein indicator in another conical flask.
3. Mix methyl acetate to 0.5N HCl, shake well, pipette out 5 cc of this into conical flask containing ice pieces and indicator. Titrate immediately with 0.1N NaOH till pale pink colour persists. Note down the burette reading at zero minute (V_0).
4. Titrate, 5 cc of the same reaction mixture at every 10 minutes till 50 minutes.
5. Calculate the value of ‘*k*’ using the given formula.
6. Find the log (a-x) value up to 4th decimal.
7. Plot a graph of log(a-x) v/s time ‘t’ and calculate ‘*k*’ from the slope.

Set-II,_ 5 cc methyl acetate + 0.5N H_2SO_4

Repeat the entire experiment with 5 cc methyl acetate and 0.5N H_2SO_4 as above and calculate the value of ‘*k*’.

Observations:

1) The titre value after completing the reaction V_∞ = ------cc.

2) Initial concentration, a = $(V_\infty - V_0)$ = ------cc.

Time (min.)	Vol. of NaOH (cc)	x =$(V_t - V_0)$	a-x =$(V_\infty - V_t)$	log (a-x)	$k = \frac{2.303}{t}\log\frac{a}{a-x}$ (min^{-}
0	V_0 =				
10	V_t =				
20	V_t =				
30	V_t =				
40	V_t =				
50	V_t =				
∞	V_∞				
				Mean '*k*'	

Calculations: 1) **Formula method:** $k = \frac{2.303}{t}\log\frac{a}{a-x}$ (min^{-}

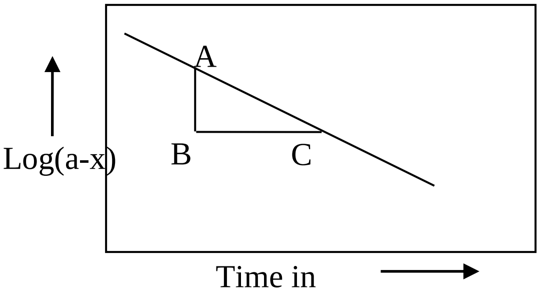
2) **Graphical Method:** Plot a graph of log(a-x) v/s time 't' and calculate '*k*' from the slope.

Nature of the graph:

$\text{Slope} = -\frac{AB}{BC} = -k/2.303$

-k = 2.303 x slope

k = ------------min^{-1}



3) **Relative strength** $= \frac{k_1}{k_2}$

Where, k_1 is mean velocity constant for HCl and k_2 is mean velocity constant for H_2SO_4.

Results:

Acids	Formula method	Graphical method
0.5N HCl	k_1 =------- min^{-1}	k_1 =-------- min^{-1}
0.5N H_2SO_4	k_2 =------ -min^{-1}	k_2 =------- min^{-1}
Relative strength = k_1/k_2		

Remarks: The degree of ionization of HCl is more than H_2SO_4, hence the 'k' value in the Set I is greater than that of H_2SO_4. Hence, k_1/k_2 is always more than unity.

Note: Students may be asked to titrate 5.0 cc of reaction mixture of set-I solution after performing set-II solution for V_∞ = Burette reading for 0.1 N NaOH.

Expt.No.8 ADSORPTION

Aim : To study the adsorption of acetic acid by activated charcoal and verify Freundlich adsorption isotherm.

Apparatus: Burette, pipette, conical flask with stopper, etc.,

Chemicals: Activated charcoal, 0.1N NaOH, 0.5N acetic acid, phenolphthalein indicator, etc.

Theory: Adsorption is purely a surface phenomenon, the amount of gas or solute from solution adsorbed will greatly depend on surface area, temperature, concentration etc. Freundlich proposed an empirical relation for the variation of amount of solute/gas adsorbed with change in concentration of solution at constant temperature. The empirical relation is shown below:

$x/m = kC^{1/n}$ or $\log x/m = \log k + 1/n \log C$

Where, ‘x’ is the amount of adsorbate,

‘m’ is amount of adsorbent ‘C’ is concentration,

‘k and n’ are constants depending upon the nature of adsorbate and adsorbent.

‘1/n’ being less than unity.

Procedure:

1. Prepare five different concentrations of acetic acid in separate 250cc conical flasks by using 0.5N acetic acid solution as shown in the table -1. To each flask add 1 gram of exactly weighed activated charcoal by using glazed papers. Stopper the bottles well and shake them one after another for 5 minutes and place them in water bath for about half an hour.
2. Mean while titrate 5cc of original acetic acid solution against 0.1N NaOH using phenolphthalein indicator. Note down the burette reading. From this, calculate the concentration of acetic acid before adsorption (C_0) for all the five flasks.
3. Filter the contents of the flask through a dry filter paper into a small dry conical flask neglecting the first portion of the filtrate of about 1-2 cc because the filter paper alters the strength of solution by process of absorption.
4. After the filtration, titrate 5cc of clear solution from each of the five flasks against 0.1N NaOH using phenolphthalein indicator. Determine the equilibrium concentrations of acetic acid (C_e) in solution.
5. Find the log X and log C_e from logarithm table only; and plot a graph of log X vs. log C_e, which gives a straight line. Calculate k from intercept and n from slope.

Observations :- Table-1:

Bottle No.	**1**	**2**	**3**	**4**	**5**
0.5N acetic acid (cc)	50	40	30	20	10
Water (cc)	0	10	20	30	40

Table-2:

Volume of 0.1N NaOH required for 5 cc CH_3COOH before addition of charcoal

V_x = ----------cc.

Bottle No.	Titre readings		Conc. of acid		Amount of acid adsorbed by 1 gram of charcoal $X=C_0-C_e$	logX	$\log C_e$
	Before adsorption (V)	After adsorption (V_e)	Before adsorption (C_0)	After adsorption (C_e)			
1							
2							
3							
4							
5							

Calculation: Calculation of titre readings (before adsorption) for bottles

For Bottle-1: 50 cc of acetic acid is present :100% i.e. $V= (V_x \times 100) / 100$,

For Bottle-2: $V= (V_x \times 40) / 50$, **For Bottle-3:** $V= (V_x \times 30) / 50$,

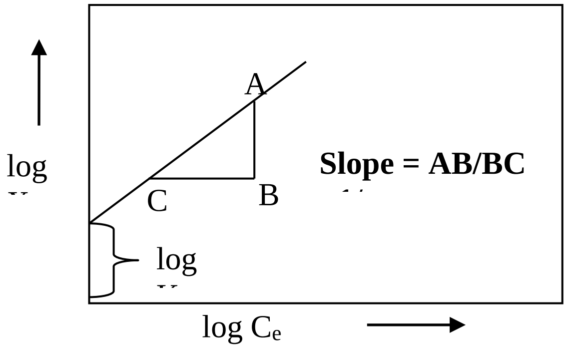
For Bottle-4: $V= (V_x \times 20) / 50$, **For Bottle-5:** $V= (V_x \times 10) / 50$.

Calculation of concentration of acetic acid (C_0 & C_e)

$C_0 = (V \times 0.1) / 5$ $C_e = (V_e \times 0.1) / 5$

Nature of graph.

n = 1/ slope &
log K = intercept
K= antilog (intercept)



Results: 1) Value of n = ------ and 2) Value of K = ----

Conclusion: **1.** The graph of log X v/s log C_e, which gives a straight line.

2. Value of n > 1, indicates that the adsorption of acetic acid on charcoal follows Freundlich adsorption isotherm.

Expt.No.9 ATOMIC PARACHOR (SURFACE TENSION)

Aim: Determination of the Surface Tension and Parachor of toluene, xylene and n-hexane.

Calculate the atomic Parachor of Carbon and Hydrogen.

Chemicals: Liquids: Toluene, Xylene and n-hexane.

Apparatus: Trauts Stalagmometer with pinchcock, beaker, etc.,

Theory: Surface tension is one of the physical properties of the liquid. It is defined as the force in dynes acting on a surface at right angles to any line of unit length. This can be determined by drop number method using Stalagmometer. The surface tension can be calculated according to the following expression:

$$\gamma_L = \frac{n_w \times d_L}{n_L \times d_w} \times \gamma_w$$

Where γ_w = surface tension of water (72 dyne/cm)

γ_L = surface tension of liquid

n_w = no. drops of water, n_L = no.of drops for liquid,

d_w = density of water, d_L = density of liquid.

The magnitude of surface tension is used to calculate the parachor of liquid series, which are additive and constitutive properties. The parachor is calculated using the following equation.

$$P = \frac{M\,\gamma^{1/4}}{d}$$

Where, P = parachor, d = density

γ = surface tension of liquid & M= Mol.Wt. of liquid.

When the parachor are calculated for the liquids of given homologous series, parachor values of difference in the atoms / group can be calculated.

Procedure:

1. Clean the Stalagmometer by ether / acetone. Dry it thoroughly by blowing hot air from rubber air blower and attach a clean and dry rubber with a screw clip in the middle to the upper end of Stalagmometer.
2. Clamp the apparatus exactly in a vertical position, lower end should be slightly inside the edge of the beaker to avoid disturbance while falling the drops by air.
3. Loosen the screw clip and suck the liquid taken in a small beaker, so that it is well above the upper round mark (care must taken to see the liquid does not enter the rubber tube). Close the tubes with the screw clips.
4. Adjust the flow of liquid using the screw clip, so that the number of drops falling from the flat end is between 20-25 drops per minute. Do not disturb this adjustment.
5. Suck the liquid again little above the upper mark and count the number of drops when the liquid flows from the upper round mark to the lower mark.

6. Repeat the same procedure for at least three times and calculate the average number of drops for the liquid.
7. Rinse the stalagmometer with ether / acetone and blow the air to dry before using for new liquid.
8. Take similar readings for other liquids under study and finally for distilled water.
9. Calculate the surface tension and parachor of each liquid by using the suitable formula and finally the parachor of CH_2 from the difference in the parachor of toluene and xylene.
10. From this, calculate the atomic parachor of ‘H’ and ‘C’.

Observations:

Liquids	No. of drops of liquids	Mean(n)	Density g/cc	Surface tension (dyne/cm)	Molar. mass	Parachor (P)
Toluene	i ii iii		0.8660		92	P_T=
Xylene	i ii iii		0.8811		106	P_X=
Hexane	i ii iii		0.6548		86	P_{Hx} =
Water	i ii iii		1.0		18	

Calculation :

1) Surface tension of liquid : $\gamma_L = \frac{n_w \times d_L}{n_L \times d_w} \times \gamma_w$

2) Calculation of parachor: $P = \frac{M \gamma^{1/4}}{d}$

3) Parachor of CH_2 group = $P_X - P_T = P(CH_2)$ =--------

3. Calculation of atomic parachor of hydrogen:

$P(C_6H_{14}) = 6\,P(CH_2) + 2P(H)$

$2P(H) = P(C_6H_{14}) - 6P(CH_2)$

$P(H) = \{ P(C_6H_{14}) - 6P(CH_2)\} / 2$ =--------

4. Calculation of atomic parachor of carbon:

$P(C) = P(CH_2) - 2 \times P(H)$ = ------

Results:

Surface Tension of (dyne/cm)	Toluene	Xylene	Hexane
Parachor of carbon		Parachor of hydrogen	

Theoretical Values

Surface Tension of (dyne/cm)	Toluene	Xylene	Hexane
Parachor of carbon	4.8	Parachor of hydrogen	17.1

Expt.No.10 PERCENTAGE COMPOSITION (VISCOSITY)

Aim: To determine the coefficient of viscosity of liquids A, B and C (C: mixture of A & B) by Ostwald's Viscometer. Calculate the percentage composition of unknown mixture.

Chemicals: Pure liquids A and B and four mixtures of known percentage composition by mass and mixture 'C' of unknown composition.

Apparatus: Ostwald's Viscometer, beaker, specific gravity bottle, weight box, etc.,

Theory: Viscosity is a resistance exerted by a liquid against the displacement of its own molecules. It is expressed in terms of coefficient of viscosity (η). It is defined as 'the force acting on unit area to maintain unit difference of velocity between two parallel layers of liquid 1 cm apart'. It can be calculated using Poiseuille's equation when η of other liquid is known.

$$\eta_l = \frac{\eta_w \text{ x } t_1 d_l}{t_w\, d_w} \text{ ---- poise}$$

Where 't_L' is time of flow of liquid,

't_w' is time of flow of water

'd_L' is density of liquid & 'd_w' is density of water

Experimentally, this can be determined with the help of Ostwald's viscometer by taking water as a reference liquid having 'η_w' , the viscosity coefficient 0.0089 poise. 'η' is an additive property. Hence it is used to calculate the percentage composition of liquid mixtures of unknown composition

Procedure:

1. Clean the Viscometer with acetone or ether and dry it thoroughly by blowing hot air from rubber air blower.
2. Clamp the Viscometer in a perfectly vertical position.
3. Attach a clean piece of rubber tube to the narrow arm of the viscometer.
4. Take 10 cc of the liquid 'A' with the help of a pipette into the wider arm of viscometer.
5. Suck the liquid by a rubber tube into the capillary side bulb up to a little above the upper mark. Allow the liquid to flow down through the capillary and at the same time record carefully the time required for the liquid to flow from upper mark to the lower mark on the capillary arm using stopwatch. Take at least three readings with each liquid and find the mean time in second (t_L).
6. Rinse the Viscometer with ether / acetone and blow the air to dry before using for new liquid.
7. Repeat the same procedure for remaining liquids and lastly find the time of flow for water (t_w).
8. Determine the density of liquid C using a specific gravity bottle. Calculate the η for all the liquids using the given formula.

9. Plot the graph ‘η’ against percentage composition that gives a straight line. From the graph, read the composition of the mixture ‘C’.
10. Percentage composition of can also be calculated using the given formula.

Observation:

1) Determination of Density of C:

1. Weight of empty of specific gravity bottle = w_1 = -----g
2. Weight of specific gravity bottle + liquid C = w_2 = -----g
3. Weight of specific gravity bottle + water = w_3 = -----g
4. Weight of liquid C = $w_2 - w_1$ = -----g
5. Weight of water = $(w_3 - w_1)$ = -----g

$$\text{Density of liquid C} = \frac{\text{Weight of liquid C} = (w_2 - w_1)}{\text{Weight of water} = (w_3 - w_1)}$$

2) Determination of η and percentage composition:

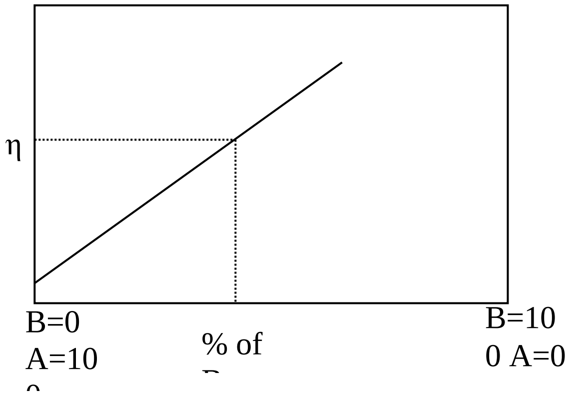
Liquids	**A**	**B**	**C**	**M$_1$**	**M$_2$**	**M$_3$**	**M$_4$**	**H$_2$O**
Percentage composition	100	100		A=40 B=60	A=50 B=50	A=60 B=40	A=70 B=30	
Density (g/cc)	**0.874**	**1.579**		**1.212**	**1.138**	**1.075**	**1.034**	**1.00**
Time of Flow ‘t’(in sec.) i ii iii								
Mean (t)								
	η_A=	**η_B=**	**η_C=**	**η_{M1}=**	**η_{M2}=**	**η_{M3}=**	**η_{M4}=**	**η_w=0.0089**

Calculation:

i) $\eta_l = \dfrac{\eta_w \times t_1 d_l}{t_w\, d_w}$ ---- poise

ii) Percentage composition of ‘B’ in ‘C’ = $\dfrac{100\,(\eta_C - \eta_A)}{(\eta_B - \eta_A)}$

Nature of the graph



Results: 1) Viscosity coefficient of ‘A’ =……………….Poise

2) Viscosity coefficient of ‘B’ =……………...Poise

3) Percentage composition of B in C by formula method =

4) Percentage composition of B in C by graphical method =

Expt.No.11 DISTRIBUTION

Aim: To study the distribution of benzoic acid between water and toluene.

Chemicals: Benzoic acid, toluene, 0.1N NaOH solution and phenolphthalein.

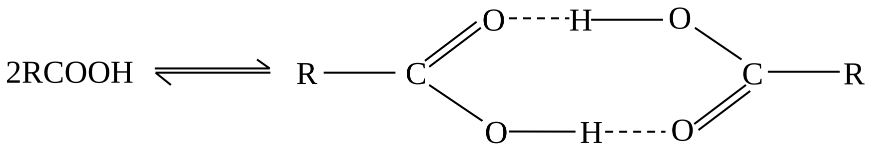
Apparatus: Conical flasks, burette, rubber cork, pipette, etc.,

Theory: When a particular solute is added in two immiscible solvents which are in contact with each other, the solute itself distributes between these two solvents such that, the ratio of the solute concentration in both the solvents remains constant at a constant temperature. This is called Nernst distribution law.

i.e., $$K = \frac{\text{Concentration of solute in solvent-1 } (C_1)}{\text{Concentration of solute in solvent-2 } (C_2)}$$

Where 'K' is a constant known as distribution co-efficient.

When a solute undergoes association or dissociation the law needs modification. For example, when a solute like benzoic acid or acetic acid (mono carboxylic acids) is added to two immiscible solvents such as water and toluene, the solute undergo dimerisation in non polar solvents like toluene due to hydrogen bonding. The dimerisation is represented below:



Hence, K will not remain constant, but the modified law $K^I = C_1 / \sqrt{C_2}$ is constant. The dimerization can be proved experimentally.

Procedure

1. Label the four cleaned having well Stoppard bottles as 1, 2, 3 & 4.
2. Mix water, toluene & benzoic acid from the measuring cylinder to these bottles as per the table-1.
3. Stopper the bottles tightly and shake vigorously for about 30 minutes.
4. Keep the bottles in water bath for 15 minutes to attain the lab. Temperature. Two distinct layers will be separated. Upper will be that of organic layer (toluene) and lower will be aqueous layer.
5. Pipette out 25 cc of aqueous layer from bottle 1 (lower layer) to a clean conical flask and titrate against 0.1 N NaOH solution using phenolphthalein as indicator. Similarly, titrate the aqueous layer for bottle 2, 3 & 4.
6. Separate the toluene layer (upper layer) using separating funnel from bottle No. 1 and pipette out 5 cc of this toluene layer. Add 10 cc water and 2 drops phenolphthalein. Titrate against 0.1 N NaOH solution.
7. Similarly, separate and titrate the toluene layer for bottle number 2, 3 and 4 as above.

8. Calculate the concentration of benzoic acid in aqueous layer (C_1) and toluene layer (C_2) and also calculate $K= C_1 / C_2$ and $K^I = C_1 / \sqrt{C_2}$.

Observations:

Table-1: Preparation of solution mixtures**.**

Bottle No.	Volume of water (cc)	Volume of toluene (cc)	Wt. of benzoic acid.(g)
1	80	20	0.5
2	80	20	1.0
3	80	20	1.5
4	80	20	2.0

Table-2: Determination of K and K^I values**.**

Bottle No.	Burette reading of 0.1N NaOH		Concentration of benzoic acid		**$K= C_1/C_2$**	**$K^I=C_1/\sqrt{C_2}$**
	Aqueous layer (25 cc)	Toluene layer (5 cc)	Aqueous layer (C_1)	Toluene layer (C_2)		
1						
2						
3						
4						

Calculations:

1). $$C_1 = \frac{\text{Volume of 0.1N NaOH (B.R) x Normality of NaOH}}{\text{25 (volume of aqueous layer)}}$$

2). $$C_2 = \frac{\text{Volume of 0.1N NaOH (B.R) x Normality of NaOH}}{\text{5 (volume of toluene layer)}}$$

3). $$K = \frac{C_1 \text{ (Aqueous layer)}}{C_2 \text{ (toluene layer)}}$$

4). $$K_a = \frac{C_1}{\sqrt{C_2}}$$

Result : K^I remain constant.

Conclusion: The values of K^I are found to be constant for all 4 different concentrations of benzoic acid. Hence, benzoic acid is existed in dimeric form in toluene layer and in equilibrium with monomer in the aqueous layer.

Expt.No.12 HEAT OF SOLUTION

Aim: To determine of Heat of solution of KNO_3 by solubility method.

Chemicals: Conc. H_2SO_4, 0.5N NaOH solution, KNO_3 crystals and phenolphthalein.

Apparatus. Thermos flask, 1/10th °C thermometer, magnifying lens, measuring cylinder, etc.,

Theory: Heat of solution is defined as the, the quantity of heat change when one mole of the solute is dissolved in a specified amount of solvent.

$$KNO_3\,(S) + \text{Water} \longrightarrow KNO_3\,(aq): \quad \Delta H_{sol} = Q\ kJ.$$

Solubility of KNO_3 is endothermic in nature. Hence, solution loses heat. The loss of heat can be determined experimentally using calorimeter as under:

A known amount of potassium nitrate is dissolved in given volume of water, the amount of heat absorbed is determined. The amount of heat absorbed, when one mole of potassium nitrate dissolved in one dm^3 water is calculated. It is the heat of solution (ΔH_{sol}).

In the determination of quantity of loss in calorimeter, there will be a loss of heat due to the absorption of heat by the thermometer, wall of the calorimeter, stirrer, water etc. This loss of heat is called water equivalent of calorimeter. It can be measured by measuring the quantity of heat liberated 'Q'for a given concentration of H_2SO_4.

Procedure

I. Determination of water equivalent of calorimeter: *w* (Calorimetric constant).

1. Take 98 cc distilled water in a thermos flask provided with a stirrer and a thermometer and kept at a room temperature.
2. Start stopwatch and temperature is noted for every minute up to 5 minute ($T_1\ ^0C$). Add 2cc of conc. H_2SO_4 at 5.5 minute, stirred well and note down the temperature at 6th min.
3. Record the temperature continuously at the interval of one minute up to 15th minute ($T_2\ ^0C$).
4. Plot the graph of temperature v/s time and calculate the difference in temperature (ΔT_w).
5. Titrate the content of H_2SO_4solution in the calorimeter by pipetting out 10cc to 100 cc conical flask and titrate against 0.5N NaOH solution using phenolphthalein indicator.
6. Calculate the normality and find out the amount of heat liberated (Q) by the calculated normality of H_2SO_4 solution using standard graph.
7. Water equivalent of calorimeter (*W*) can be calculated using the formula: $W = Q/\Delta T_w$

II) Determination of Heat of solution of KNO_3.

1. Take 100 cc of water in a thermos flask, fitted with a thermometer and stirrer.
2. Start stopwatch and note down the temperature for every minute upto 5 minutes. Add 2g KNO_3 crystals at 5.5 minute , stir well and note down the temperature at 6th minute.

3. Record the temperature continuously at the interval of one minute up to 15th minute.
4. Plot the graph of temperature v/s time and find the decrease in difference (ΔT_s).
5. Calculate heat of solution using the given formula.

Observation

Table-1: Determination of water equivalent of calorimeter or Calorimetric constant (***W***).

98 cc H_2O + 2 cc H_2SO_4 (mix at 5.5 min.)

Time (min)	1	2	3	4	5	5.5	6	7	8	9	10	11	12	13	14	15
Temp	←T₁ (water) →					**Mixing**	← T₂ (Solution) →									
(⁰C)						-----										

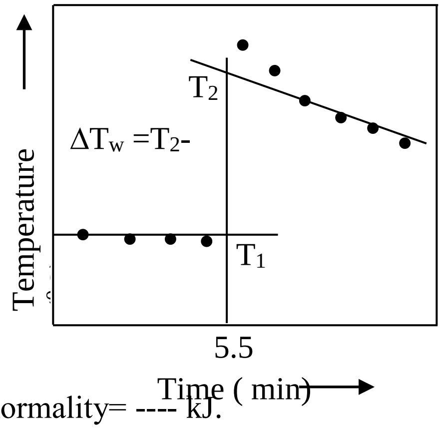
Determination of normality of H_2SO_4 solution:

10 cc of the above H_2SO_4 solution is pipette out in a conical flask and titrated against 0.5 N NaOH using phenolphthalein indicator.

$$(N_1V_1)_{H_2SO_4} = (N_2V_2)_{NaOH}$$

$$N_1 = \frac{0.5 \text{ x } V_2}{10} = \text{-------N}$$

Fig.-1.Determination of ΔT_w



Find out the 'Q' from the standard graph for corresponding normality= ---- kJ.
Calculations of water equivalent of calorimeter = $Q/\Delta T_w$ = ------k J/⁰C.

3) Determination of heat of solution.

100 cc H_2O + 2g KNO_3 (mix at 5.5 min.).

Time (min)	1	2	3	4	5	5.5	6	7	8	9	10	11	12	13	14	15
Temp	← T₃ →					**Mix**	← T₄ →									
(⁰C)						-----										

Heat of Solution (ΔH_{sol}) =

$$\frac{1000 \text{ x } \boldsymbol{W} \text{ x } \Delta T_s}{(\text{Normality x Volume})_{KNO_3 \text{ soln}}}.$$

(2 g KNO_3 in 100 H_2O = 0.198 N)

$$= \frac{1000 \text{ x } \boldsymbol{W} \text{ x } \Delta T_s}{0.198 \text{ x}100}$$

= ------x kJ mol^{-1}.

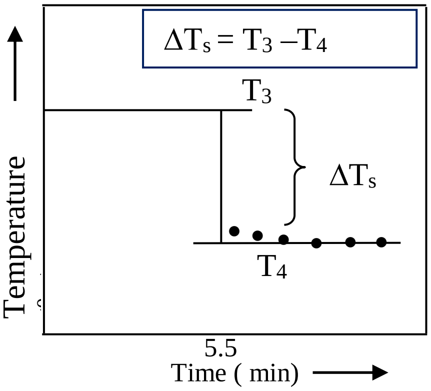
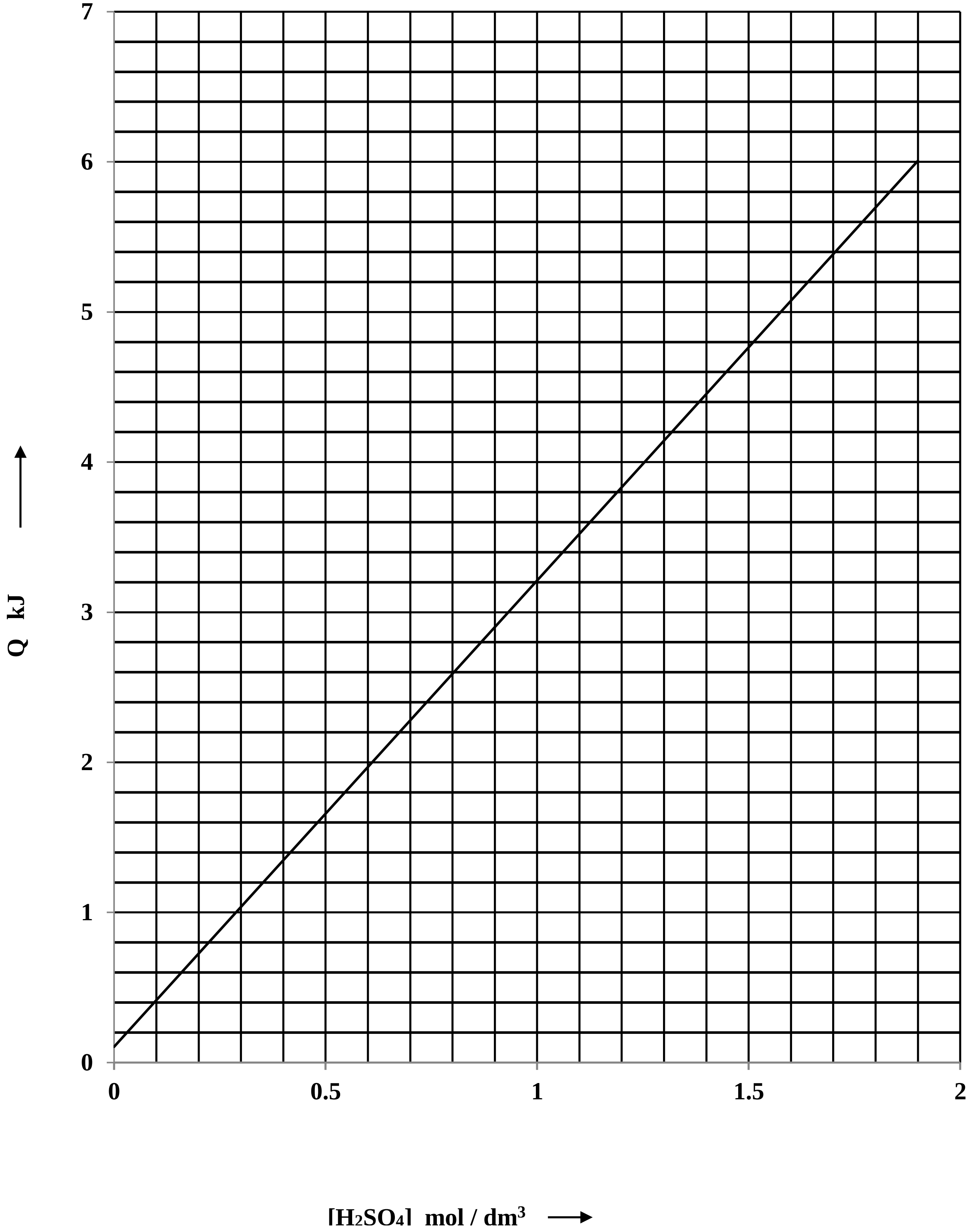


Fig.-2.Determination of ΔT_s

Result: Heat of solution of KNO_3 = ------kJ mol^{-1}.
Theoretical value: Heat of solution of KNO_3 = +35.65kJ mol^{-1}

Standard gragh for determination of water equivalent of calorimeter

Quantity of heat liberated by H_2SO_4 at various concentrations in kJ



B.Sc. IV Sem
Inorganic Experiments

Total No of hours /week : 04Hrs. Pract. : 40 Marks
Total No. of Hours : 52Hrs. I. A. : 10 Marks

Chemistry Practicals : 50 marks

Distribution of Practical Marks

1. Internal Assessment	: 10 marks
2. Experiments	: 40 marks

Distribution of 40 marks of Experiments

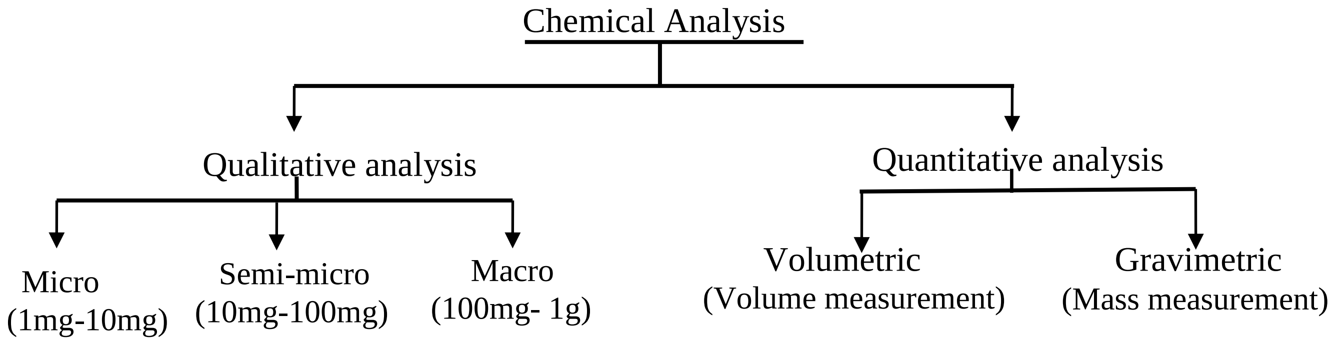
1. Preliminary tests & presentation	: 08 marks (6 + 2)
2. Positive radicals(Gp test+C.T)	: 12 marks [(2+4) ×2]
3. Negative radicals(Gp test+C.T)	: 10 marks [(2+3) ×2]
4. Journal	: 05 marks
5. Viva Voce	: 05 marks
Total	**: 40 marks**

1.Chemical Analysis

Chemical analysis plays an important role in the study of composition or constituents of substances or materials. The chemical analysis comprises of quantitative and qualitative analysis.

a) Quantitative analysis is concerned with the determination of respective amounts of any of the chemical substances (determination of how much of a particular substance is present in a sample).

b) Qualitative analysis deals with the detection or identification of constituents (elements or molecules etc.) present in an unknown compound or mixture of compounds.



Inorganic salts are a class of compounds made up of positively and negatively charged components or species called ions. Positively charged component is positive ion and negatively charged component is negative ion. Positive ions are also called cations or basic radicals as they are derived from bases viz., Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+} and Fe^{3+} etc. and the negative ions are also called anions or acid radicals derived from acids viz., Cl^-, Br^-, I^-, SO_4^{2-}, CO_3^{2-} , $PO_4^{3-}$and BO_3^{3-} etc..

Qualitative analysis of salt mixtures is based on following physico- chemical principles.

Explanation regarding Solubility, solubility product, common ion effect and applications of these in physico-chemical principles of separation of cations into groups in qualitative analysis of in-organic salts

Solubility: This is weight in grams of the substance, which dissolves in 100 gram of the solvent to form a saturated solution at a particular temperature. It can also be defined as the number of moles of solute required just to prepare one dm^3 of the saturated solution at a given temperature or it is the number of moles of the solute dissolved in one dm^3 of the saturated solution.

Solubility of some substances in 100g. of water at 20°C.

Substances	Solubility at 20°C	At 30°C
$BaCl_2$	35.7 g	38.2 g
$CuSO_4$	20.1 g	25.0 g
KI	104.0 g	158.0 g
$BaSO_4$	2.4×10^{-4}g	2.85×10^{-4}g

Solubility of the substance changes with the temperature. As it is seen above, there is an increase in the solubility with increase in temperature. However, in only a few compounds like anhydrous Na_2SO_4, $CaSO_4$ etc. the solubility decreases with increase in temperature.

Solubility of the substances varies with the solvents also. The substances whose solubility is less than 0.01 moles per litre are known as sparingly soluble salts.

An expression for the solubility of sparingly soluble salt can be given by considering a saturated aqueous solution of the type 'BA' in which the following equilibrium exists;

$$\underset{\text{Solid}}{BA} \rightleftharpoons \underset{\substack{\text{solid dissolved}\\ \text{in saturated solution}}}{[BA]} \rightleftharpoons \underset{\text{ions}}{B^+ + A^-}$$

No other species of B & A than these represented are present in the solution, the molar solubility is given by $S = [BA] = [B^+] = [A^-]$

Here 'S' = solubility of BA

Solubility product: There are many ionic solids that are sparingly soluble in water.

For ex: AgCl, $BaSO_4$, Ag_2SO_4 & $Al(OH)_3$ etc.

Solubility product:

Definition: The solubility product is defined as the product of molar concentration of the constituent ions raised to the appropriate power according to the balanced equation.

A sparingly soluble salt dissolves slightly in water forming saturated solution consequently equilibrium is established between the undissociated ionic solid and ions of the dissolved salt.

For ex. In case of AgCl, we have the equilibria

$$\underset{\textbf{Solid}}{AgCl} \rightleftharpoons \underset{\textbf{in solution}}{AgCl} \rightleftharpoons \underset{\textbf{ions}}{Ag^+ + Cl^-}$$

By applying the law of mass action we get, $K = \dfrac{[Ag^+]\,[Cl^-]}{[AgCl]}$

$K[AgCl] = [Ag^+]\,[Cl^-]$; [AgCl] is taken as unity □it is solid.

$K_{sp} = [Ag^+]\,[Cl^-]$ is known as solubility product of AgCl which is constant at a given temperature.

For a sparingly salt 'BA' equilibrium for saturated solution is

$$\underset{\text{Solid}}{BA} \rightleftharpoons \underset{\text{in solution}}{BA} \rightleftharpoons \underset{\text{ions}}{B^+ + A^-}$$

By applying the law of mass action we get $K_{eqm}\ \dfrac{[B^+]\,[A^-]}{[BA]}$

Where 'K_{eqm}' is equilibrium constant.

$$[B^+]\,[A^-] = K_{eqm}\,[BA] = \text{constant}$$

For a general case the sparingly soluble salt of the type $\mathbf{B_mA_n}$

$$\underset{\text{Solid}}{B_mA_n} \rightleftharpoons \underset{\text{ions}}{mB^{b+} + nA^{a-}}$$

$$\mathbf{[B^{b+}]^m\,[A^{a-}]^n = K_{sp}}$$

When the product of the ionic concentration (ionic product) exceeds the solubility product, the salt will precipitate. When the product of ionic concentration is lesser than the solubility product the salt will dissolve till the K_{sp} value is reached. When $\mathbf{[B^+]\,[A^-]}$ = **ionic product** = **solubility product,** solute remains dissolved in solution.

If $[B^+]\,[A^-] > K_{sp}$ salt precipitates

$[B^+]\,[A^-] < K_{sp}$ salt dissolves – solution unsaturated

The molar solubility is given by $S = [BA] = [B^+] = [A^-]$

Here S = solubility of BA

Solubility product expression

An expression which relates solubility of an electrolyte(Salt) and its solubility product.

i) BA type electrolyte(Salt) : $BA_{(s)} \rightleftharpoons B^+ + A^-$

$[B^+] =$ S mol dm^{-3} $[A^-]=$ S mol dm^{-3} $k_{sp} = [B^+]\,[A^-] = S.S$ or $\mathbf{k_{sp} = S^2}$

ii)B_2A type electrolyte(Salt): $B_2A_{(s)} \rightleftharpoons 2B^+ + A^{2-}$

$[B^+] =$ 2S mol dm^{-3} $[A^{2-}]=$ S mol dm^{-3} $k_{sp} = [B^+]^2\,[A^{2-}] = (2S)^2.S$ or $\mathbf{k_{sp}= 4S^3}$

iii) BA_2 type electrolyte(Salt): $BA_{2\,(s)} \rightleftharpoons B^{2+} + 2A^-$

$[B^{2+}] =$ S mol dm^{-3} $[A^-] =$ 2S mol dm^{-3} $k_{sp} = [B^{2+}]\,[A^-]^2= S.(2S)^2$ or $\mathbf{k_{sp} = 4S^3}$

Common ion effect: On dissolving a weak electrolyte like CH_3COOH, NH_4OH etc. an equilibrium gets established between the ions and undissociated molecules

$$\underset{\text{undissociated}}{CH_3COOH} \rightleftharpoons \underset{\text{dissociated ions}}{CH_3COO^- + H^+} \qquad \underset{\text{undissociated}}{NH_4OH} \rightleftharpoons \underset{\text{dissociated ions}}{NH_4^+ + OH^-}$$

A solution strong electrolyte containing a common ion is added, the concentration of the common ion increases. e.g. If NH_4Cl is added to a solution of NH_4OH, NH_4Cl is being a strong electrolyte dissociates completely and provides a large number of NH_4^+ ions (common ions).

$$NH_4OH \rightleftharpoons \boxed{NH_4^+} + OH^-$$

$$NH_4Cl \longrightarrow \boxed{NH_4^+} + Cl^-$$

According to Le-chatelier's principle with the increase in concentration of NH_4^+ ions, the equilibrium of dissociation of NH_4OH gets shifted towards the left. Thus the dissociation of NH_4OH is suppressed.

The suppression of dissociation of a weak electrolyte by the addition of a strong electrolyte that produces a common ion is called "**common ion effect".**

The common cations are divided in to six groups – Group I, II, III, IV, V & VI.

These metal cations of any group are precipitated by a particular group reagent. The identification of cations within each group is based on specific characteristics of each group.

Role of solubility, solubility product & common ion effect in the precipitation & separation of various cations in different groups in qualitative analysis

Group I:(Pb^{2+}, Ag^{+} & Hg^{+} or Hg_2^{2+})- Group Reagent is Dilute HCl

The group I cations get precipitated as their chlorides such as $PbCl_2$, AgCl & Hg_2Cl_2 on adding dilute HCl to the original solution. Chlorides of the cations of Group I have low solubility products and their ionic products exceed easily solubility products resulting in their precipitation.

On the other hand chlorides of the cations of succeeding groups are not precipitated in group I as they have very high solubility products which are not exceeded by their ionic products.

Group II: (Hg^{2+},Pb^{2+},Bi^{3+},Cu^{2+},Cd^{2+}, $As^{3+ \text{ or } 5+}$,$Sb^{3+ \text{ or } 5+}$,$Sn^{2+ \text{ or } 4+}$) -Group Reagent is H_2S in presence of dil. HCl

These cations are precipitated as their sulphides by passing H_2S through their solution containing hydrochloric acid. The dissociation of H_2S is suppressed due to the presence of H^+ ions from HCl(common ion effect). The concentration of S^{2-}, $[S^{2-}]$ is inversely proportional to the concentration of the hydrogen ion & hence the $[S^{2-}]$ ion can be varied by varying concentration of H^+ ion.

$H_2S \rightleftharpoons H^+ + HS^-$ $HS^- \rightleftharpoons H^+ + S^{--}$ or $H_2S \rightleftharpoons 2H^+ + S^{2-}$

Therefore, $[S^{2-}]$ is very low in such solution. Cations of group II precipitate as their sulphides because their solubility products are extremely low. i.e. ionic product easily exceeds solubility products even if $[S^{2-}]$ is very low. On the other hand, CoS, NiS, ZnS & MnS do not precipitate because they have large solubility products. Ionic product of these salts can exceed solubility products only if the solution contains a large number S^{2-} ions.

Group II	Group IV
$CuS = 5 \times 10^{-37}$	$CoS = 3 \times 10^{-26}$
$HgS = 3 \times 10^{-53}$	$NiS = 1.4 \times 10^{-24}$
$CdS = 7 \times 10^{-28}$	$ZnS = 1.2 \times 10^{-23}$
$PbS = 3 \times 10^{-28}$	$MnS = 5 \times 10^{-14}$
$Bi_2S_3 = 1.6 \times 10^{-72}$	

Group III : (Fe^{3+}, Al^{3+} & Mn^{2+}) Group Reagent is NH_4OH

In the Group III, NH_4OH is added to the solution in presence of NH_4Cl, which gives NH_4^+ ions. Due to common ion effect, dissociation of the weak base NH_4OH gets further suppressed.

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$

Group III	Succeeding groups
$Al(OH)_3 = 2 \times 10^{-32}$	$Zn(OH)_2 = 4.5 \times 10^{-17}$
$Mn(OH)_2 = 4.5 \times 10^{-17}$	$Mg(OH)_2 = 4.5 \times 10^{-17}$
$Fe(OH)_2 = 1.5 \times 10^{-11}$	$Sr(OH)_2 = 3.2 \times 10^{-4}$
$Fe(OH)_3 = 6.8 \times 10^{-38}$	

$[OH^-]$ of the solution is extremely low. Still, $Al(OH)_3$ & $Fe(OH)_3$ readily precipitate out because their solubility products are very low. They are easily exceeded even in solutions of low $[OH^-]$. But Zn^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+}, Ba^{2+}, Ca^{2+}, Sr^{2+} & Mg^{2+} etc. do not precipitate as their hydroxides because the ionic product does not exceed the solubility product for these compounds in presence of NH_4Cl & therefore remain in solution.

Group IV: (Co^{2+}, Ni^{2+}, Zn^{2+} & Mn^{2+}) Group Reagent is H_2S in presence of NH_4OH

H_2S is passed in to the solution in the presence of NH_4OH. Dissociation of H_2S is favored in alkaline solutions, as the OH^- ions of the solution remove H^+ ions obtained during dissociation of H_2S as H_2O. $H_2S \rightleftharpoons 2H^+ + S^{2-}$

As a result $[S^{2-}]$ is very high in such a solution. Ionic product exceeds the solubility product for CoS, NiS, ZnS & MnS and hence they precipitate.

Group V : (Ba^{2+},Ca^{2+}& Sr^{2+}) Group Reagent is $(NH_4)_2CO_3$ in presence of NH_4Cl & NH_4OH

When $(NH_4)_2CO_3$ is added in presence of NH_4Cl & NH_4OH to the solution containing Ba^{2+}, Ca^{2+} & Sr^{2+} they get precipitated as carbonates.

$NH_4Cl \longrightarrow NH_4^+ + Cl^-$ $\quad NH_4(CO_3)_2 \rightleftharpoons NH_4^+ + CO_3^{2-}$

Due to the common ion effect, ionization of $(NH_4)_2CO_3$ is suppressed in presence of NH_4Cl & NH_4OH. However, the low concentration of CO_3^{2-} ions is sufficient to exceed the solubility products of carbonates of cations of group IV(Ba^{2+}, Ca^{2+}& Sr^{2+}) while Mg^{2+} ion remain in solution as the solubility product of $MgCO_3$ is very high.

Group VI : (Mg^{2+}, Na^+, K^+) -No Group Reagent

The compounds of Mg^{2+}, Na^+, K^+(& NH_4^+) are water soluble. There is no common reagent that can precipitate them together. They are tested individually. NH_4^+ is tested in the preliminary tests itself or in the beginning to avoid the confusion which may occur by the addition of different salt solutions containing NH_4 during the cation separation in to groups.

Groups	Radicals of the group	Group reagents	Salts precipitated	Colours of ppt
First group	Pb^{2+}	Dil.HCl	$PbCl_2$	White
Second group	Pb^{2+} & Cu^{2+}	H_2S in acidic medium of HCl	PbS,CuS	Black
Third group	Fe^{2+},Fe^{3+} , Al^{3+} & Mn^{2+}	NH_4OH in presence of NH_4Cl	$Fe(OH)_2$, $Fe(OH)_3$ $Al(OH)_3$, $Mn(OH)_2$	$Fe(OH)_2$ -Dirty green, $Fe(OH)_3$-Reddish brown $Al(OH)_3$, -white gelatinous $Mn(OH)_2$-Buff
Fourth group	Ni^{2+} , Co^{2+} Mn^{2+}, Zn^{2+}	H_2S in alkaline medium of NH_4OH in presence of NH_4Cl	NiS, CoS MnS,ZnS	NiS & CoS -Black MnS- Pink(Buff) ZnS – White or grey
Fifth group	Ba^{2+} Ca^{2+}	$(NH_4)_2CO_3$ presence of NH_4OH and NH_4Cl	$BaCO_3$ $CaCO_3$	$BaCO_3$ & $CaCO_3$- white
Sixth group	Mg^{2+},Na^+ & K^+	**No reagent**		
		For Mg^{2+}, Na_2HPO_4 is used in presence of NH_4OH and NH_4Cl	$MgNH_4PO_4$	$MgNH_4PO_4$ - White

Identification of acid and basic radicals present in a salt mixture is carried in the semi-micro scale, in which few milligrams or pinch of solid is used for each test. The solution of reagent is added only in drops. The residues are separated from the solution by centrifugation.

Semi-micro qualitative analysis of mixtures involves the following stages

I. Preliminary Tests

II. Identification of Acid radicals (negative radicals) in solution.
  a) Preparation of sodium carbonate extract or prepared solution (P.S.) for testing the presence of an acid radical.
  b) Confirmatory test for acid radical.

III. Identification of Basic radicals (positive radicals) in solution.
  a) Preparation of original solution (O.S.)
  b) Separation of basic radicals in to groups using common reagent for each group.
  c) Confirmatory test for each basic radical from the group it belongs.

IV. Report

2.Semi - Micro Qualitative Analysis of Inorganic Salt Mixtures

Sl. No.	Tests	Observations	Inference
I PRELIMINARY TESTS			
1. a)	Appearance	Crystalline Amorphous Mixed	Water soluble salts may be present Water insoluble salts may be present Partially water soluble salts may be present.
b)	Colour	Blue Green-Bluish green Yellow Pink Colourless	**Cu^{2+} ,Co^{2+}(**anhydrous) salts may be present **Ni^{2+},Cu^{2+},Fe^{3+}** salts may be present **Fe^{2+}**, sulphides of metals may be present Mn^{2+}, Co^{2+} hydrated salts may be present Coloured salts **Ni^{2+},Cu^{2+},Fe^{3+}** Mn^{2+} etc. absent & NH_4^+,K^+,Na^+,Mg^{2+},Ca^{2+}, Zn^{2+},Ba^{2+}, Al^{3+}etc. may be present.
2.	**Heat Test:** heat the mixture in a dry test-tube & observe.	a) Decrepitates	Crystalline salts may be present.
		b) Water vapour condenses on cooler part	Compounds with water of crystallization may be present.
		c) Gas evolved	
		i) Colourless gas with ammonia smell.	**NH_4^+**salts may be present.
		ii) Colourless gas with smell of rotten eggs.	**S^{2-}** salts may be present.
		iii) Reddish brown fumes	**Br^- or NO_3^-**salts may be present.
		iv) Violet vapours & Blue- black sublimate	**I^-** salts may be present
		d) A Sublimate –White	**NH_4^+** Salts may be present.
		e) Residue: i) Fusible	Alkali salts like **Na^+,K^+** etc may be present
		ii) Infusible and White both when hot & cold	**Ca^{2+},Ba^{2+},Al^{3+}, Mg^{2+}** etc may be present.
		Infusible residue and Yellow when hot and white when cold	**Zn^{2+}** salts may be present.
		Dark coloured infusible residue	**Cu^{2+},Fe^{2+}, Fe^{3+},Mn^{2+}& Ni^{2+}** etc. may be present.
3.	Dil. H_2SO_4 Test Mixture + 5drops of	Gas with brisk effervescence is evolved	**CO_3^{2-}** may be present.

	dil.H_2SO_4 (warm if no action is observed in cold.)	b) Colourless gas with smell of rotten eggs turning lead acetate paper black	**S^{2-}** may be present.
	***C.T. for CO_3^{2-}**		
	Mixture + 5drops of dil.H_2SO_4 heat & pass the gas through lime waer.	Colourless & odourless gas with brisk effervescence turning lime water milky.	**CO_3^{2-}** is present & **confirmed.**
4.	**Conc. H_2SO_4 Test** Mixture + 5drops of Conc.H_2SO_4 First observe in cold	i) Colourless gas with pungent smell, dense white fumes with a glass rod dipped in ammonium hydroxide solution	**Cl^-** may be present
		ii) Reddish brown fumes, with pungent smell (In cold condition**)**	**Br^-** may be present
		iii) Violet vapours.	**I^-** may be present
	Then slowly heat & observe. Cool the T.T. thoroughly & then pour the contents in to the sink.	iv) Reddish brown fumes with pungent odour evolved on heating (But solution. in test tube. is not brown) & colour intensified on adding Cu-turnings.	**NO_3^-** may be present
5.	**Flame Test** Make a small loop at an end of Platinum wire & clean it by dipping in Conc.HCl & heating till no colour is imparted to the flame. Place 2-3 drops Conc.HCl on a watch glass + pinch of Mixture & make a paste. Take thepaste on a loop of clean Platinum wire & heat it by holding at the base of a non luminous flame & observe.	Golden yellow	**Na^+** may be present
		Violet/Lilac	**K^+** may be present
		Brick red	**Ca^{2+}** may be present
		Apple green (yellowish green)	**Ba^{2+}** may be present
		Greenish Blue	**Cu^{2+}** may be present

II) Identification of Acid radicals (Negative radicals) in Solution.

Preparation of Sodium Carbonate Extract: About 200 mg of mixture and 400-500 mg of solid Na_2CO_3 are added to 5-6 ml of distilled water and is boiled for 5-10 min in a conical flask with a funnel at the mouth to reduce the loss due to evaporation. The solution is centrifuged and the centrifugate is known as sodium carbonate extract(S.E.) or prepared solution (P.S.) and is used for the identification of Negative radicals.

***Note :** Carry out C.T. only when radical is present*

Sl.No	Tests	Observations	Inference
1	**Test for SO_4^{2-}** S.E.(5 drops) + dil.HCl (5 drops) Boil, cool + $BaCl_2$ solution(3 drops)	**White ppt. insoluble in Dil. HCl even on boiling**	**SO_4^{2-}** is present
2	**Test for Halides**: S.E. (5 drops) + dil. HNO_3 (5 drops) Boil, Cool + $AgNO_3$(1-2 drops) Transfer a little ppt.to a test-tube containing NH_4OH(5drops) (Carry out respective C.T.s for Halides)	i) White curdy ppt easily soluble in NH_4OH.	**Cl^-** is present.
		i) Pale yellow ppt. sparingly soluble in NH_4OH	**Br^- is present**
		ii) yellow ppt insoluble in NH_4OH.	**I^-** is present
3	**Test for NO_3^-** S.E. (5 drops) + conc.HCl(5 drops) + $MnCl_2$ solution(2-3 drops) boil.	Brown colouration	NO_3^- is present
4	**Test for oxalate($C_2O_4^{2-}$)** S.E.+ dil. Acetic acid, boil to remove CO_2 + $CaCl_2$ solution & heat.	White ppt.	**$C_2O_4^{2-}$** is present
5	**Test for Phosphate (PO_4^{3-})** **S**.E. or O.S. (2 drops) + Ammonium molybdate solution excess (7-8drops) + Conc. HNO_3(5 drops)	Canary yellow ppt. in cold or on warming	**PO_4^{3-} is present**
Confirmatory Tests for Negative ions(Acid Radicals)			
1	**C.T. for S^{2-}**(sulphide) ion		
2.	Mixture + Dil. H_2SO_4 + lead acetate	Black ppt.	**S^{2-}** is present and <u>confirmed.</u>
	C.T. for SO_4^{2-} (Sulphate) ion		
3.	S.E (5 drops)+ dil. acetic acid(5 drops) boil, cool + Lead acetate(3 drops). Transfer little ppt.to a test-tube containing ammonium acetate & heat.	White ppt. soluble in hot ammonium acetate	**SO_4^{2-}** is present & **confirmed**
	<u>C.T. for Cl^-</u> (Chloride) ion		
4.	Mixture + solid $K_2Cr_2O_7$ + Conc. H_2SO_4 in a test tube, heat gently. Pass the brown vapours in to a test tube containing NH_4OH + Acetic acid + lead acetate solution.	Reddish brown vapours give yellow ppt with lead acetate.	**Cl^-** is present & **confirmed**
	Mix.+ MnO_2 in a dry test tube + Conc. H_2SO_4 & Heat gently.	Yellowish green gas turns litmus paper red & then bleaches it.	**Cl^-** is present & **Confirmed.**
5.	**C.T.for Br^- (Bromide) ion** :		
6.	Mix.+ MnO_2 in a dry test tube + Conc. H_2SO_4 & Heat gently.	Reddish brown fumes of bromine evolved	**Br^-** is present & **confirmed.**

7.	**Orange globule Test** **S.E.** (5 drops)+ dil. HNO_3, (or a drop of conc. HNO_3) till acidic, boil cool + 2drops of $CHCl_3$ or CCl_4 + Chlorine water & shake well.	Orange globule	**Br^-** is present & **confirmed.**
	C.T. for I^- (Iodide)		
8.	**Violet globule Test** **S.E.** (5 drops) + dil. HNO_3(or a drop of conc. HNO_3) till acidic, boil cool +2 drops of $CHCl_3$ or CCl_4 + Chlorine water shake well.	Violet globule	**I^-** is present & **confirmed.**
	C.T. for NO_3^-(Nitrate ion) Brown ring Test		
9	**S.E.** (5 drops)+ 2 ml conc H_2SO_4, mix thoroughly, cool, + freshly prepared saturated soln. of $FeSO_4$ (4-5 drops) along the side of the test-tube & allow to stand for 2-3 min without disturbing.	Brown ring at the junction of two layers.	**NO_3^-** is present **and confirmed**
10	**C.T. for oxalate($C_2O_4^{2-}$)**		
	S.E (5 drops)+ Dil.H_2SO_4 (5 drops)+ a drop of very dil.$KMnO_4$ solution.	Pink colour of $KMnO_4$ decolorizes	**$C_2O_4^{2-}$** is present & confirmed.
11	**C.T. for Phosphate (PO_4^{3-})**		
	S.E. or O.S. (5 drops) + 1-2 drops of Magnesia mixture solution ($MgSO_4$+NH_4Cl+NH_4OH)	White crystalline ppt.	**PO_4^{3-} is confirmed**
	Special tests(For mixture containing Cl^-& Br^- or Cl^-& I^-)		
	Special test for chloride in presence of bromide		
12	**S.E.** (5 drops)+ dil. HNO_3, till acidic +conc HNO_3 boil till the soln. becomes colourless, cool, + $AgNO_3$.	Brown vapours White curdy ppt. soluble in NH_4OH.	**Br^- is present and confirmed.** **Cl^- is present and confirmed.**
13	**Special test for chloride in presence of iodide**		
	S.E. (5 drops) + dil HNO_3 till acidic, warm and cool + $AgNO_3$ (3 drops) Centrifuge & treat the ppt. with NH_4OH.	Violet vapours White curdy ppt. soluble in NH_4OH	**I^-** is present and **confirmed** **Cl^-** is present and **confirmed.**

Therefore the negative radicals found are i) ---, ii)---.

III) Identification of Positive Ions (Basic radicals)

Preparation of a solution for cation analysis

Solution used for cation analysis is called original solution. For the preparation of original solution (O.S.) the selection of the suitable solvent is necessary. This may be done by choosing one of the following reagents in the order given -

a) **Dist.Water b) Dil. HCl c) Conc.HCl d) Conc.HNO_3** d) **Aquaregia**(1:3:: Conc.HNO_3 : HCl).

A) Solubility Test:

Solubility of the mixture may be tried with reagents first in cold; if not soluble then contents should be heated.

Sl.No.	Tests	Observations	Inference
1	Mix.+ Dist.H_2O,heat	Soluble	Water soluble compounds present & O.S. is prepared in dist. Water.
2	Mix.+ Dil.HCl	Soluble	Water insoluble compounds present & O.S. is prepared in….

Preparation of original solution(O.S.):

The original solution is prepared by dissolving 100 mg of mixture in 5ml of------- (Solvent) taken in a beaker in cold/hot condition. (Centrifuge if necessary). This solution is called original solution (O.S.) and used for the identification of positive radicals.

NOTE:

1. If the mixture is containing CO_3^{2-} as one of the –ve radicals, prepare original solution in minimum quantity of dil. HCl, boil to expel CO_2, cool and dilute with distilled water.
2. If the mixture is containing **S^{2-} or BO_3^{3-}** as one of the –ve radicals, prepare original solution by dissolving the mixture in minimum quantity of conc.HCl & evaporating to dryness repeatedly in an evaporating dish, cooling and then extracting with dist.water or dil. HCl.
3. If **$C_2O_4^{2-}$** is present in the mixture, then prepare the original solution by dissolving 100 mg of mixture in 1-2 ml aquaregia(1.5 ml conc.HCl + 0.5 ml conc.HNO_3) taken in an evaporating dish and evaporate to dryness. The residue is treated with dil. HCl in cold/hot condition. (Centrifuge if necessary). Dilute the solution with distilled water and is use for the identification of positive radicals.
4. When the mixture does not give a clear solution with water, dil.HCl or conc.HCl & instead gives voluminous ppt. it indicates Pb^{2+} is present. Then treat the mixture with minimum quantity of Conc. HNO_3 and evaporate it to dryness and the residue is extracted with dist. water to get O.S.**]**

Sl.No.	Tests	Observations	Inference
a)	**Test for NH_4^+** Mixture or O.S.(5 drops)+ NaOH(5 drops), Boil & hold a wet litmus paper at the mouth of test-tube	Gas with ammonia smell having alkaline action on litmus paper & white dense fumes with glass rod dipped in conc. HCl.	NH_4^+ may be present
b)	**C.T. for NH_4^+:** **(**If NH_4^+ is present) Mixture or O.S.(5 drops)+ NaOH(5 drops), + NaOH + Nessler's Reagent#	Brown ppt. or solution	NH_4^+ is present and confirmed
	#Preparation of Nessler's Reagent: 2drops $HgCl_2$+ KI drop wise till scarlet ppt. formed just redissolves + NaOH excess till alkaline)		

B) SEPARATION OF CATIONS IN TO GROUPS

<table>
<tr><td colspan="6">O.S. + dil.HCl if precipitation, centrifuge.(if No precipitation proceed with next groups)</td></tr>
<tr><td rowspan="5">Ppt.
Group I
present
White
ppt.-
Pb^{2+}</td><td colspan="5">O.S. /Centrifugate + dil.HCl +H_2S if precipitation, centrifuge.</td></tr>
<tr><td rowspan="4">Ppt.
Group
II
Present
Black
ppt.
Pb^{2+} &
Cu^{2+}</td><td colspan="4">O.S./Centrifugate (if II group present, boiled off H_2S + NH_4Cl(solid) + NH_4OH excess if precipitation, centrifuge.
[Use the residue for the analysis of group-III & centrifugate for next groups]
#If phosphate is present follow Table-3</td></tr>
<tr><td rowspan="3">Ppt.
Group III
Present
White
gelatinous
ppt. –Al^{3+}
*Dirty
green–Fe^{2+}
Reddish
brown –
Fe^{3+}
Buff-Mn^{2+}</td><td colspan="3">O.S./Centrifugate+NH_4Cl(solid) + NH_4OH excess warm +H_2S if precipitation, centrifuge.</td></tr>
<tr><td rowspan="2">Ppt.
Group IV
Present
Black-Ni^{2+}
& Co^{2}
Grey/white
–Zn^{2+}
Buff
/Pink–
Mn^{2+}</td><td colspan="2">O.S./Centrifugate boil off H_2S & concentrate to small volume + NH_4Cl(solid) + NH_4OH excess +$(NH_4)_2CO_3$ warm if precipitation, centrifuge.</td></tr>
<tr><td>Ppt.
Group V Present
White ppt.-Ba^{2+},Ca^{2+}</td><td>O.S./Centrifugate:
Evaporate to dryness.
Residue obtained .
Group VI
Mg^{2+}, K^+, Na^+ present</td></tr>
</table>

(Very important note: If *Fe^{2+} is present & dirty green ppt. obtained, in III group then it is oxidized completely to Fe^{3+} by boiling the O.S. with few drops Conc.HNO_3 before going to IV group then add NH_4Cl(solid) + NH_4OH & centrifuge)

Note:

1. Once the ppt. is obtained in a group, entire O.S. is used to precipitate the radicals completely by adding the group reagents and centrifugation. The residue is analysed in accordance with the particular group and the centrifugate is used for next group analysis.
2. To eliminate II& IV group radicals completely, pass H_2S repeatedly to the hot centrifugate (2-3 times).

Table - 1 Analysis of Group I(Pb^{2+})

White ppt. - Pb^{2+}

Boil the white ppt. with 2-3 ml of distilled water and the soln. tested as follows.

Sl.No.	Tests	Observations	Inference
1	*C.T. for Pb^{2+}*		
	Solution + K_2CrO_4	Yellow ppt.	**Pb^{2+}** is present and confirmed
	Solution + KI Above yellow ppt.+ H_2O, Heat to get clear soln. Cool & observe.	Yellow ppt. Golden yellow flakes	**Pb^{2+}** is present and confirmed

Table - 2 Analysis of Group II(Pb^{2+} & Cu^{2+})

Black ppt.- Pb^{2+} or Cu^{2+}

Treat the ppt. with Dil.HNO_3 (or a drop of Conc. HNO_3) and heat for 2-3min with stirring.

Colourless solution - Pb^{2+} Blue coloured solution –Cu^{2+}

Sl.No.	Tests	Observations	Inference
1	**C.T. for Pb^{2+}**		
	Colourless Solution+ K_2CrO_4	Yellow ppt.	**Pb^{2+}** is present and confirmed
	Colourless Solution+ KI Above yellow ppt.+H_2O Heat to get clear soln. Cool & observe.	Yellow ppt. Golden yellow flakes	**Pb^{2+}** is present and confirmed
2	**C.T. for Cu^{2+}**		
	Blue Solution+ NH_4OH	Bluish white ppt. changes to Deep blue solution	**Cu^{2+}** is present and confirmed
	Blue Solution + Acetic acid + Potassium ferrocyanide	Reddish brown ppt. (Chocolate colour ppt.)	**Cu^{2+}** is present and confirmed
	Blue Solution+ KI	Brown ppt.	**Cu^{2+}** is confirmed

Table- 3 (Phosphate separation- Basic acetate method)

Phosphate radical is not decomposed by heating with mineral acids, it remains as soluble salt in acidic medium but forms insoluble phosphates with cations in alkaline medium. hence must be removed before proceeding to III group onwards as the separation of cations is carried out in alkaline medium by NH_4Cl & NH_4OH. Phosphate is removed by basic acetate method as follows.

If Group II is present, take the centrifugate from Group II and boil to remove H_2S completely (test with lead acetate paper). If GroupI & II are absent then take original solution directly.

If Fe^{2+} is present oxidize it by boiling with 4-5 drops of conc.HNO_3 and use O.S. / Centrifugate for phosphate separation.

O.S. / Centrifugate : Add 0.5 g NH_4Cl & excess of NH_4OH till precipitate obtained is dissolved in minimum amount of dilute acetic acid. Add about 1 cc of saturated ammonium acetate. Precipitate obtained at this stage contains phosphates of III group. Centrifuge the solution and use the residue for the analysis of Group III. The centrifugate contains phosphates of IV,Vand VI groups. The phosphate in the centrifugate is removed by adding excess of neutral chloride to precipitate ferric phosphate and centrifuge. The centrifugate if yellow is treated with NH_4OH to precipitate iron as ferric hydroxide and reject the ppt. The centrifugate is used to test for IV,V & VI group radicals.

Table- 4 Analysis of Group III(Al^{3+}, Fe^{2+}, Fe^{3+},Mn^{2+})

White gelatinous ppt. -**Al^{3+},** Dirty green ppt.- **Fe^{2+}** , Reddish brown ppt.- **Fe^{3+}**
White ppt.turning brown or buff ppt. -**Mn^{2+}**
Boil to dissolve the ppt. in 1-2 ml of dilute HCl and test the solution as follows.

Sl.No	Tests	Observations	Inference
1	**C.T. For Al^{3+}**		
	Solution + pinch of solid NH_4Cl, Boil.	White gelatinous ppt.	**Al^{3+}** is present & confirmed
	Solution + 2% Aluminon solution	Red colour	**Al^{3+}** is present & confirmed
2	**C.T. For Fe^{2+}**		
	Solution + conc. HNO_3 boil,cool + KCNS	Blood red colouration	**Fe^{2+}** is present and confirmed.
	Solution + Potassium ferricyanide	Dark (Turnbull) Blue colour ppt.	**Fe^{2+}** is present and confirmed
	Solution + Potassium ferrocyanide	White ppt. rapidly turns blue	**Fe^{2+}** is present and confirmed
3	**C.T. For Fe^{3+}**		
	Solution + KCNS	Blood red colouration	**Fe^{3+}** is present and confirmed
	Solution + Potassium ferricyanide	No ppt.(brown colouration)	**Fe^{3+}** is present and confirmed
	Solution + Potassium ferrocyanide	Deep(Prussian) blue ppt.	**Fe^{3+}** is present and confirmed

4	**C.T. for Mn^{2+}**		
	Solution + PbO_2 excess + 1-2 ml conc.HNO_3 Boil for few minutes & add some water, cool and allow to stand for few minutes & observe the colour of supernatant liquid.	Violet colour to the supernatant liquid	**Mn^{2+}** is present and confirmed

Table- 5 Analysis of Group IV(Ni^{2+},Co^{2+},Zn^{2+}, Mn^{2+})

	Black ppt.- **Ni^{2+}**or **Co^{2+}** White or grey or cement colured - ppt.-**Zn^{2+},** Buff coloured ppt, **-Mn^{2+}** Boil the ppt. with dil.HCl to dissolve and test the solution obtained as follows.		
Sl.No.	Tests	Observations	Inference
1	**C.T. for Ni^{2+}**		
	Solution + dimethyl glyoxime + NH_4OH till alkaline	Scarlet red coloured ppt.	**Ni^{2+}** is present and confirmed
2	**C.T. for Co^{2+}**		
	Solution + Pinch of KNO_2 + acetic acid	Yellow ppt.	**Co^{2+}** is present & confirmed
	Solution + Pinch of solid $Na_2S_2O_3$ & shake.	Blue colouration	**Co^{2+}** is present & confirmed
	Solution + isoamyl alcohol + Pinch of solid NH_4SCN & shake.	Blue colouration to alcohol layer	**Co^{2+}** is present & confirmed
3	**C.T. for Mn^{2+}**		
	Solution + PbO_2 excess + 1-2 ml conc.HNO_3 Boil for few minutes & add some water, cool and allow to stand for few minutes & observe the colour of supernatant liquid.	Violet colour to the supernatant liquid	**Mn^{2+}** is present &confirmed
4	**C.T. for Zn^{2+}**		
	Solution + NaOH	White ppt. soluble in excess of NaOH.	**Zn^{2+}** is present and confirmed.
	Solution + acetic acid till acidic + Potassium ferrocyanide	White ppt.	**Zn^{2+}** is present and confirmed

Table- 6 Analysis of Group V(Ba^{2+},Ca^{2+})

White ppt. - **Ba^{2+} or Ca^{2+}** Use a part of ppt. for flame test & Boil to dissolve remaining part of ppt. in 1 ml acetic acid & test.			
Sl.No.	Tests	Observations	Inference
	Solution+ Potassium chromate soln.	**Yellow ppt.** No Yellow ppt.	**Ba^{2+}** is present. **Ca^{2+}** is present.
1	**C.T. for Ba^{2+}**		
	Flame Test: White ppt.+ Conc.HCl to make a paste & dip a clean Platinum wire in it & heat by holding it to the edge of	Yellowish or Apple green colour to the flame	**Ba^{2+}** is present and confirmed

	non luminous flame		
2	C.T. for Ca^{2+}		
	Solution + NH_4OH till alkaline + Ammonium oxalate soln.	White ppt.	**Ca^{2+}** is present and confirmed
	Flame Test Dissolve the above white ppt. in Conc. HCl to make a paste & dip a clean Platinum wire in it & heat by holding it to the edge of non-luminous flame	Bright red colouration to the flame	**Ca^{2+}** is present and confirmed.

Table- 7 Analysis of Group VI(Mg^{2+}, K^+ & Na^+)

Evaporate the centrifugate to dryness in an evaporating dish. Use a part of residue for flame test & Dissolve remaining residue in a minimum quantity of distilled water & test the solution for Mg^{2+},K^+ and Na^+ as below.

Sl.No.	Tests	Observations	Inference
	Solution+NH_4OH + sodium hydrogen phosphate solution with a glass rod. scratch inside the test-tube.	White crystalline ppt. No ppt.	**Mg^{2+}** is present **Na^+ or K^+** present
1	**C.T. for Mg^{2+}**		
	Hypoiodite test : I_2 in KI solution + NaOH drop wise till to get yellow colour + test solution.	Reddish brown colour or ppt.	**Mg^{2+}** is present and confirmed
	Solution + Magneson soln.+NaOH	Blue colour or ppt.	**Mg^{2+}** is present and confirmed
2	**C.T.for K^+**		
	Solution + sodium cobalti nitrite (0.5 drops cobalt nitrate solution + pinch(0.1g) of $NaNO_2$ solid + 3 drops of acetic acid)	Yellow ppt.	**K^+** is present and confirmed.
	Solution (5 drops)+Picric acid (3-4 drops)	Yellow ppt.	**K^+** is present and confirmed.
	Flame test Treat the **residue** with 1 drop Conc. HCl & perform flame test	Violet colour imparted to the flame	**K^+** is present and confirmed.
	C.T. for Na^+		
3	Solution +Potassium pyroantimonate ($K_2H_2Sb_2O_7$), scratch the inside of the test-tube with glass rod.	White ppt.	**Na^+** is present and confirmed
	Flame test Treat the **residue** with 1 drop Conc. HCl & perform flame test	Golden yellow colour imparted to the flame colour	**Na^+** is present and confirmed

Therefore the positive radicals found are 1) --------, 2)--------.
Conclusion: Hence the given inorganic mixture contains

Acid radicals		
Basic radicals		

3. Determination of Dissolved Oxygen present in water by Winkler's method

Aim: To determine the dissolved oxygen present in water by Winkler's method.

Principle: Presence of dissolved oxygen is essential to maintain the higher forms of biological life in water. Low oxygen in water can kill fish and other organisms present in water.
When manganous sulphate is added to the water sample containing alkaline potassium iodide, manganese hydroxide is formed. This is oxidized to basic manganic oxide by dissolved oxygen(DO) present in the water sample. When sulphuric acid is added basic manganic oxide liberates iodine, which is equivalent to DO originally present in the water sample. The liberated iodine is titrated with a standard sodium thiosulphate solution using starch as indicator.

Chemicals required:

1. Manganous sulfate solution(48 g of $MnSO_4$ in 100 ml of H_2O)
2. Alkaline potassium iodide (36 g NaOH & 10 g KI in 100 ml of H_2O)
3. 0.025N sodium thiosulphate solution (1.550 g $Na_2S_2O_3$ in 250 ml of H_2O)
4. Concentrated H_2SO_4, Starch indicator

Procedure:

Collect the water sample in 200/250 ml bottle taking care to avoid contact with air. The bottle should be completely filled. Add 1.0 ml of manganous sulfate solution by pipette, dipping the end below the surface. Add 1.0 ml alkaline potassium iodide solution. Insert the stopper and mix thoroughly by shaking. After precipitate has settled, add 2.0 ml Conc. H_2SO_4. Dissolve the precipitate completely by shaking and titrate with sodium thiosulphate using starch as an indicator. Record the ml of titrant used.

Chemical Reactions

$$MnSO_4 \rightleftharpoons Mn^{2+} + SO_4^{2-}$$

$$\text{Alk. KI} \rightleftharpoons K^{+} + I^{-} + OH^{-}$$

$$Mn^{2+} + 2OH^{-} \longrightarrow Mn(OH)_2$$

white ppt. indicates oxygen absent

$$Mn^{2+} + 2OH^{-} + \tfrac{1}{2}O_2 \longrightarrow MnO_2 + H_2O$$

Brown ppt. indicates oxygen present

This refers to fixation phenomenon

$$MnO_2 + 4H^{+} + 2I^{-} \longrightarrow I_2 + Mn^{2+} + 2H_2O$$

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

Observations:

1. Solution in burette = 0.025N $Na_2S_2O_3.5H_2O$ solution
2. Solution in Bottle = Reaction mixture
3. Indicator = Starch solution
4. Colour change = Blue to colourless near the end point.
5. Burette readings = -----ml

Calculations

$$\text{Oxygen in mg/litre} = \frac{V_1 - V_2 \times \text{normality of } Na_2S_2O_3.5H_2O(0.025) \times 8 \times 1000}{\text{Volume of water sample}(200)}$$

Oxygen in ppm = 0.8 × B.R.× normality of $Na_2S_2O_3.5H_2O$ = -----

4. Determination of Chemical oxygen demand (COD)

Aim: To determine the Chemical oxygen demand(COD) of given water sample.

Chemical oxygen demand test is widely used for measuring the pollution strength of waste water and it is the amount of oxygen used in the oxidation of organic matter. All organic compounds with a few exceptions can be oxidized to carbon dioxide and water by the action of strong oxidizing agents like sulphuric acid.

Principal : When the waste water sample is refluxed with known excess of $K_2Cr_2O_7$ in a 50% H_2SO_4 solution in presence of Ag_2SO_4 (as catalyst) and Hg_2SO_4(to eliminate interferences due to chloride) the organic matter of the sample is oxidized to H_2O, CO_2 and NH_3. The excess dichromate remaining unreacted, in the solution is titrated with a standard solution of FAS. Similar procedure is followed for blank titration using the same amount of distilled water instead of waste water.

Chemicals required:

1. Standard potassium dichromate solution (0.25 N – 3.0625 g $K_2Cr_2O_7$ in 250 ml)
2. Standard FAS solution (0.25 N – 24.5 g FAS in 250 ml)
3. Sulphuric acid (1 g Ag_2SO_4 + 1g Hg_2SO_4), Ferroin indicator

Procedure: **Standardisation of FAS solution:**

50 ml of water sample is taken in a round bottom flask and kept in the water bath. 75 ml of conc.H_2SO_4 containing 1g each of Ag_2SO_4 and Hg_2SO_4 is added carefully, with shaking after each addition. 25 ml of 0.25 N $K_2Cr_2O_7$ is added to it with a pipette. The water condenser is attached to the flask and the contents are refluxed for 2 hours, cooled and washed the condenser with distilled water. Transferred the contents to a 500 ml conical flask and diluted to 300 ml, titrated against the standard 0.25 N ferrous ammonium sulphate solution using ferroin indicator till the colour changes from orange to greenish. The burette reading is noted as V_1 ml.

Blank titration: The above procedure is repeated with 50 ml of distilled water instead of water sample and the B.R. is noted as V_2 ml.

Chemical Reactions

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$\text{Organic matter(C \& H)} + 3\,[O] \longrightarrow CO_2 + H_2O$$

$$2FeSO_4 + 4H_2SO_4 + 3[O] \longrightarrow Fe_2(SO_4)_3 + 4H_2O$$

Observations:

1. Solution in burette = 0.25 N FAS solution
2. Solution in conical flask = Reaction mixture
3. Indicator = Ferroin
4. Colour change = orange red to greenish.
5. Burette readings i) Blank titre reading V_1= -----ml
   ii) Main titre reading V_2 = -----ml

Calculations:

$$\mathrm{COD(mg/litre\ or\ dm^3)ppm} = \frac{V_1 - V_2 \times \text{normality of FAS} \times 8 \times 1000}{\text{Volume of water sample(50)}}$$

5. Reactions involved in the test of acidic & basic radicals

Reactions acidic radicals(Anion) X = CO_3^{2-}, S^{2-}, Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-}, $C_2O_4^{2-}$ and PO_4^{3-}

Carbonate CO_3^{2-}

$$MCO_3 + H_2SO_4 \longrightarrow MSO_4 + H_2O + CO_2\uparrow \quad \text{(colourless gas with effervescence)}$$

$$\underset{\text{Lime water}}{Ca(OH)_2} + CO_2 \longrightarrow \underset{\text{Milkyness(calcium carbonate)}}{CaCO_3\downarrow} + H_2O$$

$$MCO_3 + 2HCl \longrightarrow \underset{\text{Soluble}}{MCl_2} + H_2O + CO_2\uparrow \text{(O.S. Preparation)}$$

<u>Sulphide(S^{2-})</u>

$$MS + H_2SO_4 \longrightarrow MSO_4 + H_2S \quad (\uparrow\text{gas with smell of rotten eggs})$$

$$MS + \underset{\text{lead acetate}}{Pb(CH_3COO)_2} \longrightarrow \underset{\text{black ppt.(Lead sulphide)}}{PbS\downarrow} + M(CH_3COO)_2$$

$$MS + 2HCl \longrightarrow MCl_2 + H_2S\uparrow \text{ (O.S. preparation)}$$

Chloride (Cl^-)

$$MCl + H_2SO_4 \longrightarrow M_2SO_4 + \underset{\text{Colourless gas}}{2HCl\uparrow}$$

$$HCl + NH_4OH \longrightarrow \underset{\text{White dense fumes}}{NH_4Cl} + H_2O$$

$$MCl + AgNO_3 \longrightarrow \underset{\text{White curdy ppt . (silver chloride)}}{AgCl\downarrow} + MNO_3$$

$$AgCl\downarrow + 2NH_4OH \longrightarrow \underset{\text{diammine Silver(I) chloride}}{[Ag(NH_3)_2]Cl} + 2H_2O$$

$$MCl + MnO_2 + 2H_2SO_4 \longrightarrow M_2SO_4 + MnSO_4 + 2H_2O + \underset{\text{Greenish-yellow gas}}{Cl_2\uparrow}$$

Chromyl Chloride Test:

$$MCl + K_2Cr_2O_7 + 3H_2SO_4 \longrightarrow \underset{\textbf{Chromyl Chloride Test:}}{2CrO_2Cl_2\uparrow} + 3H_2O + K_2SO_4$$

$$2CrO_2Cl_2 + 4NH_4OH \longrightarrow M_2CrO_4 + 2NH_4Cl + 2H_2O$$

$$M_2CrO_4 + Pb(CH_3COO)_2 \longrightarrow \underset{\text{yellow ppt.(Lead chromate)}}{PbCrO_4\downarrow} + 2CH_3COONH_4$$

Bromide(Br^-)

$2MBr + 2H_2SO_4 \longrightarrow M_2SO_4 + H_2O + SO_2 + Br_2\uparrow$

Reddish brown gas

$2MBr + AgNO_3 \longrightarrow AgBr\downarrow + MNO_3$

Pale yellow ppt (silver bromide)

$AgBr\downarrow + 2MOH \longrightarrow [Ag(NH_3)_2]Br + H_2O$

diammine silver(I)bromide

$2MBr + Cl_2 + CCl_4 \longrightarrow 2MCl + Br_2$ (organic layer)

(Chlorine Water)

Orange

$2MBr + MnO_2 + 2H_2SO_4 \longrightarrow M_2SO_4 + MnSO_4 + 2H_2O + Br_2\uparrow$

Reddish brown gas

Iodide(I^-)

$2MI + 2H_2SO_4 \longrightarrow M_2SO_4 + 2H_2O + SO_2\uparrow + I_2\uparrow$

$2MI + AgNO_3 \longrightarrow AgI\downarrow + MNO_3$

Violet vapours

$2MI + Cl_2 + CCl_4 \longrightarrow 2MCl + I_2$ (organic layer)

(Chlorine Water)

violet

Nitrate (NO_3^-)

$MNO_3 + H_2SO_4 \longrightarrow MSO_4 + 2HNO_3$

$4HNO_3 \xrightarrow{\text{slow}} 4NO_2\uparrow + 2H_2O + O_2\uparrow$

Reddish brown gas(nitrogen dioxide)

$Cu + 4HNO_3 \xrightarrow{\text{Fast}} Cu(NO_3)_2 + 2H_2O + 2NO_2$

blue solution(cupric nitrate) Reddish brown gas

Brown ring test

$MNO_3 + H_2SO_4 \longrightarrow MSO_4 + 2HNO_3$

$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + 4H_2O + 2NO\uparrow$

$FeSO_4 + NO \longrightarrow FeSO_4.NO$

Nitroso ferrous sulphate (Brown ring)

Sulphate (SO_4^{2-})

$MSO_4 + BaCl_2 \longrightarrow BaSO_4\downarrow + MCl_3$

barium chloride White ppt.(barium sulphate)

$MSO_4 + Pb(CH_3COO)_2 \longrightarrow 3PbSO_4\downarrow + M(CH_3COO)_3$

lead acetate White ppt. (lead sulphate)

$PbSO_4\downarrow + 2CH_3COONH_4 \longrightarrow Pb(CH_3COO)_2 + (NH_4)_2SO_4$

ammonium acetate

Oxalate ($C_2O_4^{2-}$)

$M_2(C_2O_4) + H_2SO_4 \longrightarrow MSO_4 + H_2C_2O_4$

$H_2C_2O_4 + KMnO_4 \longrightarrow K_2SO_4 + MnSO_4 + H_2O + CO_2\uparrow$

$M_2(C_2O_4) + CaCl_2 \longrightarrow CaC_2O_4\downarrow + 2\ MCl$

white ppt.(calcium oxalate)

Phosphate (PO_4^{3-})

$M_3PO_4 + 3(NH_4)_2MoO_4 \longrightarrow (NH_4)_3PO_4 + 2M_2MoO_4$

$M_2MoO_4 + 2HNO_3 \longrightarrow H_2MoO_4 + 2MNO_3$

Molybdic acid

$H_2MoO_4 \longrightarrow H_2O + MoO_4$

$(NH_4)_3MoO_4 + 12\ MoO_4 + 6H_2O \longrightarrow (NH_4)_3MoO_4.12\ MoO_3.\ 6H_2O$

canary yellow ppt.(ammonium phospho molybdate)

$M_3PO_4 + MgSO_4 + NH_4OH \xrightarrow{NH_4Cl} Mg\ NH_4PO_4\downarrow + MCl$

white crystalline ppt.(Magnesium ammonium phosphate)

Reactions for basic radicals (cations)

M= Pb^{2+}, Cu^{2+}, Al^{3+}, Fe^{2+}, Fe^{+3}, Mn^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Ca^{2+}, Ba^{2+}, Mg^{2+}, Na^{+}, K^{+} and NH_4^{+}.

Ammonium radical - NH_4^+

$NH_4\,X + NaOH \longrightarrow NaX + H_2O + NH_3\uparrow$ (colourless gas)

$NH_3 + HCl \longrightarrow NH_4Cl\uparrow$ (white dense fumes)

$NH_4X + 2K_2[HgI_4] + 4NaOH \longrightarrow IHg\text{-}O\text{-}HgNH_2\downarrow + 3NaI + 4KI + NaX + 3H_2O$

Nessler's reagent [pot.tetra iodo mercurate (II)] Brown ppt. [Mercury (II)amido – iodide]

Group I. Pb^{2+} Lead(Pb^{2+})

$PbX + HCl \longrightarrow PbCl_2\downarrow + HX$

white ppt.(lead chloride)

$PbX + KI \longrightarrow PbI_2\downarrow + KX$

yellow ppt.(Lead iodide)

Group II Pb^{2+}, Cu^{2+}

Lead(Pb^{2+})

$PbX + H_2S \xrightarrow{dilHCl} PbS\downarrow + HX$

Black ppt.

$$PbX + K_2CrO_4 \longrightarrow PbCrO_4\downarrow + 2KX$$

yellow ppt.(Lead chromate)

Copper Cu^{2+}

$$CuX + H_2S \xrightarrow{dilHCl} CuS\downarrow + HX$$

Black ppt.

$$CuX + 2NH_4OH \longrightarrow Cu(OH)_2\downarrow + NH_4X$$

blusih white ppt.

$$Cu(OH)_2 \ + 2NH_4OH \longrightarrow [Cu(NH_4)_2]X$$

deep blue solution(diammine copper(II) complex)

$$2CuX + K_4[Fe(CN)_6] \longrightarrow Cu_2[Fe(CN)_6]\downarrow + 4\,KX$$

chocholate brown ppt.(copper hexacyanoferrate(II) complex)

$$2CuX + 4\,KI \longrightarrow Cu_2I_2\downarrow + I_2 + KX$$

brown ppt.(cuprous iodide)

Group III Al^{3+}, Fe^{2+} , Fe^{3+} , Mn^{2+}

(Aluminum) Al^{3+}

$$Al\,X + 6NH_4OH \xrightarrow{NH_4Cl} 2Al(OH)_3\downarrow + 3(NH_4)_2X \qquad X = SO_4^{2-}\ \ Cl^-$$

White gelatinous ppt.

$$Al_2(SO_4)_3 + 6NaOH \longrightarrow 2Al(OH)_3\downarrow + 3(NH_4)_2SO_4$$

White gelatinous ppt.

$$Al(OH)_3\downarrow + NaOH \longrightarrow NaAlO_2 + 2H_2O$$

Excess soluble (Sod.aluminate)

(Ferrous ion) Fe^{2+}

$$\mathbf{FeX} \ \mathbf{+}\ 6NH_4OH \xrightarrow{NH_4Cl} 2Fe(OH)_2\downarrow + 3(NH_4)_2X \qquad X = SO_4^{2-}\ \ Cl^-$$

Dirty green ppt.(Ferrous hydroxide)

$$FeX + K_3[Fe(CN)_6] \longrightarrow KFe_2[Fe(CN)_6]\downarrow + 4\,KX$$

potassium ferricyanide Dark blue ppt. (iron hexacyanoferrate(II) complex)Turnbull blue

$$FeX + K_4[Fe(CN)_6] \longrightarrow Fe_2[Fe(CN)_6]\downarrow + 4\,KX$$

potassium ferrocyanide white ppt. turns to blue ppt.(iron hexacyanoferrate(II) complex)

(Ferric ion)Fe^{3+}

$$FeX + 6NH_4OH \xrightarrow{NH_4Cl} 2Fe(OH)_3\downarrow + 3(NH_4)_2X \qquad X = SO_4^{2-}\ \ Cl^-$$

Reddish brown ppt.(Ferric hydroxide)

$$FeX + 3KSCN \longrightarrow Fe(SCN)_3 \ + KX$$

blood red colouration(Ferric thiocyanate complex)

$4FeX + K_3[Fe(CN)_6] \longrightarrow Fe_4[Fe(CN)_6]_3\downarrow + 4\,KX$

potassium ferricyanide soluble Brown coloration (iron hexacyanoferrate(III) complex)

$FeX + 3K_4[Fe(CN)_6] \longrightarrow Fe_4[Fe(CN)_6]\downarrow + 4\,KX$

potassium ferrocyanide blue ppt.(iron hexacyanoferrate(II) complex) Prussian Blue

Manganese(Mn^{2+})

$MnX + 2NH_4OH \xrightarrow{NH_4Cl} Mn(OH)_2\downarrow + (NH_4)_2X + 2H_2O$

white ppt.

$2Mn(OH)_2 + (O) \longrightarrow 2MnO(OH)_2\downarrow + 2H_2O$

white ppt. brown ppt.

$MnX + H_2S + 2NH_4OH \xrightarrow{NH_4Cl} MnS\downarrow + (NH_4)_2SO_4 + 2H_2O$

Buff colour or Pink ppt

$MnS + 2HCl \longrightarrow MnCl_2 + H_2S\uparrow$

$MnCl_2 + 2PbO_2 + 4HNO_3 \longrightarrow 2Pb(NO_3)_2 + 2H_2O + HMnO_4$

(solution) lead dioxide pink or violet colour(permanganic acid)

Group IV Ni^{2+}, Co^{2+}, Mn^{2+}, Zn^{2+}

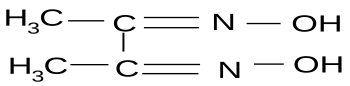
Nickel(Ni^{2+})

$NiX + H_2S + 2NH_4OH \xrightarrow{NH_4Cl} NiS\downarrow + (NH_4)_2SO_4 + 2H_2O$

black ppt (Nickel sulphide)

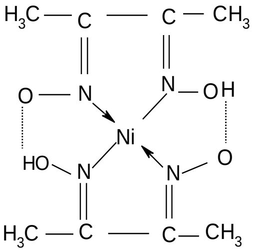
$NiS + 2\,HCl \longrightarrow NiCl_2$

Ni^{2+} +2



Dimethyl glyoxime

$\xrightarrow{NH_4OH}$



Scarlet red ppt.
Ni(DMG) complex

Cobalt (Co^{2+})

$CoX_2 + H_2S + 2NH_4OH \xrightarrow{NH_4Cl} CoS\downarrow + 2NH_4X + 2H_2O$

black ppt (cobalt sulphide)

$CoX + 2\,KNO_2 \longrightarrow Co(NO_2)_2 + KX$

$Co(NO_2)_2 + 4\,KNO_2 \longrightarrow K_4[Co(NO_2)_6]$

potassiumhexanitiritocobaltate(II)

$K_4[Co(NO_2)_6] + 2\,CH_3COOH + (O)$ **air** $\longrightarrow 2K_3[Co(NO_2)_6]\downarrow + 2CH_3COOK + H_2O$

pot.cobalti nitrite yellow ppt. pot hexanitritocobaltate(III)

Zinc (Zn^{2+})

$$MnX + H_2S + 2NH_4OH \xrightarrow{NH_4Cl} ZnS\downarrow + (NH_4)_2SO_4 + 2H_2O$$

white or grey ppt. (Zinc sulphide)

$$ZnS + 2HCl \longrightarrow MnCl_2 + H_2S\uparrow$$

$$ZnSO_4 + 2NaOH \longrightarrow Zn(OH)_2 + Na_2SO_4$$

White ppt

$$Zn\mathbf{X} + K_3[Fe(CN)_6] \longrightarrow Zn_2[Fe(CN)_6] + 4\,KX$$

white ppt.(zinc hexacyanoferrate(II) complex)

Group V (Ba^{2+}, Ca^{2+})

Barium (Ba^{2+})

$$BaCO_3 + 2HCl \longrightarrow BaCl_2 + H_2O + CO_2\uparrow \text{ (O.S. Preparation)}$$

Soluble

$$BaCl_2 + (NH_4)_2CO_3 \xrightarrow[NH_4Cl]{NH_4OH} BaCO_3\downarrow + 2NH_4Cl$$

(Original solution) White ppt

$$BaCO_3 + 2\,CH_3COOH \longrightarrow Ba(CH_3COOH)_2 + H_2O + CO_2\uparrow$$

soluble barium acetate

$$Ba(CH_3COOH)_2 + K_2CrO_4 \longrightarrow BaCrO_4\downarrow + 2\,CH_3COOK$$

yellow ppt.(barium chromate)

Calcium (Ca^{2+})

$$CaCO_3 + 2HCl \longrightarrow CaCl_2 + H_2O + CO_2\uparrow \text{(O.S. Preparation)}$$

Soluble

$$CaCl_2 + (NH_4)_2CO_3 \xrightarrow[NH_4Cl]{NH_4OH} CaCO_3\downarrow + 2NH_4Cl$$

(Original solution) White ppt

$$CaCO_3 + 2\,CH_3COOH \longrightarrow Ca(CH_3COOH)_2 + H_2O + CO_2\uparrow$$

soluble barium acetate

$$CaCl_2 + (NH_4)_2C_2O_4 \longrightarrow CaC_2O_4\downarrow + 2NH_4Cl$$

(Original solution) White ppt.(calcium oxalate)

Group VI Mg^{2+}, Na^{+} K^{+}

Magnesium(Mg^{2+})

$$MgX + Na_2HPO_4 + NH_4OH \xrightarrow{NH_4Cl} Mg\,NH_4PO_4\downarrow + Na_2X$$

white crystalline ppt.(Magnesium ammonium phosphate)

$$MgX + 2\,NaIO \longrightarrow Mg(IO)_2\downarrow + Na_2X$$

sodium hypoiodite Brown ppt. (magnesium hypoiodite)

Sodium (Na^+)

$2NaX + K_2H_2Sb_2O_7 \longrightarrow Na_2H_2Sb_2O_7\downarrow + 2KX$

potassium pyroantimonate white ppt. (sodium pyroantimonate)

Potassium (K^+)

$3KX + Na_3[Co(NO_2)_6] \longrightarrow K_3[Co(NO_2)_6] + \downarrow 2NaX$

sod. hexanitritocobaltate(III) yellow ppt. pot hexanitritocobaltate(III)

(sodium cobalt nitrite)

$KX + C_6H_2(NO_2)_3OH \longrightarrow C_6H_2(NO_2)_3OK\downarrow + HX$

Picric acid Pot.picrate (yellow ppt.)

Reference Books

1. Vogel's Qualitative Inorganic Analysis (7th edn.)– revised by G.Svehla
2. Advanced Inorganic Analysis –Dr.Subhash Rastogi, Dr.Satish Agarwala& Dr.Keemti Lal
3. A Text book of Analytical Chemistry –H.D.Tayal & A.K. Manglik
4. Practical Chemistry –Pandey,Bajpai & Giri
5. Textbook of Chemical Analysis-Raju & Mathur
6. Analytical Chemistry-Dhrubha Charan Dash
7. Lab Experiments in Organic Chemistry –Arun. Sethi, New Age International Ltd. New Delhi. 2006

B.Sc. V Sem: Paper - I

QULITATIVE ANALYSIS OF SOLID-SOLID ORGANIC BINARY MIXTURE

Total No of Hours/Week	: 04 Hours	Practical	:40 Marks
Total No of Hours	: 52 Hours	IA	:10 Marks

CONTENTS

Qualitative analysis of solid – solid organic mixtures: Identification of nature and separation of mixture (in semi micro scale). Characterization of any one separated compound through Preliminary tests, Element test, Physical constant, Functional Group test and preparation of suitable derivative and its physical constant.

Acids: Salicylic, Cinnamic, Phthalic and Anthranilic acid.

Phenol: α –naphthol, β–naphthol.

Base: p-toluidine, m-nitroaniline and p-nitroaniline.

Neutral: Naphthalene, Acetanilide, Diphenyl, Benzamide, Benzophenone and m-dinitrobenzene.

Instructions

In a batch of ten students, not more than two students should get the same mixture in the practical examination. Viva questions may be asked on any of the experiments prescribed in the practical syllabus. During practical examination chart may be referred whenever necessary.

DISTRIBUTION OF MARKS	
Nature and separation	2 +3
Prelimanary tests	02
Element test	04
Physical constant	03
Functional Group test	04
Identification and Structure	03
Preparation of derivative	03
Physical constant of derivative	03
Systematic Presentation	03
Journal	05
Viva voce	05
TOTAL	**40**

SEPARATION OF SOLID-SOLID BINARY ORGANIC MIXTURE AND SPOTTING (QULITATIVE ANALYSIS) OF ORGANIC COMPOUND

INTRODUCTION

The purpose of organic qualitative analysis is to spot a given organic substance and to substantiate its nature by performing a set of reaction/s with it. The whole process of this analysis is based on the two important concepts, namely, Homologous series and Functional group. A compound belonging to a particular class will exhibit characteristic reactions of the atoms or group of atoms present in it. Compounds of a particular homologous series show a similarity in chemical reactions and gradation in physical properties.

The process of identifying unknown compound/s is analogous to solving a puzzle. An organic chemist can often identify a sample in a very short time by performing solubility tests and some simple tests of functional group/s. Millions of known organic compounds are easily and effectively classified into a limited number of groups based on their functional group. Part of the challenge of organic qualitative analysis lays in borderline cases and possible exceptions to the general rules for many of the tests. One must work with an open, unprejudiced mind, ready to make, and test, preparing derivatives this lead to success in finding the identities of unknown compound under investigation. Thus obtained information will help to determine the structure of an unknown compound. This is the way things were done prior to the advancement of modern instrumentation like Spectroscopic methods advanced chemical technique/s using sophisticated equipments.

Five basic areas of experimental inquiry are useful for identifying an unknown compound. One must develop an understanding for what information can be obtained from each of them.

The five areas of inquiry are;

(I) Physical properties (II) Classification by solubility (III) Elemental analysis by sodium fusion (IV) Classification tests for functional groups (V) Synthesis of solid derivative, and authentication by its M.P.

All the areas of experimental inquiry just listed depend on what can be called the structural theory of organic chemistry. By discovering how compounds act under certain conditions, a chemist can deduce what their structures are. Once you have a large number of characteristics in hand, you can deduce the structure of a compound.

I. Physical properties

The physical appearance of an unknown will be our first information in the search to discover its identity. Simply knowing that the compound is a solid rather than a liquid at room temperature narrows the search considerably. A few solids have characteristic bright colors that may be of great significance in reaching a final answer. The physical properties of a compound that are of interest in qualitative analysis are its appearance and its melting point or boiling point etc.

II. Solubility

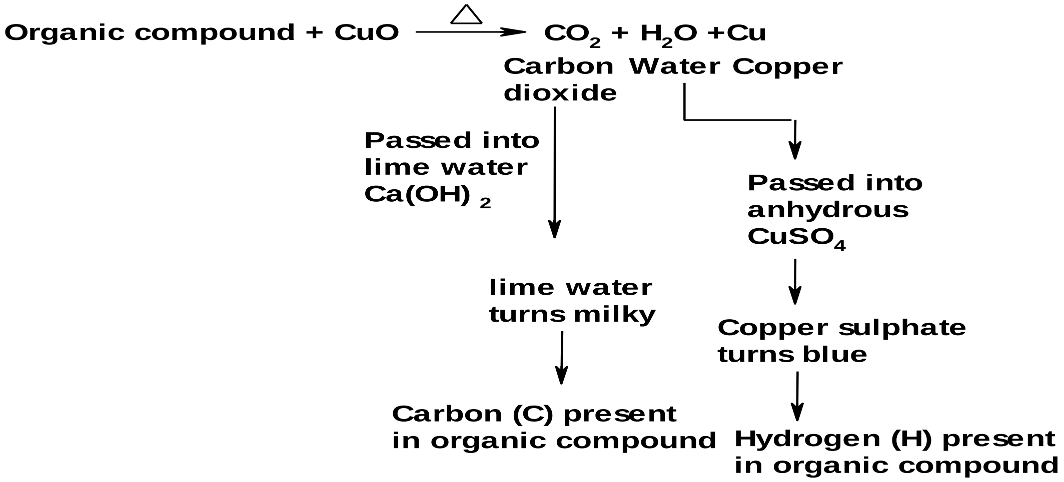
Solubility tests should be performed on every general unknown because they are quick and reliable and use only a small amount of sample. One can gather valuable information about possible functional groups through the use of the solubility classifications. Five common reagents are used for solubility tests are (1) Water (2) $NaHCO_3$ (3) NaOH (4) HCl,and (5) Concentrated H_2SO_4. Except in the case of water, solubility experiments probe the acid-base properties of organic compounds.

If a compound is an acid, you can obtain a relative measure of its acid strength by testing it against the weak base sodium bicarbonate and the stronger base sodium hydroxide. Naturally, *any organic compound that is soluble in water is also likely to be soluble in* $NaHCO_3$*, NaOH, HCl, and* H_2SO_4 *solutions* because these solutions are composed largely of water.

III. Elemental analysis

Presence of oxygen in organic compound is detected by testing for functional group containing oxygen eg- alcohol (–OH), aldehyde (–CHO), ketone (RCOR), carboxylic acid (–COOH), ester (–COOR) and nitro ($–NO_2$) etc.

Detection of Carbon and Hydrogen is generally carried in the following way.



Lassaigne's Test:

Sodium fusion converts halogens to corresponding sodium halides, which are easily detected by silver nitrate. A knowledge of the elemental composition of a substance is useful in planning the identification procedure.

$$\underset{\text{Organic compound}}{\textbf{C, H, S, N, X (covalent forms) + Na}} \longrightarrow \underset{\text{X= halogen/s}}{\textbf{NaCN, Na}_2\textbf{S, NaX}_{\textbf{(ionic)}}}$$

- **Halogen test:** Sodium extract is treated with $AgNO_3$ following reactions takes place.

$$NaX + AgNO_3 \longrightarrow AgX + NaNO_3$$
$$\text{ppt} \big\downarrow 2NH_4\,OH$$
$$[Ag(NH_3)_2\,]X + 2H_2O$$

- **Sulphur test:** $Na_2S + Pb(CH_3COO)_2 \longrightarrow PbS + 2CH_3COONa$

- **Nitrogen test:** On treatment sodium extract with freshly prepared $FeSO_4$, $FeCl_3$ in presence of H_2SO_4 a prussian blue or greenish blue coloration is observed.

$$\underset{\substack{\text{Ferrous amm.}\\ \text{Sulphate}}}{Fe(NH_4)_2(SO_4)_2} \rightarrow \underset{\substack{\text{sodium}\\ \text{ferrocyanide}}}{Na_4Fe(CN)_6} + Na_2SO_4\ 6NaCN + (NH_4)_2SO_4$$

$$Na_4Fe(CN)_6 + Fe^{+3} \longrightarrow \underset{\substack{\text{Sodium ferriferrocyanide}\\ \text{(Prussian blue)}}}{NaFe\,[Fe(CN)_6] + 3Na^+}$$

IV. Functional Groups

The functional groups are detected based on the elements present in the compound. These are categorized as follows;

C,H, and **(O)** : This category includes Alcohols, phenols, aldehydes, ketones, carboxylic acids, esters and even hydrocarbons.

C,H, (O) and **N** : This category includes Amines, amides, annilides nitro compounds and even bi-functional compounds containing nitro and amino groups of Alcohols, phenols, aldehydes, ketones, carboxylic acids, esters etc.

C,H, (O),N and **S** : This category includes thio compounds containing amino, amido, annilido and nitro groups.

C,H,(O), N , and **X** : This category includes halogen compounds containing amino, amido, annilido and nitro groups.

C,H,(O), N , S, and **X:** This category includes thio and halogen compounds containing amino, amido, annilido and nitro groups.

V. Derivative:

After detecting the functional group/s by carrying out confirmative tests, finally the compound under investigation is confirmed by preparing its solid state derivative. Thus formed derivative is confirmed by finding its melting point.

Solid – Solid type of mixtures

The organic compounds are classified in to the four groups viz., acid, phenol, base and neutral. Organic acids contain carboxylic acid group. Phenols are the class of organic compound where –OH group is directly attached to the benzene nucleus. Organic compounds containing amino (–NH_2) group are the bases. The rest of the organic compounds containing various functional groups are neutral in nature**.** Usually the following combination of mixtures of organic compounds is given for the separation.

1] Acid + Base 2] Acid + Neutral 3] Phenol + Base 4] Phenol+ Neutral 5] Base + Neutral 6] Neutral + Neutral (Acid + Phenol combination is usually not given)

After separation of the organic compound from the mixture, the individual compound is systematically analyzed. The process of analysis / identification of an organic compound is called **"organic spotting".**

Principles of separation: Solid Mixture:

Generally two components present in the mixture are of different nature. They may differ in their solubility in water, acids, and alkalies or in some common solvents. The separation of two components of the solid mixture is achieved by dissolution of one component in a solvent or a reagent leaving behind the insoluble component which is collected by filtration.

Separation of organic mixture is based on the chemical nature of components like difference in polarities, acidic or basic strength. General method developed for the separation is based on the concept of converting one of the components in to salt which being polar becomes soluble in water. For example, an acid dissolves in sodium bicarbonate solution as it forms its salt sodium benzoate and can be regenerated by neutralization of the solution with hydrochloric acid. Aniline (or bases) forms its salt aniline-hydrochloride & goes into aqueous solution when treated dilute HCl or dilute sulphuric acid. Acetanilide dissolves in hot water and can be obtained back by evaporation of the solvent.

Compounds differing in acidic strength can be separated by extraction methods. Strong acids form salts with sodium bicarbonate solution whereas the weaker acids do not react with this weak base. So, weakly acidic Phenols dissolve in sodium hydroxide solution but not in sodium bicarbonate. So it is necessary, at the beginning itself, to find out the nature of the components of a solid-solid binary mixture before starting the actual separation.

Sl.No.	Test	Observation	Inference
1	Mixture + $NaHCO_3$	Partially soluble with effervescence	One component is Carboxylic acid.
2.	Mixture + NaOH	Partially Soluble in NaOH	One component is Phenol / acid.
3.	Mixture + HCl	Partially Soluble in HCl	One component is base (an amine).

Note: If only one component is detected then the remaining component is **neutral.**

Systematic analysis of the individual compounds: The two components in their pure forms are now analysed in the following systematic manner.

1. Preliminary tests
2. Identification of nature of the compound
3. Determination of melting point or boiling point.
4. Detection of Elements
5. Detection of functional groups
6. Identification of the compound
7. Confirmation of the compound through preparation of derivative.

IDENTIFICATION OF NATURE OF SOLID-SOLID BINARY MIXTURES

Take 5 mg of solid mixture in a test tube, add reagents as given below and observe.

Sl.No	Test	Observation	Inference
1	Mixture + 1cc $NaHCO_3$ solution. Shake well & filter the above solution, (if soluble)- Residue* Filtrate + Conc. HCl, cool (add ice)	Effervescence & Partially soluble Precipitate	One component is **Carboxylic acid.** **Carboxylic acid** is present.
	If above test is negative, perform the following tests.		
2.	***Residue** from above solution or **Mixture +** 1cc NaOH solution. Shake well & filter the above solution, Filtrate + Conc. HCl, cool.(add ice)	Partially Soluble in NaOH Precipitate	One component is **Phenol**. **Phenol** is present.
3.	Mixture + 1cc HCl(1:1), Shake well & add H_2O filter the above solution, Filtrate +NaOH cool. (add ice pieces)	Partially Soluble in HCl Precipitate	One component is **base** (an amine). **Base** (an amine) is present.

If only one component is detected then the remaining component is **neutral.**

Conclusion: **N**ature / type of the given binary mixture is________________

Note: If one component of mixture is soluble both in $NaHCO_3$ and NaOH solutions gives ppt with Conc. HCl, nature of the component is **ACID.** If it is insoluble in $NaHCO_3$ but soluble only in NaOH and gives ppt with Conc. HCl then nature of the component is **PHENOL**

1. SEPARATION OF THE COMPONENTS OF A BINARY MIXTURE

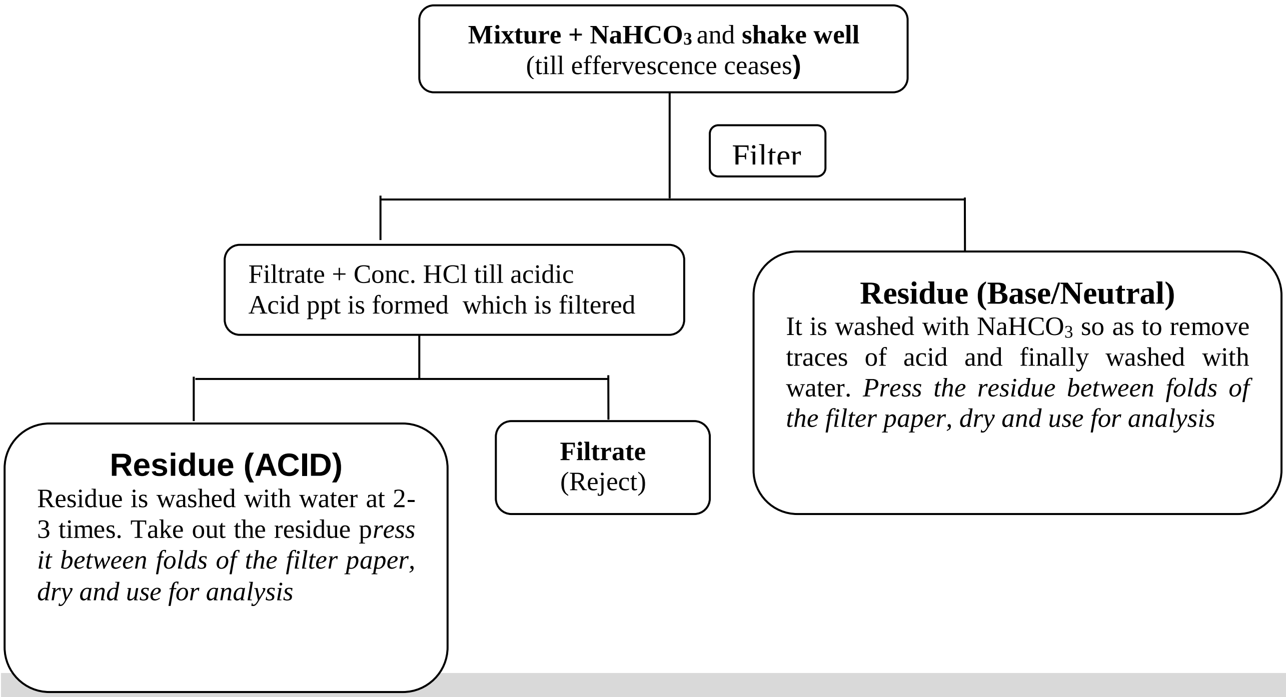
Follow the scheme according to the type of binary mixture

SCHEME - I

(ACID + BASE or ACID + NEUTRAL MIXTURE)

Separation of two components when one of the component is acid i.e., given mixture is of the type Acid + Base or Acid + Neutral

Take about 85% given mixture in a beaker. Add 10% sodium bicarbonate solution till there is no effervescence. Stir thoroughly with a glass rod and filter. Acidify the filtrate with conc. HCl. Follow below mentioned scheme.



Mixture + $NaHCO_3$ and **shake well**
(till effervescence ceases**)**

Filter

Filtrate + Conc. HCl till acidic
Acid ppt is formed which is filtered

Residue (ACID)
Residue is washed with water at 2-3 times. Take out the residue p*ress it between folds of the filter paper, dry and use for analysis*

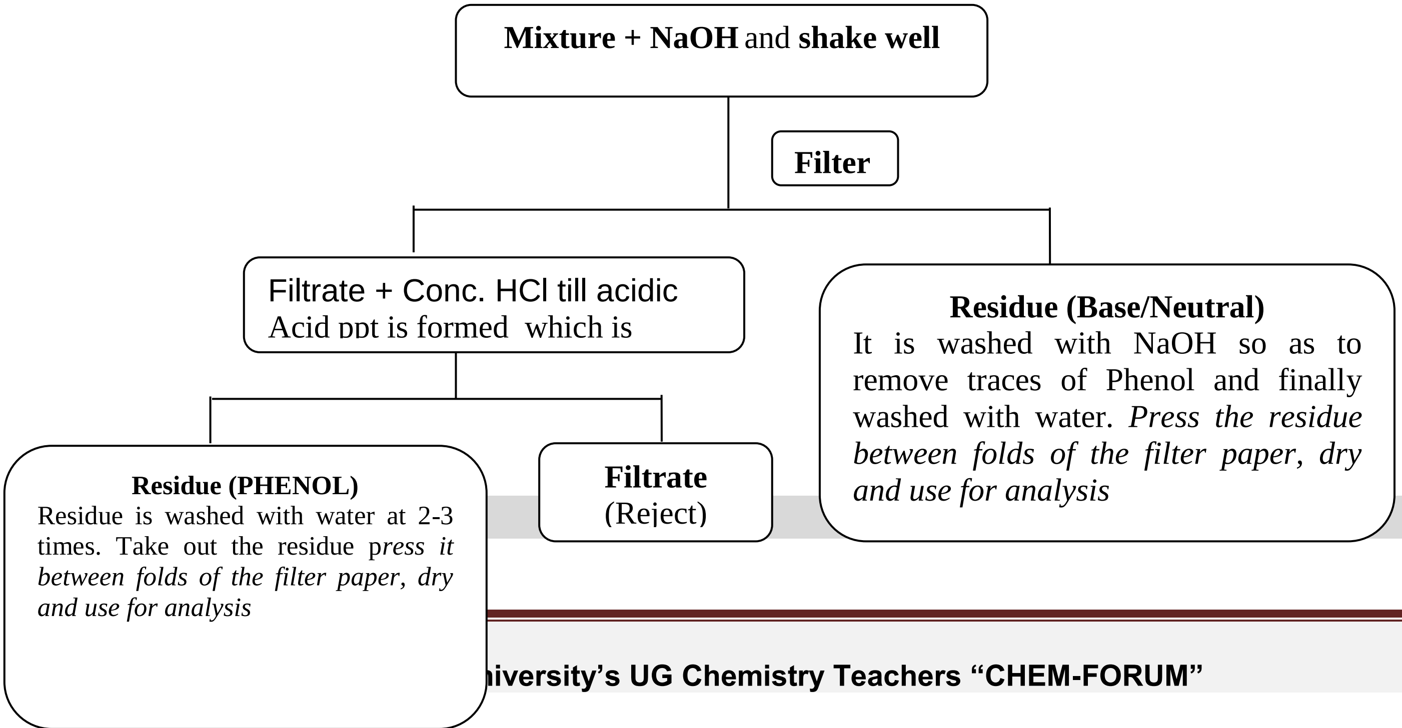
Filtrate
(Reject)

Residue (Base/Neutral)
It is washed with $NaHCO_3$ so as to remove traces of acid and finally washed with water. *Press the residue between folds of the filter paper, dry and use for analysis*

SCHEME - II

PHENOL + BASE or PHENOL + NEUTRAL MIXTURE

Take the mixture in a beaker. Add 10% NaOH solution till alkaline . Stir thoroughly with a glass rod and filter. Acidify the filtrate with conc. HCl. Follow below mentioned scheme.



Mixture + NaOH and **shake well**

Filter

Filtrate + Conc. HCl till acidic
Acid ppt is formed which is

Residue (PHENOL)
Residue is washed with water at 2-3 times. Take out the residue p*ress it between folds of the filter paper, dry and use for analysis*

Filtrate
(Reject)

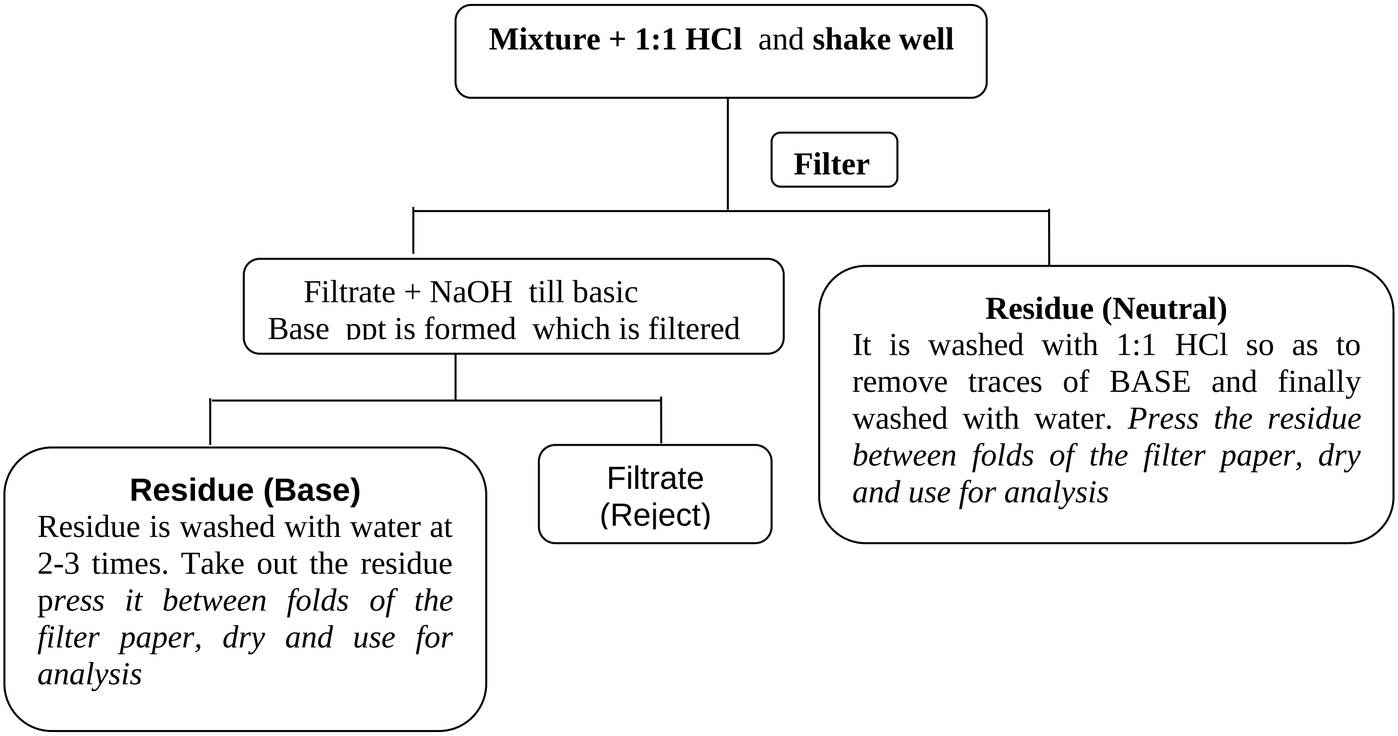
Residue (Base/Neutral)
It is washed with NaOH so as to remove traces of Phenol and finally washed with water. *Press the residue between folds of the filter paper, dry and use for analysis*

SCHEME - *III*

(BASE + NEUTRAL MIXTURE)

Separation of two components when one of the component is Base i.e., given mixture is of the type *Base + Neutral*

Take the mixture in a beaker. Add 1:1 HCl . Stir thoroughly with a glass rod and filter. Add NaOH solution to the filtrate till alkaline and filter. Follow below mentioned scheme.



Mixture + 1:1 HCl and **shake well**

Filter

Filtrate + NaOH till basic
Base ppt is formed which is filtered

Residue (Neutral)
It is washed with 1:1 HCl so as to remove traces of BASE and finally washed with water. *Press the residue between folds of the filter paper, dry and use for analysis*

Residue (Base)
Residue is washed with water at 2-3 times. Take out the residue *press it between folds of the filter paper, dry and use for analysis*

Filtrate
(Reject)

QUALITATIVE ANALYSIS OF ORGANIC COMPOUND

I. Preliminary Tests

S.N	TEST	OBSERVATION	INFERENCE
1.	**Appearance and Colour**	Colourless solids	**<u>Acids</u>** Salicylic acid, Phthalic and, Anthranilic acid may be present **<u>Neutral Compounds</u>** Naphthalene, Acetanilide, Diphenyl, Benzamide, Benzophenone
		Coloured solids	
		Cream coloured solid	Cinnamic acid
		Pink (or pale brown)	*β*-Naphthol
		Dark brown	*α*-Naphthol
		Pale yellow	*m*-dinitrobenzene
		Turmeric or greenish yellow	*m*-nitroaniline or *p*-nitroaniline may be present
		Black/dirty green shining crystals	*p*-toluidine may be present
		Cream coloured solid	Cinnamic acid
		Pink (or pale brown)	*β*-Naphthol
2.	**Odour**	Pleasant odour	Diphenyl may be present
		Moth ball smell	Naphthalene
		Cinnamon like odour	Cinnamic acid
		Phenolic odour	Naphthols
		Fishy odour	Amines (*p*-toluidine, *m*-nitroaniline or *p*-nitroaniline)
		Odour of bitter almonds	*m*-dinitrobenzene
		No Characteristic odour	Carboxylic acids (Except cinnamic acid)
3.	**<u>Beilstien's Test</u>** Heat a loop of copper wire till it does not impart green colour to the flame. Cool, and take substance on loop of copper wire and then heat it.	(a) Burns with sooty flame	Aromatic compounds present
		(b) Burns with non-sooty flame	Aliphatic compounds present
		(c) Green edged flame after the initial sooty flame has vanished	Halogenated compounds present ***Exception:*** Urea gives a green flame due to cyanide of copper formed and not due to halogen

Conclusion: The given compound____________________________ (Aliphatic / Aromatic)

SOLUBILITY TEST (IDENTIFICATION OF NATURE OF THE COMPOUND)

II. Solubility Test

Take a little compound in a test tube and test the solubility in the following solvents.

a) Compound(0.5 g) + water (1ml) Shake well and test with litmus paper ***If insoluble*** **Compound** + water + heat Shake well and test with litmus paper	Soluble in cold water and solution is acidic to litmus. (blue to red) Sparingly soluble in cold but soluble in hot water and solution is acidic to litmus. (blue to red) Soluble and the solution is neutral to litmus.	Carboxylic acid (Anthranilic acid) may be present Carboxylic acids(Phthalic acid, Salicylic acid, Cinnamic acid etc.) may be present Acetanilide, Benzamide may be present
b) Compound (0.5 g) + $NaHCO_3$ (1 ml)and shake well	Soluble with effervescence	Carboxylic acid is present
c) Compound (0.5 g) + NaOH (1ml) and shake well	*Dissolves in NaOH and $NaHCO_3$ and reprecipitated by adding conc. HCl Dissolves only in NaOH but not in $NaHCO_3$	Carboxylic acid is present Naphthols (phenol) present
d) Compound +1:1 HCl and shake well	Soluble and reprecipitated by adding NaOH Soluble in water & dil HCl	Bases like amines, -p-toluidine) may be present Acidic (Anthranilic acid) or neutral(acetanilide)may be present
e) Compound +Conc.H_2SO_4	**Soluble** with colour (yellow) Soluble with red colour In soluble	Ketones may be present Cinnamic acid may be present Aromatic Hydrocarbons may be present

Note: If the given compound is soluble in H_2O & acidic to litmus, and it is soluble in $NaHCO_3$ & NaOH – Acidic

- If the given compound is soluble only in NaOH & insoluble in $NaHCO_3$ & reprecipitated by adding Con.HCl- Phenol
- If the given compound is soluble only in HCl & reprecipitated by adding NaOH & insoluble in NaOH &$NaHCO_3$ – Basic.
- If the given compound is soluble in H_2O & neutral to litmus, and it is insoluble in $NaHCO_3$,NaOH &HCl or soluble/ insoluble in all –Neutral(Aromatic Hydrocarbons, amides, Anilides etc.)
- *If substance gives test with both $NaHCO_3$ solution as well as NaOH, then report as Carboxylic acid. If fails to give test with $NaHCO_3$ solution but soluble only with NaOH, report it as* ***Phenol***

II. TEST FOR SATURATION AND UNSATURATION

i. Baeyer's reagent (Alkaline $KMnO_4$) 0.2 g comp. + 2cc Na_2CO_3 solution + 2-3 drops of very dilute $KMnO_4$ solution	Decolourisation of $KMnO_4$ No decolourisation of $KMnO_4$	*Unsaturated compounds may be present Saturated compounds may be present
ii. ii **Bromine water or Bromine in Carbon tetra Chloride** 0.2 g comp. + 2cc bromine water. (If compound is water insoluble perform the test with bromine in carbon tetra chloride).	Decolourisation of Br_2 No decolourisation of Br_2	Unsaturated compounds may be present Saturated compounds may be present

* Quickly oxidisable compounds like phenols, aromatic amines. Aldehydes & ketones change purple colour to brown or black at once.

Conclusion: The given compound is ____________________ (Saturated / Unsaturated)

III. DETERMINATION OF PHYSICAL CONSTANT:

Determine physical constant (melting point) M.P. using Thiele's Apparatus or Electric melting point instrument

The melting point of the given compound is______ °C(Observed)

Literature value ……….°C

The melting point is represented in range by ± 0.2^0C. for example 156^0C - 158^0C or 157^0C - 159^0C

IV. DETECTION OF ELEMENTS:

Generally organic compounds contain Nitrogen(N), Halogen(X) and Sulphur(S) along with Carbon, Hydrogen and (Oxygen). For the detection of N, X, and S the **Lassaigne's** test is performed.

Preparation of Sodium fusion extract(S.E.)

Place a piece of dry sodium metal (*dried by pressing between folds of the filter paper*) in a fusion tube and heat till sodium melts to form shining globule. Add a pinch of an organic compound and heat slowly and then strongly until the tube becomes red hot. Plunge the tube at once in a china dish or 50 ccl beaker containing 5 cc. of distilled water. Boil the resulting contents to concentrate for about five minutes and filter the hot solution. The filtrate so obtained is called as ***Lassaigne's sodium fusion extract (S.E.).***

i. <u>Test for Nitrogen (N)</u> 1 ml of S.E. + 1ml of freshly prepared saturated $FeSO_4$ solution + 1or 2 drops NaOH, boil well, add 2 drops of $FeCl_3$, cool thoroughly and acidify with conc. HCl or dil. H_2SO_4.	Blue ppt or greenish blue coloured solution	Nitrogen present
ii. <u>Test for Sulphur (S)</u> a) *<u>Nito prusside solution test</u>* 1 ml of S.E. + 3-4 drops of fresh and very dilute sodium nitro prusside solution + 1or 2 drops NaOH solution.	Intense purple colour	Sulphur present
b) *<u>Lead acetate solution test</u>* 1 ml of S.E. is acidified with 1ml of dilute acetic acid + 2-3 drops of lead acetate solution.	Black ppt of PbS	Sulphur present
iii. <u>Test for Halogens (X)</u> 2 ml of S.E. treated with dil HNO_3 till acidic boil well, cool and add few drops of Silver nitrate ($AgNO_3$) solution.	i. White curdy ppt. readily soluble in ammonia solution.	Chlorine present
	ii. Pale yellow ppt. soluble in ammonia solution.	Bromine present
	iii. Yellow ppt. insoluble in ammonia solution	Iodine present

Conclusion : The compound contains the elements : C, H, (O) and ………

V. DETECTION OF FUNCTIONAL GROUPS:

The functional groups are detected based on the elements present in the compound and categoried into the following division; **a]** C,H,(O) **b]** C,H, (O) and N **c]** C,H, (O),N and S **d]** C,H,(O), N , and X and **e]** C,H,(O), N , S, and X

Division: I : Compounds containing elements C, H, & (O). The compounds may be Acids / Phenols / Neutral.

1. TEST FOR CARBOXYLIC ACIDS

DISTINGUISHING TESTS FOR ACIDS

<u>Neutral $FeCl_3$ Test :</u> Compound + 1 ml H_2O heat to dissolve + 3 drops of neutral $FeCl_3$ Solution and observe.	(a) Violet colour in cold disappearing by HCl	Salicylic acid present
	(b) Buff coloured ppt (warm if you do not get in cold) dissolved by ammonia or HCl .	Cinnamic acid present
	(c) Reddish brown ppt or buff coloured ppt soluble in HCl.	Phthalic acid present

Confirmatory Tests for Carboxylic Acids		
C.T. for Salicylic acid : compound + 5drops methyl alcohol + one drop of conc. H_2SO_4 warm cool and pour in cold water taken in a beaker.	Smell of oil of wintergreen (Iodex smell)	Salicylic acid is present and **confirmed**
C.T. for Cinnamic acid : To the aqueous solution of the acid + 2-4 drops of $CaCl_2$ Solution	White ppt. insoluble in acetic acid	Cinnamic acid is present and **confirmed**
C.T. for Phthalic acid : **(Flourescein test) :** Fuse a pinch of the compound with equal quantity of resorcinol, Cool + 2-3 drops of conc. H_2SO_4 warm, cool and pour in water containing 2-5 drops of NaOH taken in a beaker.	Reddish green fluorescence (red colour with a green fluorescence)	Phthalic acid is present and **confirmed**

2. TEST FOR PHENOLS

Distinguishing Tests for phenols (α – Naphthol & *β*-Naphthol)

i. **Neutral $FeCl_3$ solution Test** Sub + alcohol, shake well and add 1-2drops of neutral $FeCl_3$ solution	a) Green colour immediately changing to a white ppt. b) White ppt slowly changing to violet.	*β*-Naphthol present *α*-Naphthol present
C . T. for Naphthols ii. **Phthalein fusion Test** 0.2g sub + 0.2g Phthalic anhydride + 3drops of con. H_2SO_4 fuse the mixture in a dry test tube gently for about 5-10 minutes. Cooled and diluted with 2ml water and pour into beaker containing 10ml of 10% NaOH solution .	a) Very faint green colour with slight blue fluorescence b) Green colour	*β*-Naphthol present & Confirmed *α*-Naphthol present & Confirmed
C . T. for Naphthols 0.1 g. of substance + 5ml of 10% NaOH solution + Few drops chloroform + Copper turnings and warm gently	a) Blue colour to the solution b) Blue colour changes to green-brown on exposure	*β*-Naphthol present & Confirmed *α*-Naphthol present & Confirmed

3. TEST FOR NEUTRAL COMPOUNDS (KETONES AND AROMATIC HYDROCARBONS) (Benzo phenone, *Naphthalene and Diphenyl*)

Test for Ketone (Benzo Phenone) (a) Sub + Conc. H_2SO_4. (b) Sub + Dry sodium metal (rice grain size) fuse on gentle heating	Yellow solution Deep blue colour	Benzophenone present Benzophenone present and confirmed
2,4 – DNP Test Take Compound in a TT, add ethyl alcohol + Brady's reagent (2,4,DNP) warm on water bath. (*take orange ppt. as derivative)	Orange ppt.	Benzophenone is confirmed
Test for Hydrocarbons 0.1 g. of substance + 0.5cc of Conc. H_2SO_4	Insoluble	Hydrocarbon Present (Naphthalene or Diphenyl may be present . confirmed on the basis of their M.P.s)
C.T. for Naphthalene Compound + benzene + Picric acid in benzene,mix& shake well	Yellow ppt.	**Naphthalene** is present and confirmed
C. T. Dipehenyl Compound (0.5g) + 2 ml of fuming HNO_3 (or 1 cc of con. H_2SO_4 + 1 cc of Con. HNO_3) in a conical flask. Heat for 5 minutes, cool and pour it into ice cold water.(* take white ppt. as derivative)	White ppt.	(Biphenyl) Diphenyl is present and confirmed

Division II: Compounds containing elements C, H, (O) & N. The compounds may be Acids/ Bases / Neutral.

Test for Acids (Anthranilic acid)

i. Sub + $NaHCO_3$ solution	Soluble with effervescence	Acid (-COOH) present
ii. Test for $–NH_2$ Group by Diazotisation: ***Diazotization test*** ***Diazotization:*** 0.1g Comp. + 3 times conc. HCl in a test tube and cool in ice cold water + add few drops of ice cold solution of sodium nitrite($NaNO_2$). Add an ice cold solution of β-Naphthol in NaOH to the above solution.	Orange dye stuff	**'$-NH_2$'** (primary amino) group present.
iii. Comp. + Alcohol	Soluble with blue fluorescence	Anthranilic acid present

iv. C.T. for Anthranilic acid Mix a small amount of substance with equal amount of $CaCl_2$ and heat gently. Dissolve the product in 2 ml. of alcohol.	Red coloured solution exhibiting violet fluorescence on standing	Anthranilic acid present and Confirmed
v. 0.1g Sub + $ZnCl_2$ fuse by gentle heating dissolve the product in alcohol	Yellow colour	Anthranilic acid present and Confirmed

TEST FOR BASES: (*p*- Toluidine or *p*-Nitroaniline or *m*-Nitro aniline)

Sub + 1:1 HCl	Soluble and re precipitation with NaOH	Base present
Test for $-NH_2$ Group by Diazotisation: ***Diazotization test*** ***Diazotization:*** 0.1g Comp. + 3 times conc. HCl in a test tube and cool in ice cold water + add few drops of ice cold solution of sodium nitrite($NaNO_2$). Add an ice cold solution of β-Naphthol in NaOH to the above solution.	Orange Red dye	**$-NH_2$ group** is present Amine is present (p-Toluidine or Nitro aniline)
Test for $-NO_2$ group : Mulliken's Test **(Neutral Reduction test) :** Dissolve the Compound (0.3 g) in 0.5 ml of hot 50% aqueous alcohol + 5-6 drops of 10% $CaCl_2$ + pinch of Zn dust. Boil the mixture for a minute. Filter and test the filtrate with Tollen's reagent (To silver nitrate add NaOH. Then add NH_4OH till the ppt. first formed dissolves)	A black ppt. or grey ppt.	**$-NO_2$ group** is present (Nitro anilines present)
C.T. FOR NITRO ANILINES:		
Dissolve the Compound in (0.2 g) 0.5 ml acetone + titanous chloride reagent,(0.5 ml) warm the mixture very gently.	Discharge of Mauve colour of the titanous chloride	*m*- & *p*-Nitro aniline is present and confirmed
Further these m- & p-Nitro anilines are confirmed by their melting points.		
C.T. FOR NITRO *P*-TOLUIDENE		
0.5g sub + 3-4 drops of dilute HCl. +2 ml water + 2-3 drops of $FeCl_3$ solution.	A pale yellow colour changing to red	*p*- Toluidene present and confirmed

TEST FOR NEUTRAL COMPOUNDS

COLOURLESS (BENZAMIDE & ACETANILIDE), M-DINITROBENZENE(YELLOW)

Compound + Water warm	Soluble in hot water	-Anilides(Acetanilide) &(Benzamide) present
Compound + NaOH, Warm	Smell of NH_3 No smell of NH_3 *(Fishy odour of aniline)*	Amide is present **(Benzamide)** Anilides(Acetanilide) is present
Confirmatory tests for Benzamide, Acetanillide or m-dinitrobenzene		
<u>C.T. for Benzamide</u> Boil the compound with dilute NaOH for 5 minutes, cool and acidify with dilute H_2SO_4	White ppt. of benzoic acid	Benzamide is present and confirmed
C.T. for Acetanilide Compound **+** dilute HCl, heat to dissolve, then cool in ice + ice cold solution aq. $NaNO_2$ solution + ice cold solution β-Naphthol in excess NaOH.	Bright Red ppt.	Acetanilide present and confirmed

Division –III: Compounds containing elements C, H, (O) & Halogens.

i. <u>Beilstien,s Test</u> Heat a loop of copper wire till it does not impart green colour to the flame. Cool, and take substance on loop of copper wire and then heat it.	Green edged lame after the initial sooty flame has vanished	Halogen present
<u>Test for Hydrocarbons</u> 0.1 g. of substance + 0.5 cc of Conc. H_2SO_4	Insoluble	Halogenated hydrocarbon Present

Note: as per the syllabus halogen compounds are not included

VI. BROAD INFERENCE

S.N	Particulars	Inference
1.	Nature	:
2.	Aliphatic / Aromatic	:
3.	Saturated / Unsaturated	:
4.	Physical Consatant (Melting point)	M.P. = ____°C Literature ____°C
5	Elements present	
6	Functional group (s) present	:
7	Name of the compound	:
8	Molecular formula	:
9	Structural formula	:
10	Name of the Derivative	:
11	Structural formula of the Derivative	:
12	Physical Constant (Melting point) of the derivative	M.P. = ____°C Literature ____°C

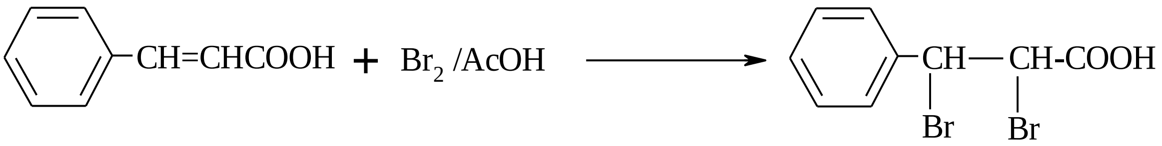
PREPARATION OF DERIVATIVES

A derivative may be defined as a chemical compound obtained by the chemical reaction of a substance, generally retaining the structure of parent substance.

Preparation of a derivative constitutes the last and of course confirmatory step in systematic identification of an organic compound since the identification of organic compound is said to be correct if the melting point of the derivative coincides with the melting point given in the literature for the same derivative of the same compound.

1. Dibromo derivative of Cinnamic Acid (2,3-Dibromo-2-phenyl propionic acid)

Dissolve about 0.5g of cinnamic acid in 5 ml. of glacial acetic acid in a 100 ml. beaker or conical flask and add excess (5-6ml) of solution of bromine in acetic acid in small lots with constant shaking. Allow the reaction mixture to stand for about 10 min. and dilute with water. Filter, and wash the product with water and dry. Recrystallise from hot water and determine its M.P.

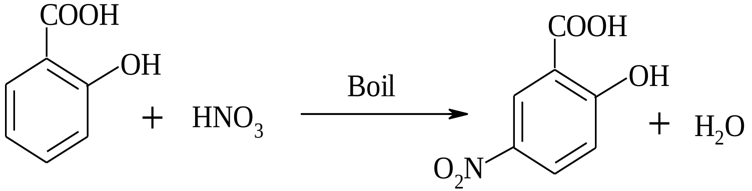


3-phenyl-prop-2-ene-1-oic acid
(Cinnamic acid)

2,3-Dibromo-3-phenylpropanoic acid

2. 5-Nitro Salicylic acid from Salicylic acid

Dissolve the compound (0.5 gm) in hot water and add 0.5 ml of dilute HNO_3 and boil for 5 minutes. Yellow solution is obtained Pour it into the ice – cold water taken in a beaker. Solid separates. Filter, and wash the product with water and dry. Recrystallise from hot water and determine its M.P.

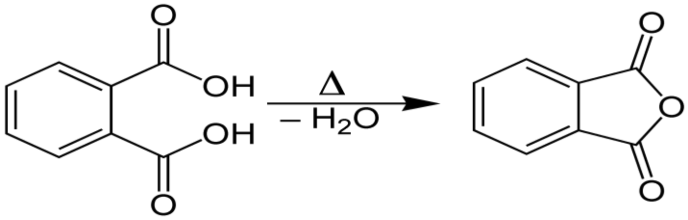


Salicylic acid

5-Nitro salicylic acid

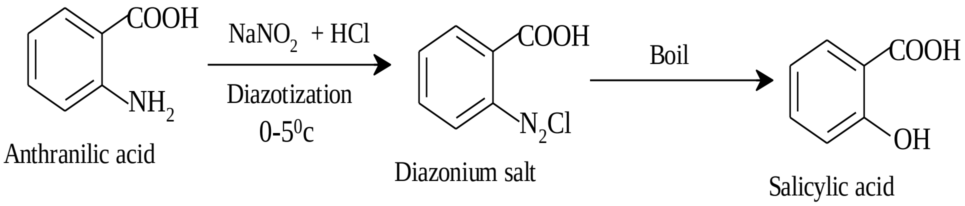
3. Phthalic acid to Phthalic anhydride

Take 0.5g Phthalic acid in a china dish covered with filter paper having a hole in the middle. Place an inverted funnel on the filter paper, lightly plug the nozzle with cotton or filter paper, and heat the dish on a sand bath. On sublimation the acid converts into Phthalic anhydride which collects on the inner side of the funnel. Collect the crystals of phthalic anhydride and determine its M.P.



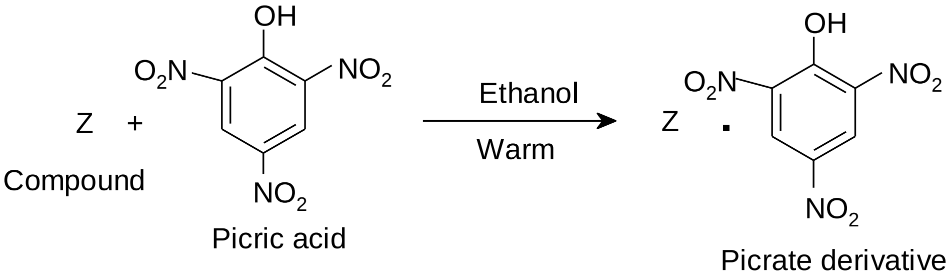
4. Anthranilic acid to Salicylic acid

Diazotise anthranilic acid as follows: Dissolve 0.5g of acid in about 4ml of 1:1 HCl and cool thoroughly. To this solution, add $NaNO_2$ solution drop by drop till a drop of the solution just tints the starch – iodide paper blue, showing a slight excess of HNO_2.Boilthe solution until the evolution of nitrogen ceases. Cool and shake thoroughly, Salicylic acid separates out easily. Dry and recrystallise from hot water, determine its the M.P.



5. Picrate derivative for α-Naphthol , β-Naphthol and Naphthalene

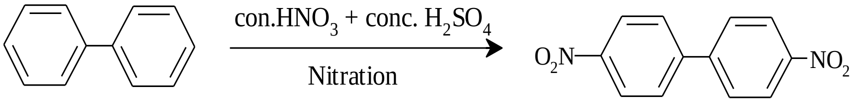
Dissolve 0.5 to 1 g of the given substance (α – naphthol or β – naphthol or naphthalene) in ethanol. Add 2-3 ml of saturated solution of picric acid in the ethanol. Picrate derivative separates out on mixing. In case no solid separates on mixing , heat the reaction mixture on hot water bath. Cool thoroughly. Filter the product, recrystallize from alcohol(if necessary), dry and determine its M.P.



Z = α – Naphthol or β – Naphthol or Naphthalene whichever is given

6. *p,p'*- Dinitro diphenyl from Diphenyl

Dissolve 0.5g. substance in 3ml of conc H_2SO_4 add 2ml. conc HNO_3. Shake well and place the test tube in a gently boiling water bath for about 5-10 minutes with occasional shaking. Pour the reaction mixture in 50ml, ice cold water with constant stirring. Filter, dry and recrystallise from aqueous alcohol and determine its M.P.

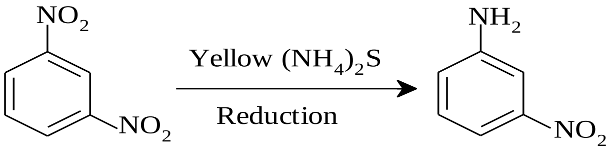


Diphenyl

p,p'-Dinitro diphenyl

7. *m*-Nitroaniline from *m*-Dinitrobenzene

Dissolve 0.5g. of m-Dinitrobenzene in 25ml. of boiling water. To the boiling solution, add yellow ammonium sulphide till the yellow colour is persistent. Boil further for five minutes. Filter while hot. On cooling, yellow needles of *m*-Nitro aniline separates out. Recrystallise from hot water and determine its M.P.

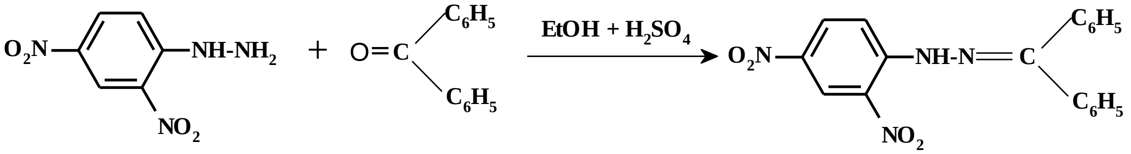


m-Dinitro benzene

m-Nitro aniline

8. 2,4-Dinitrophenylhydrazone derivative from Benzophenone

Take 0.5 g of benzophenone in a dry test tube and dissolve it in few drops of water or ethanol. Add $1cm^3$ of 2,4 – DNP solution. Heat the mixture on water bath for few minutes and cool it in ice. Orange or red crystalline precipitate separates out. Filter, dry and take the melting point.



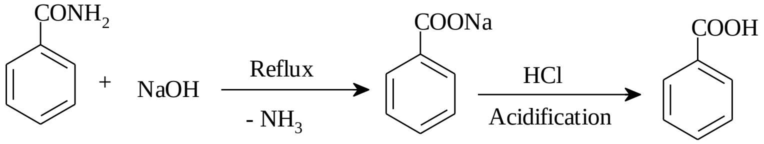
2,4-Dinitro phenyl hydrazine

Benzophenone

Benzophenone-2,4-Dinitro phenyl hydrazone

9. Benzoic acid from Benzamide

Take 0.5g. of benzamide in a 100ml. R.B. flask or conical flask and add 6-7ml. of 25% NaOH solution. The flask is fitted with reflux (air) condenser. Reflux the contents until all ammonia has been driven off (it takes about half an hour) and then cool. Add concentrated hydrochloric acid drop wise till the reaction mixture is strongly acidic and the benzoic acid separates out as a derivative. Filter and recrystallise from hot water. Determine melting point.



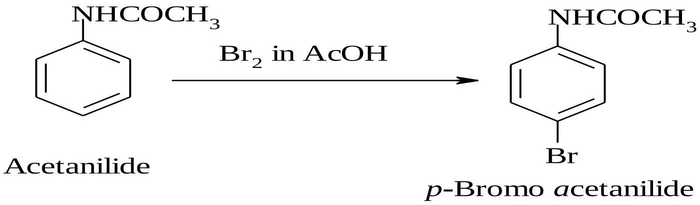
Benzamide

Sodium benzoate

Benzoic acid

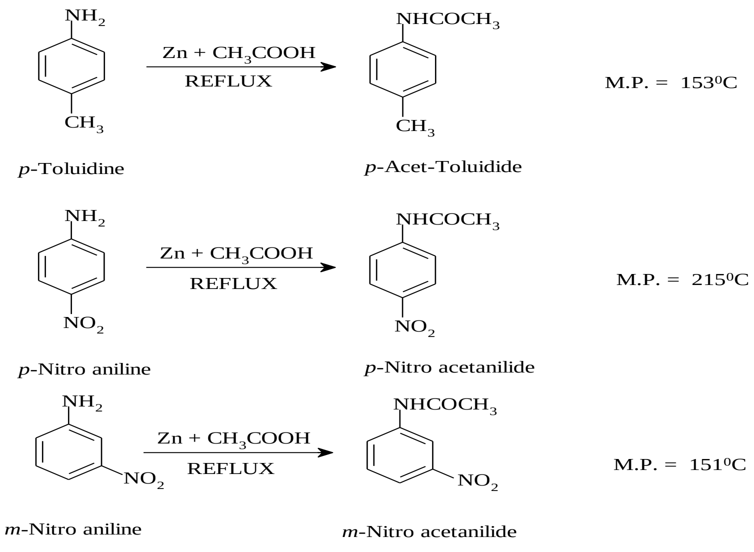
10. *p*-Bromo acetanilide from Acetanilide

1g. of acetanilide is dissolved in 5ml. glacial acetic acid in a 100 or 50ml. conical flask. To this add bromine in acetic acid in small quantities till colour of bromine persists to solution. The mixture is allowed to stand for 10-15 minutes and then poured into ice cold water with constant stirring and filter the product, wash with cold water and recrystallise from 25% ethanol. Determine the melting point.



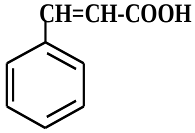
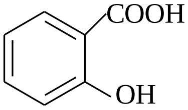
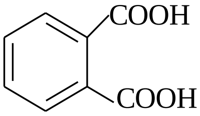
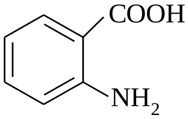
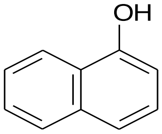
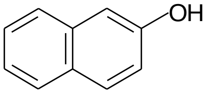
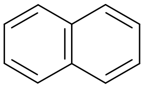
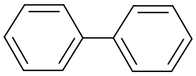
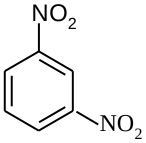
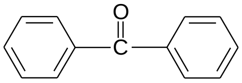
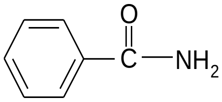
11. Acetyl derivative.

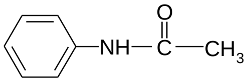
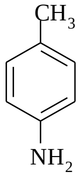
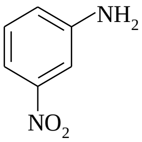
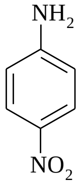
A mixture of *p*-toluidine or *m*-nitroaniline or *p*-nitroaniline (1g) and zinc dust (0.5 g) in acetic acid (5 ml) in a 100 ml round bottom flask was heated over a gentle flame using water condenser. Heating was continued for about 30min. The reaction mixture was then carefully poured in cold water (20 ml) in a 100 ml beaker with cooling and vigorous stirring. The shining crystals of respective anilides were separated slowly. After 15 min. the anilide crystals were collected by filtration. The solid crystals were washed over the Buchner funnel with water and the products dried and take melting point.



Picrate derivative can also be performed for nitro anilines. Procedure is remained same to that of Naphthols.

Name, Structure and M.P. of derivatives of Organic Compounds

Compound	Melting point range (^{0}C)	Molecular Formula	Structural formula	Derivative Melting point
1. Cinnamic acid	133 -134	$C_6H_5CH=CH\text{-}COOH$		2,3 – Dibromo-3- phenyl propionic acid (**194-195^{0}C**)
2. Salicylic acid	157 - 158	$C_6H_4(OH)COOH$		5-Nitro salicylic acid (**230-231^{0}C**)
3. Phthalic acid	193-213	$C_6H_4(COOH)_2$		Phthalic anhydride (**127-128^{0}C**)
4. Anthranilic acid	148-149	$C_6H_4(NH_2)COOH$		Salicylic acid **(157-158^{0}C)**
PHENOLS				
5. α-Naphthol	93 - 94	$C_{10}H_7OH$		Picrate derivative (**189-190^{0}C**)
6. β-Naphthol	121-122	$C_{10}H_7OH$		Picrate derivative (**156-158^{0}C**)
NEUTRALS **1. Hydrocarbons**				
7. Naphthalene	79-80	$C_{10}H_8$		Picrate derivative Naphthalene picrate (**149-151^{0}C**)
8. Diphenyl	70-72	$C_{12}H_{10}$		*p,p'*- Dinitro diphenyl (**233-234^{0}C**)
9. *m*-Dinitrobenzene	89-90	$C_6H_4(NO_2)_2$		*m*-Nitroaniline (**114-115^{0}C**)
2. KETONES				
10. Benzo phenone	48-49	$C_6H_5\text{-}CO\text{-}C_6H_5$		2,4 – Dinitrophenyl hydrazone (**238-239^{0}C**)
3. AMIDES				
11. Benzamide	128-129	$C_6H_5\text{-}CONH_2$		Benzoic acid (**122-123^{0}C**)

4. ANILIDES				
12. Acetanilide	114-115	$C_6H_5NHCOCH_3$		*p*-Bromoacetanilide **(166-167^{0}C)**
BASES				
13. *p*-Toluidine	43-44	$C_6H_4(CH_3)NH_2$		*p*-Acet-toluidide (**153-154^{0}C**)
14. *m*-Nitroaniline	113-114	*o*-$C_6H_4(NO_2)NH_2$		*m*-Nitroacetanilide (**154-155^{0}C**)
15. *p*-Nitroaniline	147-148	*p*-$C_6H_4(NO_2)NH_2$		*p*-Nitroacetanilide (**255-257^{0}C**)

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2. Organic Experiments VIIth Edition Louis F. Fieser Late Professor Emeritus Harvard University Kenneth L Williamson Mount Holyoke College
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5. Advanced Practical Organic Chemistry – N.K.Vishnoi
6. Practical Chemistry -.O.P.Pandey,D.N.Bajpai & S.Giri
7. A hand book of Analytical Chemistry– Subhash & Satish
8. Elementary Practical Chemistry–G.D.Sharma, Arun Bahl
9. Practical Organic Chemistry – V. K. Ahluvalia, Dhingra & Gulati

DISTRIBUTION OF MARKS	
Nature and separation	2 +3
Prelimanary tests	02
Element test	04
Physical constant	03
Functional Group test	04
Identification and Structure	03
Preparation of derivative	03
Physical constant of derivative	03
Systematic Presentation	03
Journal	05
Viva voce	05
TOTAL	**40**

B.Sc V Semester: Paper-II

EXPERIMENTS IN PHYSICAL CHEMISTRY

Total No of Hours/Week : 04 Hours Practical:40 Marks
Total No of Hours : 52 Hours IA :10 Marks

Expt. No	Experiments	Page No.
1	Determination of concentration of HCl solution by conductometric titration using the standard NaOH solution.	163
2	Determination of concentration of CH_3COOH solution by conductometric titration using the standard NaOH solution.	165
3	Determination of dissociation constant of acetic acid conductometrically	167
4	Verification of Beer-Lambert's law by colorimetric method and calculation of molar extinction coefficient of cuprammonium sulphate complex.	169
5	Determination of concentration of strong acid by potentiometric titration against standard solution of 0.1 N NaOH.	171
6	Preparation of buffer solutions using 0.2 M acetic acid and 0.2M sodium acetate with help of Henderson equation and determination of their mole ratio using pH-meter.	173
7	Determination of equivalent conductance of strong electrolyte (NaCl) at infinite dilution (λ_∞).	175
8	Verification of the Beer Lambert's Law by colorimetric method and determination of unknown concentration of ferric (Fe^{3+}) ions.	177
9	To determine conductometrically the 2nd order rate constant for soaponification of ethyl acetate.	179
10	Determination of pH of the following biological Juices. (i)Milk (ii) Orange Juice (iii) Lime water (iv) citrus acid solutions (vi) $NaHCO_3$.	181
11	Determination of critical solution temperature of the partially miscible liquids (Water and Phenol).	183

Scheme of Marking:

Accuracy	= 18
Proper Technique and Presentation	= 03
Calculation (Calculation + Graph)	= 09 (5+4)
Viva voce	= 05
Journal	= 05
Total	**= 40 Marks**

NB: 1. Scientific calculators are not allowed.
2. Use A4 size graph sheets.

Expt No 1: CONDUCTOMETTIC TITARATION (Acid-Base)

Aim: To titrate conductometrically the given solution of HCl (approx 0.1 N) against standard NaOH solution and determine the strength and amount of the acid solution.

Chemicals: 0.5N NaOH and approx 0.1N HCl solution.

Apparatus: Conductivity meter, conductivity cell (1.0 or 0.5 cm) , Micro burette, Pipette etc.

Theory: Conductivity of a solution depends on the mobility as well as number of ions. The H^+ ions have greater mobility than any other ions. When a strong acid is titrated against NaOH, the $[H^+]$ decreases there by conductance decreases till all the H^+ ions are neutralized. Further, addition of NaOH increases the conductance due to a second highest mobile ion, OH^- that is not consumed after neutralization. Hence, for any strong acid against strong base titration, a plot of conductance against volume of alkali gives two straight lines. Intersection of these lines will be the end point or neutralization point.

Reaction: $HCl + NaOH \longrightarrow NaCl + H_2O$

Procedure:

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 25cc of given HCl solution into a 100 cc beaker.
5. Place the conductivity cell and connect to the terminals of the conductivity meter.
6. Add about 20 cc distilled water to the beaker (if the electrodes are not completely immersed) and stir well with glass rod. Note down the conductance of solution in mS.
7. Rise up the electrodes and add 0.5cc of the NaOH solution from micro burette carefully and stir the solution. There may be a slight heating effect due to neutralization and hence, wait for 30 seconds to cool. Note down the conductance.
8. Continue the titration by adding 0.5cc at a lot up to 10cc and record the conductance for every addition.
9. Plot a graph of conductance against volume of NaOH added. Find out the end point from the intersection of two lines. Calculate the normality and amount of HCl.

Observations:

Volume of HCl solution taken =25.0cc

Volume of NaOH added	Observed conductance (mS)
0.0 cc	
0.5 cc	
1.0 cc	
..	
..	
10.0 cc	

Calculations:

i. Normality of acid solution : $N_1 V_1 = N_2 V_2$

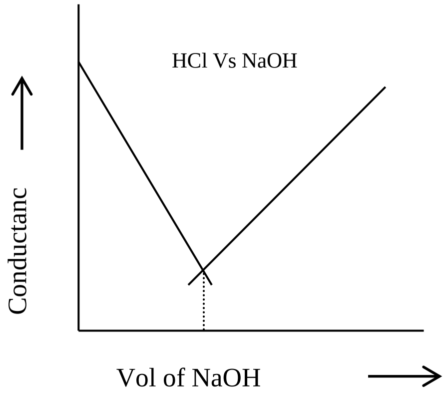
$$N_{HCl} = \frac{N_{NaOH} \text{ x End point from graph}}{V_{HCl}}$$

= …………..N

ii. Amount of HCl = N_{HCl} x equivalent mass of HCl

= ……………g/dm^3

Nature of the Graph



Results:-

1. Normality of HCl =………….N
2. Amount of HCl =………… g/dm^3

Note: 1. Preserve the conductivity cell always in distilled water

Expt No 2: CONDUCTOMETTIC TITARATION (Acid-Base)

Aim: To titrate conductometrically the given solution of CH_3COOH (approx 0.1 N) against standard NaOH solution and determine the strength and amount of the acid solution.

Chemicals: 0.5N NaOH, approx. 0.1N CH_3COOH solution.

Apparatus: Conductivity meter, conductivity cell (1.0 or 0.5 cm) , Micro burette, Pipette etc.

Theory: Conductivity of a solution depends on the mobility as well as number of ions. The H^+ ions have greater mobility than any other ions. When a strong acid is titrated against NaOH, the $[H^+]$ decreases there by conductance decreases till all the H^+ ions are neutralized. Further addition of NaOH increases the conductance due to a second highest mobile ion, OH^- that is not consumed after neutralization.

In case of weak acid like CH_3COOH, the free H^+ ions are not there in sufficient numbers. Hence, the addition of NaOH increases the conductance gradually due to formation of CH_3COONa till neutralization and further addition of NaOH leads to a rapid increase in conductance due to unused OH^- ions

Hence, for any weak acid against strong base titration, a plot of conductance against volume of alkali gives two straight lines. Intersection of these lines will be the end point or neutralization point.

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

Procedure:

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 25cc of given CH_3COOH solution into a 100 cc beaker.
5. Place the conductivity cell and connect to the terminals of the conductivity meter.
6. Add about 20 cc distilled water to the beaker (if the electrodes are not completely immersed) and stir well with glass rod. Note down the conductance of solution in mS.
7. Rise up the electrodes and add 0.5cc of the NaOH solution from micro burette carefully and stir the solution. There may be a slight heating effect due to neutralization and hence, wait for 30 seconds to cool. Note down the conductance.
8. Continue the titration by adding 0.5cc at a lot up to 10cc and record the conductance for every addition.

9. Plot a graph of conductance against volume of NaOH added. Find out the end point from the intersection of two lines. Calculate the normality and amount of acid.

Observations:

Volume of CH_3COOH solution taken =25.0cc

Volume of NaOH added	Observed conductance (mS)
0.0 cc	
0.5cc	
1.0 cc	
..	
..	
10.0 cc	

Calculation:

i. Normality of acid solution: $N_1 V_1 = N_2 V_2$

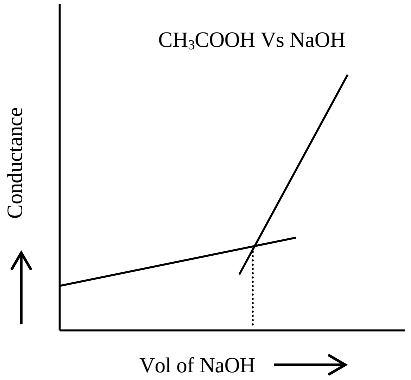
$$N_{CH3COOH} = \frac{N_{NaOH} \times \text{End point from graph}}{V_{CH3COOH}}$$

= …………….N

ii. Amount of CH_3COOH = $N_{CH3COOH}$ x equivalent mass of CH_3COOH

= ………… g /dm^3

Nature of the Graph



Results:-

1. Normality of CH_3COOH =…………….N
2. Amount of CH_3COOH =……………. g /dm^3

Note: 1. Preserve the conductivity cell always in distilled water

Expt No. 3 CONDUCTOMETERY ($K_{a\,HAC}$)

AIM: To determine the dissociation constant K_a of acetic acid conductometrically.

Chemicals: 0.1N KCl, 0.1N CH_3COOH solution

Apparatus: Conductivity meter, conductivity cell (1.0 cm), beaker, glass rod etc.

Theory: Since acetic acid is weak electrolyte, it partially ionizes in aqueous solution. Hence, acetic acid solution shows a low conductance. As dilution increases, specific conductance of CH_3COOH decreases, but both equivalent conductance and degree of dissociation (α) will increase with dilution. According to Ostwald's dilution law, the degree of dissociation (α) of weak electrolyte is inversely proportional to square root of initial molar concentration of electrolyte. For strong electrolytes, α is almost equal to 1, but in case of weak electrolytes α is less than 1. However, dissociation constant of CH_3COOH (K_a) always remains constant for any dilution.

Procedure:

A) Determination of cell constant :

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 50cc of 0.1N KCl solution in to 100cc beaker.
5. Place the conductivity cell in the beaker and connect it to the terminals of conductivity meter.
6. Note down the conductance of the solution in mS.
7. Calculate the cell constant.

B) Determination of equivalent conductivities:

1. Pipette out 50cc of 0.1N CH_3COOH solution into 100cc clean beaker containing the conductivity cell and note down the conductance in mS.
2. Dilute this solution to 0.05N by withdrawing 25 cc of the above solution and adding 25cc distilled water with pipette, stir well and note down the conductance in mS.
3. Similarly, dilute the above solution to 0.025 N and 0.0125N, and record the conductance for every dilution.
4. Calculate the specific conductance, equivalent conductance, degree of ionization and dissociation constant K_a.

Observations:

A) Determination of cell constant :

1. Observed conductance of 0.1N KCl soln. = ………x 10^{-3} S.
2. Specific conductance of 0.1N KCl soln. at room temperature = 0.01288 S cm^{-1}
3. Determination of cell constant:

$$\text{Cell constant} = \frac{\text{Specific conductance of 0.1N KCl}}{\text{Observed conductance}}$$

$$= \frac{0.01288}{\text{Observed conductance}}$$

$$= \text{-------------}\ cm^{-1}$$

B) Determination of equivalent conductivities:

The equivalent conductance of CH_3COOH at infinite dilution ie **λ_∞ =387Scm^{2}eqv**$^{-1}$

Concentration of solution (C)	Observed conductance (S)	Specific conductance, κ = Cell constant × Observed conductance (Scm^{-1})	Equivalent conductance $\lambda_c = \frac{1000 \times \kappa}{C}$ (Scm^2eqv^{-1})	Degree of dissociation $\acute{\alpha} = \lambda_c / \lambda_\infty$	$K_a = \frac{C.\acute{\alpha}^2}{(1-\acute{\alpha})}$
0.1 N	x 10^{-3}				
0.05N	x 10^{-3}				
0.025N	x 10^{-3}				
0.0125N	x 10^{-3}				
	Average of K_a				

Result:

The average value of Ka of CH_3COOH =…………

Conclusion

1. As dilution increases equivalent conductance increases.
2. As dilution increases degree of dissociation increases.
3. As dilution increases magnitude of dissociation constant remains same.

Note: 1. The theoretical value of $Ka_{CH3COOH}$ = 1.8 x 10^{-5} mol /dm^3

2. Preserve the conductivity cell always in distilled water

Expt. No.4 COLORIMETRY

Aim: To Verify the Beer-Lambert's law by colorimetric method and calculate the molar extinction coefficient of Cu^{+2} in cuprammonium sulphate complex.

Chemicals: solid $CuSO_4$, ammonia and Con.H_2SO_4.

Apparatus: Colorimeter, cells, test tubes, test tube stand etc.

Theory: Suppose an intensity of light absorption of a colored solution at a suitable wavelength for various known concentration is determined, using Beer-Lambert's law the unknown concentration of same solution can be determined by measuring absorption at the same wavelength.

Beer-Lambert's states that when beam of light is passed through a coloured solution, decrease in intensity of transmitted light is directly proportional to thickness as well as path length of the solution.

Mathematically, Beer-Lambert's law can be written as

$$I = I_o.e^{-kCd}$$

$$\ln I_o/I = k\,C\,d$$

where,

I_o = Intensity of the incident light
I = Intensity of transmitted light
C= concentration of a solution
d = thickness of a solution
k = absorption coefficient

or $$\log I_o/I = \frac{k.\,C.d}{2.303}$$ or $$OD = \frac{k.\,C.d}{2.303} = \text{Optical Density}$$

or $$OD = \varepsilon\, Cd$$

where, OD = Optical Density, i.e., amount of light absorbed by solution of known concentration& known thickness.

$$\varepsilon = \frac{k.}{2.303}$$ = molar extinction coefficient (constant) when 'C' in mol /dm^3

This principle is extensively used in colorimetric estimation. When the thickness of a solution is 1 cm, a slope from the plot of OD Vs Concentration gives ε. The ε of Cu^{2+} in a colored complex can be determined calorimetrically.

Reaction: The complex between Cu^{2+} & NH_3 is formed as follows:

$$Cu^{2+} + 4\,NH_3 \rightleftharpoons \underset{\text{Cuprammonium complex ion}}{[Cu(NH_3)_4]^{2+}}$$

Procedure:

1. Prepare 0.01 M $CuSO_4$ solution in 100cc volumetric flask by weighing out 250mg and 2M NH_4OH solution by measuring out 13.7 cc NH_3 from measuring cylinder to 100 cc volumetric flasks as explained in observation.
2. Prepare cuprammonium sulphate complex solution of different concentrations in 08 separate test tubes by adding required quantity of 0.01N $CuSO_4$, 2M NH_4OH and distilled water as given in table 2.
3. **Selection of suitable filter**: Inserting the cell containing solution No.1 in the colorimeter, adjust the OD to zero. Now using solution No. 5 of moderate concentration, observe the OD for a filter having lower wave length.

4. Similarly, observe the OD of the same solution for remaining filters of higher wave lengths by setting OD zero to solution No. 1 for every filter. The filter which gives the maximum OD is suitable one.
5. **Determination of OD of solutions**: Using the suitable filter selected, find out the OD for all the prepared solutions.
6. Plot a graph of OD against concentration, which gives a straight line passing through the origin. This is the verification of Beer-Lambert's law.
7. From the slope of graph, calculate the molar extinction coefficient of complex.

Observations:

1. **Preparation of 0.01M$CuSO_4$:**
   250 mg of $CuSO_4.5H_2O$ is dissolved in few cc of distilled water and taken into 100cc volumetric flask. Two to three drops of Conc. H_2SO_4 are added to prevent the hydrolysis. Then it is diluted to 100cc which gives 0.01M $CuSO_4$.
2. **Preparation of 2M NH_4OH:**
   13.7 cc of liquor ammonia is measured out with measuring cylinder and diluted to 100cc with distilled water in a volumetric flask.

Table No.1 : Selection of Suitable filter

Solution No. 04

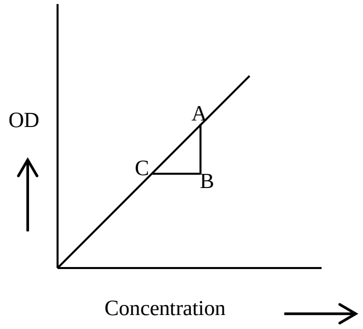
Filter No.(Wavelength)									
OD									

Remark: The Filter No. ____ gives the maximum OD. Hence it is selected as suitable filter.

Table No.2 : Determination of OD of Solutions

Sol. No.	Vol. of $CuSO_4$ Soln.(cc)	Vol. of distilled water (cc)	Vol. of NH_4OH (cc)	Concentration of Cu^{2+} in mol/dm^3	OD
1	0.0	8.0	2.0	0.000	
2	1.0	7.0	2.0	0.001	
3	2.0	6.0	2.0	0.002	
4	3.0	5.0	2.0	0.003	
5	4.0	4.0	2.0	0.004	
6	5.0	3.0	2.0	0.005	
7	6.0	2.0	2.0	0.006	
8	7.0	1.0	2.0	0.007	
9	8.0	0.0	2.0	0.008	

Nature of the graph



$$\text{Slope} = \frac{AB}{BC} = \varepsilon l \quad \text{where } l = \text{length(constant=1cm)}$$

$\therefore\ \varepsilon = \text{Slope}$

Result: ε=……dm^3mol^{-1} cm^{-1} at……..nm wavelength

Expt No.5 POTENTIOMETRIC TITRATION (Acid-Base)

Aim: To determine the concentration of strong acid (HCl) by potentiometric titration against standard solution of 0.1N NaOH solution.

Chemicals: 0.1N NaOH, approx 0.1N HCl, Quinhydrone. Saturated solution of KCl etc.

Apparatus: Potentiometer, calomel electrode, platinum electrode etc.

Theory: If HCl is titrated against NaOH potentiometrically, one should select an electrode reversible to hydrogen ions. Quinhydrone is one such electrode and when an inert electrode like platinum is inserted in its solution, a potential develops. It can be given as:

$E_{Qin} = E^o + 0.0591 \log [H^+]$ at 25°C.

When this is connected to reference electrode like calomel, a cell is set up. The EMF of the cell depends on $[H^+]$. On adding small quantities of NaOH, the EMF will change slowly first and rapidly at the end point and again becomes slow after crossing the end point. The graph of EMF v/s volume of NaOH added gives a reverse sigmoid curve passing through X-axis, which is the equivalence point. The differential plot of $\Delta E/\Delta V$ vs. volume of NaOH added also gives a sharp end point

Procedure:

1. Pipette out 25cc of given HCl solution in 100cc beaker and add a pinch of solid quinhydrone and stir the solution with a glass rod. Keep it for a while.
2. Fill the burette with exact 0.1N NaOH solution.
3. Switch on the potentiometer and standardize the potentiometer by inserting two banana plugs into the sockets at channel I or II and adjust the EMF to 1.018V with the calibration screw. Remove these plugs after standardization.
4. Place the platinum electrode in the above quinhydrone solution and calomel electrode in another beaker having about 50 cc saturated KCl solution. Connect the solutions internally through KCl salt bridge and electrodes externally to the terminals of Potentiometers at either channel I or II at which it is standardized i.e., calomel electrode to negative and platinum electrode to positive terminals of the potentiometer. Record the EMF at 0.0 volume.
5. Start titration by adding 1.0cc of 0.1N NaOH at a lot with constant stirring and record the EMF every addition. Meanwhile, observe the rapid change in EMF at certain volume of NaOH where there will be an equivalence point (approximate) and EMF will be in –ve value. Continue the additions for another 04 readings after –ve EMF are obtained.
6. Plot a graph of EMF v/s volume of NaOH added which gives a reverse sigmoid curve and $\Delta E/\Delta V$ v/s volume of NaOH added gives a peak which is the exact point of equivalence.

Observations

a) Representation of cell

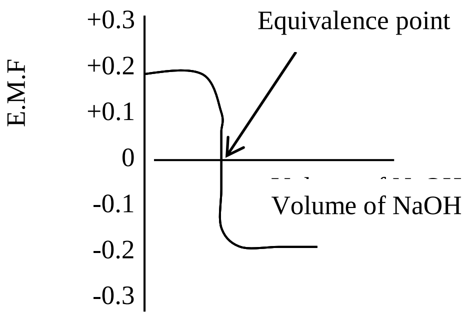
$$\overset{-}{Hg} \;/\; Hg^{+} \;//\; H^{+} \;/\; \text{Quinhydrone} / \overset{+}{Pt}$$

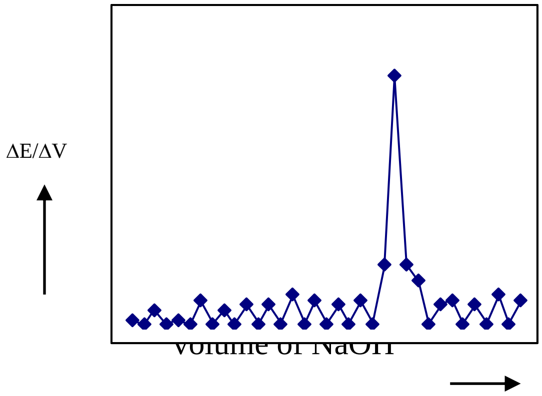
Table: Volume of HCl taken =25.0cc

Volume of 0.1N NaOH (V cc)	EMF (E) in Volts	ΔV	ΔE	$\Delta E/\Delta V$
0.0				
1.0				
2.0				
.				
.				
.				
-				
-				

Nature of the graph (graph-I)

Differential curve (graph-II)





Calculation

From graph-I

1. $$N_{HCl} = \frac{N_{NaOH}\ X \text{ equivalence point from graph}}{V_{HCl}} = \frac{0.1 \ \ x \text{ equivalence point}}{25}$$

2.Amount of HCl **=** N_{HCl} x equivalent mass of HCl=……….g / dm^3

From graph-II

1. $$N_{HCl} = \frac{N_{NaOH}X \text{ equivalence point from graph}}{V_{HCl}} = \frac{0.1 \ x \text{ equivalence point}}{25}$$

2. Amount of HCl =N_{HCl} x equivalent mass of HCl= ………. g / dm^3

Result:

1. The concentration of the given HCl solution = graph-I………& graph-II ……N
2. Amount of HCl = graph-I………& graph-II …… g / dm^3

Expt.No.6 pH metry (Buffer solutions)

Aim: To prepare buffer solutions using 0.2M acetic acid and 0.2M sodium acetate with help of Henderson equation and determine the mole ratio of solution of unknown pH using pH-meter.

Chemicals: 0.2N acetic acid, 0.2N sodium acetate

Apparatus: 12 test tubes, gradated pipette, pH meter, glass electrode etc

Theory: A solution whose pH does not practically change on addition of small amounts of acids or alkalis is called as a buffer solution. Buffer capacity means, the amount in gram equivalent of an acid or base which is to be added in order to change the pH of buffer solution by unity.

A buffer solution generally consists of a mixture of a weak acid and one of its salt solution or a mixture of a weak base and one of its salt solution. The pH of such buffer solution remains practically constant by addition of small amounts of acids or base because it is controlled by chemical equilibrium. The buffer solutions of required pH can be prepared by using Henderson –Hasselbalch equation:

$pH = pK_a + \log [salt]/[acid]$ for acidic pH solution.

The pH meters are used to determine the pH of solution using glass electrode.

Procedure:

1. Prepare the standard solutions of 0.2M acetic acid and 0.2M sodium acetate in 100cc volumetric flasks.
2. Arrange 11 clean test tubes (20cc capacity) and label them from 1 to 11.
3. Calculate the volume of 0.2M acetic acid and 0.2M sodium acetate solutions required to prepare buffer solution of given pH in 10cc using Henderson equation. Record such calculated volumes in table.
4. Add required volume of 0.2M acetic acid and 0.2M sodium acetate solutions to each test tube as calculated above. The total volume should be 10 cc.
5. Standardize the pH meter as mentioned on the instrument. (May be using the standard solutions of known (minimum) pH=4.0 and known (maximum) pH=9.2 with the help of glass electrode or with the adjustment by screw driver).
6. 10cc of each buffer solution is taken in another big test tube separately in which the glass electrode is dipped and later on connected to the pH meter.
7. Record the pH of these buffer solutions.
8. Measure the pH of unknown composition of buffer solution.
9. Calculate the mole ratio of the unknown composition.

Preparation of standard solution

1. **0.2 M Acetic acid solution (normality of glacial acetic acid in bottle sample = 17)**
   Measure out 1.2 cc glacial acetic acid to 100 cc volumetric flask and dilute up to the mark with distilled water, which gives 0.2 M acetic acid solution.
2. **0.2 M sodium acetate (Mol. Mass = 82)**
   Dissolve 1.64 g of sodium acetate in water and transfer to 100 cc volumetric flask , and dilute up to the mark with distilled water, which gives 0.2 M sodium acetate solution.

Observations

Sl. No. of test tubes	Required pH	Vol. of 0.2M acetic acid (cc)	Vol.of 0.2M Sodium acetate (cc)	Total Volume (cc)	Observed pH
1	3.45	9.5	0.5	10.0	
2	3.75			10.0	
3	4.14			10.0	
4	4.38			10.0	
5	4.57			10.0	
6	4.74	5.0	5.0	10.0	
7	4.92			10.0	
8	5.11			10.0	
9	5.35			10.0	
10	5.70			10.0	
11	6.02	0.5	9.5	10.0	
Unknown				10.0	

1. **Calculation of volume of acid and salt solution to be mixed to prepare a solution of required pH**.
   Volume of acid = (proportionate of acid/ total volume ratio) x 10 cc
   Similarly, Volume of salt = (proportionate of salt / total volume ratio) x 10 cc
2. **Ex., Required pH = 6.02 (When pH > pK_a)**

pH = pK_a + log [salt]/ [acid] K_a of acetic acid =1.8×10^{-5}
6.02 = 4.7447 + log [salt]/ [acid]
6.02- 4.7447 = log [salt]/ [acid]
1.2753 = log [salt]/ [acid]
Anti log(1.2753) = [salt] / [acid] = Volume of salt solution / Volume of acid solution
18.8495 = Volume of salt solution / Volume of acid solution
i.e., 18.8495 = proportionate of salt solution
i.e., 1.000 = proportionate of acid solution
19.8495 = Total volume ratio
Volume of salt solution = (18.8495 /19.8495) x 10 cc = 9.5 cc
Similarly, Volume of acid solution = (1.00 / 19.8495) x 10 cc = 0.5 cc

3. **Ex., Required pH = 3.45 (When pH < pK_a)**

, pH = pK_a + log [salt]/ [acid] K_a of acetic acid =1.8×10^{-5}
3.45 = 4.7447 + log [salt]/ [acid]
3.45 - 4.7447 = log [salt]/ [acid]
-1.2947 = log [salt]/ [acid]
Anti log(-1.2947) = [salt]/ [acid] = Volume of salt solution /Volume of acid solution
0.05073 = Volume of salt solution /Volume of acid solution
i.e., (1/0.05073) = **19.710 = Volume of acid solution /Volume of salt solution**
i.e., **19.710 =** proportionate of acid solution
1.00 = proportionate of salt solution
20.710 = Total volume ratio
Volume of salt solution = (1.00 / **20.710**) x 10 cc = 0.5 cc
Similarly, Volume of acid solution = (**19.710** / **20.710**) x 10 cc = 9.5 cc

4. **Calculation of mole ratio of unknown composition:**

pH= pK_a + log [salt]/ [acid] K_a of acetic acid =1.8×10^{-5}
log [salt] / [acid] = (pH - pK_a)
[salt] / [acid] = anti log (pH - pK_a)

When Concentration of Acid is equal to that of salt, mole ratio can be considered as volume ratio.

[salt] / [acid] = Volume of salt solution /Volume of acid solution

Result: The pH of the unknown composition solution = ……………
Mole ratio of unknown composition solution =…………….

Expt. No.7 CONDUCTOMETERY (Infinite dilution)

Aim: To determine the equivalent conductance of strong electrolyte (NaCl) and equivalent conductance at infinite dilution ($\lambda\infty$).

Chemicals: 0.1M KCl, 0.2N NaCl solution

Apparatus: Conductivity meter, conductivity cell(1.0 cm), Beaker (100cc), Volumetric flask etc.

Theory: The equivalent conductance of the strong electrolytes approaches a limiting value as concentration is diminished. This quantity is called the equivalent conductance at "infinite dilution" and represented by λ_∞ wherein inter ionic interaction is negligible. It is presumably a measure of the conducting power of 1 gm equivalent of the electrolyte when it is completely split up into ions in the solution. The determination of λ_∞ involves an extrapolation of the measured equivalent conductance in dilute solution to zero concentration. This is one of the best methods of Kohlrausch law.

Procedure:

A) Determination of cell constant :

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 50cc of 0.1N KCl solution in to 100cc beaker.
5. Place the conductivity cell in the beaker and connect it to the terminals of conductivity meter.
6. Note down the conductance of the solution in mS.
7. Calculate the cell constant.

B) Determination of equivalent conductivities:

1. Pipette out 50cc of 0.2N NaCl solution into 100cc clean beaker. Place the conductivity cell and note down the conductance in mS.
2. Dilute this solution to 0.1N by withdrawing 25 cc of the above solution and adding 25cc distilled water with pipette, stir well and note down the conductance in mS.
3. Similarly, dilute the above solution to 0.05 N, 0.025 N and 0.0125N, and record the conductance for every dilution.
4. Calculate the specific conductance, equivalent conductance, degree of ionization and dissociation constant K_a.
5. Plot a graph of λ_c against $\sqrt{C}$ which gives a straight line. Intercept of the straight line gives a value of λ_∞.

Observations:

A) Determination of cell constant :

1. Observed conductance of 0.1N KCl soln. = ………$x 10^{-3}$ S.

2. Specific conductance of 0.1N KCl soln. at room temperature = 0.01288 S cm^{-1}

3. Determination of cell constant:

$$\text{Cell constant} = \frac{\text{Specific conductance of 0.1N KCl}}{\text{Observed conductance}}$$

$$= \frac{0.01288}{\text{Observed conductance}}$$

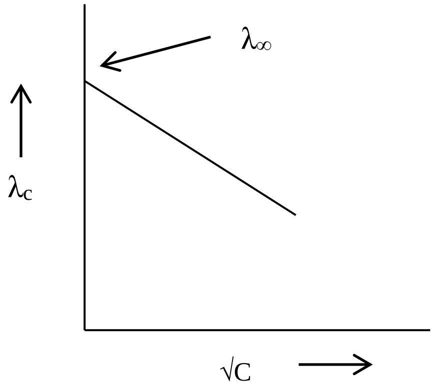
= **-------------** cm^{-1}

B). Determination of equivalent conductivities: (λ_c)

Concentration of the solution , C	Observed conductance (S)	Specific conductance κ=cell constant × Observed conductance (S cm^{-1})	Equivalent conductance $\lambda_c = \frac{1000 \times \kappa}{C}$ (Scm^2eqv^{-1})	$\sqrt{C}$
0.2N	$x 10^{-3}$			
0.1N	$x 10^{-3}$			
0.05N	$x 10^{-3}$			
0.025N	$x 10^{-3}$			
0.0125N	$x 10^{-3}$			

1) **Determination of equivalent conductivity at infinite dilution** (λ_∞)

Plot the graph of λ_c v/s $\sqrt{C}$, it gives a straight line



The intercept of the straight line, λ_∞ =…… Scm^2eqv^{-1}

Result:

The equivalent conductance of NaCl at infinite dilution $\lambda_{\infty NaCl}$ =……. Scm^2eqv^{-1}

Note: Theoretical value of λ_∞ for NaCl = 126.7 Scm^2eqv^{-1}

Expt No.8 COLORIMETRY

Aim: Verification of the Beer Lambert's Law by colorimetric method and determination of unknown concentration of ferric (Fe^{3+}) ions.

Chemicals: 0.001M $Fe_2(SO_4)_3\ (NH_4)_2\ SO_4.24H_2O$(Ferric alum) and 2% KCNS,

Apparatus: Colorimeter, cells, test tubes, test tube stand etc.

Theory: Suppose an intensity of light absorption of a colored solution at a suitable wavelength for various known concentration is determined, using Beer-Lambert's law the unknown concentration of same solution can be determined by measuring absorption at the same wavelength.

Beer-Lambert's states that when beam of light is passed through a coloured solution, decrease in intensity of transmitted light is directly proportional to thickness as well as path length of the solution

Mathematically, Beer-Lambert's law can be written as

$$I = I_o.e^{-kCd}$$

$$\ln I_o/I = k\,C\,d$$

where,
I_o = Intensity of the incident light
I = Intensity of transmitted light
C= concentration of a solution
d = thickness of a solution
k = absorption coefficient

$$\text{or} \quad \log I_o/I = \frac{k.\,C.d}{2.303} \quad \text{or} \quad OD = \frac{k.\,C.d}{2.303} = \text{Optical Density}$$

$$\text{or} \quad OD = \varepsilon\, Cd$$

where, OD = Optical Density, i.e., amount of light absorbed by solution of known concentration& known thickness.

$\varepsilon = \frac{k.}{2.303}$ = molar extinction coefficient (constant) when 'C' in mol dm^3

This principle is extensively used in colorimetric estimation. When the thickness of a solution is 1 cm, a slope from the plot of OD Vs Concentration gives ε. The [Fe^{3+}] in a colored complex can be estimated colorimetrically.

Reaction: The complex between Fe^{3+} & CNS^- ions are formed as follows:

$$Fe^{3+} + nCNS^- \rightleftharpoons [\,Fe(CNS)]^{(3-n)+}$$

Ferric thiocyanate complex ion

Procedure:

1. Prepare a stock solution of 0.0001M ferric alum solution by diluting10 cc of given 0.001M Ferric alum solution in to 100 cc.
2. Prepare ferric thiocyanate complex solutions of different concentrations in 08 separate test tubes by adding required volume of 0.0001M ferric alum solution , 2% KCNS solution and distilled water as given in table 2.
3. **Selection of suitable filter**: Inserting the cell containing solution No.1 in the colorimeter, adjust the OD to zero. Now using solution No. 5 of moderate concentration, observe the OD for a filter having a lower wave length.

4. Similarly, observe the OD of the same solution for remaining filters of higher wave lengths by setting OD zero to solution No. 1 for every filter. The filter which gives the maximum OD is suitable one.
5. **Determination of OD of solutions**: Using the suitable filter selected, find out the OD for all the prepared solutions.
6. Plot a graph of OD against concentration of Fe^{3+}, which gives a straight line passing through the origin. This is the verification of Beer-Lambert's law.
7. Determine the concentration of Fe^{3+} in unknown composition.
8. From the slope of graph, calculate the molar extinction coefficient of complex.

Observations:

Table No.1 Selection of filter using solution no 4

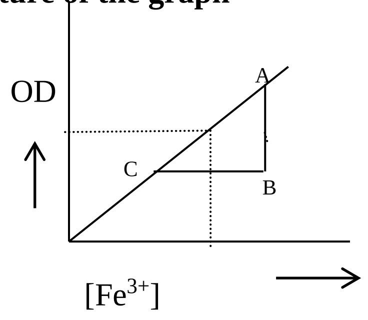
Filter No							
OD							

Remarks: The Filter no.-------nm gives maximum OD. Hence it is selected as suitable filter.

Table No.2 Determination of OD of Solutions

Sl.No.	0.0001M Ferric alum (cc)	Distilled water (cc)	2% KCNS	Concentration of Fe^{3+} in mol/dm^3	OD
1	0.0	8.0	2.0	0.00000	
2	1.0	7.0	2.0	0.00001	
3	2.0	6.0	2.0	0.00002	
4	3.0	5.0	2.0	0.00003	
5	4.0	4.0	2.0	0.00004	
6	5.0	3.0	2.0	0.00005	
7	6.0	2.0	2.0	0.00006	
8	7.0	1.0	2.0	0.00007	
9	8.0	0.0	2.0	0.00008	
10	Unknown composition				

Nature of the graph



Calculation of Molar extinction coefficient:

$$\varepsilon = \frac{\text{Slope}}{\text{length}} = \frac{\text{Slope}}{1} = \frac{AB}{BC} = \text{……..} \quad dm^3\,mol^{-1}\,cm^{-1}$$

Because, l (length) = 1 cm

Result: Concentration of Fe^{3+}=----------- mol/dm^3

Expt No.9 SAPONIFICATION OF ETHYL ACETATE - SECOND ORDER REACTION

Aim: To determine the second order rate constant for hydrolysis of ethyl acetate by sodium hydroxide conductometrically.

Chemicals: 0.1M Ethyl acetate, 0.1M NaOH and 0.1N CH_3COONa solution.

Apparatus: Conductometer, conductivity cell, beaker, glass rod, burette, pipette etc

Theory : Hydrolysis of ethyl acetate by NaOH is a second order reaction as order with respect to each reactant is one.

$$CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH$$

$$\text{Rate} = k\ [CH_3COOC_2H_5]\ [NaOH]$$

When both the concentrations are equal (a=b), the 'k' can be calculated by using integrated rate equation:

$$\boldsymbol{k} = \frac{1}{t}\,\frac{x}{a(a-x)} \quad \text{dm}^3\ \text{mol}^{-1}\ \text{min}^{-1}$$

Where, 'x' is concentration of reactant disappeared at a given time, t.

'a-x' is concentration of reactant remained at a given time, t.

When a known volume of above reaction mixture is titrated against standard acid, the volume of acid consumed at t= 0 is 'a' and volume at definite interval of time, t is 'a-x'. Hence, x = a - (a-x). Since the reaction is second order, the 'k' for all the interval of time should be constant.

Progress of the reaction can also be followed conductometrically. During reaction, fast moving OH^- ions are replaced by slow moving CH_3COO^- ion, so that the overall conductance of the solution will decrease progressively. Hence, by measuring decrease in the conductance of NaOH at regular interval of time, the velocity constant can be determined.

i.e. $a \ \alpha \ (C_o-C_\infty)$

$x \ \alpha \ (C_o-C_t)$

$a-x \ \alpha \ (C_t-C_\infty)$

$$k = \frac{1}{t}\,\frac{(C_o-C_t)}{a\ (C_t-C_\infty)} \quad \text{dm}^3\ \text{mol}^{-1}\ \text{min}^{-1}$$

where, C_o, C_t and C_∞ are conductance of the solution at the beginning, after time 't' and after completion of the reaction 't_∞' respectively.

Procedure: 20 cc of 0.1M NaOH + 20 cc of 0.1M $CH_3COOC_2H_5$ + 40 cc water

1. Pipette out 20 cc of 0.1M NaOH and 20 cc of 0.1M $CH_3COOC_2H_5$ in two separate clean and dry conical flasks. Add 40cc water to NaOH solution, stopper both the flasks and keep them in water bath to attain room temperature.
2. Keep the conductivity cell in 100 cc beaker, and connect to the terminals of the conductometer.
3. When the content attains room temperature, pour NaOH and $CH_3COOC_2H_5$ solutions to the above 100cc beaker, stir it properly and note down the conductance immediately by **starting the stopwatch simultaneously**. This is zero minute conductance (C_o).
4. Note down the conductance of the reaction mixture at every 1 minutes till 10 minutes and thereafter for 15, 20, 25, 30, 35 , 40 and 45 minutes (C_t).

5. Measure the conductance of the reaction mixture after 2 hrs from mixing time, (it completes in 2hrs) which gives C_∞ or alternatively find out the conductance of 0.025M CH_3COONa which also gives C_∞.
6. Calculate the second order velocity constant, '***k***' using a suitable equation.
7. Plot a graph of (C_o-C_t) / (C_t-C_∞) against time, which gives a straight line, the slope of the straight line is '***k a***'.

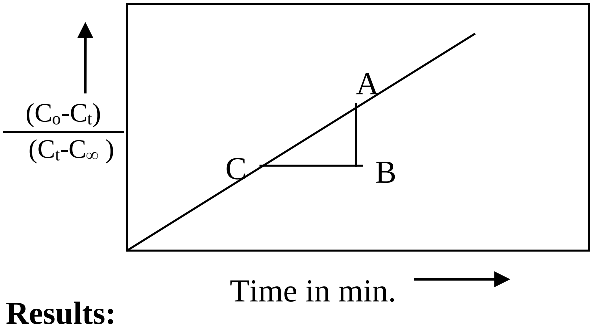
Observation : **Reaction mixture:** 20 cc 0.1M NaOH + 20 cc 0.1M $CH_3COOC_2H_5$ + 40cc water

Time in minute	Conductance in mS (C)	(C_o-C_t)	(C_t-C_∞)	$\frac{(C_o-C_t)}{(C_t-C_\infty)}$	$k = \frac{1}{t\,a}\frac{(C_o-C_t)}{(C_t-C_\infty)}$ $dm^3\ mol^{-1}\ min^{-1}$
0	C_o=	----	-----	-----	------
1	C_t=				
2					
3					
.					
,					
,					
10					
15					
20					
25					
30					
35					
40					
45					
2hrs	C_∞=				
				Average 'k'=	

Calculations:

1. Calculation of initial concentration $a = \frac{M_{NaOH} \times V_{NaOH}}{\text{Total volume}} = \frac{\mathbf{0.1 \times 20}}{\mathbf{80}} = 0.025 mol\ dm^{-3}$
2. Calculation of velocity constant $\mathbf{k} = \frac{\mathbf{1}}{\mathbf{t\,a}}\frac{(C_o-C_t)}{(C_t-C_\infty)}\ dm^3\ mol^{-1}\ min^{-1}$

Nature of the graph:



Slope = AB/BC

k = slope / a

k = slope / 0.025

Results:

1. Mean value of ***k*** by formula method = …………………… $dm^3\ mol^{-1}\ min^{-1}$
2. Value of ***k*** by graphical method =…………………… $dm^3\ mol^{-1}\ min^{-1}$

Expt. 10 pH metry (pH of biological juice)

Aim: To determine the pH of the following biological juices

i) Milk ii) Orange juice iii) Lime water iv) Citric acid v) $NaHCO_3$

Chemicals: Raw milk, Orange fruits, Lime, Citrus fruits, Baking soda

Apparatus: pH meter, glass electrode etc.

Theory: Biological juices like milk and fruit juices will be in good quality only when they have desirable pH values. They may spoil due to change in their pH values. Milk is one of the deliberately flavored, easily changed foods. It is an excellent culture medium for many kinds of microorganisms, being high in moisture nearly neutral in pH. When milk sours, it is usually considered to be as spoiled. On proteolysis (hydrolysis of proteins) milk may turn up into acidic or alkaline in nature. The spoilage of milk and thus change in its pH value occurs due to the microbial action of bacteria like lactostreptococci and micrococci, thermodurics etc. Low temperature always favors to maintain good quality of milk i.e below 7.2^{o}C.

Further, in fruit juices under normal course of changes an alcoholic fermentation occurs at ordinary temperature. It leads to change in their pH value followed by spoilage. Low temperatures help to maintain the desirable pH and thus minimize the spoilage.

The dissolution of **Quick lime** or Lime (CaO) in water is regarded as highly exothermic in nature. Its cold and filtered clear solution [$Ca(OH)_2$] i.e lime water shows alkalinilty.

Similarly, the **Baking soda** ($NaHCO_3$) is lass soluble in water. Its solution shows the alkalinity. pH of all such solutions can be ascertained before usage using pH meter.

Procedure:

1. Standardize the pH meter as mentioned on the instrument. (May be using the standard solutions of known (minimum) pH=4.0 and known (maximum) pH=9.2 with the help of glass electrode or with the adjustment by screw driver).
2. Collect the raw milk (not dairy milk), filtered orange juice, citrus fruit, supernatant solution of lime, baking soda, etc
3. Take 25 cc of each juice or solution into a beaker. Dip the glass electrode into it.
4. Connect the glass electrode to the terminals of pH meter and record the pH and then tabulate.
5. Write the nature of the solution or the juice (Acidic/Alkaline)

Observations:

Biological juices or solutions	pH	Acidic/Alkaline
Raw milk		
Orange juice		
Lemon juice		
Lime water		
Baking soda		

Result:

1. Raw milk is found to be slightly acidic, where as orange juice and Lemon juice are highly acidic.
2. Lime water and Baking soda are found to basic in nature.

Note:

1. Raw milk should be used for the test but not dairy milk.
2. Fruits should be squashed, juice should be filtered and pure juice may be used for the test.
3. For lime water , 56 gm of Lime (CaO) should be dissolved in one litre of water. It gives milk of lime. Cool and filter it to get clear solution of lime water. Take this lime water solution for the test.
4. For baking soda , 84 gm of baking soda ($NaHCO_3$) should be dissolved in one litre of water. Filter it to get a clear solution. Take this solution for the test.
5. Preserve the glass electrode always in wet condition by keeping it in sat. KCl. Solution.

Expt. No.11 PARTIAL MISCIBILITY OF LIQUIDS

Aim: To determine the critical solution temperature of two partially miscible liquids (water and phenol).

Chemicals: Pure Phenol.

Apparatus: 09 hard glass test tubes, glass ring stirrer, air jacket (large tube), thermometer ($1/10^{th}$ °C)

Theory: Critical solution temperature is a temperature at which, two partially miscible liquids become completely miscible. Phenol and water are such a pair partially miscible liquids at room temperature. They become miscible at a particular temperature with a particular composition by weight. In order to determine the mutual solubility curve of this system a synthetic method is followed. Known amounts of different composition of two liquids are warmed slowly and temperature is recorded at which the two layers just disappear, that is turbidity just disappears. The temperature is once determined at the appearance of turbidity when the system is cooled. The mean of the two temperatures gives the miscibility temperature of two liquids in that proportion.

Procedure:

1. Take eight thoroughly **cleaned** and dried hard glass test tubes and label them as 1 to 8.
2. Since phenol is hygroscopic and caustic in nature, add it from burette 8,7…….1. cc to the each of the above test tubes and 1,2,3,……..8 cc of distilled water in each test tube as shown in table.
3. Insert the thermometer and a small glass ring stirrer into one of these tubes. Mount the tube in a large glass tube (as air jacket) suspended in large beaker containing water.
4. Heat the water in the beaker slowly with constant stirring so as to have the uniform temperature. Stir well the phenol solution and record the temperature when the turbidity just disappears.
5. Remove the source of heating and allow the water in the beaker to cool slowly with constant stirring. When the turbidity begins to appear due to partial miscibility, record the temperature. The average of two readings gives the temperature of complete miscibility of the two liquids.
6. Repeat the determination more cautiously allowing the temperature to change more slowly in the vicinity of the above temperature.
7. In the same way determine the temperature of complete miscibility for other phenol solutions.
8. Plot a graph of the miscibility temperatures and the composition of the solution in percentage (weight / weight). The smooth curve will be the parabolic in shape, the maximum of which gives the critical solution temperature of phenol and water system.

Observations:

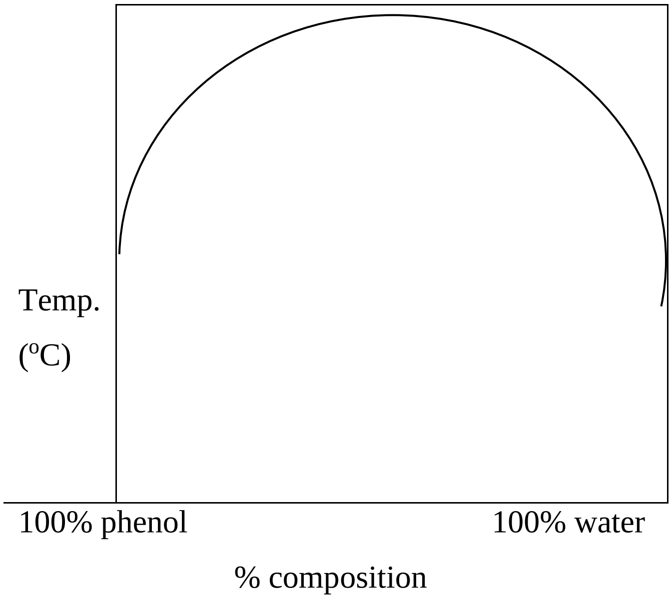
a. Density of Phenol= 0.8 g/cc
b. Density of Water = 1.0 g/cc

Test tube No.	Volume of Water	Volume of Phenol	Phenol (Wt/V) gram	Total weight of mixture (gram)	% of phenol in water (Wt/Wt)	Temperature At which turbidity (two layers) disappears (°C)	Temperature at which turbidity appears (°C)	Mean Temp (°C)
1	2cc	8cc	6.4	8.4	73.8			
2	3cc	7cc	5.6	8.6	65.1			
3	4cc	6cc	4.8	8.8	54.5			
4	5cc	5cc	4.0	9.0	44.4			
5	6cc	4cc	3.2	9.2	34.8			
6	7cc	3cc	2.4	9.4	25.5			
7	8cc	2cc	1.6	9.6	16.7			
8	9cc	1cc	0.8	9.8	08.2			

Calculation

1. Weight of phenol =Density x volume of phenol =……g
2. Percentage of phenol (Weight / Weight) = (Weight of phenol x 100) / Total weight of mixture
3. The value of CST = --------- °C
4. Percentage of phenol at CST=

Nature of Graph



Result:

1. Experimental value of CST = --------- °C
2. Percentage of phenol at CST-………

Note: 1. Theoretical value of CST = 66°C
2. Theoretical Percentage of phenol at CST= 34

B.Sc. VI Sem : Paper – I
Inorganic Experiments

Total No of hours /week : 04Hrs.	Pract. : 40 Marks
Total No. of Hours : 52Hrs.	I. A. : 10 Marks

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Introduction to Inorganic Gravimetric Analysis

II. Inorganic Volumetric Analysis

Introduction to Inorganic Volumetric Analysis

III. Inorganic preparations

Preparation of the following Compounds

Gravimetric Analysis

The quantitative chemical analysis that involves the determination of the constituent by weighing is called the gravimetric analysis. i.e. The process of isolation and weighing an element or a definite compound of the element in an as pure form as possible. A large portion of the determination in gravimetric analysis is concerned with the transformation of the element or radical to be determined in to pure stable compound, which can be readily converted into a form suitable for weighing. The weight of the element or radical may be readily calculated by knowing the formula of the compound and atomic weight of the constituent elements.

Gravimetric analysis involves the following steps

1. Preparation of solution of the known amount of substance
2. Separation of the substance to be estimated from solution as ppt.- Precipitation
3. Digestion or heating the precipitate.
4. Filtration and washing the precipitate.
5. Drying and ignition of the precipitate.
6. Weighing dried or ignited precipitate.
7. Calculation of the constituents.

Distribution of marks of Gravimetric Experiments

Accuracy	: 16 marks
Proper Technique & Calculation	: 04 ,,
Practical Record	: 05 ,,
Viva Voce	: 05 ,,
Tour report or Project Report	: 10 ,,
Total	**40 Marks**

Deduction of Marks for Accuracy

± 6 mg	16 marks
± 8 mg	14 marks
± 10 mg	12 marks
± 12 mg	10 marks
± 14 mg	08 marks
± 16 mg	06 marks
Above ± 16 mg	00 marks

Gravimetric Determinations

1. Determination of Barium as $BaSO_4$

Aim: To determine the amount of Barium as $BaSO_4$ present in a given solution.

Apparatus: Watch glass, Beaker, Funnel, silica crucible, desiccators etc.

Chemical: $BaCl_2$ solution, Conc. HCl, 5% H_2SO_4 Solution etc.

Outline: Barium from the solution is precipitated as barium sulphate by the addition of H_2SO_4 to the acidified hot solution. The precipitate is digested, filtered washed with water till it is free from sulphate and chloride ions. It is then dried, ignited and weighed as $BaSO_4$.

Procedure:

1. Dilute the given barium chloride solution up to the mark with distilled water in 250 cc volumetric flask.
2. Shake it well and pipette out 25 cc in to a clean 250 cc beaker with the help of a pipette.
3. Introduce a glass rod in to the beaker at this stage and it should not be taken out till the precipitate is completely transferred to filter paper.
4. Dilute it to about 100 cc with distilled water (Add about 75 cc distilled water) and 1-2cc conc. HCl. Heat the solution to boiling.
5. Heat about 20 cc of 5% solution of H_2SO_4 in a small beaker and add it to hot barium solution drop wise with constant stirring till the precipitation of **$BaSO_4$** is complete.
6. Cover the beaker partially with a watch glass and digest the precipitate on a sand bath for about 45 minutes for **Digestion** of the precipitate (i.e. heating the solution just short of boiling without actually boiling which granulates the precipitate).
7. Allow the precipitate to settle. Test the supernatant solution by adding a few drops of 5% sulphuric acid solution for complete precipitation.
8. Clean the crucible by heating the crucible with 1 cc conc.HNO_3 by placing on wire gauze and cool, wash with water, wipe out with clean cloth. Heat it by placing on pipe-clay triangle supported by tripod stand for 10 minutes with non-luminous (Blue)flame. Cool on tripod stand for 10 minutes and transfer it with a clean pair of tongs to desiccator. After thorough cooling weigh the crucible and note down the weight(W_1).
9. Take Whatman's filter paper no. 41 circle and fold it along a diameter to form a semicircle. Fold again symmetrically so as to form four folds. Open one fold on side & three on the other side to form a cone. Attach filter paper cone properly to the funnel in such a way that it touches the funnel only along its edges leaving the filter paper cone hanging & moisten it with few drops of distilled water.

10. Decant the supernatant solution carefully through Whatman's filter paper no. 41 without disturbing the precipitate.
11. Wash the precipitate with hot distilled water (100-120 ml) for several times in the beaker itself and then transfer it quantitatively to the filter paper, using the policeman to detach the particles from the sides of the beaker.
12. Wash the precipitate again with hot water till the filtrate is free from sulphate and chloride ions (test the filtrate with dil. $BaCl_2$ & $AgNO_3$ solutions which should not give white ppt. with both).
13. Drain the filter paper thoroughly. Dry the precipitate partially on a hot air cone and incinerate it along with the filter paper in a previously weighed crucible.
14. First heat slowly till the precipitate dries and filter paper gets charred then strongly till the residue becomes white. Then heat strongly either in a burner or electrical Incinerator or Muffle furnace. If the crucible is blackened due to preliminary heating, by turning the crucible repeatedly and carefully ignited by the flame until all the carbon has been burned off: the crucible becomes white as before. The white residue is $BaSO_4$.
15. Heat the crucible for another 10 minutes, cool, desiccate and weigh. Note down the weight of the residue. Again heat it for 10 minutes, cool, desiccate and weigh it again and note down the weight of the residue. (Heating, cooling and desiccating and weighing the crucible is repeated till a constant weight is obtained.)
16. From the weight of $BaSO_4$ obtained calculate the amount of barium present in the given solution.

Equations: $$\mathbf{BaCl_2 + H_2SO_4 \longrightarrow \underset{\text{barium sulphate}}{BaSO_4\downarrow} + 2HCl}$$

1 gram mole of $BaSO_4 \equiv$ 1 gram mole of Ba

233.42 g $BaSO_4 \equiv$ 137 gram mole of Ba

1 g. $BaSO_4 \equiv 0.5887$ g Ba

Observations: 1. Weight of the empty crucible = W_1 = ----g
2. Weight of the crucible + residue = W_2 = ----g
3. Weight of the residue ($BaSO_4$) = $W_2 - W_1$ = ----g

Calculations

1. Weight of the residue ($BaSO_4$) = $W_2 - W_1$ = ---- g
2. Amount of Barium present in 25cc of the given solution = X = ($W_2 - W_1$) × 0.5887=… g
3. Amount of Barium present in 250 cc of the given solution = X × 10 = ..'a' ..g
4. Amount of Barium present in $1dm^3$ of the given solution =X × 40 = **…**g Or 'a'× 4 = **...**g

Result:

1.Weight of the residue ($BaSO_4$) _____ g
2.Amount of Barium present in 25 cc of the given solution _____g
3.Amount of Barium present in 250 cc of the given solution______g
4.Amount of Barium present in $1dm^3$ of the given solution _____g

2. Determination of Aluminium as Al_2O_3

Aim: To determine the amount of aluminium(Al) as Al_2O_3 in the given solution.

Apparatus: Watch glass, beaker, funnel, silica crucible, desiccator etc..

Chemicals: $AlCl_3$ solution, NH_4Cl, 1:1 NH_4OH, 1% NH_4NO_3 etc.

Outline: Aluminium is precipitated as $Al(OH)_3$ by adding NH_4OH to the hot & dilute solution of potash alum in presence of ammonium chloride at a pH of 4-6. NH_4Cl controls the pH of the solution by exerting buffer effect and also assists the coagulation of the initially colloidal precipitate. $Al(OH)_3$ is filtered, washed with hot 1% NH_4NO_3, dried, ignited and weighed as Al_2O_3.

Procedure:

1. Dilute the given aluminium solution up to the mark with distilled water in the 250 cc volumetric flask and shake it well.
2. Pipette out 50 cc of the diluted solution in to a clean 250 cc beaker and dilute to about 100 cc with distilled water.
3. Add 1-2g of NH_4Cl and few drops of methyl red indicator and heat it boiling.
4. Precipitate the Al by adding 1:1 NH_4OH solution drop wise with constant stirring till the colour of the solution changes to distinct yellow.
5. Stir and boil it for a minute and allow the precipitate to settle for few minutes.
6. Decant the supernatant solution immediately through a Whatman's filter paper No. 41 moistened with 1% NH_4NO_3 solution.
7. Take Whatman's filter paper no. 41 circle and fold it along a diameter to form a semicircle. Fold again symmetrically so as to form four folds. Open one fold on side & three on the other side to form a cone. Attach filter paper cone properly to the funnel in such a way that it touches the funnel only along its edges leaving the filter paper cone hanging & moisten it with few drops of 1% NH_4NO_3 solution.
8. Wash the precipitate at least twice with hot 1% NH_4NO_3 solution in the beaker itself till the filtrate is free from sulphate ions (test with $BaCl_2$ solution & it should not give white ppt.or turbidity) but need not be free from chloride. (Filtration becomes difficult in the cold condition due to the clogging of pores of the filter paper by gelatinous $Al(OH)_3$ & hence filter the ppt. when solution is hot.) Wash it again with hot 1% NH_4NO_3 solution.
9. Partially dry the precipitate on a hot air cone and incinerate it along with the filter paper in a previously weighed crucible.
10. Clean the crucible by heating the crucible with 1 cc conc.HNO_3 by placing on wire gauze and cool (this operation is omitted when it is free from dark color), wash with water and wipe out with clean

cloth. Heat it by placing on **pipe-clay triangle** supported by **tripod stand** for 10 minutes with non-luminous (Blue)flame. Cool on tripod stand for 10 minutes and transfer it with a clean **pair of tongs** to **desiccator**. After thorough cooling weigh the crucible and note down the weight(W_1).

11. First heat gently to char the filter paper with a low flame and then slowly increase the flame. Ignite the crucible to red hotness till the residue becomes white, for about 30 minutes. Cool, desiccate and weigh.
12. First heat slowly till the precipitate dries and filter paper gets charred then strongly till the residue becomes white. Then heat strongly either in a burner or electrical Incinerator or Muffle furnace. If the crucible is blackened due to preliminary heating, by turning the crucible repeatedly and carefully ignited by the flame until all the carbon has been burned off: the crucible becomes white as before.
13. Repeat the process of heating, cooling and weighing till a constant weight is obtained.
14. The white residue is Al_2O_3. From the weight of the residue obtained, calculate the amount of Al in the given solution.

Equations:

$$Al_2(SO_4)_3 + 6NH_4OH \xrightarrow{NH_4Cl} 2Al(OH)_3\downarrow + 3(NH_4)_2SO_4$$

$$2Al(OH)_3 \xrightarrow{\Delta} Al_2O_3 + 3H_2O$$

$$1\text{ g mole of } Al_2O_3 \equiv 2Al$$

$$102\text{ g of } Al_2O_3 \equiv 54\text{ g Al}$$

$$1\text{ g of } Al_2O_3 \equiv 0.529\text{ g Al}$$

Observations

1. Weight of the empty crucible = W_1 = -----g.
2. Weight of the crucible + residue = W_2 = -----g.
3. Weight of the residue (Al_2O_3) = $W_2 - W_1$ = -----g.

Calculations:

1. Weight of the residue (Al_2O_3) = ($W_2 - W_1$) = -----g.
2. Amount of Al present in the given diluted 50 cc solution = X =($W_2 - W_1$) × 0.529 = ----g
3. Amount of Al present in the given diluted 250 cc solution = X × 5 = ---g. = ..'a'g
4. Amount of Al present in the given diluted 1 dm^3 solution = X × 20 = 'a'× 4 = ---g.

Result:

1. Weight of the residue (Al_2O_3) _____ g
2. Amount of Al present in 50 cc of the given solution _____g
3. Amount of Al present in 250 cc of the given solution _____g
4. Amount of Al present in 1dm^3 of the given solution _____g

3. Determination of Iron as Fe_2O_3

Aim: To determine the amount of iron as Fe_2O_3 present in a given solution.

Apparatus: Watch glass, Beaker, Funnel, silica crucible, desiccator etc.

Chemical: Iron solution, conc. HC1, conc. HNO_3, NH_4Cl, 1:1NH_4OH, 1% NH_4NO_3 etc.

Outline: Iron is precipitated as $Fe(OH)_3$ by the addition of 1:1NH_4OH to the hot and diluted iron solution in presence of NH_4Cl. The precipitate, $Fe(OH)_3$ is washed with hot 1% NH_4NO_3 solution, filtered and dried. It is then ignited and weighed as Fe_2O_3.

Procedure:

1. Dilute the given iron solution up to the mark in 250 cc volumetric flask with distilled water.
   Shake it well and pipette out 50 cc of the diluted solution in to a clean 250 cc beaker.
2. Add 1 cc of conc. HC1 & 1-2 cc of conc. HNO_3 and boil till the solution becomes deep yellow or orange in colour. This is to oxidize any unoxidised Fe^{2+} to Fe^{3+}. (**This operation may be omitted if the solution is already deep yellow in colour).**
3. Dilute it to about 100 cc with distilled water (Add about 50 cc distilled water). Add 1-2 g of NH_4Cl and heat it boiling.
4. Precipitate the iron as $Fe(OH)_3$ by adding 1:1 NH_4OH solution(approximately 1-1/⅓ TT) drop wise till the reddish brown precipitate is obtained & beaker smells ammonia after stirring.
5. Stir and boil it for a minute and allow the precipitate to settle for few minutes.
6. Take Whatman's filter paper no. 41 circle and fold it along a diameter to form a semicircle. Fold again symmetrically so as to form four folds. Open one fold on side & three on the other side to form a cone. Attach filter paper cone properly to the funnel in such a way that it touches the funnel only along its edges leaving the filter paper cone hanging
7. & moisten it with few drops of 1% NH_4NO_3 solution.
8. Decant the supernatant solution through funnel attached with Whatman's filter cone.
9. Wash the precipitate at least twice with hot 1% NH_4NO_3 solution in the beaker itself (Filter the ppt. in the hot condition only).
10. Transfer the precipitate to the filter paper and wash it again with hot 1% NH_4NO_3 solution (test with $AgNO_3$) till it is free from chloride.
11. Partially dry the precipitate on a hot air cone and incinerate it along with the filter paper in a previously weighed crucible.
12. Clean the crucible by heating the crucible with 1 cc conc.HNO_3 by placing on wire gauze and cool (this operation is omitted when it is free from dark color), wash with water and wipe out with clean cloth. Heat it by placing on **pipe-clay triangle** supported by **tripod stand** for 10 minutes with non-

luminous (Blue)flame. Cool on tripod stand for 10 minutes and transfer it with a clean **pair of tongs** to **desiccator**. After thorough cooling weigh the crucible and note down the weight(W_1).

13. 13. First heat slowly till all the filter paper is charred and then strongly for about 30 minutes.If the crucible is blackened due to preliminary heating, by turning the crucible and carefully ignited by the flame until all the carbon has been burned off: the crucible becomes white as before.
14. Heat the crucible for another 10 minutes, cool, desiccate and weigh. Again heat it for 10 minutes, cool, desiccate and weigh it again and note down the weight of the residue.
15. After cooling and desiccating, weigh it to a constant weight.
16. Residue is Fe_2O_3 (dark brown). From the weight of the residue obtained, calculate the amount iron in the given solution.

Equations

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

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

$$1 \text{ g mol of } Fe_2O_3 \equiv 2Fe$$

$$\text{i.e. } 159.7 \text{ g mol of } Fe_2O_3 \equiv 111.7 \text{ g mol Fe}$$

$$1\text{g of } Fe_2O_3 \equiv 0.6993 \text{ g Fe.}$$

Observations

1. Weight of the empty crucible = W_1 = -----g.
2. Weight of the crucible + residue = W_2= -----g.
3. Weight of the residue (Fe_2O_3) = $W_2 - W_1$ = -----g.

Calculations:

1. Weight of the residue (Fe_2O_3) = ($W_2 - W_1$) = -----g.
2. Amount of Fe present in 50 cc of the given solution = X = (W_2 - W_1) × 0.6993 g
3. Amount of Fe present in 250 cc of the given solution = X × 5 **= ---**g =‘a’ g
4. Amount of Fe present in the given diluted 1 dm^3 solution = X × 20 =‘a’× 4 = ---g.

Result:

1. Weight of the residue (Fe_2O_3) _____g
2. Amount of Fe present in 50 cc of the given solution _____ g
3. Amount of Fe present in 250 cc of the given solution _______g
4. Amount of Fe present in the given diluted 1 dm^3 solution _______g

4. Determination of lead as $PbSO_4$

Aim: To determine lead as lead sulphate in the given solution of lead nitrate or lead acetate.
Apparatus: Watch glass, beaker, funnel, silica crucible & desiccator etc.
Chemicals required :Lead solution, Dil. Sulphuric acid, rectified spirit etc.
Outline: Lead is precipitated as lead sulphate by the addition of cold and dil. sulphuric acid to the cold and dilute solution of lead nitrate or acetate. Precipitation is completed by the addition of rectified spirit. The white precipitate of lead sulphate is filtered, washed with 1% cold solution of sulphuric acid till free from sulphate. Dried, ignited and weighed as $PbSO_4$.

Procedure:

1. Dilute the given lead nitrate (acetate) solution up to the mark in 250 cc volumetric flask with distilled water.
2. Shake it well and pipette out 25 cc of the diluted solution into a clean 250 cc beaker.
3. Dilute it to about 100 cc with distilled water (Add about 75 cc distilled water).
4. Add cold 5% solution of H_2SO_4 (about 20 cc) drop wise with constant stirring and allow the precipitate to settle and add few more drops of dilute sulphuric acid to the supernatant liquid.
5. Finally complete the precipitation by the addition of 50 cc of rectified spirit and keep it for half an hour.
6. Decant the supernatant solution through a Whatman's filter paper No. 42 moistened with 1% cold solution of sulphuric acid.
7. Wash the precipitate at least twice with 1% cold solution of sulphuric acid in the beaker itself.
8. Transfer the precipitate to the filter paper and wash it again with 1% cold solution of sulphuric acid (test with $AgNO_3$) till it is free from chloride.
9. Then wash with 5% rectified spirit till free from sulphate ions (no white ppt. with $BaCl_2$). Drain the filter paper thoroughly.
10. Partially dry the precipitate on a hot air cone and incinerate it along with the filter paper in a previously weighed crucible.
11. First heat slowly till all the filter paper gets charred and then strongly for about 30 minutes (**use low flame& single burner**). After cooling and desiccating, weigh it to a constant weight.
12. White residue obtained is $PbSO_4$. From the weight of the residue obtained, calculate the amount of lead in the given solution.

Equations:

$$Pb(NO_3)_2 + H_2SO_4 \longrightarrow PbSO_4 \downarrow + 2HNO_3$$

1 gram mole of $PbSO_4 \equiv$ 1 gram mole of Pb.
303. 27 g $PbSO_4 \equiv$ 207.21 gram mole of Pb.
1 g $PbSO_4 \equiv$ 0.6833 g Pb.

Observations:

1. Weight of the empty crucible = W_1 = ----g
2. Weight of the empty crucible + residue = W_2 = ----g
3. Weight of the residue ($PbSO_4$) = $W_2 - W_1$ = ----g

Calculations

1. Weight of the residue ($PbSO_4$) = $W_2 - W_1$ = ---- g
2. Amount of lead present in 25cc of the given solution = X = ($W_2 - W_1$) × 0.6833 g
3. Amount of lead present in 250cc of the given solution = X × 10 = 'a' ..g
4. Amount of lead present in 1dm^3 of the given solution = X × 40 **=** **---** g Or 'a'×10 **= ...**g

Result:

1. Weight of the residue ($PbSO_4$) ---- g
2. Amount of lead present in 25cc of the given solution ____ g
3. Amount of lead present in 250cc of the given solution ____ g
4. Amount of lead present in 1dm^3 of the given solution ____g

Volumetric Analysis

Volumetric analysis is quantitative chemical analysis carried out by determining the volume of solution (whose concentration is accurately known) required to react with a measured volume of a solution of unknown concentration. It involves the estimation of a substance in solution either by precipitation, neutralisation, oxidation and reduction by means of a solution of known strength.

Types of titration:

1. Neutralization titration/Acid-base titration
2. Redox titration
3. Iodometric and iodimetric titration
4. Precipitation titration
5. Complexometric titration

……………………………………………………………………………………………

Distribution of marks of Volumetric Experiments

Accuracy (Titre values 2 × 5**)**	: 10 marks
Proper Technique	: 02 ,,
Calculation	: 02 ,,
Practical Record	: 05 ,,
Viva Voce	: 05 ,,
Tour report or Project Report	: 10 ,,
Total	**: 34 Marks**

Deduction of Marks for Accuracy for Volumetric Experiments

± 0.2 cc	10 marks
± 0.4 cc	08 marks
± 0.6 cc	06 marks
± 0.8 cc	04 marks
Above ± 0.9 cc	00 marks

Distribution of marks of Preparation of complex Experiments

Yield of the Complex	: 06 marks

Deduction of Marks for Accuracy for Preparation Experiments

Error in yield less than 10%	06 marks
11-15%	05 marks
16-20%	04 marks
21-25%	03 marks
26-30%	02 marks
More than 30%	00 marks

Volumetric Determinations

1. Determination of percentage of iron in haematite ore or Solid Fe_2O_3.

Aim: To extract Iron(III) from haematite ore or solid Fe_2O_3 and to determine the percentage of iron in the solution using standard $K_2Cr_2O_7$ solution (internal indicator method).

Apparatus: Burette, Pipette, Conical flask, volumetric flask, funnel, beaker etc.

Chemicals: Haematite ore (solid Fe_2O_3), Conc.HNO_3, Conc.HCl, $K_2Cr_2O_7$, $HgCl_2$, $SnCl_2$, 2N H_2SO_4, Phosphoric acid, Diphenylamine indicator etc.

Outline: This is a redox titration, in which ferric iron present in the solution is prepared by dissolving Haematite ore or solid Fe_2O_3 sample in acidic solution. It is first reduced to ferrous state by adding $SnCl_2$ in hot condition and then oxidized to ferric state again by titrating against a standard solution of $K_2Cr_2O_7$ in presence of sulphuric acid and phosphoric acid using diphenylamine indicator.

Procedure:

1. Weigh about 1 g of haematite ore (solid Fe_2O_3) in a watch glass. Transfer it completely to a 250cc beaker. Add few ccs of 1:1 HCl (about 7-8cc), 2cc Conc.HCl and 1cc Conc.HNO_3.
2. Evaporate the solution on a sand bath to a volume of 1-2cc, but not to dryness. Add about 50cc of distilled water and heat on the sand bath for 8-10 minutes in order to dissolve all soluble material.
3. Stir it well and filter if necessary through Whatman filter paper no.42. Transfer the filtrate and washings to 250 cc volumetric flask.
4. Dilute the Fe^{3+}solution in 250cc volumetric flask up to the mark with distilled water and shake it well.
5. Weigh 0.613g potassium dichromate (mol.wt.=298.18; Eq.wt.= 49.06) crystals on a watch glass. Transfer the crystals to beaker with the help of little distilled water and dissolve them completely.
6. Transfer the solution obtained to the 250 cc volumetric flask, using a funnel. Wash the beaker 2-3 times and collect washings in the same volumetric flask. Make the solution up to the mark with distilled water. Shake it well for uniform concentration and fill it in the burette.
7. Pipette out 25 cc of Fe^{3+} solution into a clean 250 cc conical flask. Add 2 cc of Conc.HCl & heat the solution to boiling. To the hot solution add $SnCl_2$ drop wise with constant shaking until the solution just turns colourless and then add a drop more.
8. Cool the solution rapidly under tap (cold water). Add 10 cc of $HgCl_2$ in one lot. A silky white precipitate should be obtained (Reject if black precipitate or excess precipitate is obtained).
9. Add 20 cc(2 test-tube full) of 2N H_2SO_4, 5cc(1/2 TT) of phosphoric acid and 2-3 drops of diphenylamine indicator.

10. Titrate the solution against standard $K_2Cr_2O_7$ solution till colour of the solution changes from pale green to deep violet colour which remains permanent on shaking.
11. Record the burette reading and repeat the titration till three readings agree closely. The amount of ferric iron in given ore or solution is calculated using the accurate readings.

Observations:

Part A

I. **Weighing of haematite ore (solid Fe_2O_3)**

1. Weight of empty watch glass = W_1 = ---g
2. Weight of watch glass + haematite ore (solid Fe_2O_3) = W_2 = ---g
3. Weight of haematite ore (solid Fe_2O_3) = W_2 - W_1 = …. g

II. **Preparation of standard 0.05N $K_2Cr_2O_7$solution**

Molecular weight of $K_2Cr_2O_7$ = 298.18 Equivalent weight = 49.03

Amount of $K_2Cr_2O_7$ crystals to be weighed to prepare 250 cc of 0.05N solution

$$\mathbf{w = \frac{N\times E\times V}{1000} = \frac{0.05\times 49.03\times 250}{1000} = 0.613\ g} \quad \textbf{or}$$

For 100 cc of 0.05N solution

$$\mathbf{w = \frac{N\times E\times V}{1000} = \frac{0.05\times 49.03\times 100}{1000} = 0.245\ g}$$

III. **Weighing of $K_2Cr_2O_7$ crystals**

1. Weight of empty watch glass = W_1 = ---g
2. Weight of watch glass + $K_2Cr_2O_7$ crystals = W_3 = ---g
3. Weight of $K_2Cr_2O_7$ crystals= W_3 - W_1 = ………**0.613/0.245 g**

Part B

1. Solution in burette = 0.05N $K_2Cr_2O_7$ solution
2. Solution in conical flask = 25 cc Fe^{3+}solution(hot) + $SnCl_2$(till just colourless) cooled + 10cc $HgCl_2$, (silky white ppt) + 20 cc H_2SO_4 + 5cc phosphoric acid
3. Indicator – Diphenylamine
4. Colour change- pale green to violet.
5. **Reactions**

$$Fe_2O_3 + 6HCl \longrightarrow 2FeCl_3 + 3H_2O$$

$$FeCl_3 + SnCl_2 \longrightarrow FeCl_2 + SnCl_4$$

$$2HgCl_2 + SnCl_2 \longrightarrow \underset{\text{white silky ppt.}}{Hg_2Cl_2\downarrow} + SnCl_4$$

$$K_2Cr_2O_7 + 6FeCl_2 + 7H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + Fe_2(SO_4)_3 + 4FeCl_3 + 7H_2O$$

6. Burette Readings:

Burette reading in cm^3	Accurate reading in cm^3	
	I	II
Final burette reading		
Initial burette reading	0.0	0.0
Difference		

Calculations

1. Normality of Fe^{3+}solution $= \frac{\mathbf{0.05 \times B.R.}}{\mathbf{25}}$

2. Gram per litre = Normality × equivalent mass of Fe^{3+} $(= \frac{\text{Atomic mass}}{3} = \frac{55.85}{3} = 18.62)$

3.Amount of Fe^{3+} present in the diluted 250cc solution (given sample ore or solid Fe_2O_3)

$$= \frac{\text{normality } \times \text{equivalent mass}(55.85)}{4} = \cdots (x)g$$

4. Percentage of $Fe^{3+}$$= \frac{x \times 100}{\text{mass of ore}} = \frac{x \times 100}{1} = .. \%$

Result:

1.Amount of Fe^{3+} present in the diluted 250 cc solution(given sample ore or solid Fe_2O_3) =….g

3. Percentage of Fe^{3+} =…….%

2. Determination of percentage of copper in brass.

Aim: To extract Cu and Zn from brass and to determine the percentage of copper in the solution using standard $Na_2S_2O_3$ solution.

Chemicals required: brass, Conc.HNO_3,urea, $Na_2S_2O_3$, KI, Na_2CO_3, acetic acid etc.

Principle: The main constituents of Brass alloy are Copper & Zinc with small quantity of tin, lead & iron. The percentage composition of a typical brass is Cu-50-90, Zn-20-40, Sn-0-6, Pb-0-2, Fe-0-1. Sample of brass is dissolved in $HNO_3$to get the solution of Cu and Zn. Cu is estimated by iodometric titration method in which iodine is liberated when KI solution is added to Cu^{2+}solution that is titrated against sodium thiosulphate solution using starch solution as an indicator.

Procedure:

1. Weigh about 1 g of the brass into a watch glass and transfer it to a 250cc beaker. Add 1-2 cc of con. HNO_3.
2. Slowly heat the beaker covered with a watch glass.
3. When the vigorous reaction is over, add about 1 g of urea. When a pasty mass is obtained add about 50 cc of distilled water and boil for 2 minutes to destroy the oxides of nitrogen and dissolve all the soluble material.
4. Stir it well and filter if necessary through the filter paper. Collect the filtrate and washings in 250 cc beaker.
5. Transfer the brass solution, into a **100cc/250cc** volumetric flask.
6. Weigh **1.240/3.102g** of Sodium thiosulphate (mol.wt.= Eq.wt. = 248) crystals and transfer to beaker with the help of little distilled water and dissolve them completely.
7. Transfer the solution obtained to the **100/250 cc** volumetric flask, using a funnel. Wash the beaker 2-3 times and collect washings in the same volumetric flask.
8. Make the solution up to the mark with distilled water. Shake it well for uniform concentration, and fill it into the burette.
9. Dilute the brass solution (containing Cu^{2+}) in **100cc /250cc** volumetric flask up to the mark with distilled water. Shake it well and pipette out 25 cc in to a clean 250 cc conical flask.
10. **Add Na_2CO_3 solution drop wise to the solution till a slight ppt. is obtained. Add 1:1 acetic acid until the precipitate just dissolves and add two more drops.**
11. Add one test tube full 10% KI solution, cover the conical flask with a watch glass and allow the mixture to stand for one minute.
12. Titrate slowly with standard sodium thiosulphate solution till colour of the solution changes from reddish brown to pale yellow and then add 1cc of starch solution. The solution turns blue. Continue

the addition drop wise till the dark blue colour just disappears. There is a tendency for the blue colour to return, so that the first complete disappearance of the blue colour for about 10 seconds is taken as the end point.

13. Record the burette readings and repeat the titration till three concurrent readings are obtained.
14. Calculate the amount of copper in the brass solution (sample) using the accurate readings.

Observations I. weighing of sample of brass

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

II. Preparation of standard 0.05N $Na_2S_2O_3.5H_2O$ solution

Molecular weight = Equivalent weight = 248

Amount of $Na_2S_2O_3.5H_2O$ crystals to be weighed to prepare 100/250 cc of 0.05N solution

For 100cc $$\mathbf{w} = \frac{\mathbf{N\times E\times V}}{\mathbf{1000}} = \frac{\mathbf{0.05\times 248\times 100}}{\mathbf{1000}} = \mathbf{1.240\ g}$$

For 250cc $$\mathbf{w} = \frac{\mathbf{N\times E\times V}}{\mathbf{1000}} = \frac{\mathbf{0.05\times 248\times 250}}{\mathbf{1000}} = \mathbf{3.102\ g}$$

III. Weighing of $Na_2S_2O_3.5H_2O$ crystals

1. Weight of the empty watch glass = W_1 = ----g
2. Weight of the watch glass + $Na_2S_2O_3.5H_2O$ crystals = W_3 = ----g
3. Weight of the $Na_2S_2O_3.5H_2O$ crystals = $W_3 - W_1$ = ----g

IV. Titration

1. Solution in burette: 0.05N $Na_2S_2O_3.5H_2O$ solution
2. Solution in conical flask: 25 cc of Cu^{2+} solution + 1 test tube of 10% KI
3. Indicator: Starch solution
4. End point: Blue to colourless near the end point.
5. Reactions

$$Cu^{2+} + 2KI \longrightarrow 2\,K^{+} + CuI_2(\text{unstable})$$

$$2CuI_2 \longrightarrow Cu_2I_2 + I_2(\text{liberated iodine})$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow \underset{\text{Sodium tetra thionate}}{Na_2S_4O_6} + \underset{\text{Sodium iodide}}{2NaI}$$

6. Burette Readings:

Burette reading in cm^3	Accurate reading in cm^3	
	I	II
Final burette reading		
Initial burette reading	0.0	0.0
Difference		

IV. Calculations

1. Normality of Cu^{2+} solution $= \frac{\textbf{Normality of Na}_2\mathbf{S_2O_3}\times \textbf{B.R.}}{\mathbf{25}}$

2.Gram per liter of Cu^{2+} = Normality **of Cu^{2+}** × Eq.mass of Cu^{2+} (63.5) = x. g

3. Amount of Cu^{2+}present in the diluted 250cc solution (given sample of alloy or solution) $= \frac{\mathbf{x}}{\mathbf{4}} = \ldots$

g = ‘y’ g OR

Amount of Cu^{2+}present in the diluted 100cc solution (given sample of alloy or solution) $= \frac{\mathbf{x}}{\mathbf{10}} = \ldots$ **g**

= ‘y’ g

4. Percentage of Cu^{2+}== $\frac{\mathbf{y \times 100}}{\textbf{mass of alloy}} =$ %

Results:

1. Normality of Cu^{2+} solution = ……… g

2. Amount of Cu^{2+}present in the diluted 250cc brass solution (given sample of alloy or solution)__g

3. Percentage of Cu^{2+} =……..g %

3. Determination of percentage of calcium in limestone

Aim: To extract calcium from limestone and to determine the percentage of calcium in the solution by oxalate method.

Apparatus: Burette, Pipette, Conical flask, volumetric flask, funnel, beaker etc.

Chemicals : $CaCO_3$, 5% ammonium oxalate, NH_4OH, $KMnO_4$,Oxalic acidetc.

Outline : Limestone when treated with 1:1HCl to forms $CaCl_2$ solution which on treating with ammonium oxalate solution it gives white ppt. of calcium oxalate. CaC_2O_4 is dissolvedin dil. H_2SO_4& then titrated against standard $KMnO_4$ solution.

Procedure:

1. Weigh about 0.100/0.500 g of the limestone in a watch glass and transfer it to a 250 cc beaker. Add 10 cc of 1:1 HCl to it and slowly boil till the evolution of effervescence of CO_2 ceases. When all the limestone is dissolved, add little distilled water.
2. Transfer the solution obtained into a **100/250 cc** volumetric flask and dilute up to the mark with distilled water.
3. Shake well and pipette out 25 cc diluted solution in to a 250 cc beaker. Dilute to about 100 cc (by adding about 75 cc dist.water).
4. Add few drops of methyl red indicator and boil the solution. Add 25 cc of hot 5 % ammonium oxalate solution and dilute NH_4OH solution until the solution is neutral or slightly alkaline (colour changes from red to yellow).
5. Calcium is precipitated as calcium oxalate. Test for complete precipitation by adding slight excess amount of oxalate solution.
6. Allow the precipitate to settle for about 1 hr and filter it through filter paper No.1 and wash with hot water. **Reject the filtrate.**
7. In the mean while standardize given $KMnO_4$ solution as follows: Prepare **0.05** N oxalic acid solution by dissolving **0.315**g oxalic acid crystals in water & diluting up to the mark in **100 cc** volumetric flask.
8. Pipette out 25 cc of oxalic acid solution into a clean 250 cc conical flask. Add 20 cc(1½TT) of 2N H_2SO_4 & heat the solution (60-70°C) just short of boiling. Titrate the hot solution against standard $KMnO_4$ till colour changes from colourless to light pink. Note down the burette reading & by using it calculate the exact normality of $KMnO_4$ solution.
9. Dissolve the precipitate $CaC_2O_4.2H_2O$ in 5 cc of 1:8 H_2SO_4 (alternatively the solution may be collected by piercing a hole in the filter paper with a glass rod & washings of the ppt. & even

transfer the filter paper through a funnel in to a conical flask) and collect the solution in a conical flask.

10. Add 50 cc dilute (2N) H_2SO_4 and heat to boiling. Titrate the hot solution against standard $KMnO_4$ till colour changes from colorless to light pink.
11. Note down the burette reading. Using this calculate the percentage of calcium in the given sample of limestone.

Observations I. Weighing of limestone

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

II. Preparation of standard 0.05N $H_2C_2O_4.2H_2O$ solution

Molecular weight of $H_2C_2O_4.2H_2O$ = 126   Equivalent weight = 63

Amount of $H_2C_2O_4.2H_2O$ crystals to be weighed to prepare **100 of 0.05N** solution

$$w = \frac{\mathbf{N \times E \times V}}{\mathbf{1000}} = \frac{\mathbf{0.05 \times 63 \times 100}}{\mathbf{1000}} = \mathbf{0.315\ g}$$

III. Weighing of $H_2C_2O_4.2H_2O$ crystals

1. Weight of empty watch glass = W_1 = ---g
2. Weight of watch glass + Oxalic acid crystals = W_3 = ---g
3. Weight of Oxalic acid crystals = $W_3 - W_1$ = 0.315 g

IV. Part B: Standardization of $KMnO_4$ solution

1. Solution taken in burette = 'a'N **$KMnO_4$** solution
2. Solution taken in conical flask = 25 cc of 0.05N oxalic acid solution+20 cc 2N H_2SO_4
3. Indicator – $KMnO_4$ itself
4. Colour change- Colourless to light pink.
5. **Reactions**

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(\emptyset)$$
$$[H_2C_2O_4 + (\emptyset) \longrightarrow CO_2\uparrow + H_2O] \times 5$$
$$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow$$

Burette Readings:

Burette reading in cm^3	Accurate reading in cm^3	
	I	II
Final burette reading		
Initial burette reading	0.0	0.0
Difference		

Exact Normality of $KMnO_4$ = 'a' = $\frac{0.05 \times 25}{B.R}$

V. Part B Determination of Ca

1. Solution taken in burette = *a*(..) N $KMnO_4$ solution
2. Solution taken in conical flask = 25 cc Ca^{2+} solution + 50 cc 2N H_2SO_4
3. Indicator –$KMnO_4$ itself
4. Colour change- Colourless to light pink.
5. **Reactions**

$$\begin{array}{l}
[CaC_2O_4 + H_2SO_4 \longrightarrow CaSO_4 + H_2C_2O_4] \times 5 \\
2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(\emptyset) \\
[H_2C_2O_4 + (\emptyset) \longrightarrow CO_2\uparrow + H_2O] \times 5 \\
\hline
2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow
\end{array}$$

Burette Readings:

Burette reading in cm^3	Accurate reading in cm^3	
	I	II
Final burette reading		
Initial burette reading	0.0	0.0
Difference		

VIII. Calculations

1. 25 cc of Ca^{2+} solution required -------cc of *a*(..) N $KMnO_4$ solution
2. Normality of $Ca^{2+} = \frac{\textbf{Normality of } \mathbf{KMnO_4}\mathbf{(aN)} \times \mathbf{B.R.}}{\mathbf{25}} =' \mathbf{x'N}$
3. Gram per litreof Ca^{2+} = '**x**' × equivalent mass of Ca^{2+}(20.04)=.. (y) g
4. Amount of Ca^{2+}present in the diluted 100/ 250cc solution (given sample of limestone)

   For 100c

$$= \frac{\mathbf{x \times equivalent\ mass}(20.04)}{\mathbf{10}} \ \mathbf{or}\ \frac{\mathbf{y}}{\mathbf{10}} = .(\mathbf{z}). =..\mathbf{g}$$

   For 250cc

$$= \frac{\mathbf{x \times equivalent\ mass}(20.04)}{\mathbf{4}} \ \mathbf{or}\ \frac{\mathbf{y}}{\mathbf{4}} = .(\mathbf{z}). =..\mathbf{g}$$

5. Percentage of Ca^{2+} present in given sample of limestone

$$= \frac{\mathbf{z \times 100}}{\textbf{mass of limestone}} = \ \ldots.\%$$

Results:

1. 25 cc of Ca^{2+} solution required 1----cc 2. ----cc(B.R.) of *a*(..) N$KMnO_4$ solution
2. Amount of Ca^{2+}present in the diluted 100 cc solution (given sample of limestone)= ---g
3. Amount of Ca^{2+}present in the diluted 250cc solution (given sample of limestone)= ---g
4. Percentage of Ca^{2+} present in given sample of limestone=……. %

Preparation of Complexes

1. Preparation of bis(dimethylglyoximato)nickel(II)

Aim: To prepare bis(dimethylglyoximato)nickel(II)

Chemicals required: $NiSO_4$, Dilute ammonia, 1% alcoholic solution of DMG.

Procedure:

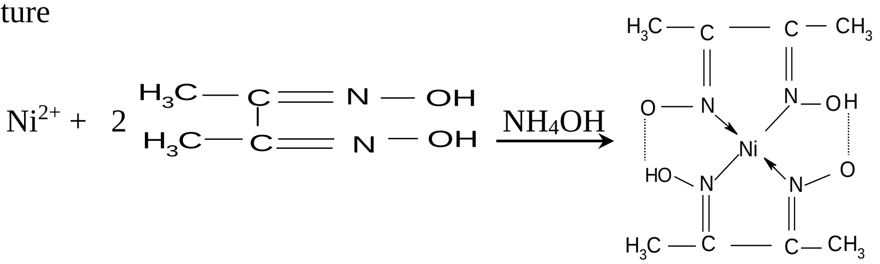
1. Dissolve the given sample (say 0.200g) of nickel salt in 50 cc of distilled water taken in a clean 100 cc beaker.
2. Add 2 cc of dilute (1:1) HCl and heat the solution to 70-80°C.
3. Add a slight excess of 1% alcoholic solution of dimethyl glyoxime (10-12 cc), immediately followed by dilute ammonia solution drop wise until the solution is slightly ammoniacal, stir well and allow to stand on the steam bath for 30 minutes.
4. Test the solution for complete precipitation when scarlet red has settled down. Allow the precipitate to stand for one hour while cooling at the same time.
5. Filter the cold solution through previously weighed Gooch (sintered glass) crucible dry it to 110-120° C for 40-45 minutes.
6. Allow to cool in a desiccator and weigh. Note down the yield.

Observations

1. Yield –…..g

2.Colour – Scarlet red

3. Structure



2.Preparation of trans-potassiumdiaquabis(oxalato)chromate(III)

Aim: To prepare trans potassiumdiaquobis(oxalato)chromate (III)

Chemicals required: Oxalic acid & Potassium dichromate

Procedure:

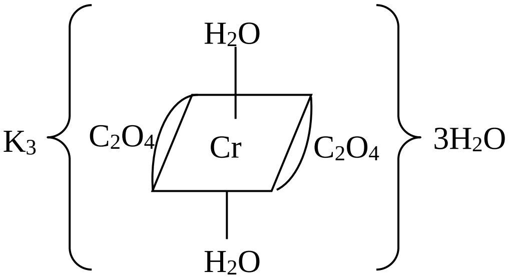
1. Dissolve oxalic acid dihydrate (6 g) in the minimum amount of boiling water(10-15 ml) in 250 cc beaker.
2. Add in small portion a solution of $K_2Cr_2O_7$(2 g) dissolved in the minimum quantity of hot water.
3. Cover the beaker while the violent reaction proceeds.
4. Evaporate the solution to about one half of its original bulk and then allow spontaneous evaporation at room temperature to proceed until the solution is reduced about one third of its original bulk.
5. Filter off the crystals. Wash with cold water and alcohol. Record the yield.

Observations

1. Yield = ……..g
2. Colour – Violet
3. Reaction

$$7H_2C_2O_4.2H_2O + 3K_2Cr_2O_7 \longrightarrow 2K_3[Cr(C_2O_4)_2(H_2O)_2].3H_2O + 6H_2O + 6CO_2\uparrow + 7O_2\uparrow + 2Cr_2O_3$$

Structure



3. Preparation of tris(thiourea)copper(I)sulphate monohydrate.

Aim: To prepare tris(thiourea) copper (I) sulphate monohydrate

Chemicals required: Thiourea and coppersulphate

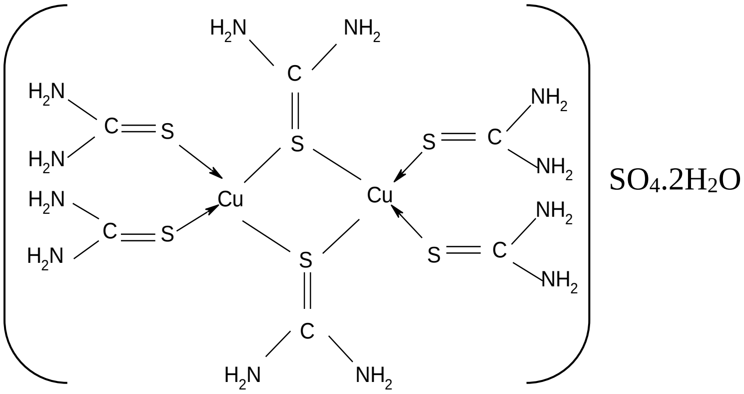
Procedure:

1. Dissolve thiourea (1.0 g) in 7-8 ml(appro.3/4th TT) of distilled water in a test-tube & warm to prepare a solution.
2. Prepare a solution of copper sulphate by dissolving 1.0g in 7-8(appr.3/4th TT) ml of distilled water in a small beaker. Cool both the solutions.
3. **Add copper sulphate** solution slowly **to the thiourea** solution drop wise with constant stirring.(Add a few ml of cold 10% thiourea solution if the color of copper sulphate solution is not discharged completely).
4. Stir the mixture vigorously. Allow to stand for about 20-30 minutes.
5. Filter the white precipitate. Wash the precipitate with water & with alcohol and dry it.
6. Weigh the product & record the yield.

Observations

1. Yield = ….g
2. Colour – colourless
3. Reaction

$$2CuSO_4 + 6\,S = C(NH_2)_2 \twoheadrightarrow [Cu\{(NH_2)CS(NH_2)\}_3]_2\,SO_4.2H_2O$$



4. Preparation of sodiumtris(oxalato)ferrate(III).

Aim: To prepare sodiumtris(oxalato)ferrate(III).

Chemicals required: Ferric chloride, Oxalic acid & NaOH etc.

Procedure:

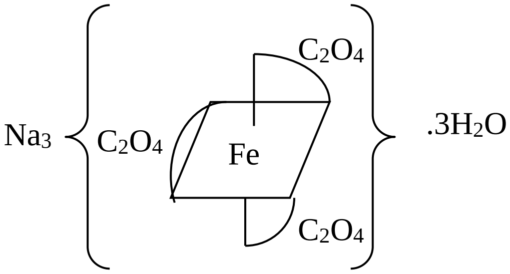
1. Dissolve Ferric chloride (1 g) in 3-5 ml of distilled water in a 250 ml beaker to prepare a solution.
2. Prepare sodium hydroxide(5g) solution in 10 ml of distilled water in a test-tube.
3. Add 5 ml of sodium hydroxide solution to ferric chloride solution in small quantities & stir. The reddish brown ferric hydroxide(hydrated ferric oxide) is obtained. Filter it through a filter paper.
4. Prepare a solution of oxalic acid (2.0g) in 5ml of hot water.
5. Add 5ml of NaOH to oxalic acid solution to obtain sodium oxalate solution.
6. Heat 10-15 ml sodium oxalate solution and pour it on ferric hydroxide precipitate to dissolve it and transfer it to the same beaker by piercing a hole in the filter paper cone and then transfer completely and heat.
7. Filter the *solution if necessary* and concentrate green filtrate or solution in a beaker by heating slowly on sand bath and then in an evaporating dish to get green crystals.
8. Filter off the crystals. Wash with cold water and alcohol. Record the yield.

Observations

1. Yield = ………g
2. Colour – Green
3. Reaction

$$FeCl_3 + 3NaOH \longrightarrow Fe_2O_3 . xH_2O \downarrow + 3NaCl$$

$$Fe_2O_3 + 3Na_2C_2O_4 + 3H_2C_2O_4 \longrightarrow 2Na_3[Fe(C_2O_4)_3].3H_2O$$



IV : Ion Exchange Process:

Separation of Mg(II) and Fe(II) by ion exchange method using anion exchange resin.

Aim: To separate of Mg(II) and Fe(II) by ion exchange method using anion exchange resin and determine the same.

Chemical required: resin (amberlite), 2NNaOH, 2N HCl, HNO_3, EDTA, $K_2Cr_2O_7$

Theory: The term ion exchange means the exchange of ions of like sign between a solution and insoluble solid phase. The solid material is known as ion exchanger, which must have an open permeate molecular structure so that ions and solvent molecules can move freely in and out. Usually the ion exchangers are highly cross-linked organo polymers, which are insoluble in water and in organic solvents and having active ions those exchangers reversibly with other ions in a surrounding solution without any appreciable physical change occurring in the material. Depending upon the nature of the active ions present on the ion exchanger they have been classified in to cation exchanger and anion exchanger. Cation exchanger resin is a highly cross-linked sign molecular weight polymer containing sulphonic, carboxyl, phenolic etc. groups as an integral part of the resin and an equivalent amount of active cations.

Anion exchange resin is a polymeric cation and contains active anions such as chloride, hydroxyl, sulphate etc

Procedure:

Conditioning the resin: The anion exchange resin is taken in a beaker. Wash thoroughly with 2N HCl then by distilled water and finally by 2N NaOH and again by glass distilled water. This process is repeated several times.

Column preparation: The above conditioned resin is taken in a clean burette fill up to 12-14 inches. Initially the column is thoroughly washed with glass-distilled water by passing the water through the column. This is then followed by passing about 80-100 cc of 2N HCl through the column. The rate of elution of drops is adjusted to about 60-70 drops/min.

Separation of Mg(II) and Fe(II):

1. Dilute the given solution of Mg(II) & Fe(II) up to the mark with 2N HCl solution.
2. Pipette out 10 cc of the solution and add to the column. Collect the eluted Mg(II) ions in a 500 cc conical flask. Elute the column with 100cc 2N HCl. Collect the elute in the same flask. The solution in flask containing Mg(II) ions but Fe(II) forms $FeCl_4^{2-}$ which undergoes exchange phenomenon with anion exchange of the resin so that it remains in the resin bed.
3. Add 50 cc of glass distilled water to the ion exchange column and collect the eluent in another 500cc conical flask. Then elute by 100 cc of 0.025N HNO_3.

4. The eluent is collected in the second conical flask, which is containing Fe(II) ions. It should be noted that the resin bed should not be allowed to dry during the experiment.

Method of determination:

i. Take the Mg(II) solution present in first conical flask. As it is strongly acidic in nature, it is neutralized with 2N NaOH. Add 2N NaOH drop by drop to Mg(II) solution till to get a slight precipitate.
ii. Add 2-3 cc of buffer solution (pH=10) and 4-5 drops of eriochrome black –T indicator to Mg(II) solution. Titrate it against standard 0.1M EDTA solution till the colour changes from wine red to blue.
iii. Titrate another solution containing Fe(II) solution against standard solution of $K_2Cr_2O_7$ in presence of sulphuric acid using diphenylamine indicator and phosphoric acid.
iv. With the help of burette readings, calculate the amount of Mg(II) and Fe(II) ions in the solution.

Reference Books:

1. A Text Book of Quantitative Inorganic Analysis (3rd edition) - A.I. Vogel
2. A hand book of Analytical Chemistry – Subhash & Satish & Lal
3. Elementary Practical Chemistry –G.D.Sharma and Arun Bahl
4. Practical Inorganic Chemistry –Preparation, Reaction and Instrumental methods – Geofrey& Haydn Sutcliff (For preparations of complexes)

B.Sc. VI :Paper- II

Organic Experiments

SEPARATION AND QULITATIVE ANALYSIS OF LIQUID-LIQUID ORGANIC BINARY MIXTURES

Total No of hours/week : 4Hrs
Total No. of Hours : 54 Hrs

Total No of hours/week : 4Hrs
Total No. of Hours : 54 Hrs

CONTENTS
Separation of organic liquid binary mixture by distillation. Characterization of any one separated compound through preliminary tests, element test, physical constant, functional group test and preparation of suitable derivative and its physical constant. Low Boiling Liquids : Ethyl acetate, Acetone, Toluene, Chlorobenzene. High Boiling Liquids: Phenol, Aniline, Nitrobenzene, Benzaldehyde, Acetophenone, Bromobenzene.

Instructions:

In a batch of ten students, in the practical examination, five students may be given experiment number 1-6 (binary mixture) and remaining five students may be given physical experiments. In a batch of five students in the practical examination, not more than two students should get the same experiment.

SCHEME FOR PRACTICAL EXAMINATION

DISTRIBUTION OF MARKS	
Separation	03
Preliminary tests	02
Nature	02
Element test	04
Physical constant	03
Functional Group test	04
Identification and Structure	03
Preparation of derivative	03
Physical constant of derivative	03
Systematic Presentation	03
Journal	05
Viva voce	05
TOTAL	40

NOTE: In a batch of ten students, not more than two students should get the same mixture in the practical examination. Viva questions may be asked on any of the experiments prescribed in the practical syllabus. During practical examination chart may be referred whenever necessary.

SEPARATION OF ORGANIC LIQUID BINARY MIXTURE BY DISTILLATION

Principles of separation:

Liquid-Liquid binary mixture: If mixture is a given is homogeneous, it is possible that both components are liquids or one liquid and other solid that has dissolved in the liquid on mixing.

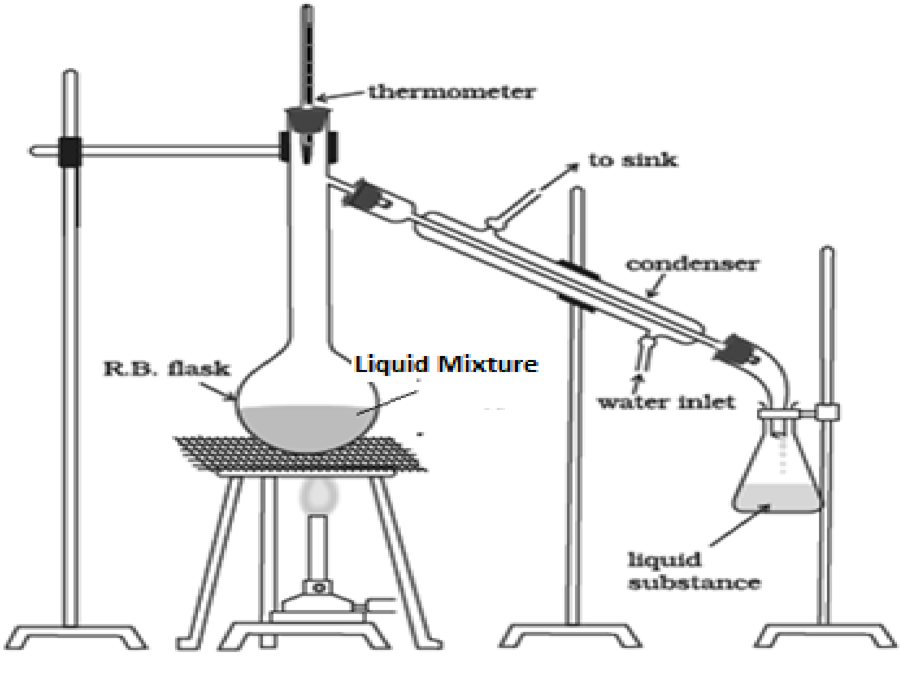
When both components are liquid, the mixture is separated by fractional distillation. The distillation should be carried out slowly and carefully.

Scheme for actual separation of organic liquid- liquid mixture:

DISTILLATION OF A LIQUID MIXTURE

Place 15-20 cc of an unknown liquid mixture (say *10cc P + 10 cc Q*) that is to be purified by simple distillation and for which the boiling point range is to be determined.

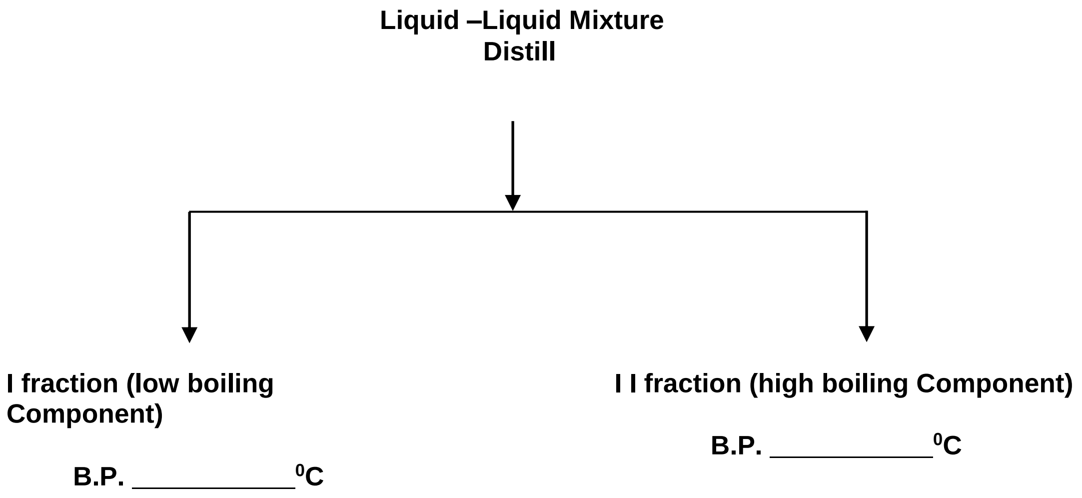
Step 1: Assemble the distillation apparatus (simple or fractional). Transfer the unknown liquid to a 50 cc round bottom flask (*this will be the distilling pot*). Add one boiling chip, and proceed to distill the liquid into a 10 cc graduated cylinder (*this will be the receiver*). Check the position of the thermometer (*the bulb of the thermometer must be below the arm of the distillation head*) and make sure that the bottom of the distillation pot touching the heating surface of the heating set. Now fix the condenser along with rubber tubes for water circulation. As shown in figure.



Step-2: Slowly turn on the water for condenser, and begin heating. Adjust the heating set to maintain a distillation rate of one drop per second. As the lower boiling component is distilled, the boiling point of the mixture in the distillation flask will increase. *Record the temperature after the first drop is collected and again after every 2 ml of distillate is collected. Collect at least 10 ml of distillate in a separate test tube labelled as Low boiling fraction* (component P). PRESERVE IT.

Step 3: Collect the next 10 ml of distillate, again recording the temperature after every 1 ml of distillate. Collection of last portion of distillate should continue until the temperature remains constant. If the distillation flask is approaching dryness, remove the heat source immediately and after cooling, transfer the distillate and any remaining liquid from the flask to the third test tube (component Q). KEEP IT.

Determine the boiling point range of the first fraction of the collected liquid and the third portion of the collected liquid. Identify the unknowns by their boiling points using the possible boiling points of compounds by referring the literature.



Qualitative analysis of Organic Compounds

After separation (distillation) of the organic compound from the binary mixture, the individual compound is systematically analyzed. The process of analysis / identification of an organic compound is called "organic spotting". The purpose of organic qualitative analysis is to spot a given organic substance and to substantiate its nature by performing a set of reaction/s with it. The frame work for qualitative analysis of the given organic compound will proceeds follows.

I) PRELIMINARY TESTS

S.N	Test	Observation	Inference
1.	State	Liquid	*Low boiling liquids*; Acetone, Ethyl acetate may be present. *High boiling liquids*; Aniline, Phenol,Acetophenone, Nitrobenzene,Toluene, Benzaldehyde, bromobenzene, Chlorobenzene may be present.
2.	Colour	Colourless Yellow Reddish/Brown	Benzaldehyde, Acetone, Acetophenone, Ethyl acetate, Toluene, Chlorobenzene may be present. Nitrobenzene, bromobenzene may be present. Phenol,Aniline may be present
3.	Odour	Phenolic Fishy Pleasant /Fruity Bitter almond	Phenol Amines (Aniline) may be present. Acetone, Acetophenone, Ethyl acetate, Bromobenzene, Chlorobenzene Benzaldehyde, Nitrobenzene
4.	Beilstein's Test : Heat a loop of copper wire till it does not impart green colour to the flame. Cool, and dip in the liquid and then heat it.	Burns with non-sooty flame Burns with sooty flame Burns with sooty flame followed by green edged flame	Aliphatic compound Aromatic compound Halogenated aromatic compound
Therefore, the given compound is --------------			
5.	Solubility Test		
i	Liq. + Water	Miscible in cold solution acidic to litmus	Acetic acid may be present

		Miscible in cold and neutral to litmus Immiscible	Acetone and ethylacetate may be present Phenol, aniline, toluene, chlorobenzene, benzaldehyde etc; may be present.
ii	Liq. + $NaHCO_3$ Solution	Miscible with effervescence	Acids present
	Above Sol. + dil HCl	Reappearance of oily drops or turbidity	Acid confirmed
iii	Liq. + NaOH	Miscible	Phenol present
	Above Sol. + dil HCl	Reappearance of oily drops or turbidity	Phenol confirmed
iv	Liq. + 1:1 HCl	Miscible	Base present
	Above Sol. + NaOH	Reappearance of oily drops or turbidity	Base confirmed
(if all the above tests are negative the nature of the given compound is NEUTRAL) Note: A.S. = Above solution Conclusion: The given compound is____________________________ (Acid/Phenol/Base/Neutral)			
6.	Test for Un-saturation.		
i	Br_2 water test: 2-3 drops of liquid + few drops of Br_2 water. If it doesn't give test treat with Bromine in carbon tetra chloride	Decolourisation of Br_2 water No decolourisation	Unsaturated compound Saturated compound
ii	Alkaline $KMnO_4$ test : Dissolve the compound in hot water + few drops of very dilute alkaline $KMnO_4$ solution	Decolourisation of $KMnO_4$ solution No decolourisation	Unsaturated compound present Saturated compound

Conclusion: The given compound is____________________________ (Saturated/Unsaturated)

II. **Determination of physical constant:**

Using Thiel's tube the *boiling point* of given compound under investigation is determined.

Boiling point of the compound is ……….. ^{0}C

III. Detection of Elements:

Generally organic compounds contain Nitrogen (N), Halogen (X) and Sulphur (S) along with Carbon, Hydrogen and (Oxygen). For the detection of N, X, and S the Lassaigne's test is performed.

Lassaigne's Test :

Take a small piece of clean and dry Sodium metal in a fusion tube and heat it slowly till the metal fuses. Cool and add 2-3 drops of liquid under investigation. Heat continuously till the fusion tube becomes red hot. Plunge the red hot fusion tube into about10 ml of distilled water taken in an evaporating dish. Break the fusion tube with a glass rod and boil the mixture for about 5 min and filter. The filtrate is called Sodium Extract (S.E) and use it for the test for Nitrogen, Halogen/s and Sulphur.

Test for Nitrogen : 1 cm^3 of S.E. + 1 cm^3 of freshly prepared $FeSO_4$ + 1 drop of NaOH soln. Boil and cool. Add a few drops of $FeCl_3$ and acidify with Conc. H_2SO_4 or Conc. HCl.	Green or blue colouration. (Prussian blue colour)	Nitrogen present
Test for halogens : 1 cm^3 of S.E. + dil HNO_3 boil and cool + $AgNO_3$ solution.	a) Curdy white ppt. easily soluble in NH_4OH b) Pale yellow ppt. sparingly soluble in NH_4OH c)Yellow ppt insoluble in NH_4OH	Chlorine is present Bromine is present Iodine is present
Test for Sulphur : S.E. (2ml) + 2-3 drops of sodium nitroprusside solution.	Violet colouration	Sulphur present

Conclusion: The elements present in the compound are C, H, (O) and ....

IV. Detection of Functional Group

It can be done on the basis of elements present in the compound, its nature and they are divided into following divisions.

Division I - C, H, & (O)

Division II – C, H, (O) & N

Division III – C, H, (O) and Halogen

The given compound contains the elements C,H (O) & … The compound belongs to the division ………

V) DETECTION OF FUNCTIONAL GROUPS

Division I - C, H, & (O)

[Phenols, Neutral (Aldehydes, Ketones, Esters & Aromatic Hydrocarbons)].

B) Test for Phenols

i)Br_2 Water Test : Dissolve the given Compound in water or in acetic acid + Bromine water and observe	White ppt	Phenol is present
ii) Alcoholic $FeCl_3$ Test : Dissolve the given Compound in water or in acetic acid + alcoholic $FeCl_3$ solution and observe.	Violet Colouration	Phenol is present

Confirmatory tests for Phenols)		
i)**PhthaleinFusionTest:** Compound (1-2 drops) + a pinch of phthalic anhydride + 2 drops of conc. H_2SO_4, heat gently, cool, pour it in a beaker containing water and NaOH (5 drops)	Red (Pink) Colour	Phenol is present and confirmed
ii) **Leiberman's Nitroso Test** Compound (2-3 drops) + $NaNO_2$, heat gently, cool + Con. H_2SO_4 (5 drops)	A deep green to blue solution is formed at first which turns red when poured in to water containing few drops of NaOH	Phenol is present & confirmed.
C) Test for neutral compounds containing C,H& (O) (Aldehydes, Ketones & Esters)		
Brady's reagent Test: Compound + 2,4:DNP	Yellow crystalline ppt.	Benzaldehyde or (Ketones) Acetone or Acetophenone present.
Schiff's reagent test: Compound(1 drop)+Schiff's reagent(2-3 drops) and shake the mixture well. Keep for a while.	Pink colouration	Benzaldehyde is present.
	No Pink colouration	Acetophenone is present.
C.T. for Benzaldehyde:		
Silver mirror test (*Tollen's reagent test) : Compound(1 drop) +Tollen's reagent. Warm the mixture on a water bath without disturbing.	Silver mirror or grey ppt.	Benzaldehyde is present & confirmed
*Preparation of Tollen's reagent : Mix equal volume of 10% aqueous $AgNO_3$ (1 ml) & dil NaOH (1 ml) Add dilute NH_4OH drop wise till the brown ppt. just dissolves to get a clear solution.		
Ketones – Acetone & Acetophenone		
Aliphatic compound-Acetone, Aromatic compound-Acetophenone		
Compound (1-2 drops) + Sodium Nitroprusside solution(5 drops) + few drops of NaOH.	Red colouration Red coluration changes to blue on adding acetic acid	Acetone is present Acetophenone is present
<u>**C.T. for Acetophenone**</u>		
Brady's reagent Test: Compound + 2,4:DNP	Yellow ppt.	Acetophenone is present and confirmed.

C.T. for Acetone		
ii) Iodoform test: Compound(3-4 drops) + I_2 in KI solution till yellow colour persists + NaOH, heat the solution gently.	Yellow ppt.	Acetone is present and confirmed.
Esters - Ethyl Acetate		
Compound (5drops) + 1-2 drops of phenolphthalein and one drop of very dil. NaOH (Diluted 10 times), heat	Pink colour is formed, which disappears on heating due to the free acid formed by the hydrolysis of esters.	Ethyl acetate is present
C.T. Ethyl Acetate		
Feigl Test : 1-2 drop of compound + Hydroxylamine hydrochloride Solution(5 drops) + 5 drops of KOH in methanol solution. Boil for a minute, cool & acidify with dil HCl. + 1-2 drops of $FeCl_3$	Violet colouration	Ethyl acetate is present and confirmed
Test for Neutral compounds containing C & H only (Aromatic Hydrocarbons)		
Hydrocarbons(Toluene)		
Compound + Conc.H_2SO_4	Insoluble	Toluene is present
C.T. for Toluene		
Compound + Picric acid in Benzene shake well.	Yellow ppt.*	Toluene is present & confirmed
*Take it as picrate derivative with M.P. = 88°C		
Division II - C, H, (O) & N (Bases & Neutral compounds)		
Base – Amines (Aniline)		
Compound + dil HCl	Dissolves completely and reprecipitated by NaOH	Base (Amine) is present
Compound (2-3 drops)+ $K_2Cr_2O_7$ (pinch)+ conc.H_2SO_4(3-4 drops) shake well.	Blue or Black colour	Anilne is present.

C T. for Aniline (Test for -NH_2 group)		
Azo-dye test: Compound + con. HCl (1:1) cool in ice + 10% ice cold $NaNO_2$ solution + 2-naphthol in NaOH.	Orange Red dye	Aniline present and confirmed (-NH_2 group is present)
Neutral -- Nitro Benzene		
Mulliken's Test (Neutral reduction test : Dissolve the Compound (4 drops) in a hot 50% aqueous alcohol + 5-6 drops of 10 % $CaCl_2$ + pinch of Zn dust Boil the mixture for a minute. Filter and test the filtrate with Tollen's reagent.	A black ppt.or grey ppt.	Nitro(-NO_2) group (Nitro benzene) is present.
C.T. for Nitro Benzene		
Compound(5 drops) + Glacial acetic acid(1 ml) + pinch of Zn dust, Boil cool, & add water(1 ml) +NaOH till alkaline + Sodium nitroprusside (2-3 drops)	Violet colouration	Nitro benzene is present and confirmed
DIVISION – III (C, H and Halogens(Br or Cl) (Bromobenzene or Chlorobenzene)		
Test for Bromobenzene		
Beilstein's Test: (Test for aliphatic or aromatic) Heat a small piece of copper foil in a non-luminous flame using pair of tongs until it imparts no colour to the flame. Cool, dip into the given organic compound and again hold it to the flame and observe	Burns with Sooty(smokey) flame followed by green edged flame	Bromobenzene or Chlorobenzene present
Compound + Alcoholic $AgNO_3$ & mix & warm	Pale yellow ppt. A white curdy ppt.	Bromobenzene is present Chlorobenzene is present
C.T. for Bromobenzene		
Compound (4 drops) + 2 ml of fuming HNO_3 (or 1 ml of con. H_2SO_4 + 1 ml of Con. HNO_3) Heat for 5 minutes, cool and pour it into water.	Yellow solid	Bromobenzene is present and confirmed

C.T. for Chlorobenzene		
Compound (4 drops) + 2 ml of fuming HNO_3 (or 1 ml of con. H_2SO_4 + 1 ml of Con. HNO_3) Heat for 5 minutes, cool and pour it into water containing ice pieces.	*Yellow solid	Chlorobenzene is present and confirmed
*Take it as derivative p-nitro-chlorobenzene with M.P.=83 ^oC		

VI. BROAD INFERENCE

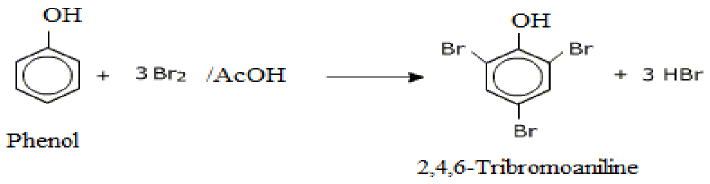
S.N	**Particulars**	**Inference**
1.	Nature (Acid/ base/phenol/Neutral)	
2.	Aliphatic / Aromatic	
3.	Saturated or Unsaturated	
4.	Physical constant of compound	Observed B.P = …^0C Literature B.P =…. ^0C
5.	Elements present	
6.	Functional group	
7.	Molecular formula of the compound	
8.	Structural formula of the compound	
9.	Name of the compound	
10.	Name of the derivative	
11.	Structure of the derivative	
12.	Physical constant of the derivative	Observed MP = …^0C Literature MP =…. ^0C

PREPARATION OF DERIVATIVES

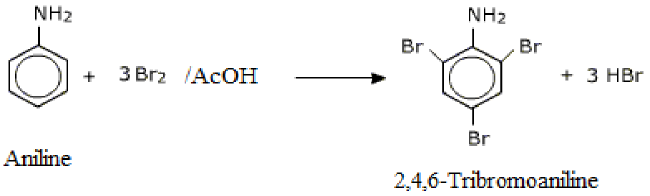
1. Bromo derivative for Phenol and Aniline

Dissolve about 1ml of aniline or phenol in acetic acid and take this content in 100 c.c. conical flask. Add strong bromine solution (bromine in acetic acid) until, after shaking, the liquid is pale yellow. Add 50 c.c. water, cool and shake vigorously. Filter and wash the bromo-derivative with water. Recrystallise the product from alcohol.

For Phenol

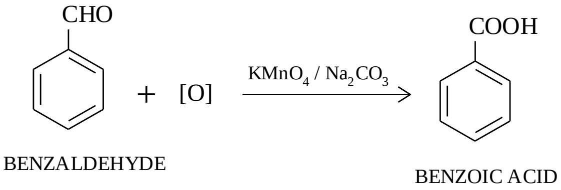


For Aniline



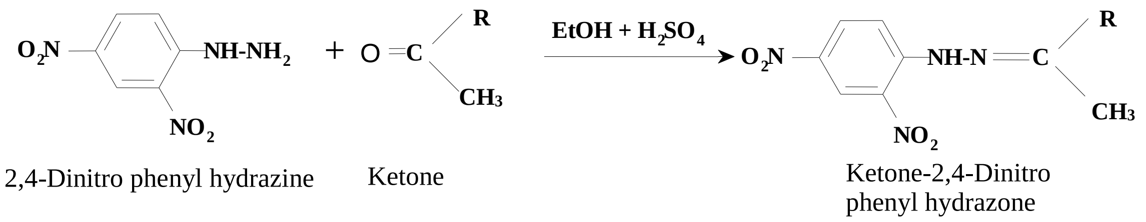
2. Benzoic acid from Benzaldehyde

Take 1ml of benzaldehyde in a 100ml. conical flask and add about 10ml. of 10% Na_2CO_3 and boil the solution by placing boiling chips. To, the boiling solutions add about 15ml of $KMnO_4$ gradually till the solution contains a little excess of potassium permanganate. Filter of the precipitated hydrated MnO_2 and few drops of SO_2 water to remove excess of $KMnO_4$. Filter and acidify the filterate, on cooling, the acid precipitates. Recrystallise from hot water.



3. 2,4-D.N.P – derivative for Acetone and acetophenone

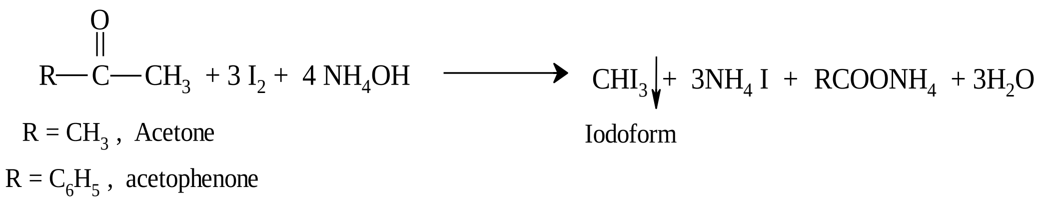
Take about 5 ml of 2,4-DNP solution in a test tube. Add 5-6 drops of the given liquid (acetone or Acetophenone) shake well and warm it for few minutes. Cool and filter the precipitate thus formed. Recrystallise it from alcohol.



If R = CH_3 ; Acetone and R = C_6H_5 Acetophenone

4. Iodoform derivative for Acetone and Acetophenone

To about 5-6 drops of the liquid add 10ml NH_4OH. Add iodine solution drop by drop till the solution is distinctly yellow. Warm gently on water bath. When iodoform a yellow crystalline solid , separates in short time. Filter, dry and take M.P.



$$R-\overset{\overset{\large O}{\|}}{C}-CH_3 + 3I_2 + 4NH_4OH \longrightarrow \underset{\text{Iodoform}}{CHI_3}\downarrow + 3NH_4I + RCOONH_4 + 3H_2O$$

R = CH_3 , Acetone

R = C_6H_5 , acetophenone

5. Iodoform derivative for Ethyl acetate

Hydrolyse 1ml of ethyl acetate with 50 ml of 10% NaOH by gently boiling under reflux for 1 hour. A mixture of ethyl alcohol and sodium acetate are formed. Completion of hydrolysis is indicated by the formation of a homogeneous solution.

Take about 1 ml of above hydrolysed solution, add 10% of potassium iodide solution and 5ml of freshly prepared sodium chlorite solution. Warm for few minutes and cool. Yellow crystals of iodoform are produced. Filter and collect it as derivative.

HYDROLYSIS OF ESTER

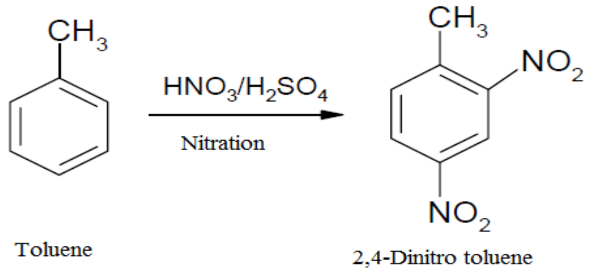
$$\underset{\text{Ethyl acetate}}{CH_3COOC_2H_5} + NaOH \longrightarrow \underset{\text{Sodium acetate}}{CH_3COONa} + \underset{\text{Ethanol}}{CH_3CH_2OH}$$

IODOFORM REACTION

$$\underset{\text{ethanol}}{CH_3CH_2OH} \xrightarrow{I_2\ /\ NaOH} \underset{\text{ethanal}}{CH_3CHO} \xrightarrow{I_2\ /\ NaOH} CI_3CHO \longrightarrow \underset{\text{iodoform}}{CHI_3} + HCOOH$$

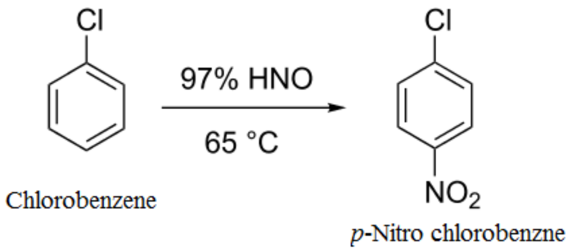
6. 2,4-Dinitrotoluene from toluene

To 5ml of nitrating mixture (1:1 Conc. H_2SO_4 + Fuming Nitric acid), add 1ml of toluene in small lots with shaking after each addition. Cool in ice –water, by maintaining temperature 10^0C. Heat for two minutes and pour into about 50 ml. of cold water. Filter, wash and crystallise from alcohol.



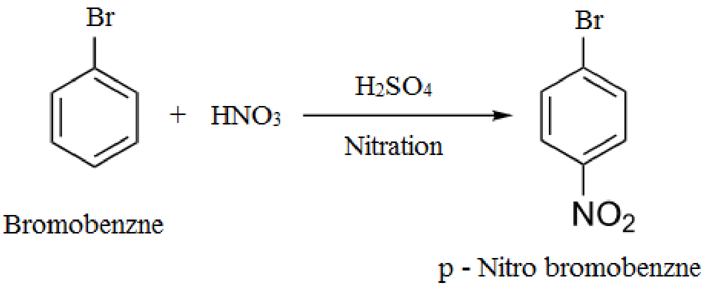
7. *p*-Nitrochlorobenzene from Chlorobenzene

4-5 drops of chlorobenzne + 2ml of fuming nitric acid. Heat for 5-10 minutes on water bath and pour into 10 ml. water. Separated solid is Filter and dry. Recrystallise from ethanol.



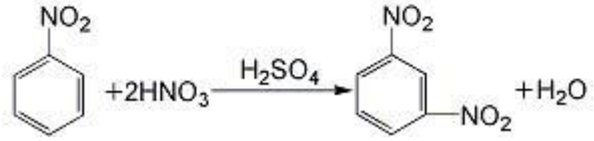
8. *p*-Nitrobromobenzene from Bromobenzene

4-5 drops of Bromobenzne + 2ml of Conc. HNO_3 and Conc. H_2SO_4 shake well, and Heat for 2 minutes on water bath and pour into 10 ml. water. Separated solid is Filter and dry. Recrystallise from ethanol.



9. *m*-Dinitrobenzene from Nitrobenzene

4-5 drops of nitrobenzene dissolved in 1 ml of Conc. H_2SO_4 in a dry test tube and add a mixture of 1ml of Conc. HNO_3 and 1ml of Conc. H_2SO_4 and add few drops of fuming nitric acid shake well. Heat for 2 minutes at 100^0C and pour into finely crushed ice in a beaker. Cool thoroughly and scratch by means of a glass rod when the oily suspension solidifies. Filter and recrystallise from alcohol.

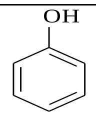
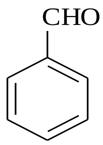
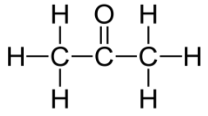
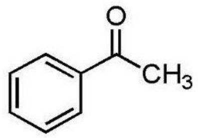
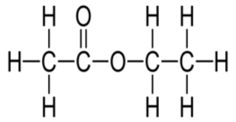
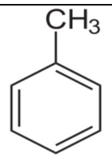
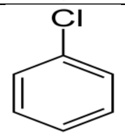
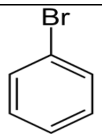
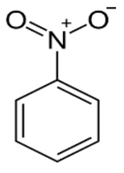
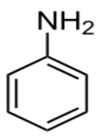


References:

1. A Text book of Practical Organic Chemistry- By Arthur I .Vogel, IVth Edn. ELBS, 1978 Longman Group Ltd.
2. Organic Experiments VIIth Edition Louis F. Fieser Late Professor Emeritus Harvard University Kenneth L Williamson Mount Holyoke College

   D. C.HEATH AND COMPANY Lexington, Massachusetts Toronto
3. Systematic Lab experiments in Organic Chemistry- ArunSethi
4. Practical Organic Chemistry – Nadkarni and Kulkarni
5. Advanced Practical Organic Chemistry – N.K.Vishnoi
6. Practical Chemistry -.O.P.Pandey,D.N.Bajpai & S.Giri
7. A hand book of Analytical Chemistry– Subhash & Satish

Name, Structure and M.P. of derivatives of Organic Compounds

Substance	B.P. (^{0}C)	Mol. Formula	Str. Formula	Derivative (in M.P.)
1. Phenol	183-184	C_6H_5OH		2,4,6-Tribromo phenol **(95-97^{0}C)**
2. Benzaldehyde	179-180	C_6H_5 CHO		Benzoic acid **(120-122^{0}C)** Or 2,4-D.N.P derivative **(237-239^{0}C)**
3. Acetone	56-58	CH_3-CO-CH_3		Iodoform **(119-121^{0}C)** or 2,4-D.N.P derivative **(126-128^{0}C)**
4. Acetophenone	202-204	C_6H_5-CO-CH_3		Benzoic acid **(120-122^{0}C)** or 2,4-D.N.P derivative **(249^{0}C)**
5. Ethyl acetate	77-79	CH_3-$COOC_2H_5$		Iodoform **(119-120^{0}C)**
6. Toluene	110-112	C_6H_5-CH_3		2,4-Dinitrotoluene **(70-72^{0}C)**
7. Chlorobenzne	132-134	C_6H_5-Cl		*p*-Nitrochlorobenzne **(83-84^{0}C)**
8. Bromobenzne	155-157	C_6H_5-Br		*p*-Nitrobromobenzne **(126-127^{0}C)**
9. Nitrobenzene	209	C_6H_5-NO_2		*m*-Dinitrobenzne **(90-92^{0}C)**
10. Aniline	184	C_6H_5-NH_2		2,4,6-Tribromoaniline **(119-121^{0}C)**

B. Sc. VI Sem : Paper – II

EXPERIMENTS IN PHYSICAL CHEMISTRY

Total No of Hours/Week : 04 Hours	Practical:40 Marks
Total No of Hours : 26 Hours	IA :10 Marks

Expt. No	Experiments	Page No.
1	Determination of the concentrations of given acids in a mixture of HCl+CH_3COOH conductometrically using the standard(0.5N) NaOH	226
2	Determination of solubility of sparingly soluble Salt, $BaSO_4$ / $PbSO_4$ conductometrically.	228
3	Determination of redox potentials of Fe^{3+}/Fe^{2+} using of $FeSO_4.7H_2O$ solution (≈ 0.1N) by potentiometric titration against the standard solution of $K_2Cr_2O_7$ (0.1N).	230
4	Determination of the solubility and solubility product of sparingly soluble salts (AgCl) potentiometrically.	232
5	Determination of the percentage composition of unknown mixture of A & B liquids using Abbe's refractometer by formula method.	234
6	Determination of the percentage composition of unknown mixture of A & B liquids using Abbe's refractometer by graphical method.	236
7	Determination of pK_a of acetic acid potentiometrically.	238

Scheme of Marking:

Accuracy	= 18
Proper Technique and Presentation	= 03
Calculation (Calculation + Graph)	= 09 (5+4)
Viva voce	= 05
Journal	= 05
Total	= 40 Marks

NB: 1. Scientific calculators are not allowed.

2. Use A4 size graph sheets.

Note: Paper –II has experiments related to Physical and Organic exercises with equal weightage. Hence, they are to be allotted on the basis of lot (For a batch of ten students, five Physical and five organic)

Expt No 1: CONDUCTOMETRIC TITRATION (Acid -Base)

Aim: To determine the concentrations of given acids in a mixture (HCl + CH_3COOH) conductometrically using standard NaOH

Chemicals: ~ 0.1N HCl, ~ 0.1N CH_3COOH and 0.5N NaOH.

Apparatus: Conductometer, conductivity cell, glass rod, pipette, burette, beaker etc.

Theory: This type of titration is just a combination of two separate titrations viz. HCl against NaOH and CH_3COOH against NaOH. By adding alkali to the mixture, conductivity of the solution decreases due to replacement of H^+ ions from strong acid. It then increases slowly as the weak acid converts into salt and finally rises steeply as the excess of alkali is added.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

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

A graph of conductivity against volume of alkali gives three straight lines and the intersection of these lines give end points. It must be noted that the first end point will be that of HCl (strong acid) and second is for CH_3COOH (weak acid).

Procedure:

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 25cc each of 0.1N HCl & 0.1 N CH_3COOH into a 100cc beaker.
5. Place the conductivity cell and connect to the terminals of the conductivity meter.
6. Note the conductance of the solution before adding the alkali in mS.
7. Rise up the electrodes and add 0.5cc of the NaOH solution from micro burette carefully and stir the solution with glass rod. There may be a slight heating effect due to neutralization and hence, wait for 30 seconds to cool. Note down the conductance.
8. Now add 0.5cc of 0.5 NaOH solutions every time from the burette into the above solution with stirring. Note the conductance of the solution for every addition.
9. Continue the titration by adding 0.5cc at a lot up to 15cc and record the conductance for every addition.
10. Plot a graph of conductance against volume of NaOH added. Find out the end point from the intersection of lines.
11. First inter section will be the end point for HCl (strong acid) and second for CH_3COOH (weak acid).

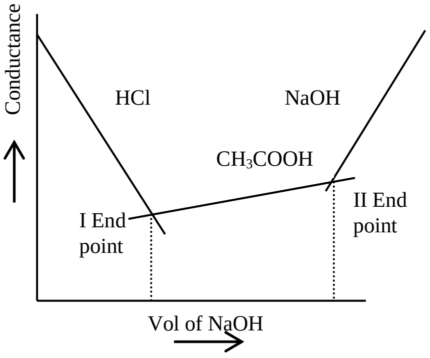
12. Calculate the normality and amount of acids.

Observation:

Total volume of acid mixture = 25cc 0.1N HCl + 25cc 0.1 N CH_3COOH=50.0cc

Volume of alkali added (cc)	Observed Conductance (mS)
0.0	
0.5	
1.0	
1.5	
2.0	
.	
.	
.	
15.0	

Nature of the Graph



Calculation:

(i) For HCl (I End point)

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{0.5 \times \text{ B.R from graph}(V_1)}{50}$$

(ii) For CH_3COOH (II End point)

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{0.5 \times \text{B.R from graph}}{50\text{cc}} = \frac{0.5 \text{ x } (V_2 - V_1)}{50}$$

(iii) The amount of HCl = Normality of HCl × Equivalent mass of HCl

= --------x 36.5

=- --------g/dm^3

(iv) The amount of CH_3COOH = Normality of CH_3COOH × Equivalent mass of CH_3COOH

= -------- x 60

= ---------g/dm^3

Results:

1. Normality of HCl = ………….N
2. Amount of HCl = ………… g/dm^3
3. Normality of CH_3COOH = ……………N
4. Amount of CH_3COOH = ………… g/dm^3

Expt. No. 02: CONDUCTOMETERY (Solubilty-$BaSO_4$)

AIM:- To determine the solubility and solubility product of Sparingly soluble salt ($BaSO_4$) conductometrically.

Chemicals: - $BaSO_4$ powder, distilled water and 0.1N KCl.

Apparatus: Conductometer, conductivity cell, glass rod, beaker etc.

Theory: The solubility of sparingly soluble salts like AgCl, $BaSO_4$, $PbSO_4$ etc can be determined by conductometric measurements. As the solubility of the sparingly soluble salt is extremely low, a small quantity that is dissolved in saturated solution may be regarded as present at infinite dilution. Thus, its equivalent conductance λ_v may be taken as the equivalent conductance at infinite dilution λ_∞.

Thus, $\lambda_v = \lambda_\infty = \lambda_+ + \lambda_-$

Knowing the specific conductance, the solubility of sparingly soluble salt i.e $BaSO_4$ can be calculated.

Concentration of sparingly soluble salt = Solubility, $S = \frac{1000K}{\lambda^0_m}$

Where, 'λ^0_m' is molar conductance at infinite dilution.

Procedure:

A) Determination of cell constant :

1. Switch on the conductivity meter for stabilization.
2. Calibrate the conductivity meter if necessary.
3. Wash the electrode of the conductivity cell with distilled water.
4. Pipette out 50cc of 0.1N KCl solution to 100cc beaker.
5. Place the conductivity cell in the beaker and connect it to the terminals of conductivity meter.
6. Note down the conductance of the solution in mS.
7. Calculate the cell constant.

B). Determination of Solubility of $BaSO_4$

1. Measure the conductance of conductivity water (distilled water).
2. Grind 2 g of $BaSO_4$ to fine powder. Add conductivity water. Stir well and allow the solid to settle down. Decant the supernatant liquid and reject it.
3. Wash the $BaSO_4$ paste three to four times with fresh distilled water to dissolve out all the soluble impurities.
4. Add about 50cc of conductivity water to the above paste; warm the solution gently with stirring for about 5 minutes (solution becomes saturated).

5. Allow the heavier particles to settle down by cooling to room temperature. Decant the solution and determine the conductance.
6. Repeat the same procedure for two more times for the same paste and record the conductance of the solution as above.

Observations:

A) **Determination of cell constant :**

1. Observed conductance of 0.1N KCl soln. = ……x 10^{-3} S.
2. Specific conductance of 0.1N KCl soln. at room temperature = 0.01288 S cm^{-1}
3. Determination of cell constant:

$$\text{Cell constant} = \frac{\text{Specific conductance of 0.1N KCl}}{\text{Observed conductance}} = \frac{0.01288}{\text{Observed conductance}} = \text{-------------}\ cm^{-1}$$

B). Determination of Solubility of $BaSO_4$

1. Observed conductance of conductivity water, C_1=……x 10^{-6} S
2. Equivalent conductance of $BaSO_4$ at infinite dilution

$$\lambda_\infty = \lambda_{Ba^{2+}} + \lambda_{SO_4^{2-}} = 64.3+80.0 = 144.3$$

3. Molar Conductance of $BaSO_4$ at infinite dilution

$$\lambda^0_m = \lambda_\infty \times 2 = 144.3 \times 2 = 288.6$$

Sl.No	Observed Conductance of solution (C_2)	Actual conductance = C_2-C_1	Specific Cond (κ) = Cell const × Actual Conductance	Solubility, S = $\frac{1000\,\kappa}{\lambda^0_m}$ (mol / dm^3)
1				
2				
3				
Average value of S				

Calculations:

1. Solubility product (K_{sp}) of $BaSO_4$ = (Solubility)^2 = …………. mol^2/dm^6
2. The solubility of $BaSO_4$ in g/ dm^3 = solubility x molar mass of $BaSO_4$
   = ------- x 233.3

Result:

1. Solubility of $BaSO_4$ = ------------ g/ dm^3
2. Solubility product (K_{sp}) of $BaSO_4$ = …………. mol^2/dm^6

Note: Expected value of solubility of $BaSO_4$ = 2.5×10^{-4} mol / dm^3

Expt No. 3 POTENTIOMETRIC TITEATION (Redox)

Aim: Determination of redox potentials of Fe^{3+}/Fe^{2+} using of $FeSO_4.7H_2O$ solution (≈0.1N) by potentiometric titration against the standard solution of $K_2Cr_2O_7$ (0.1N).

Chemicals: 0.1N $K_2Cr_2O_7$, ≈ 0.1N $FeSO_4$ and 4N H_2SO_4.

Apparatus: Potentiometer, calomel electrode, platinum electrode, burette, pipette, beaker etc.

Theory: When an inert electrode is inserted in a solution containing $Fe^{2+}$and Fe^{3+} ions, potential develops. Its oxidation potential can be given as:

$$E\ (Fe^{3+/}Fe^{2+}) = E^o(Fe^{3+}/Fe^{2+}) + 0.0594\ \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

If a reference electrode like calomel is connected to the platinum (indicator) electrode dipped in the Fe^{2+} solution containing H^+, a cell is set up which can be represented as below:

$$^{-}\ Hg\ /\ Hg^+\ //\ Fe^{3+}\text{-}\ Fe^{2+}\ /\ Pt\ ^{+}$$

Therefore, $E_{cell} = (E^o(Fe^{3+}/Fe^{2+}) - E^0_{Cal}) + 0.0594\ \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$

EMF of the cell depends on the ratio of $[Fe^{3+}] / [Fe^{2+}]$. When the potassium dichromate solution is added, Fe^{2+} ions are oxidized to Fe^{3+} and thereby EMF will increase slowly. At the end point, there will be a sharp change due to the conversion of all the Fe^{2+} ions to Fe^{3+} ions. Graph of EMF v/s volume of $K_2Cr_2O_7$ gives a sigmoid curve (S-type) and $\Delta E/\Delta V$ v/s volume of $K_2Cr_2O_7$ added gives a sharp peak, the equivalence point. From the sigmoid curve, the half equivalence point can be determined.

Therefore, $E_{cell} = (E^o(Fe^{3+}/Fe^{2+}) - E^0_{Cal})$

Reaction: $Cr_2O_7^- + 14\ H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7\ H_2O$

$$6Fe^{2+} \longrightarrow 6Fe^{3+} + 6e^-$$

$$6Fe^{2+} + Cr_2O_7^{2-} + 14\ H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7\ H_2O$$

Procedure:

1. Pipette out 25cc of given ferrous sulfate solution in 100cc beaker and add 25 cc of 4N H_2SO_4 to it. Fill the burette with 0.1N $K_2Cr_2O_7$ solution.
2. Switch on the potentiometer and standardize the potentiometer by inserting two banana plugs into the sockets at channel I or II and adjust the EMF to 1.018V with the calibration screw. Remove these plugs after standardization.
3. Place the platinum electrode in the above ferrous sulfate solution and calomel electrode in another beaker having about 50 cc saturated KCl solution. Connect the solutions internally through KCl salt bridge and electrodes externally to the terminals of Potentiometers at either channel I or II at which

it is standardized i.e., calomel electrode to negative and platinum electrode to positive terminals of the potentiometer.

4. Record the EMF at 0.0 volume.
5. Start the titration by adding 1.0cc of 0.1N $K_2Cr_2O_7$ at a lot with stirring and record the EMF every time. Meanwhile, observe the rapid change in EMF at certain volume of $K_2Cr_2O_7$ where there will be an equivalence point (approximate). Continue the addition up to 32cc.

6. Plot the graphs of ;

- i. $\Delta E/\Delta V$ v/s volume of $K_2Cr_2O_7$ added which gives a sharp peak (graph-I). Find out the equivalence point.
- ii. EMF v/s volume of $K_2Cr_2O_7$ added which gives a sigmoid curve (graph-II) and

7. From the equivalence point, find out the EMF for **half equivalence** point from sigmoid curve (graph-II). Calculate the redox potentials of Fe^{3+}/Fe^{2+} using a suitable equation.

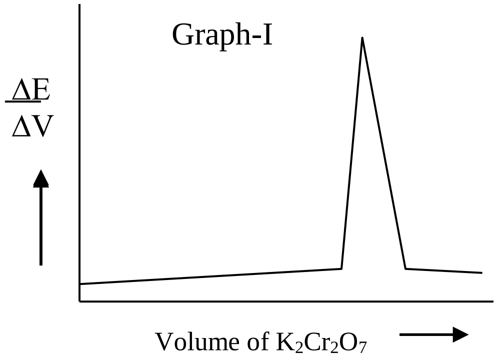
Observations:

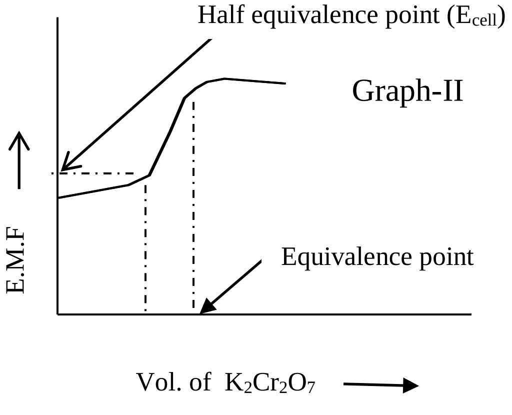
Solution in beaker = 25cc $FeSO_4 . 7H_2O$ + 25cc 4N H_2SO_4

Table

Volume of 0.1N $K_2Cr_2O_7$ (V cc)	EMF in Volts (E)	ΔV	ΔE	$\frac{\Delta E}{\Delta V}$
0.0		-----	------	-----
1.0				
2.0				
-				
-				
-				
32.0				

Nature of the graph





Calculation

1. Normality of Ferrous sulphate

   $N_1V_1=N_2V_2$ $N_1 = (0.1\times \text{BR from graph}) / 25$

2. Redox potential (E^o)of Fe^{3+}/Fe^{2+} = $E_{cell} + E^0_{calomel}$

   Where, E_{cell} = EMF at half equivalence point (from graph)

   $E^0_{calomel}$ = 0.2422V

Result : Redox potential (E^o) of Fe^{3+}/Fe^{2+} = -------------------V

Expt No.4 POTENTIOMETRY (Solubility product)

AIM: To determine the solubility and solubility product of sparingly soluble salts (AgCl) potentiometrically.

Chemicals: 0.01M KCl, 0.01M $AgNO_3$, Saturated NH_4NO_3 / KNO_3 solution.

Apparatus: Two silver electrodes, potentiometer, NH_4NO_3 / KNO_3 salt bridge etc.

Theory:

The following cell is constructed:

$^{(-)}Ag/\ 0.01M\ KCl\ //\ sat.\ AgCl//Sat.NH_4NO_3//0.01M\ AgNO_3/Ag^{(+)}$

Above cell is a concentration cell reversible with respect to silver ions. One Ag electrode is in connection with a solution of Ag^+ ions of known concentration, (0.01M) by assuming $AgNO_3$ be completely ionized at this dilution. Other electrode is in contact with a solution of much lower concentration of Ag^+ ions (furnished by the ionization of sparingly soluble salt AgCl formed by adding a drop of $AgNO_3$ solution to KCl), which has unknown concentration. EMF (E_{cell}) of the cell can be determined using equation.

$$E_{cell} = \frac{2.303\ RT}{nF} \log \frac{(a_{\ Ag+})_r}{(a_{\ Ag+})_l} = 0.0592 \log \frac{(a_{\ Ag+})_r}{(a_{\ Ag+})_l}$$

$$E_{cell} = 0.05921 \log \frac{0.01}{(a_{\ Ag+})_l} = 0.05921 \log \frac{0.01}{[Ag^+]}$$

Thus, knowing the value of $E_{cell,}$ one can easily calculate the value of $[Ag^+]$ from which solubility product (K_{sp}) can also be calculated.

Procedure:

1. Pipette out 25cc of the given 0.01M KCl solution in to 100cc beaker and add 2-3 drops of $AgNO_3$ solution to form a precipitate of AgCl, which gives a saturated solution of AgCl in 0.01M KCl solution.
2. Pipette out 10 cc of 0.01M $AgNO_3$ solution in another 100cc beaker.
3. Switch on the potentiometer and standardize the potentiometer by inserting two banana plugs into the sockets at channel I or II and adjust the EMF to 1.018V with the calibration screw. Remove these plugs after standardization
4. Place two sensitized silver electrodes in these two separate beakers containing respective solutions. Connect them internally with the salt bridge filled with saturated ammonium nitrate or potassium nitrate solution.

5. Connect the silver electrodes to the terminals of the potentiometer as noted in the cell convention.
6. Observe the EMF of the cell (E_{cell}) from the potentiometer.
7. Add 10 cc of H_2O to second beaker already contains 0.01 M $AgNO_3$ to make 0.005M $AgNO_3$ and determine its EMF.
8. Similarly, add 20 cc H_2O to the above beaker to get 0.0025 M $AgNO_3$ and determine its EMF and also for 0.00125M solution.

Observations:

Concentration of $AgNO_3$ (M)	Cell EMF (V)		$[Ag^+]$	K_{sp} =	Solubility S=
0.01		$\text{Antilog}\left(\frac{EMF}{(0.05921)}\right)$			
0.005					
0.0025					
0.00125					
				Average, K_{sp} =	Average, S=

Calculations

1. The concentration of Ag^+ ions in 0.01 M KCl solution, [Ag+]

$$[Ag^+] = \frac{[AgNO_3]}{\text{Antilog}\left(\frac{EMF}{(0.05921)}\right)}$$

2. The concentration of Cl^- ions in this solution is ≈ 0.01 mol / dm^3
3. Therefore, the solubility product of AgCl, K_{sp} = $[Ag^+][Cl^-]$
= $[Ag^+] \times 0.01$
= -------- mol^2/dm^6
4. Similarly calculate K_{sp} for 0.005M & 0.0025 M $AgNO_3$ solutions
5. Calculate the solubility of AgCl(for all K_{sp}) $S=\sqrt{K_{sp}}$= --------- mol /dm^3

Results:

1. Average solubility(S) of AgCl = ---------- mol / dm^3
2. Average K_{sp} value of AgCl at 0.01 M$AgNO_3$ = ---------- mol^2/dm^6

Note: 1. The silver electrodes can be sensitized by dipping into 1:1 HNO_3 solution containing a little $NaNO_3$ till gas formation starts.

2. Theoretical K_{sp} value of AgCl = 2.0×10^{-10} mol^2/dm^6

3. Theoretical value of solubility of AgCl = 1.76×10^{-5} mol / dm^3

Exp. No. 5 REFRACTOMETRY (Formula Method)

Aim: To determine the percentage composition of unknown mixture of A & B liquids using Abbe's Refractometer (formula method)

Apparatus: Abbe's refractometer and accessories.

Chemicals: Two pure liquids A and B and one mixture 'C' of their unknown composition.

Theory:

Refractive index of a liquid varies with the temperature as well as wavelength of light used. Therefore, this cannot be used satisfactorily for comparing the constitutions of the substance. But the Lorentz and Lorenz expression is used as given bellow.

$R=\frac{n^2-1}{n^2+2} \times \frac{1}{d}$ where 'n' is RI and 'd' is density

'R' is specific refraction, which is additive as well as constitutive property. It is also used to calculate the percentage of unknown composition of binary mitxture

Percentage Composition of 'A' in 'C' $=\frac{100(R_c - R_b)}{(R_a - R_b)}$

Where 'R_a' is specific refraction of 'A'

Where 'R_b' is specific refraction of 'B'

Where 'R_c' is specific refraction of 'C'

Procedure:

1. Open the prism box and clean the prism surface with ether. Blow the air to dry the prism.
2. Introduce 0.5 cc of liquid between the prisms and close the prism box.
3. Focus the mirror to reflect the light in to prism box. Turn the right hand knob till the color fringes are seen. Then make it sharp (partly black and partly white) by rotating the RHS knob, compensator.
4. With the help of LHS knob, adjust the line of demarcation at the point of intersection of cross wire. Observe the refractive index (RI) directly from the scale up to fourth decimal place.
5. Similarly, determine the RI for all the given liquids. Take at least three independent settings for each liquid and use the average of these.
6. Determine the density of liquid A, B & C at the temperature in which the RI is being determined.
7. Calculate the specific refraction for each of the various liquids by using Lorentz and Lorenz formula.
8. Calculate the percentage of liquid A in liquid mixture C.

Observations I :

Liquid		**A**	**B**	**C**
Percentage Composition		100	100	-
Density, g/cc				
Settings (n_1)	i			
(n_2)	ii			
(n_3)	iii			
Mean (n)				
Specific Refraction (R)				

Observations II:

1. Wt. of empty Sp. Gravity bottle = W_1 g
2. Wt. of empty Sp. Gravity bottle+ liquid = W_2 g
3. Wt. of empty Sp. Gravity bottle+ water = W_3 g
4. Wt. of water = $(W_3 - W_1)$ g
5. Wt. of liquid = $(W_2 - W_1)$ g

Calculations:

$$\text{Density , d} = \frac{\text{Wt. of liquid}}{\text{Wt. of water}}$$

1) Specific refraction, $R = \frac{\boldsymbol{n}^2 - 1}{\boldsymbol{n}^2 + 2} \times \frac{1}{\boldsymbol{d}}$ where ‘n’ is RI and ‘d’ is density.

2) Percentage Composition of ‘A’ in ‘C’ $= \frac{100(R_c - R_b)}{(R_a - R_b)}$

Where ‘R_a’ is specific refraction of ‘A’

Where ‘R_b’ is specific refraction of ‘B’

Where ‘R_c’ is specific refraction of ‘C’

Result:

1. Percentage composition of ‘A’ in ‘C’ = ----------
2. Percentage composition of ‘B’ in ‘C’ = ----------

Exp. No. 6 REFRACTOMETRY (Graphical Method)

Aim: To determine the percentage composition of unknown mixture of A & B liquids using Abbe's Refractometer (graphical method).

Apparatus: Abbe's refractometer and accessories.

Chemicals: Two pure liquids A and B and four mixtures of known percentage composition by weight of A and B and one mixture 'C' of unknown composition.

Theory:

Refractive index of a liquid varies with the temperature as well as wavelength of light used. Therefore, this cannot be used satisfactorily for comparing the constitutions of the substance. But the Lorentz and Lorenz expression is used as given bellow.

$$R=\frac{\boldsymbol{n}^2-1}{\boldsymbol{n}^2+2}\times\frac{1}{\boldsymbol{d}}$$ where 'n' is RI and 'd' is density

'R' is specific refraction, which is additive as well as constitutive property. It is also used to calculate the percentage of unknown composition of mixture by plotting a graph of specific refraction against percentage composition of the given known mixture.

Procedure:

1. Open the prism box and clean the prism surface with ether. Blow the air to dry the prism.
2. Introduce 0.5 cc of liquid between the prisms and close the prism box.
3. Focus the mirror to reflect the light in to prism box. Turn the right hand knob till the color fringes are seen. Then make it sharp (partly black and partly white) by rotating the RHS Knob, compensator.
4. With the help of LHS knob, adjust the line of demarcation at the point of intersection of cross wire. Observe the refractive index (RI) directly from the scale up to fourth decimal place.
5. Similarly, determine the RI for all the given liquids. Take at least three independent settings for each liquid and use the average of these.
6. Determine the density of liquid 'C' at the temperature at which the RI is being determined.
7. Calculate the specific refraction for each of the various liquids by using Lorentz and Lorenz formula.
8. Plot a graph 'R' against composition, which gives a straight line. From the graph, read the composition of the mixture of unknown composition.

Observations I :

Liquid		**A**	**B**	**C**	**M$_1$**	**M$_2$**	**M$_3$**	**M$_4$**
%age Composition		100	100	-	A=40 B=60	A=50 B=50	A=60 B=40	A=70 B=30
Density, g/cc		0.873	1.579		1.212	1.138	1.075	1.034
Settings (n_1)	i							
(n_2)	ii							
(n_3)	iii							
Mean (n)								
Specific refraction, R								

Observations II:

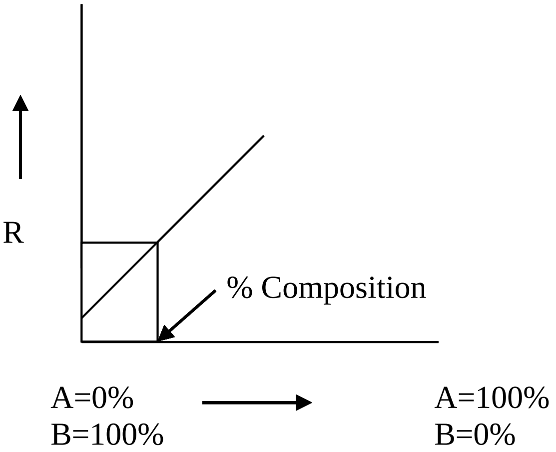
1. Wt. of empty Sp. Gravity bottle = W_1 g
2. Wt. of empty Sp. Gravity bottle+ liquid C = W_2 g
3. Wt. of empty Sp. Gravity bottle+ water = W_3 g
4. Wt. of water = $(W_3 - W_1)$ g
5. Wt. of liquid C = $(W_2 - W_1)$ g

Calculation:

1. Density , d_c = $\dfrac{\text{Wt. of liquid, C}}{\text{Wt. of water}}$
2. Specific refraction, R = $\dfrac{n^2 - 1}{n^2 + 2} \times \dfrac{1}{d}$

Where ,
'n' is RI and
'd' is density of liquid, C

Nature of graph



Result: From the graph

1. Percentage composition of 'A' in 'C' = ----------
2. Percentage composition of 'B' in 'C' = ----------

Expt No.7 POTENTIOMETRY (pK_a of CH_3COOH)

AIM: To determine the pK_a of acetic acid potentiometrically.

Chemicals: 0.2N NaOH, approx 0.2N CH_3COOH, Quinhydrone etc.

Theory: Dissociation of an acid may be represented by the equation

$$CH_3COOH \longrightarrow CH_3COO^- + H^+$$

When the acid is half neutralized, the concentration of acid and the concentration of its salt in the solution will be equal. For a weak acid the concentration of undissociated acid molecules can be assumed to be unchanged, by a small fraction which dissociates to provide the hydrogen ions. The salt can be regarded as fully dissociated and hence the concentration of CH_3COO^- ions can be assumed to be equal to that of the salt formed into any stage of titration.

For the half neutralization stage:

$$K = \frac{[CH_3COO^-]\,[H^+]}{[CH_3COOH]} = \frac{[CH_3COO^-]}{[CH_3COOH]} \times [H^+]$$

$$K = [H^+] \text{ Or } pK = pH$$

Thus, the dissociation constant may be calculated from $[H^+]$ or pH values for the half neutralized solution of the acid at different stages of titration and the corresponding values at the CH_3COO^- ion concentrations.

Procedure:

1. Pipette out 25cc of the given CH_3COOH solution in a beaker and add a pinch of solid quinhydrone and stir the solution with a glass rod. Keep it for a while.
2. Fill the burette with exact 0.2N NaOH solution.
3. Switch on the potentiometer and standardize the potentiometer by inserting two banana plugs into the sockets at channel I or II and adjust the EMF to 1.018 volts with the calibration screw. After standardization remove these plugs.
4. Place the platinum electrode in the above quinhydrone solution and calomel electrode in another beaker having about 50 cc saturated KCl solution. Connect the solutions internally through KCl salt bridge and electrodes externally to the terminals of Potentiometers at either channel I or II at which it is standardized i.e., calomel electrode to negative and platinum electrode to positive terminals of the potentiometer. Record the EMF at 0.0 volume.
5. Start titration by adding 1.0cc of 0.2N NaOH at a lot with constant stirring and record the EMF every time. Meanwhile, observe the rapid change in EMF at certain volume of NaOH where there

will be an equivalence point (approximate) and EMF will be in –ve value. Continue the additions for another 04 readings after –ve EMF are obtained.

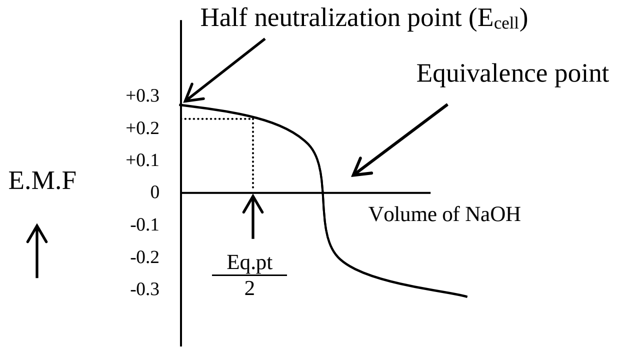
6. Plot a graph of EMF v/s volume of NaOH added which gives a reverse sigmoid curve. Find out the equivalence point and EMF at half neutralization point(E_{cell}).

Observations

Solution in beaker = 25 cc 0. 2N CH_3COOH + quinhydrone

Volume of 0.2N NaOH added	EMF (V)
0.0	---
1.0	
2.0	
….	
….	
…	

Nature of the graph



∴ EMF for half neutralization of an acid solution from the graph (E_{cell}) =…… volts

Calculations

We have,

$$pH = \frac{0.456 - E_{cell}}{0.0592} = \ldots\ldots\ldots$$

At half neutralization point, pH = pK_a

$$\therefore \quad K_a = \text{antilog}(-pK_a)$$
$$= \ldots\ldots$$

Result: The experimental K_a value for the CH_3COOH = ………………

Note:

1. The theoretical K_a value for the CH_3COOH = 1.8×10^{-5} mol/ dm^3
2. Use logarithm table for calculation of K_a from - pK_a otherwise, candidate shall lose marks reserved for calculations.
3. Keep the Pt wire in 1:1 HNO_3 for a while before using.

Physical Chemistry Experiments: Instruments based

Some of the experiments in Physical chemistry are based on instruments. Generally, simple instruments like conductometer, pH meter, potentiometer, colorimeters etc are used. These instruments are designed with electronic circuits. Maintenance of such instruments is very much necessary. Supplier will provide the instruction manual. Manual will have all the necessary instructions about maintenance of the instruments and electrodes. However, following are the tips to maintain and check the working conditions of the instruments.

1. Switch on the instruments at least 10 minute prior to the use for stabilization.
2. If no light glows in the instruments, check the fuse and replace.
3. Check the colorimeter for water to get OD zero for filter No. 510 nm. If not to zero, please change the lamp.
4. Calibrate conductometer and potentiometer frequently.
5. Keep away the instruments from moisture and acid fumes.
6. Use soft tissue paper for removal of liquid from the surface of prism in Refractometer otherwise surface of prism spoils.
7. Do not wipe out the surface of colorimetric cell with ordinary filter paper, use tissue paper. To get reproducible result, focus the cell to light in cell holder by keeping every time the cell with the same face by marking in the cell with marker.

Maintenance of electrodes

1. **Platinum Electrode**

❖ Should be dipped in dil. HNO_3 for 2 hours in alternative day.

❖ If not used for long period - preserve in dry condition after cleaning with dil. HNO_3 with air tight packing.

❖ Before reusing – Keep it in distilled water for 2 hrs.

2. **Conductivity Cell**

❖ Always preserve in distilled water.

❖ If not used for long period - preserve in dry condition after cleaning with dil. H_2SO_4 with air tight packing.

❖ Before reusing – Keep it in distilled water/ dil. H_2SO_4 for 2 hrs.

3. **Combined Glass Electrode**

❖ Always preserve in 4 N KCl / 0.01N HCl / Buffer soln. of pH = 4.0

❖ Never preserve in water but never allow drying.

- If not used for long period – Do not preserve in dry condition but keep the glass bulb in black boot wetted with 4 N KCl. Check weekly the wetting condition and recharge.
- Before reusing – Keep it in 4 N KCl / 0.01N HCl / Buffer soln. of pH = 4.0 for 10 minutes.
- Fill the side tube with 4 N KCl solutions if necessary.
- Do not wipeout the glass bulb from ordinary filter paper. Use soft tissue paper otherwise membrane spoils.

4. **Calomel Electrode**

- Always preserve in Saturated KCl. Never preserve in water.
- If not used for long period - preserve in dry condition after cleaning with dil. HNO_3 with air tight packing.
- Fill the side tube with Saturated KCl solutions if necessary.

Replatinization of Conductivity cell

- Remove the old coating by keeping the cell in dilute aqua-regia.
- Further, clean with warm dil. Potassium dichromate in 5% H_2SO_4 followed by water.
- Coating:
  - i. 01 g Platinum Chloride +0 .007g Lead Acetate 30 ml of 50% HCl.
  - ii. Keep the cell and connect to 3 - 4 V Battery, pass the current with 30mA for 2 minutes.
  - iii. Interchange the terminals and repeat the same.
  - iv. Clean the electrodes with 5 % H_2SO_4 followed by distilled water.

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Chem – Forum : 2017-18

Commonly used Laboratory Reagents

Front shelf reagents and their concentrations: 13 Reagents

Sl. No.	Name of the acid	Required Normality	Method of Preparation
1.	Hydrochloric acid (HCl)	5N	Dilute 455ml of conc. HCl to 1000cc with distilled water.
2.	Nitric acid (HNO_3)	5N	Dilute 310ml of conc.HNO_3 to 1000cc with distilled water.
3.	Sulphuric acid (H_2SO_4)	5N	Add 318mlof conc.H_2SO_4 slowly and with constant stirring into about 500 cc of distilled water. Cool it and make up to 1000cc by distilled water.
4.	Ammonium chloride NH_4Cl	5N	Dissolve 265g in water and dilute to 1000cc.
5.	Ammonium hydroxide (NH_4OH)	5N	Dilute 335ml of conc. ammonia solution to 1000cc by distilled water.
6.	Ammonium carbonate $(NH_4)_2CO_3$	4N	Dissolve 190gms of the commercial sample in about 100ml of conc.NH_3 solution and dilute with water to 1000cc.
7.	Ammonium oxalate $(NH_4)_2C_2O_4H_2O$	0.5N	Dissolve 35gms in water and dilute to 1000cc .
8.	Barium chloride $BaCl_2.2H_2O$	0.5N	Dissolve 61g in water and dilute to 1000cc.
9.	Mercuric chloride $HgCl_2$	0.2N	Dissolve 54g in water and dilute to 1000cc.
10.	Sodium hydroxide solution (NaOH)	5N	Dissolve 200g of ordinary sample of NaOH (appx.90% NaOH by weight)in water. Cool it and dilute to make the solution 1000cc.
11.	Disodium hydrogen phosphate $Na_2HPO_4.12H_2O$	0.3N	Dissolve 110gms in water and dilute to 1000cc.
12.	Calcium sulphate $CaSO_4.2H_2O$	0.03N	3g of the salt dissolved and diluted to 1000cc allow to settle and decant.
13.	Acetic acid (CH_3COOH)	4N	Dilute 230cc of glacial acetic acid to 1000cc with distilled water.

Note: Concentration of all acids and sodium hydroxide can also be reduced to half however; concentration of other reagents should be maintained without any deviation.

Side shelf Reagents and their concentrations

Sl. No.	Name of the acid	Required Normality	Method of Preparation
1.	Ammonium acetate CH_3COONH_4	4N	Dissolve 231gms in water and dilute to 1000cc.
2.	Ammonium sulphate $(NH_4)_2SO_4$	2N	Dissolve 132gms in water and dilute to 1000cc.
3.	Ammonium thiocyanate NH_4CNS	0.5N	Dissolve 38gms in water and dilute to 1000cc.
4.	Calcium chloride $CaCl_2.6H_2O$	0.5N	Dissolve 55gms in water and dilute to 1000cc.
5.	Cobalt nitrate $CO(NO_3)_2.6H_2O$	0.3N	Dissolve 44gms in water and dilute to 1000cc..
6.	Copper sulphate $CuSO_4.5H_2O$	0.5N	Dissolve 125gms in water containing about 3ml of conc.H_2SO_4 and dilute to 1000cc..
7.	Ferric chloride $FeCl_3.6H_2O$	0.5N	Dissolve 135gms in water containing about 20ml of conc.HCl and dilute to 1000cc.
8.	Ferrous sulphate $FeSO_4.7H_2O$	0.5N	Dissolve 140gms of water containing 7ml of conc.H_2SO_4 and dilute.
9.	Lead acetate $Pb(CH_3COO)_2.3H_2O$	0.5N	Dissolve 95gms in water and dilute to 1000cc..
10.	Potassium chromate K_2CrO_4	0.5N	Dissolve 48.5gms in water and dilute to 1000cc.
11.	Potassium cyanide KCN (Poisonous)	0.5N	Dissolve 32.5gms in water and dilute.
12.	Potassium ferriccyanide $K_3[Fe(CN)_6]$	0.5N	Dissolve 55gms in water and dilute to 1000cc.
13.	Potassium ferrocyanide $K_4[Fe(CN)_6]$.3H2O	0.5N	Dissolve 53gms in water and dilute to 1000cc.
14.	Potassium iodide KI	0.5N	Dissolve 3.2gms in water and dilute to 1000cc.
15.	Potassium permanganate $KMnO_4$	0.1N	Dissolve 3.2gms in water and dilute to 1000cc.. Filter through glass wool.
16.	Potassium thiocyanate KCNS	0.5N	Dissolve 49gms in water and dilute.
17.	Silver nitrate $AgNO_3$	0.1N	Dissolve 17gms in water and dilute to 1000cc..
18.	Sodium acetate $CH_3COONa.3H_2O$	3N	Dissolve 408gms in water and dilute. To 1000cc
19.	Stannous Chloride $SnCl_2.2H_2O$	0.5N	Dissolve 56gms in about 100ml of conc.HCl and dilute with water or dil HCl to 1000cc..
20.	Alcoholic KOH(10%)	-	10 g KOH in 100cc 50% alcohol
21.	Bromine water	-	Dilute 10-15 cc liq.Bromine with 1000cc water. Or 5-6 cc liq. Br_2 in 50 cc water

			containing 20 g KBr. Dilute to 200 cc.
23	Br_2 in acetic acid	-	20 cc Liq.Br_2 in 80 cc glacial acetic acid
24	$KMnO_4$		1 g in 100 cc water(1%) : 6g in 100 cc water(6%)
25	Alkaline $KMnO_4$	-	3 g in 100 cc water containing 10 g Na_2CO_3
26	$FeSO_4$ solution(fresh)	-	15 g mixted with 1-2 cc con.H_2SO_4 & dilute with 100 cc water.
27	Neutral $FeCl_3$ solution	-	10 g in 100 cc water
28	Alcoholic $FeCl_3$ solution	-	10g in 100 cc alcohol
29	$CaCl_2$ solution		Dissolve 55 g of hydrated $CaCl_2$ in 1000cc water.
30	$NaNO_2$ solution (10%)	-	10 g in 100 cc water.
31	Sodium nitroprusside		Dissolve 3g of compound in 1000cc in water .
32	Brady's reagent (2,4 DNP)	-	Add 4g 2,4 dinitro phenyl hydrazine to 8 cc con.H_2SO_4 add slowly with shaking & cooling 70cc Methyl alcohol & make up to 100cc with water.
33	Iodine solution (for iodoform)	-	20 g KI & 10 g Iodine in 100 cc water.
34	Picric acid	-	5 g in 100 cc alcohol or 100cc benzene or glacial acetic acid
35	β-naphthol	-	10 g of compound in 100cc 10% NaOH.
36	Schiff's reagent	-	Rosaniline hydrochloride (0.2g) is dissolved in 100 cc water. & solution is saturated with SO_2 when colour of solution just vanishes.
37	Hydroxylamine hydrochloride	-	5 g in 100 cc methyl alcohol.
38	Tollen's reagent	-	2-3 drops of dil.NaOH (1%) solution to 5 cc $AgNO_3$. White ppt. obtained is dissolved in ammonia.
39	Aluminon	-	0.1% solution in distilled water (use freshly prepared)
40	Dimethyl glyoxime	-	1% solution in ethanol
41	Magneson	-	0.5% in 0.2N NaOH (pinch in 100cc solution containing 0.800 g of NaOH)
42	5% Picric acid	-	Dissolve 5g of picric acid in 100 cc Ethanol
43	Calcium hydroxide solution($Ca(OH)_2$)	0.04N	Place 2-3 grams of quick lime in 1000cc of distilled water and allow it to stand for some time. Now decant it. The clear supernatant liquid lime water.

INDICATORS			
1	Phenolphthalein	-	Dissolve 0.5g of Phenolphthalein in 50 cc of ethanol and add 50 cc of distilled water with constant stirring, filter, if necessary
2	Methyl orange	-	Dissolve 50 mg of methyl orange in distilled water and diluted to 100 cc filter, if a ppt. is formed
3	Methyl red	-	Dissolve 0.1 g of methyl red in 100 cc of hot distilled water and cool, filter, if a ppt. is formed.
4	Starch	-	Add distilled water to about o.5 g of soluble starch (A.R) taken in a beaker, stir with a glass rod and heat to make transparent paste. Pour into it 100 cc of boiling distilled water with constant stirring and cool
5	Eriochrome Black-T	-	Dissolve 0.2 g of solid dyestuff in 15 cc triethanolamine and 5cc ethanol or 0.5 g of the dyestuff in 100 cc rectified spirit.
6	Potassium chromate	-	Dissolve 5g of A.R. Potassium chromate in 100 cc distilled water.

Precautions in preparation of Reagents:

1. Wear apron.
2. Gas Masks.
3. Use Goggles.
4. Use hand glows.
5. Be away from the flame.
6. Be close to sink for water support.
7. Use distilled water for preparation of reagents.
8. Confirm that the laboratory having first aid box.
9. Never pour hot / cold sulphuric acid in to sink.
10. Use fuming chamber for preparation of Br_2, and other volatile solutions.
11. **For diluting sulphuric acid solutions:** Add concentrated sulphuric acid to water slowly by cooling under tap. Never add water to concentred sulphuric acid for dilution of sulphuric acid.
12. Never use tap water for preparation of Sodium hydroxide and ammonium hydroxide solution at any condition.
13. Preserve conc. / dil. ammonia, Sodium hydroxide and lime water in well stopped bottles.
14. Do not keep conc. / dil. ammonia and hydrochloric acid bottles adjacent to each other.
15. Preserve the silver nitrate, potassium ferricyanide, iodine, potassium permanganate solutions etc in amber colour bottle.

Universal Method of Numbering the General Reagent Bottles in Chemistry Laboratory

SL.NO	Name of Reagents	Sl.No	Names of Reagent
1	Hydrochloric acid(dil)	32	Iodine(I_2)
2	Nitric acid (dil)	33	Lime water
3	Sulphuric acid (dil)	34	Lead acetate
4	Ammonium chloride	35	Methanol
5	Ammonium hydroxide	36	β-Naphthol in NaOH
6	Ammonium carbonate	37	Potassium chromate
7	Ammonium oxalate	38	Potassium dichromate
8	Barium chloride	39	Potassium iodide
9	Mercuric chloride	40	Potassium permanganate
10	Sodium hydroxide	41	Potassium ferrocyanide
11	Sodium hydrogen phosphate	42	Potassium ferricyanide
12	Calcium sulphate	43	Potassium pyroantimonate
13	Acetic acid	44	Picric acid
14	Acetic anhydride	45	Potassium sulphocyanide
15	Aniline	46	Sodium nittroprusside
16	Ammonium acetate	47	Sodium nitrite
17	Ammonium molybdate	48	Sodium bicarbonate
18	Ammonium sulphate	49	Silver nitrate
19	Brady'sreagent	50	Bromine in HAC
20	Chlorine water	51	Pyridine
21	Copper sulphate	**SOLIDS**	
22	Cobalt nitrate	52	Ferrous sulphate
23	Calcium chloride	53	Lead dioxide(PbO_2)
24	Chloroform	54	Manganese dioxide(MnO_2)
25	Ethanol	55	Oxalic acid
26	Fehling's solution A	56	Potassium dichromate ($K_2Cr_2O_7$)
27	Fehling's solution B	57	Soda lime
28	Ferric chloride	58	Phthalic anhydride
29	Ferrous sulphate	59	Zinc dust
30	Ferric chloride(Neutral)	60	β-Naphthol
31	Schiff's reagent	61	Resorcinol

General Chemicals in liquid form having specific normality in the supplied bottles

Sl. No.	Name of the acid	Percent by weight	Approximate specific gravity	Concentration
1.	Hydrochloric acid (HCl)	36.5	1.18	11N
2.	Nitric Acid (HNO_3)	70	1.42	16N
3.	Sulphuric Acid (H_2SO_4)	98	1.84	36N
4.	Aceticacid (glacial) (CH_3COOH)	99.6	1.05	17N
5.	Ammonia (NH_3)	28	0.88	15N

Specific Reagents for Inorganic Experiments

B.Sc. I-Semester

Preparation of Stock Solution

- Direct stock solution may be supplied for standardisation.
- For determination of the amount of substance, known volume of the solution may be given in 250 cc of volumetric flask and students should dilute the solution OR direct solution may be given.

Sl. No.	Name of solution	Concen tration	Method of preparation
1.	Sodium hydroxide (NaOH)	0.5 N	Dissolve 20 g of NaOH in 1000cc distilled water.
	Sodium hydroxide (NaOH)	0.1N	Dissolve 4 g of NaOH in 1000cc distilled water.
	Sodium hydroxide (NaOH)	0.05 N	Dissolve 2 g of NaOH in 1000cc distilled water.
2	Hydrochloric acid (HCl)	0.5 N	Dissolve 45 cc of conc. HCl in 1000cc distilled water.
	Hydrochloric acid (HCl)	0.05 N	Dissolve 4.5 cc of conc. HCl in 1000cc distilled water
	Hydrochloric acid (HCl)	0.1N	Dissolve 9.0 cc of conc. HCl in 1000cc distilled water.
3	Na_2CO_3 and NaOH mixture:	-	26.5g Na_2CO_3 + 20 g of NaOH in 1000cc distilled water.
	Na_2CO_3 and NaOH mixture:	-	13.5g Na_2CO_3 +10 g of NaOH in 500cc distilled water.
4.	Na_2CO_3 and $NaHCO_3$ mixture:	-	26.5g Na_2CO_3 + 42 g of $NaHCO_3$ in 1000cc distilled water.
	Na_2CO_3 and $NaHCO_3$ mixture:	-	13.5g Na_2CO_3 + 21 g of $NaHCO_3$ in 1000cc distilled water.
5	Potassium permanganate $KMnO_4$:	0.05 N	Dissolve 1.6g AR $KMnO_4$ in distilled water. Keep it over night, filter through glass wool. Dilute to 1000cc. Store in brown coloured bottle. (Standardize the solution with sodium oxalate)
6	oxalic acid and sulphuric acid mixture:	-	31.5 g of oxalic acid is dissolved in minimum quantity of water, to this add 14cc of sulphuric acid and diluted up to 1000cc.
	oxalic acid and sulphuric acid mixture:	-	16 g of oxalic acid is dissolved in water, to this 7cc of sulphuric acid is added and diluted to 5000cc.
7	Mohr's solution (FAS): Ferrous ammonium sulphate	0.5N	Make a paste of 196.9g of FAS with 10 cc of Sulphuric acid and dilute up to 1000cc with distilled water.
	Mohr's solution (FAS): Ferrous ammonium sulphate	0.05N	Make a paste of 19.6g of FAS + 10 cc of Sulphuric acid and dilute up to 1000cc with distilled water.
8.	Potassium dichromate $K_2Cr_2O_7$	0.05 N	2.5 g $K_2Cr_2O_7$ dissolved in 1000cc of distilled

			water (if necessary add 1cc of sulphuric acid)
9.	Fe^{+2} and Fe^{+3} ion mixture :	-	49 g of FAS +20 g of $FeCl_3.6H_2O$ + 10cc sulphuric acid make a paste dilute up to 500cc with distilled water.
10.	Sodium thiosulphate :	0.05 N	Dissolve 12.5g of Sodium thiosulphate in 1000cc distilled water
11.	EDTA:disodium etylenediammine tetra-acetate	0.05 M	Dissolve 18.6g of disodium etylenediammine tetra-acetate in 1000cc distilled water
12	Zinc sulphate solution:	0.5 M	143.7 g of $ZnSO_4.7H_2O$ +10cc of sulphuric acid dilute up to 1000cc with distilled water.
	Zinc sulphate solution:	0.05 M	14. g of zinc sulphate +5cc of sulphuric acid dilute up to 1000cc with distilled water.
13	Buffer solution:	-	17.5 g NH_4Cl + 150 cc of 17N NH_4OH solution further dilute with distilled water up to 250 cc.
14	Hard water:	-	Hard water (one ml =1 mg $CaCO_3$) Add slowly a amount of dilute HCl through a funnel to 1 g of anhydrous $CaCO_3$ (A.R grade) taken in a conical flask. Boil gently to remove CO_2.Heat to dryness on a water bath. Dissolve in distilled water and dilute up to 1000cc.
15	Iodine Solution (N/10)	-	Refer foot note
16	Alcoholic potassium hydroxide (N/2)	-	Dissolve 28 g of A.R potassium hydroxide in 1000cc of 95% alcohol(freshly distilled from KOH) . Mix thoroughly and let stand undistilled for any carbonate to settle down. Decant the clear supernatant solution and use.
17	Bromine Solution	-	Prepare a saturated solution by shaking 11cc of liquid bromine with 1000cc of distilled water.
18	Mercuric chloride	saturated	Dissolve 80 g of Mercuric chloride in a 1000cc of hot distilled water. Cool to room temperature and filter.
19	Potassium thiocynate	0.1M	Dissolve 9.7g of A.R KSCN in a 1000cc of distilled water
20	Stannous Chloride	0.1M	Dissolve 2.26 g of A.R Stannous Chloride in 5-6 ml con HCl acid and diluted to 100cc with water
	Stannous Chloride	5%	Dissolve 50g of A.R Stannous Chloride in 100 ml con HCl and diluted to 1000cc with water.
21	KI	10%	Dissolve 100g of KI in a 1000cc of boiled out distilled water.

Note: Place about 10 g of A.R KI and 2-3 cc distilled water in a large weighing bottle fitted with a ground- glass stopper. Dissolve by gentle shaking and weigh accurately, after the bottle and the solution have reached room temperature. Introduce quickly 3.2-3.5 g of A.R or resublimed iodine into the weighing bottle. Without splashing and re-stopper it. Shake and let stand for some time. After the temperature has reached equilibrium, reweigh the bottle accurately.

The difference gives the weight of pure iodine. Dilute the solution in the weight of pure iodine. Dilute the solution in the weighing bottle and quickly transfer it quantitatively to a 250cc measuring flask. Dilute up to mark with a distilled water. Stopper and keep in a dark place. The prepare solution is W X 4/126.9 N where W is weight of iodine dissolved. Make exactly N/10 by appropriate dilution.

BSc.VI SEMISTER

VOLUMETRIC ANALYSIS

SL NO	Experiment	Solution	Chemicals	Preparation
1	Experiment-1. Extraction of Iron(III) from haematite ore	Fe^{3+} solution (Haematite ore or SolidFe_2O_3 Solution) 100g of $FeCl_3.6H_2O$ or 50g of anhydrous $FeCl_3$ + 10 cc of Conc. HCl make a paste & add water to make the volume 1000cc with water. Filter if necessary.	• $SnCl_2$ solution • 5% $HgCl_2$ solution • Diphenylamine • Phosphoric acid or Na_2HPO_4.	• Dissolve 12 g of pure tin or 30 g of stannous chloride ($SnCl_2$.$2H_2O$) in 100 cc conc. HCl and add 200 cc water. • 5g of $HgCl_2$ in 100 cc water. • 1g in 100 cc Conc.H_2SO_4
2	Experiment-2. Extraction of Cu and Zn from brass	Cu^{2+} (Brass solution) 100 g of $CuCl_2.2H_2O$ + 5 cc conc.HCl dilute to 1000cc with water. OR 125 g of $CuSO_4.5H_2O$ + 5 cc conc.H_2SO_4 dilute to 1000cc with water.	• 1N NaOH • Dil. Acetic acid • 10% KI • Starch solution.	• 40 g NaOH in 1000cc water. • 100 cc acetic acid + 100 cc water • 100 g of KI in 1000cc water.
3	Experiment-3. Extraction of calcium from limestone.	To dissolve sample of limestone in minimum amount of 1:1 HCl dilute to 250 cc with distilled water.	• 5% ammonium oxalate • 1:8 H_2SO_4 • NH_4OH Solution • Methyl red indicator	• 5g ammonium oxalate in 100 cc water • 100cc H_2SO_4 + 800 cc water. • (0.02% methyl red solution in alcohol).

Inorganic Gravimetric Determination

S.No.	**Experiment Name**	**Solution Required**	**Chemicals And Materials**
1	Experiment-1. Determination of Barium as $BaSO_4$	$BaCl_2.2H_2O$-10% solution100 g $BaCl_2.2H_2O$ + 5 cc of conc. HCl diluted to 1000cc with water.	• 2N sulphuric acid solution -100 cc con. H_2SO_4 should be added slowly to 900 cc H_2O with stirring. • Whatman filter paper 40(41)
2	Experiment-2. Determination of Al as Al_2O_3	150g of $AlCl_3.6H_2O$ + 10 cc of Conc.HCl or 106 g of $Al_2(SO_4)_3$ + 10 cc of Conc.H_2SO_4 make a paste & add water to make the volume 1000cc with water.	• 1:1 ammonia solution (100 cc Conc. ammonia solution+100 cc Dist. Water) • 2% NH_4NO_3(20 g in 1000cc of dist. water). • 1-2 g NH_4Cl. Whatman filter paper 40(41). • Methyl red indicator (0.02% solution in alcohol).
3	Experiment-3. Determination of iron as Fe_2O_3	140g of $FeCl_3.6H_2O$ or 80g of anhydrous $FeCl_3$ + 10 cc of Conc.HCl make a paste & add water to make the volume 1000cc with water.	• 1:1ammonia solution (100 cc Conc. ammonia solution + 100 cc Dist.Water). • 2% NH_4NO_3(20 g in 1000cc of dist. water). • 1-2 g NH_4Cl. • Whatman filter paper 40(41).
4	Experiment-4. Determination of Lead as $PbSO_4$	100 g $Pb(NO_3)_2$+ 5 cc of conc. HNO_3 diluted to 1000cc with water or100g$Pb(CH_3COO)_2.3H_2O$ + little acetic acid diluted to 1000cc with water.	• 2N sulphuric acid solutions -100 cc conc. H_2SO_4 should be added slowly to 900 cc H_2O with stirring. • 5% sulphuric acid solution -50 cc conc. H_2SO_4 should be added slowly to 1000 cc H_2O with stirring • 50 cc Rectified spirit (800 cc alcohol + 200cc H_2O) • Whatman filter paper 40(41)

Specific Reagents for Organic Experiments

B.Sc. II semester

ORGANIC PREPARATIONS			
Sl No	Experiment Name	Chemical	Physical Constant
1	Preparation of acetanilide from aniline.	- 5cc of aniline, - 6cc of glacial acetic acid - pinch of zinc dust	M.P of Acetanilide = 114°C
2	Preparation of phthalimide from phthalic anhydride.	- 4g of phthalic anhydride - 2g of urea	M.P of phthalimide = 234°C
3	Preparation of p-nitroacetanilide from acetanilide.	- 5g of acetanilide in 5cc of glacial acetic acid - 10 cc of sulphuric acid - 6 cc of conc. nitric aci	M.P of p-nitro acetanilide = 214°C
4	Preparation of aspirin from salicylic acid.	- 5g of salicylic acid - 10 cc of glacial acetic acid - 1-2 drops of conc. H_2SO_4	M.P of aspirin = 132-137^oC
5	Preparation of 1,1'-bis-2-Naphthol from 2-Naphthol.	- 1.5g of 2-Naphthol in 20ml water - 3.0g of $FeCl_3$ in 10ml water	M.P of 1,1'-bis-2-Naphthol = 218° C.
ORGANIC ESTIMATIONS			
SL NO	**EXPERIMENT NAME**	**CHEMICALS**	**SOLUTION REQUIRED**
6	Determination of Aniline / Phenol:	- Requirements: - Conc. HCl - Approximately 0.1N Brominating Mixture. - Exact 0.1N Sodium thiosulphate solution. - 10% KI solution. Starch solution.	Dissolve 5-6 grams of Aniline / Phenol and 5-6 ml of Conc. HCl in water to obtain 1000cc of solution.
7	Determination of Acetamide:	- Requirements: - 0.1N H_2SO_4 - 2. 1N KOH - 3.Phenolphthalein Indicator	Dissolve 15 grams of Acetamide in water to obtain 1000cc of solution
8	Determination of carboxylic acids.	- Requirements: - 0.1N NaOH - Phenolphthalein Indicator	Instruct the candidates to prepare 0.1N $H_2C_2O_4.2\ H_2O$ and use it for standardization of NaOH
9	Determination of aspirin.	- Requirements: - 0.2N HCl - 0.2N NaOH - Ethanol - Phenolphthalein Indicator	Dissolve 7-8 grams of aspirin in water to obtain 1000cc of solution. OrDistribute 2 aspirin tablets to candidates

Preparation of Brominating mixture: 2.8 g of $KBrO_3$ + 15.5 g KBr dilute up to mark in 1000cc flask with distilled water.

Specific Reagents for Organic Experiments B.Sc.V & VI

Sl. No.	Reagents	Preparation of solution
1.	Dil. Acetic acid(5N)	287 cc conc.Acetic acid & dilute it with 1000 cc of water.
2.	$NaHCO_3$	100 g in 1000cc water. Or 250 g in 1 litre(3N)
3.	NaOH(10%)	100 g in 1000cc water.
4.	Conc. NaOH	225 g in 1000cc water.
5.	Alcoholic KOH(10%)	10 g KOH in 100cc 50% alcohol
6.	HCl (5N)	454 cc con.HCl diluted to 1000cc.
7.	Bromine water	Dilute 10-15 cc liq.Bromine with 1 litre water. Or 5-6 cc liq. Br_2 in 50 cc water containing 20 g KBr. Dilute to 200 cc.
8.	Br_2 in acetic acid	20 cc Liq.Br_2 in 80 cc glacial acetic acid
9.	$KMnO_4$	1 g in 100 cc water(1%) : 6g in 100 cc water(6%)
10.	Alkaline $KMnO_4$	3 g in 100 cc water containing 10 g Na_2CO_3
11.	$FeSO_4$ solution(fresh)	15 g mixted with 1-2 cc con.H_2SO_4 & dilute with 100 cc water.
12.	Neutral $FeCl_3$ solution	10 g in 100 cc water
13.	Alcoholic $FeCl_3$ solution	10g in 100 cc alcohol
14.	$CaCl_2$ solution	Dissolve 55 g of hydrated $CaCl_2$ in 1 litre water.
15.	$NaNO_2$ solution(10%)	10 g in 100 cc water.
16.	Sodium nitroprusside	Dissolve 3g of compound in 1 litre.
17.	Brady's reagent(2,4 DNP)	Add 4g 2,4 dinitro phenyl hydrazine to 8 cc con.H_2SO_4 add slowly with shaking & cooling 70cc Methyl alcohol & make up to 100cc with water.
18.	Iodine solution (for iodoform)	20 g KI & 10 g Iodine in 100 cc water.
19.	Picric acid	5 g in 100 cc alcohol or 100cc benzene or glacial acetic acid
20.	β-naphthol	10 g of compound in 100cc 10% NaOH.
21.	Schiff's reagent	Rosaniline hydrochloride (0.2g) is dissolved in 100 cc water. & solution is saturated with SO_2 when colour of solution just vanishes.
22.	Hydroxylamine hydrochloride	5 g in 100 cc methyl alcohol.
23.	Tollen's reagent	2-3 drops of dil.NaOH (1%) solution to 5 cc $AgNO_3$. White ppt. obtained is dissolved in ammonia.

Specific Reagents for Physical Chemistry Experiments

B.Sc. III semester

Sl No.	Experiment name	Solutions required
1	Viscosity	Toluene and Carbon tetrachloride
2	Surface tension (Parachor of CH_2)	Benzene as liquid A. Toluene as liquid B o-Xylene as liquid C.
3	Surface tension (atomic parachor)	Toluene , Xylene amd n-Hexane
4	Second order reaction	0.05N potassium iodide, 0.05N $K_2S_2O_8$,0.002N Na_{2}-S_2O_3 , Freshly prepared starch
5	Heat of Neutralization	0.5N NaOH, 0.5N CH_3COOH, Conc. H_2SO_4 Phenolphthalein indicator
6	First order reaction	Methyl acetate, 0.5N HCl, 0.5H_2SO_4 0.01N NaOH,, Phenolphthalein indicator
7	Heat of Solution	0.5N NaOH, Conc. $H_2 SO_4$ and KNO_3 Phenolphthalein indicator
8	Adsorption	0.1N NaOH, 0.5N CH_3COOH Phenolphthalein indicator , Charcoal
9	Distribution	Benzoic acid, 0.1N NaOH,, Phenolphthalein indicator

First order reaction

Calculation of ‘V’$_\infty$ for 5 cc of Methyl acetate

1. Mol weight of methyl acetate = 74.03 and density = 0.932 g / cc
2. Density = mass/volume : volume = 74.03/0.932 = 79.48 cc

∴ When 5 cc of Methyl acetate with 100 cc of 0.5N HCl, is used, then its concentration in the reaction mixture is

79.48 cc dissolved in 1000 cc = 1N

7.948 cc dissolved in 100 cc = 1N

∴ 5cc diluted to (100+5)cc = 5 × 105/100×7.948 =0.66N

5cc of 0.66N of **methyl acetate** when titrated with 0.1 N NaOH (volume=B.R.)

= 0.66 ×5 / 0.1 = 33 cc

V$_\infty$ = V_0 (Zero time B.R. for HCl (say for 24cc)+ 33 cc for methyl acetate)

V_∞ = 24+ 33 =57 cc for 100% hydrolysis

But 100%hydrolysis is not possible ∴ 75% may be considered.

Hence, 75% hydrolysis **V**$_\infty$ **=** $\mathbf{V_0}$ + 75% of 33

= 24+ 24.75 = 48.75 ~ 48.0 cc

∴ **V**$_\infty$ **= 48.0 cc**

Specific Reagents for Physical Chemistry Experiments

Sl.No	Name	Concentration	Preparation
1	Sodium hydroxide Eq. wt. = 40 g	1N	Dissolve 40 g in 1000 cc of distilled water.
		0.1N	Take 100 cc 1N NaOH solution + 900 cc distilled water.
		0.2N	Take 400 cc of 0.5N NaOH +600 cc of distilled water.
		0.5N	Take 1000cc of 1N NaOH + 1000cc distilled water.
2	Hydrochloric acid Lab normality =11	1N	By dissolving 100cc conc. HCl in 900cc of distilled water it gives 1000cc of 1 N HCl solution.
		0.1N	Take 100cc of 1N HCl +900cc of distilled water it gives 0.1N HCl solution.
		0.5N	Take 1000cc of 1N HCl +1000cc distilled water it gives 2000cc of 0.5N HCl solution.
3	Sodium thiosulphate Mol.wt.=249	0.1N	Dissolve 24.9mg of $Na_2S_2O_3$ in 1000cc of distilled water it gives 0.1 N solution.
		0.002N	Dissolve 0.498g ~ 0.500 g in 1000cc of distilled water gives 0.002N solution.
		0.01N	Dissolve 2.49g of $Na_2S_2O_3$ in 1000cc of distilled water.
4	Potassium per sulphate Mol.wt=270	0.05N	Dissolve 6.8g of potassium per sulphate in 1000cc of distilled water it gives 0.05N solution.
5	Potassium iodide Mol.wt=166	0.05N	Dissolve 8.3 g of KI in 1000ccf distilled water.
		0.1N	Dissolve 16.6g of KI in 1000cc distilled water.
6	Potassium chloride Eq. wt. =74.56g	1N	74.56g of KCl dissolved in 1000cc distilled water
		0.1N	7.456g of KCl dissolved in 1000cc distilled water
7	Sodium chloride Eq. wt. =58.457g	1N	Dissolve 58.5g of NaCl in 1000cc distilled water
		0.1N	Dissolve 5.85g of NaCl in 1000cc of distilled water
		0.2N	Dissolve 11.7g of NaCl in 1000cc of distilled water
		0.5N	Dissolve 29.25g of NaCl in 1000cc of distilled water
8	Oxalic acid Eq. wt. =63.04g	1N	Dissolve 63.04g of oxalic acid in 1000cc of distilled water
		0.1N	Dissolve 6.304g of oxalic acid in 1000cc of distilled water
		0.5N	Dissolve 31.52g of oxalic acid in 1000cc of distilled water
9	Acetic acid Molarity=17.4/L	1N	Dissolve 57.47cc of acetic acid in 1000cc of distilled water
		0.1N	Dissolve 5.74cc of acetic acid in 1000cc of distilled water
		0.5N	Dissolve 28.73cc of acetic acid in 1000cc of distilled water

		0.2N	Dissolve 11.48cc of acetic acid in 1000cc of distilled water
10	Ferrous ammonium sulphate(FAS) Mol. wt. =392.2g	1N	Dissolve 392.2g of FAS in 1000cc of distilled water. (100 cc of conc.H_2SO_4+900ccc distilled water)
		0.1N	Dissolve 39.22g of FAS in 1000cc of distilled water. (100cc of conc.H_2SO_4+900ccc distilled water)
11	Potassium dichromate Eq. wt. =49.04g	1N	Dissolve 49.04g of potassium dichromate in 1000cc of distilled water.
		0.1N	Dissolve 4.904g of potassium dichromate in 1000cc of distilled water.
12	Sulphuric acid (98%) Lab normality=36.8	2N	Dissolve 56cc of conc.H_2SO_4 in 1000cc of distilled water.
		4N	Dissolve 112cc of conc.H_2SO_4 in 1000cc of distilled water.
		0.5N	Dissolve 14cc of conc.H_2SO_4 in 1000cc of distilled water
13	Ferric alum (Ferric thiocyanate complex)	0.001N	Dissolve 1g of Ferric alum in 50cc of conc. HCl and dilute upto1000cc with distilled water.
14	KCNS	2%	Dissolve 20g of KCNS in 1000cc of distilled water.
15	Phenolphthalein indictor	-	Dissolve 500mg of phenolphthalein powder in 500cc of absolute alcohol. Test with NaOH it gives pink colour.
16	Copper sulphate Mol. wt. =249.5	0.1M	Dissolve 24.95g of copper sulphate in 1000cc of distilled water. (Add 2 or 3 drops of H_2SO_4)
17	Sodium acetate Mol. wt. =82g	0.1M	Dissolve 8.2g of sodium acetate in 1000cc of distilled water.
		0.025M	Dissolve 2.05g of sodium acetate in 1000cc of distilled water.
18	Ethyl acetate Mol. wt. =96g	0.1M	Dissolve 9.6g of Ethyl acetate in 1000cc of distilled water.
19	Silver nitrate Mol. wt. =169.86g	0.01M	Dissolve 1.698g of silver nitrate in 1000cc of distilled water.
		0.02M	Dissolve 3.397g of silver nitrate in 1000cc of distilled water.

Preparation of liquid mixtures for Refractometry and Viscosity

Calculation of volume required to prepare the binary liquid mixture

1) Liquid ‘A’ is Benzene d=0.87g/cc 2) Liquid ‘B’ is Carbon tetrachloride d=1.59g/cc

M_1: a) 40% ‘A’ by weight: Calculation of volume of 40g of Benzene

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{40}{0.87} = \mathbf{46}\text{cc}$$

b) 60%’B’ by weight: Calculation of volume of 60g of Carbon tetrachloride

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{60}{1.59} = 37.7\text{cc}$$

M_2: a) 50% ‘A’ by weight: Calculation of volume of 50g of Benzene

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{50}{0.87} = 57.5\text{cc}$$

b) 50% ‘B’ by weight: Calculation of volume of 50g of Carbon tetrachloride

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{50}{1.59} = 31.5\text{cc}$$

M_3: a) 60% ‘A’ by weight: Calculation of volume of 60g of Benzene

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{60}{0.87} = 69.00\text{cc}$$

b) 40% ‘A’ by weight: Calculation of volume of 40g of Carbon tetrachloride

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{40}{1.59} = 25.2\text{cc}$$

M_4: a) 70% ‘A’ by weight: Calculation of volume of 70g of Benzene

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{70}{0.87} = 80.5\text{cc}$$

b) 30% ‘B’ by weight: Calculation of volume of 30g of Carbon tetrachloride

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{30}{1.59} = 18.9\text{cc}$$

Unknown ‘C’: 55% of ‘A’+45% of ’B’

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{55}{0.87} = 63.2\text{cc}$$

$$d = \frac{M}{V} \qquad v = \frac{M}{d} = \frac{45}{1.59} = 28.3\text{cc}$$

A+B=Benzene +Carbon tetra chloride

M_1=40%A+60%B =46cc+37.7cc

M_2=50% A+50%B=57.5cc+31.5cc

M_3=60% A+40%B=69.0cc+25.2cc

M_4=70%A +30%B=80.5cc+18.9cc

Unknown: Liquid ‘C’=55% Benzene+45% CCl_4=63.2cc+28.3cc

General Instructions to the Examiners

B.Sc Semesters I

Inorganic

i. In a batch of ten students at least two different acid- base and two different redox / complexo metric titration experiments may be distributed to the students.
ii. It may be reversed in the subsequent batches to cover eight experiments.
iii. Selection of experiments may be done by the students based on lot.
iv. Discourage the students to use chart but may be allowed to refer whenever necessary.
v. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.
vi. Distribute: 20, 22, 24, 26, 28 & 30 cc at random in 250 cc volumetric flask or 8, 10, 12 &14 at random in 100 cc volumetric flask.
vii. Instruct the candidates to use 25cc /10cc diluted solution for determination.
viii. Ask the candidate to prepare the required solutions in 250cc / 100cc
ix. Experiments related to reduction of Fe^{3+} to Fe^{2+}, iodimetry and determination of temporary, permanent and total hardness of water using standard EDTA method may be avoided for examination.
x. However, such experiments should be performed in regular practicals.

B.Sc.I sem Distribution of Marks:	
Accuracy Standardization titration Main titration	 10 marks 16 marks
Technique & Presentation	04 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

Deduction of Marks for accuracy	
Standardization titration	Main titration
± 0.2 cc -10 marks	± 0.2 cc -16 marks
± 0.4 cc - 08 marks	± 0.4 cc - 14 marks
± 0.6 cc - 06marks	± 0.6 cc - 12 marks
± 0.8 cc - 04 marks	± 0.7 cc - 10 marks
Above ± 0.8 cc - zero marks	± 0.8 cc - 08 marks
	± 0.9 cc - 06 marks
	above ± 1.0 – zero mark

B.Sc Semesters II
Organic
General Instructions to the Examiners

i. In a batch of ten students, for six students preparation experiments and for four students determination experiments may be given in the practical examination.

ii. Two different preparation experiments and two different determination experiments may be distributed to the students.

iii. Selection of experiments may be done by the students based on lot. Discourage the students to use chart but may be allowed to refer whenever necessary.

iv. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.

B.Sc.II sem Organic Preparation experiments Distribution of Marks	
Yield	16 marks
Nature of crystals	04 marks
Technique & Presentation	04 marks
Purity and M.P(4+2)	06 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

Deduction of Marks for accuracy in Preparation experiments
Error in yield less than 10% - 16 marks
11 - 15% = 14 marks
16 - 20% = 12 marks
21 - 25% = 10 marks
More than 30% = zero mark

B.Sc.II sem Organic Determination experiments Distribution of Marks:	
Accuracy Blank titration Main titration	 10 marks 16 marks
Technique & Presentation	04 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

Deduction of Marks for accuracy in Determination experiments:	
Blank titration	Main titration
± 0.2 cc -08 marks	± 0.2cc -12marks
± 0.4 cc- 06 marks	± 0.4 cc- 10 marks
± 0.6 cc- 04marks	± 0.6cc- 08 marks
± 0.8 cc- 02 marks	± 0.7 cc- 06 marks
Above ± 0.8 cc zero marks	± 0.8 cc- 04 marks
	± 0.9 cc- 02 marks
	above ± 0.9cc – zero mark

B.Sc Semesters III

Physical

General Instructions to the Examiners

i. In a batch of ten students, not more than two students should get the same experiment in the practical examination.
ii. Selection of experiments should be done by the students based on lots.
iii. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.
iv. During practical examination chart is not allowed, wherever necessary simple procedure may be given.
v. Determination of percentage composition by viscosity method may be avoided in examination.

Distribution of marks for accuracy- 18 marks

1. **First order and second order reactions**.
   a. For accuracy in calculation of ‘k’ by formula method: any three ‘k’ values from each set, matching with the expert values may be considered. Each carries 3 marks.
   b. For accuracy in calculation of ‘k’ from graphical method, each set carries 9 marks.
2. **Enthalpy of ionization and solution**
   a. Water equivalent of calorimeter carries 06 marks.
   b. Enthalpy of ionization or solution carries 12 marks.
   c. 06 marks shall be deducted if water equivalent of calorimeter is not in between 0.45 & 0.52 KJ.
   d. Under such condition use 0.52 KJ as water equivalent of calorimeter and calculate the enthalpy of ionization / solution.
3. **Surface tension –** Parachor of CH_2
   a. Determination of atomic parachor carries 18 marks
   b. Each liquid carries 06 marks
4. **Surface tension –atomic parachor**
   a. Determination of atomic parachor carries 18 marks
   b. 09 marks may be deducted if parachor of CH_2 is not in between 38-40.
   c. Under such condition use 39 as parachor of CH_2 and calculate the atomic parachor of ‘C’ and ‘H’.
5. **Viscosity of pure liquids**
   a. 06 marks for determination of density of liquids: each liquid carries 03 marks.
   b. 12 marks for determination of viscosity coefficient of liquids: each liquid carries 06 marks.

6. **Viscosity of liquids: percentage composition**
    a. 03 marks for determination of density of liquid ‘C’.
    b. 15 marks for determination of percentage composition of liquid ‘C’.
7. **Distribution**

    Any three ‘K^I’ values matching with the expert values may be considered: each carries 06 marks.
8. **Adsorption**
    a. Any three burette readings after adsorption matching with the expert values may be considered: each carries 06 marks.
    b. Do not take in to account of magnitude of n and K for accuracy.
9. **Determination of degree of dissociation of KCl by Landsberger’s method**.
    a. Each degree of dissociation carries 06 marks: total – 18 marks.
    b. Degree of dissociation should be decreased with increase in concentration of KCl.

B.Sc.III sem General distribution of Marks:	
Accuracy	18 marks
Technique and Presentation	03 marks
Calculation and graph(5+4)	09 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

General deduction of Marks for accuracy:
Error up to 5% = 18 marks
6 - 10% = 15 marks
11-15% = 12 marks
16-20% = 6 marks
above 20% = zero (0) mark

Deduction of Marks for accuracy in 12 + 06 System:

Error	Against marks 12	Against marks 06
up to 5%	12 marks	06 marks
6 - 10%	10 marks	05 marks
11-15%	08 marks	04 marks
16-20%	05 marks	02 marks
above 20%	zero (0) mark	zero (0) mark

B.Sc Semester IV

INORGANIC CHEMISTRY EXPERIMENTS:
General Instructions to the Examiners

i. In a batch of ten students, not more than two students should get the same mixture in the practical examination.

ii. Determination of DO, COD and BOD experiments may be avoided in examination.

iii. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.

iv. Discourage the students to use chart but may be allowed to refer whenever necessary.

Semi-micro qualitative analysis of mixtures of two simple inorganic salts containing two anions and two cations.

Anions: CO_3^{2-}, S^{2-}, Cl^-, Br^-, NO_3^-, SO_4^{2-}
Cations: Pb^{2+}, Cu^{2+},Al^{3+}, Fe^{2+}, Fe^{+3}, Mn^{2+}, Ni^{2+}, Zn^{2+},Ca^{2+}, Ba^{2+}, Mg^{2+}, Na^+, K^+ and NH_4^+.

- Mixture having Phosphate / oxalate radical may not be given.
- Salts with following combinations may be given.

Sl.No	Mixtures	Sl.No	Mixtures
1	$CaCO_3$ + NH_4Cl	15	$BaCl_2$ +$(NH_4)_2CO_3$
2	$Al_2(SO_4)_3$ + Na_2CO_3	16	$Al_2(SO_4)_3$ + $MgCO_3$
3	$MgSO_4$ + ZnS	17	KCl + ZnS
4	$CaCO_3$ + KBr	18	$Al_2(SO_4)_3$ +NaCl
5	$BaCl_2$ + KBr	19	$BaCl_2$ +NH_4Br
6	$ZnSO_4$ + $MgCO_3$	20	$MgSO_4$ +$ZnCO_3$
7	Na_2CO_3 + $FeSO_4$	21	KCl + $FeSO_4$
8	$FeSO_4$ + $ZnCO_3$	22	$MgSO_4$ + $ZnCO_3$
9	$FeSO_4$ + KNO_3	23	$FeSO_4$ + Na_2CO_3
10	$NiCO_3$ + $Ba(NO_3)_2$	24	$NiSO_4$+ KBr
11	$MnSO_4$+ KBr	25	$NiSO_4$+ Na_2CO_3
12	$CuSO_4$ + KNO_3	26	$CuSO_4$ + KBr
13	$PbCO_3$ + NH_4Br	27	$Pb(NO_3)_2$ + $(NH_4)_2CO_3$
14	$MnSO_4$+ Na_2CO_3		

B.Sc.IV sem Distribution of Marks:	
Preliminary tests and presentation (6+2)	08 marks
Negative radicals(group test + C.T)(2+3)×2	10 marks
Positive radicals (group test + C.T) 2+4)×2	12 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

B.Sc Semester V

ORGANIC CHEMISTRY EXPERIMENTS: Paper-I

(Solid- Solid Mixture)

General Instructions to the Examiners

i. In a batch of ten students, not more than two students should get the same mixture in the practical examination.

ii. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.

iii. For Separation of Organic Mixture and Qualitative Analysis of Organic compounds the binary mixtures of organic compounds may be prepared by mixing two compounds of different nature.

iv. The acids and phenol compounds should not be mixed and hence such mixture may not be given.

v. One pure organic compound from the separated mixture may given for analysis after separation.

vi. During practical examination chart may be referred whenever necessary

Acids: Salicylic, Cinnamic, Phthalic and Anthranilic acid.

Phenol: α –naphthol, β–naphthol.

Base: p-toluidine, m-nitroaniline and p-nitroaniline.

Neutral: Naphthalene, Acetanilide, Diphenyl, Benzamide, Benzophenone and m- dinitrobenzene.

NOTE:

Sl. No.	Mixture of Solid organic compounds		Nature	Mixture of Solid organic compounds	
	Compound for analysis	Compound for analysis		Compound for analysis	Compound for analysis
1.	Salicylic acid	Acetanilide	A + N	Acetanilide	Cinnamic acid
2.	p-nitroaniline	Salicylic acid	A + B	Salicylic acid	o-nitroaniline
3.	α –naphthol/ β–naphthol	Benzamide	P + N	β–naphthol	Naphthalene
4.	Benzophenone	α – naphthol/ β–naphthol	N + P	α–naphthol	Benzamide
5.	p-toluidine	Cinnamic acid	A + B	Cinnamic acid	p-toluidine
6.	m-dinitrobenzene	Phthalic acid	N + A	m-dinitrobenzene	p-nitroaniline
7.	p-toluidine	Diphenyl	B + N	Benzamide	p-nitroaniline
8.	Benzophenone	Phthalic acid	N + A	Diphenyl	Phthalic acid
9.	Anthranilic acid	m-dinitrobenzene	A+N	Anthranilic acid	m-dinitrobenzene
10.	β–naphthol	Benzophenone	P + N	β–naphthol	Benzophenone

* A new compound may be given for preparation of derivative when the given compound for analysis fails / tedious to give the derivative.

B.Sc.V Sem Paper-I :Organic Separation & Qualitative Analysis Experiments Distribution of Marks	
Nature & Separation (2+3)	05 marks
Preliminary test	02marks
Element test	04 marks
Physical Constant	03 marks
Functional Group test	04 marks
Identification & structure	03 marks
Preparation of derivative	03 marks
Physical constant of derivative	03 marks
Systematic Presentation	03marks.
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

B.Sc Semester V

PHYSICAL CHEMISTRY EXPERIMENTS: Paper -II

General Instructions to the Examiners

i. In a batch of ten students, not more than two students should get the same experiment in the practical examination.
ii. Selection of experiments may be done by the students based on lots.
iii. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.
iv. Out of 18 marks reserved for accuracy, 06 marks shall be distributed for determination of cell constant in the conductometric experiments wherever applicable.
v. During practical examination chart is not allowed, wherever necessary simple procedure may be given.
vi. pH of biological Juices may not be given in examination.

B.Sc.V sem General distribution of Marks:	
Accuracy	18 marks
Technique and Presentation	03 marks
Calculation and graph(5+4)	09 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

General deduction of Marks for accuracy:
Error up to 5% = 18 marks
6 - 10% = 15 marks
11-15% = 12 marks
16-20% = 6 marks
above 20% = zero (0) mark

Deduction of Marks for accuracy in 12 + 06 System:		
Error	Against marks 12	Against marks 06
up to 5%	12 marks	06 marks
6 - 10%	10 marks	05 marks
11-15%	08 marks	04 marks
16-20%	05 marks	02 marks
above 20%	zero (0) mark	zero (0) mark

B.Sc Semester VI

Inorganic Chemistry Experiments:Paper-I

Gravimetric and Volumetric Analysis

General Instructions to the Examiners

i. In a batch of ten students in the practical examination, five students may be given volumetric determination and preparation of complexes (if time permits) and the other five students may be given gravimetric determination.

ii. Selection of experiments may be done by the students based on lots.

iii. Viva questions may be asked on any of the experiments prescribed in the practical syllabus.

iv. Discourage the students to use chart but may be allowed to refer whenever necessary **and instruct to use electronic balance for weighing.**

v. Out of 12 experiments in Inorganic Chemistry, only 07 experiments (four gravimetric and three volumetric experiments) are to be given.

vi. Preparation of complex may be given if time permits; ***otherwise the marks allotted for preparation (Yield of the complex- 06marks) may be added to accuracy in volumetric analysis.***

Inorganic Volumetric Determination

1. **Extraction of Iron(III) from haematite ore or solid Fe_2O_3 and determination of percentage of iron in the solution using standard $K_2Cr_2O_7$ solution(internal indicator method).**

   Distribution: 20, 22, 24, 26, 28 & 30 cc at random in 250 cc volumetric flask flasks may be given. Or 10, 11, 12, 13, 14 & 15 cc at random in 100 cc volumetric flask flasks may be given. Instruct the candidates to use 25cc diluted solution for determination and to prepare 0.05N $K_2Cr_2O_7$

2. **Extraction of Cu and Zn from brass and determination of percentage of copper in the solution using standard $Na_2S_2O_3$ solution.**

   Distribution:18, 20, 22, 24, 26 & 28 cc in 250 cc at random in 250 cc volumetric flask may be given. Or 09, 10, 11, 12, 13 & 14 cc in 100 cc at random in 250 cc volumetric flask may be given.

   Instruct the candidates -

   i. To dilute Cu^{2+} or Brass solution to 250 cc in a volumetric flask and use 25cc diluted solution for determination

   ii. To prepare 0.05N $Na_2S_2O_3.5H_2O$.

3. Extraction of calcium from limestone and determination of percentage of calcium in the solution by oxalate method.

Limestone – Distribution: 0.300, 0.350, 0.400, 0.450 & 0.500 g $CaCO_3$ to candidates

Instruct the candidates -

i. To dissolve sample of limestone in minimum amount of 1:1 HCl dilute to 250 / 100cc with distilled water.

ii. To use 25cc diluted solution for determination and to prepare 0.1N $KMnO_4$.

B.Sc.VI sem : Paper-I Inorganic Volumetric & Preparation Experiments Distribution of Marks:	
Accuracy For two titre values(2×5)	10 marks
Yield of the complex	06 marks
Technique & Presentation	02 marks
Calculation	02 marks
Journal	05 marks
Viva-Voce	05 marks
Tour Report or Project Report	10 marks
Total	40 marks

Inorganic Gravimetric Determination

1. Determination of Barium as $BaSO_4$

Distribution: 22, 24, 26, 28, 30, 32, 34 & 36 cc at random in 250 cc volumetric flask may be given or. 11, 12, 13, 14, 15, 16, 17 & 18 cc at random in 100 cc volumetric flask may be given. Instruct the candidates to use 25cc diluted solution for determination.

2. Determination of Al as Al_2O_3

150g of $AlCl_3.6H_2O$ + 10 cc of Conc. HCl or 106 g of $Al_2(SO_4)_3$ + 10 cc of Conc.H_2SO_4 make a paste & add water to make the volume to 1000cc.

Distribution: 26, 28, 30, 32, 34 & 36 cc at random in 250 cc volumetric flask or 11, 12, 13, 14, 15, 16, 17 & 18 cc at random in 100 cc volumetric flask may be given.

3. **Determination of iron as Fe_2O_3**

140g of $FeCl_3.6H_2O$ or 80g of anhydrous $FeCl_3$ + 10 cc of Conc.HCl make a paste & add water to make the volume 1000cc.

Distribution: 26, 28, 30, 32, 34 & 36 cc at random in 250 cc volumetric flask may be given. Instruct the candidates to use 50cc diluted solution for determination.

4. **Determination of Lead as $PbSO_4$**

100 g $Pb(NO_3)_2$+ 5 cc of conc. HNO_3 diluted to 1000cc. or1 00g $Pb(CH_3COO)_2.3H_2O$ +
little acetic acid diluted to 1000cc.

Distribution: 26, 28, 30, 32, 34 & 36 cc at random in 250 cc volumetric flask or 13, 14, 15, 16, 17 & 18 cc at random in 100 cc volumetric flask may be given. Instruct the candidates to use 25cc diluted solution for determination.

Note: Industrial study tour report or Project work report is compulsory. The report carries 10 marks and it should be submitted in paper-I practicals.

The following projects may be considered Soil analysis, water analysis, sewage water treatment and management, ion exchange methods, chromatography, preparation and assay of organic compound of pharmaceutical importance , effect of pesticides and insecticides, milk analysis, chemical processes in; textile industries / fertilizer industries/ dairy industries / chemical industries / ceramic industries / sugar industries/ oil industries / solvent extraction / liquor industries, alternate sources of generating electric power / engine fuel (apart from these, any other projects of student interest may also be considered).

B.Sc.VI sem: Paper-I Inorganic Gravimetric experiments Distribution of Marks:	
Accuracy	16 marks
Technique and calculation	04 marks
Journal	05 marks
Viva-Voce	05 marks
Tour Report or Project Report	10 marks
Total	40 marks

Deduction of Marks for accuracy in Gravimetric experiments:
± 6 mg - 16 marks
± 8 mg - 14 marks
± 10 mg - 12 marks
± 12 mg - 10 marks
± 14 mg - 08 marks
± 16 mg - 06 marks
above ± 16 mg- zero (0) mark

B.Sc VI Semester :Paper –II

Organic Experiments(Liquid – Liquid mixture)

General Instructions to the Examiners

i. In a batch of ten students in the practical examination, five students may be given experiment number 1-6 (binary mixture) and remaining five students may be given experiment number 7-12 (physical experiments).

ii. Selection of experiments may be done by the students based on lot.

iii. In a batch of five students in the practical examination(1-6), not more than two students should get the same mixture.

iv. Viva questions may be asked on any of the experiments prescribed in the practical syllabus. Discourage the students to use chart but may be allowed to refer whenever necessary.

Instructions to the Examiners for Organic Experiments

1. The experiments are to be allotted for physical and organic by lot system.
2. For Separation of Organic Mixture and Qualitative Analysis of Organic compounds, the binary mixtures of organic compounds can be prepared by mixing two compounds one from low boiling and other from high boiling liquid as given below. And one pure organic compound is given for analysis.

   Low Boiling: ethyl acetate, acetone, toluene, chlorobenzene.

   High Boiling: phenol, aniline, nitrobenzene, benzaldehyde, acetophenone, bromobenzene.
3. Following binary liquid mixtures can be given.

Sl.No.	Mixture of liquid Organic compounds		Compound for analysis	Compound for analysis
1.	Aniline- 184^{o}C	Ethyl acetate-77^{o}C	Ethyl acetate	Aniline
2.	Acetone-56^{o}C	Nitrobenzene -209^{o}C	Acetone	Nitrobenzene
3.	Toluene-110^{o}C	Benzaldehyde-179^{o}C	Toluene	Benzaldehyde
4.	Acetone- 56^{o}C	Bromobenzene-155^o C	Bromobenzene	Acetone
5.	Acetophenone-202^{o}C	Chlorobenzene-132^o C	Acetophenone	Chlorobenzene
6.	Phenol -182^{o}C	Toluene -110^{o}C	Toluene	Phenol
7.	Acetone- 56^{o}C	Toluene -110^{o}C	Acetone	Toluene
8.	Nitrobenzene-209^{o}C	Ethyl acetate-77^{o}C	Nitrobenzene	Ethyl acetate
9.	Ethyl acetate-77^{o}C	Bromobenzene-155^o C	Ethyl acetate	Bromobenzene
10	Toluene-110^{o}C	Acetophenone-202^{o}C	Toluene	Acetophenone

4. A new compound may be given for preparation of derivative when the given compound for analysis fails / tedious to give the derivative.

B.Sc.VI sem Paper-II : Organic Separation & Qualitative Analysis Experiments Distribution of Marks	
Separation	03 marks
Preliminary test	02 marks
Nature	02 marks
Element test	04 marks
Physical Constant	03 marks
Functional Group test	04 marks
Identification & structure	03 marks
Preparation of derivative	03 marks
Physical constant of derivative	03 marks
Systematic Presentation	03 marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

B.Sc VI Semester- Paper –II

PHYSICAL CHEMISTRY EXPERIMENTS

Instructions to the Examiners for Physical Experiments

i. In a batch of five students in the practical examination(7-12), not more than two students should get the same experiment.

ii. Selection of experiments may be done by the students based on lot.

iii. Out of 18 marks reserved for accuracy, 06 marks may be distributed for determination of cell constant in the conductometric experiments wherever applicable.

iv. Charts and scientific calculators are not allowed.

B.Sc. VI Semester- Paper –II Physical Experiments Distribution of Marks:	
Accuracy	18 marks
Technique & Presentation	03 marks
Calculation & Graph (5+4)	09marks
Journal	05 marks
Viva-Voce	05 marks
Total	40 marks

Deduction of Marks for accuracy in Physical Experiments
Error up to 5 % - 18 marks
6 - 10% 15 marks
11 – 15 % 12 marks
16 - 20% 06 marks
Above 20% 00 marks
More than 30% zero (0) mark

B.Sc. Sem I

Inorganic Chemistry: Volumetric Analysis

Expt.: To determine the HCl /NaOH / Na_2CO_3 + $NaHCO_3$ / $H_2C_2O_4$ + H_2SO_4 /FAS/ Fe^{2+} / Zn^{2+} volumetrically ***(perform marked expt.)***

Standardize the given solution of ________________by preparing 100/250 cc ______0.05N/0.1N standard solution of _______________. Determine the amount of ________________ present in 100/250 cc volumetric flask bearing your table number and in $1dm^3$ using the above standardized solution.

Instructions:

1. Chart and Scientific Calculator are not allowed.
2. Dilute the given ______solution up to the mark with distilled water, shake it well and use 25 ml of the diluted solution for determination.
3. Prepare100/250 cc ….. . 0.05N/M solution of Na_2CO_3 / $H_2C_2O_4.2H_2O$/ $K_2Cr_2O_7$/ $Na_2S_2O_3.5H_2O$/ **Mohr's salt** /$ZnSO_4.7H_2O$
4. Write all the reactions involved.
5. Get all readings initialed by the examiner.
6. Present your results as below.

Results:

1. 25cc of _____solution required 1.______2._____ cc of ______solution.
2. Normality/Molarity of the prepared standard solution of ______ is _______N/M.
3. Normality/Molarity of the given standardized solution of ______ is _______N/M.
4. Amount of ____ present in given 100/250 ml solution is ______g
5. Amount of ____________present in $1dm^3$ is ______g

B.Sc. Sem II

Organic Chemistry-Estimation

Expt. No.1: To determine the amount of ANILINE/PHENOL(perform marked expt.)

You are given a solution of Aniline/Phenol in 100/250 cc volumetric flask bearing your table number. Determine the amount of Aniline/Phenol present in the given solution volumetrically by bromination method.

INSTRUCTIONS:

a) Chart and Scientific Calculator are not allowed.
b) Dilute the given solution up to the mark in the flask with distilled water and use 10/25cc of the diluted solution for the each determination.
c) **Standardization:** Standardize the given brominating solution (0.1N approx) supplied in Bottle 'A' by titrating 10/25ml of this solution against standard 0.1N sodium thiosulphate solution supplied in Bottle 'B'. Take at least two readings.
d) **Main titration:** Take at least two readings for the main titration.
e) Get all readings initialed by the examiner.
f) Present your results as below.

RESULTS:

1. 0.1N $Na_2S_2O_3$ solution required for 25 cc of the brominating solution (Blank Titration)
   i) cc ii) cc
2. 0.1N sodium thiosulphate required for excess of brominating solution added (Main Titration)
   i) cc ii) cc
3. Amount of Aniline/Phenol in the given solution …………….. g.

Expt. *No.2:* To determine the amount of *ACETAMIDE*

You are given a solution of Acetamide in 100/250 cc volumetric flask bearing your table number. Determine the amount of Acetamide present in the given solution volumetrically.

INSTRUCTIONS:

a) Chart and Scientific Calculator are not allowed.

b) Add 25 cm^3 of KOH solution to the given acetamide solution in a conical flask and boil it with reflux condenser for 1 hour.

c) **Standardization:** Dilute the KOH solution up to the mark in the flask with distilled water and use 25ml of the diluted solution for the each titration. Take at least two readings.

d) **Main Titration:** Take at least two readings for the main titration.

e) Get all readings initialed by the examiner.

f) Present your results as below.

RESULTS:

1. 0.1 N H_2SO_4 required for 25 cc of the dilute KOH solution:(Blank titration Reading)
   i) cc ii) cc
2. 0.1 N H_2SO_4 required for 25 cc of the dilute solution after hydrolysis :
   (Main titration Reading) i) cc ii) cc
3. Amount of Acetamide in the given solution …………….. g.

Expt. *No.3:* To determine the amount of Carboxylic Acid

You are given a solution of Carboxylic acid in 100/250 cc volumetric flask bearing your table number. Determine the amount of Carboxylic acid present in the given solution volumetrically**.**

INSTRUCTIONS:

1. Chart and Scientific Calculator are not allowed.
2. Dilute the given solution up to the mark in the flask with distilled water and use 25cc of the diluted solution for each determination.
3. **Standardization:** Standardize the given NaOH supplied in Bottle ‘B’ by titrating 25 cc of this solution against standard 0.1N oxalic acid solution. Take at least two readings.
4. **Main Titration:** Take at least two readings for the main titration.
5. Get all readings initialed by the examiner.
6. Present your result as below.

RESULTS:

1.____ N NaOH required for 25 cc of the 0.05N oxalic acid solution(StandardizationTitration):
i) cc ii) cc
2. ___N NaOH required for 25 cm^3 of the diluted carboxylic acid solution:
i) cc ii) cc

3. Normality of carboxylic acid ………..N.
4. Amount of carboxylic acid present in 1 dm^3 ………..g
5. Amount of carboxylic acid present in given solution ……… .g

Expt. ***No.4:*** **To determine the amount of ASPIRIN**

You are given a solution of aspirin in 100/250 cc conical flask bearing your table number. Determine the amount of aspirin present in the given solution volumetrically.

INSTRUCTIONS:

1. Chart and Scientific Calculator are not allowed.
2. To the given aspirin solution, add 25 cc of NaOH solution and warm it for 15minutes.
3. **Standardization:** Titrate 25 cc NaOH solution against standard (0.2M exact) HCl solution. Take at least two readings.
4. **Main titration:** Titrate reaction mixture containing aspirin (after hydrolysis) against standard HCl solution. Take at least two readings.
5. Get all readings initialed by the examiner.
6. Present your results as below.

RESULTS:

1. ______ N HCl required for 25 cc of the NaOH solution (Blank Titration):
   i) cc ii) cc
2. _____ N HCl required for 25 ml of the reaction mixture containing aspirin (after hydrolysis) (Main Titration): i) cc ii) cc
3. Amount of aspirin present in given solution ……… .g

<u>Note: Marks are reserved for accuracy, calculation, Journal and viva –voce.</u>

* Student can ask an additional 30 minutes to complete the practical if necessary

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Expt. **No.5***:* To Prepare acetanilide / phthalimide /1,1'-bis-2-naphthol/ p-nitroacetanilide using the appropriate starting material supplied to you in the container.***(perform marked expt.)***

INSTRUCTIONS:

a) Chart and Scientific Calculator are not allowed.
b) Follow the usual procedure.
c) Write all the reactions and mention the quantities of reagents used.
d) Recrystalise a small amount of compound with suitable solvent.
e) Record the M.P. of the recrystalised compound and get initialed by examiner.
f) Record the yield and submit the crude product in a packet bearing your table number.

B.Sc. Sem III

Physical Experiments

Chart and Scientific Calculator are not allowed in the examination.

First Order reaction

Q.No.1: To study the effect of acid strength on hydrolysis of methyl acetate using HCl and H_2SO_4

Perform the experiment as follows

Set – I : 5 cc Methyl acetate + 0.5N HCl (Reaction mixture)

- Pipette out 5cc of reaction mixture and titrate against 0.1N NaOH at the interval of 0,10, 20, 30, 40 and 50 minutes.
- Present the results in the following tabular form.

The titre value after completing the reaction V_∞ : ……..cc

Initial Concentration, a = V_∞ - V_0 = ….. cc

Table

Time (min)	Vol of NaOH (cc)	x =	a-x =	log (a-x)	k
0	V_o =				
10	V_t =				
20	V_t =				
30	V_t =				
40	V_t =				
50	V_t =				

- **Set II: Repeat the same with 5cc methyl acetate + 0.5N H_2SO_4**
- Calculate the first order rate constant 'k' for the marked:
  1. Formula method (take mean of k as k_1 for HCl and k_2 for H_2SO_4)
  2. Graphical method.
- Calculate relative strengths of both the acids.

Second order reaction

Q.No.2: To study the second order reaction between potassium iodide and potassium persulphate and show that the velocity constant is independent upon concentration by performing the experiment at different concentrations (a = b only) of KI and $K_2S_2O_8$ at least in two sets as below.

Perform the experiment as follows:

- **Set –I : 50 cc of 0.05N KI + 50 cc of 0.05N $K_2S_2O_8$ (Reaction mixture)**
- Pipette out 10cc of reaction mixture and titrate against 0.002N $Na_2S_2O_3$ at the intervals of 5,10,15, 20,25 and 30 minutes.
- Calculate initial concentration 'a' for both sets.
- Present the results in the following tabular form.

Table I

Time in minute	Titre readings 'x' in cc	a-x	$\frac{1}{a-x}$	k
5				
10				
15				
20				
25				
30				

- **Set II: Repeat the experiment with reaction mixture:**
  25 cc 0.05 N KI + 25 cc 0.05 N $K_2S_2O_8$ + 50 cc H_2O.
- Calculate the second order rate constant 'k' for the marked:
  1. Formula method
  2. Graphical method.

Compare the 'k' of both the sets and comment.

Adsorption

Q.No.3: To study the adsorption of acetic acid on activated charcoal in the following concentrations.

Perform the experiment as follows:

- Prepare the different concentrations of acetic acid as in table 1.

Table 1:

Bottle No.	1	2	3	4	5
0.5N acetic acid (cc)	50	40	30	20	10
Water (cc)	0	10	20	30	40

- Add 1 g. activated charcoal to each bottle.
- Stopper the bottles ,shake well for about 30 minutes and keep them in water bath to attain lab temperature.
- Titrate the acetic acid against **0.1N NaOH** as given in table 2.

Table 2 :

Bottle No.	Titre readings		Conc. of acid		Amount of acid adsorbed by 1 gram of charcoal $X = C_0 - C_e$	log X	log C_e
	Before adsorption	After adsorption	Before adsorption C_0	After adsorption (equilibrium) C_e			
1							
2							
3							
4							
5							

- Volume of 0.1N NaOH required for 5 cc original CH_3COOH is V = ………cc
- Calculate C_0 & C_e using titre readings.
- Plot a graph of log 'x' against log C_e and calculate 'n' and 'K'

Viscosity of Pure liquids

Q.No.4: To determine the density and viscosity coefficient of liquids A and B, given that Viscosity coefficient of water = 0.0089 poise.

Perform the above experiment using Ostwald's Viscometer:

Surface Tension

Q.No.5: To determine the surface tension of benzene, **toluene and xylene** by the drop number method and calculate the parachor of CH_2.

Given : Surface tension of water is 72 dyne / cm

Given: Mol Mass of Benzene = 78; Toluene= 92 ; Xylene=106

Given: Density of Benzene = 0.878; Toluene = 0.866; Xylene=0.881 g / cc

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Surface Tension(Atomic parachor)

Q.No.6: To determine the surface tension of toluene, xylene and n-hexane by the drop number method and and calculate the atomic parachor of Carbon and Hydrogen.

Given : Surface tension of water is 72 dyne / cm

Given: Mol Mass of Toluene= 92 ; Xylene=106 : Hexane= 86

Given: Density of Toluene = 0.866; Xylene=0.881: Hexane= 0.655 g / cc

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Distribution

Q.No.7: To study the distribution of benzoic acid between water and toluene.

Perform the experiment as follows: Prepare **solution mixtures** as given in table 1.

Table-1:

Bottle No.	Volume of water (cc)	Volume of toluene (cc)	Wt.of benzoic acid.(gram)
1	80	20	0.5
2	80	20	1.0
3	80	20	1.5
4	80	20	2.0

- Stopper the bottles; shake well for about 30 minutes keep them in water bath to attain lab temperature.
- Titrate benzoic acid in toluene and water layers against 0.1N NaOH as given in table 2. Calculate K and K^I ; and comment.

Table-2:

Bottle No.	Burette reading of 0.1N NaOH		Concentration of benzoic acid		$K= C_1/C_2$	$K^I=C_1/\sqrt{C_2}$
	Aqueous layer (25 cc)	Toluene layer (5 cc)	Aqueous layer (C_1)	Toluene layer (C_2)		
1						
2						
3						
4						

Viscosity (percentage composition)

Q.No.8:To determine the viscosity of binary liquid mixtures of Toluene & carbon tetrachloride and calculate their percentage composition of unknown mixture. Determine the density of Mixture. Given that Viscosity coefficient of water = 0.0089 poise.

Perform the experiment using Ostwald's Viscometer:

Calorimetry (Enthalpy of ionization)

Q.No.9: To determine the enthalpy of ionization of acetic acid by calorimetric method.

Perform the above experiment as follows:

- Determine the water equivalent of calorimeter using 2 cc concentrated H_2SO_4 and 98 cc of water.
- Determine the normality of H_2SO_4 by titrating against 0.5N NaOH.
- Find out the 'Q' liberated for [H_2SO_4] from the std. graph.
- Use 50 cc of 0.5N CH_3COOH and 50 cc of 0.5N NaOH for calculation of Enthalpy of ionization.

Given: Enthalpy of strong acid and strong base: - 57.32 kJ / eqv.

Ebullioscopy

Q.No.10: To determine the degree of dissociation of KCl by Landsberger's method.

Perform the experiment as follows:

Find out the boiling point of pure water and solutions of KCl having 0.5, 1.0 and 1.5g in 15 cc water. Calculate Mol.wt. and degree of dissociation for all the concentrations of KCl.

(Given that the K_b of water = 0.525)

Calorimetry (Enthalpy of Solution)

Q.No.11: To determine the enthalpy of solution of KNO_3 by calorimetric method.

Perform the experiment as follows:

- Determine the water equivalent of calorimeter using 2 cc concentrated H_2SO_4 and 98 cc water.
- Determine the normality of H_2SO_4 by titrating against 0.5N NaOH.
- Find out the 'Q' liberated for [H_2SO_4] from the std. graph.
- Use 2 g KNO_3 and 100 cc water and determine the enthalpy of Solution of KNO_3.

B.Sc. Sem IV
Inorganic Chemistry: Semi-micro Qualitative Analysis

Expt.: Semi-micro Qualitative Analysis of Inorganic salt mixture contains 04 radicals

Instructions:

1. Chart is not allowed.
2. You are given a mixture containing 04 radicals in a capsule or beaker bearing your table number. Analyze and report two basic and two acidic radicals.
3. Present the analysis systematically in the answer paper.
4. Report the results as under.

Basic radicals	Acidic radicals
1.______________________	1.______________________
2.______________________	2.______________________

B.Sc. Sem V (Paper-I)

Separation of Organic Mixture and Qualitative Analysis of Organic compound

Expt: You are given a binary mixture containing two organic solids in a beaker bearing your table number. Detect the nature of the two components and report to the examiner, separate two components after obtaining approval. Ask for the pure solid compound for analysis.

Instructions: Get the signature of the examiner for element test, physical constant, & confirmatory tests of the compound & M.P. of derivative.

NOTE: Chart is not allowed. Marks are separately reserved for actual separation, preliminary and solubility tests, physical constant, test for elements, functional groups, identification, correct name and structure, preparation of derivative & its M.P., Practical record & viva-voce.

Report:

1. Soluble in :
2. Physical constant(M.P.) :
3. Elements Present :
4. Functional group:
5. Name of the compound identified :
6. Its Structure :
7. Name of the Derivative :
8. M.P. of the Derivative :

B.Sc. Sem. V- Paper II (Physical Chemistry)

Chart and Scientific Calculator are not allowed in the examination.

Conductometric titration(HCl)

Q1. To determine the concentration of HCl solution by conductometric titration using the standard 0.5 N NaOH solution.

Perform the experiment as follows:

- Pipette out 25 cc of given HCl solution and titrate against 0.5 N NaOH solution by adding 0.5 cc at a time.
- Record the Conductance for every addition up to 10.00 cc.
- Determine the end point graphically.

B.Sc. Sem. V- Paper II (Physical Chemistry)

Conductometric titration(CH_3COOH)

Q2. To determine the concentration of CH_3COOH solution by conductometric titration using the standard 0. 5 N NaOH solution.

Perform the experiment as follows:

- Pipette out 25 cc of given CH_3COOH solution and titrate against 0.5 N NaOH solution by adding 0.5 cc at a time.
- Record the Conductance for every addition up to10.00 cc.
- Determine the end point graphically.

B.Sc. Sem. V- Paper II (Physical Chemistry)

Conductometry (Dissociation constant)

Q3.To determine the dissociation constant of acetic acid conductometrically.

Perform the above experiment as follows:

- Determine the Cell constant using 0.1 N KCl solution Pipette out 50.0cc of given 0.1 N solution to a clean beaker and determine the conductance.
- Similarly, find the conductance for 0.05, 0.025 and 0.0125 N solution by half dilution method. Given: Specific conductance of 0.1 N KCl= 0.01286 S cm^{-1}.

B.Sc. Sem. V- Paper II (Physical Chemistry)

Colorimetry (Cu^{2+})

Q4. To verify the Beer-Lambert’s law by colorimetric method and calculate the molar extinction coefficient of cuprammonium sulphate complex

Perform the experiment as follows:

- Prepare 100 cc 2M ammonia solution by measuring out 13.0 cc liquor ammonia.
- Prepare 100cc 0.01M $CuSO_4$ solution by weighing the solid $CuSO_4$.
- Use this solution to prepare different concentration of cuprammonium sulphate complexes for 10.0cc each in 8 test tubes. Select a suitable filter.
- Find O.D. for all the solutions using selected filter.
- Calculate the molar extinction coefficient of cuprammonium sulphate complex graphically.

B.Sc. Sem. V- Paper II(Physical Chemistry)

Potentiometry (Acid – Base titration)

Q5. To determine the concentration of strong acid by potentiometric titration against standard solution of 0.1 N NaOH.

Perform the experiment as follows:

- Pipette out 25 cc of given hydrochloric acid, add a pinch of quinhydrone.
- Place the platinum and calomel electrodes in the solution and titrate against NaOH by adding one cc at a time.
- Note down EMF at each addition and determine the end point from both the plots of:

1. EMF Vs. Volume of NaOH
2. $\Delta E/\Delta V$ vs Volume of NaOH

B.Sc. Sem. V- Paper II (Physical Chemistry)

Potentiometry (dissociation constant)

Q6. To determine the dissociation constant K_a of a weak acid (CH_3COOH) potentiometrically by titrating against 0.2N NaOH using quinhydrone and calomel electrodes.

Perform the experiment as follows:

- Pipette out 25 cc of given acetic acid and add a pinch of quinhydrone.
- Place the platinum and calomel electrodes in the solution and titrate against NaOH by adding one cc at a time. Note down EMF at each addition.
- Determine the end point from the plot of EMF vs volume of NaOH and calculate K_a.

Given: **$E^o_{calomel} = 0.2422$ V**

B.Sc. Sem. V- Paper II (Physical Chemistry)

Conductometry(equivalent conductance)

Q7. To determine the equivalent conductance of strong electrolyte (NaCl) at infinite dilution (λ_∞).

Perform the experiment as follows:

- Determine the Cell constant using 0.1 N KCl solution.
- Pipette out 50.0cc of given 0.2 N NaCl solution to a clean beaker and determine the conductance.
- Similarly, find the conductance for 0.1, 0.05, 0.025 and 0.0125N solutions by half dilution method. Calculate λ_c and λ_∞.
- Given : Specific conductance of 0.1 N KCl= 0.01286 S cm^{-1}.

B.Sc. Sem. V- Paper II (Physical Chemistry)

Colorimetry (Fe^{3+})

Q8. To verify the Beer Lambert's Law by colorimetric method and determine unknown concentration of ferric (Fe^{3+}) ions in its thiocyanate complex.

Perform the experiment as follows:

- **D**ilute the given 0.001M (Ferric alum) solution up to the mark in 100cc volumetric flask.
- Use this solution to prepare different concentration of ferric thiocyanate complexes using 2% KCNS for 10.0cc each in 8 test tubes. Select a suitable filter.
- Find O.D. for all the solutions and a solution of unknown concentration using selected filter.

B.Sc. Sem. V- Paper II (Physical Chemistry)

Conductometry(II order reaction)

Q9. To determine 2nd order rate constant for soaponification of ethyl acetate by following progress of the reaction conductometrically.

Perform the experiment as follows:

- Pipette out 20 cc of 0.1M NaOH and 20 cc of 0.1M $CH_3COOC_2H_5$ in two separate clean and dry conical flasks.
- Add 40cc water to NaOH solution, mix them and note down the conductance immediately.
- Find the conductance of the reaction mixture for 1,2,3,4,5,6,7,8,9,10,15,20,25,30,35 and 40minutes.
- Measure the conductance of the reaction mixture after 2 hrs from mixing time.
- Calculate the second order velocity constant, '***k***' using a suitable equation and mean k for the minutes 1 to 10 readings.
- Graphically also for the minutes 1 to 10 readings by ignoring other readings of higher minutes.

B.Sc. Sem. V- Paper II(Physical Chemistry)

Critical solution temperature

Q10. To determination of critical solution temperature of the partially miscible liquids (Water and Phenol).

Perform the experiment as follows:

- **A**dd phenol from burette 8,7…….1. cc to the each of the test tubes and 1,2,3,……..8 cc of distilled water to make 10cc in 08 different test tubes.
- Determine the temperature of each when the turbidity just disappear using water bath.

Given : Density of Phenol= 0.8 g/cc and density of Water = 1.0 g/cc

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B.Sc. Sem. V- Paper II(Physical Chemistry)

pH metry

Q.11. To prepare the acetate buffer solution . Determine the pH using pH meter and calculate the mole ratio for unknown composition..

Perform the experiment as follows

- Prepare 0.2N acetic acid and 0.2N sodium acetate in 100 cm^3 each**.**
- Prepare 10 different sets of solutions by mixing these solutions in a wide mouth test tubes for 10 cc solutions of pH 3.45, 3.75, 4.14, 4.38, 4.57, 4.74, 4.92, 5.11, 5.35, 5.70 and 6.02.
- Find out the pH of the above solutions pH metrically.
- Determine the pH of buffer solutions of unknown concentration

B.Sc. Sem VI: Paper-I

Inorganic Chemistry: Volumetric Analysis and preparation

Expt. No. 1: Determination of Cu / Ca / Fe Volumetrically & Preparation ***(perform marked expt.)***

You are given a solution/sample of brass / limestone /haematite in a volumetric flask/ bottle bearing your table number _______. Determine the amount of ____________using the standard solution of ____________volumetrically. Calculate the amount of _________ present in the given solution/sample.

If time permits, prepare ………………… from suitable starting materials and record the yield & submit the dry complex(not mandatory).

Instructions:

1. Chart and Scientific Calculator are not allowed.
2. Dilute the given ______solution up to the mark with distilled water, shake it well and use 25 cc of the diluted solution for determination.
3. Prepare 0.05N solution of $K_2Cr_2O_7$/ $H_2C_2O_4.2H_2O$/ $Na_2S_2O_3.5H_2O$
4. Use $KMnO_4$ solution after standardizing in case of **lime stone**.
5. Get all readings initialed by the examiner. Present your results as below.

Results:

6. 25cc of _____solution required 1.______2._____3.____ cc of ______solution.
7. Amount of ____ present in given solution is ______g.

Note: Marks are reserved for accuracy, presentation, technique, calculation, viva – voce and Practical record.

Q.No.2: Tour Report or Project Report

B.Sc. Sem VI : Paper-I

Inorganic Chemistry: Gravimetric Analysis

Expt. No.2: Determination of Ba^{2+}/Al^{3+}/Fe^{2+}/Fe^{3+}/ Pb^{2+} gravimetrically

(perform marked expt.)

You are given a solution of Ba^{2+}/Al^{3+}/Fe^{2+}/Fe^{3+}/ Pb^{2+} in 100/250cc volumetric flask bearing your table number. Determine gravimetrically the amount of ______ present in the given solution.

Instructions: Chart and Scientific Calculator are not allowed.

1. Dilute the given ______solution up to the mark with distilled water, shake it well and use **25/50** cc of the diluted solution for determination.
2. Get all weights initialed by the examiner
3. Present your results as below.

Results:

1. **25/50** cc of the diluted solution gave ____g of ____($BaSO_4$ /Al_2O_3 /Fe_2O_3 /$PbSO_4$)
2. Amount of ____ present in the given solution is ______g.

Note: Marks are reserved for accuracy, presentation, technique, calculation, viva –voce and Practical record

Q.No.2.: Tour Report or Project Report

B.Sc. Sem VI: Paper-II

Separation of Organic Mixture and Qualitative Analysis of Organic compound

Expt: You are given a binary mixture containing two organic liquids in a beaker bearing your table number. Separate the components by distillation and ask for the pure liquid for analysis.

NOTE: Chart is not allowed. Marks are separately reserved for actual separation, preliminary and solubility tests, physical constant, test for elements, functional groups, identification, correct name and structure, preparation of derivative & its M.P.

Report:

1. Soluble in :
2. Physical constant(B.P.):
3. Elements Present:
4. Functional group:
5. Name of the compound identified:
6. Its Structure:
7. Name of the Derivative :
8. M.P. of the Derivative :

B.Sc. Sem VI : Paper-II
Physical Experiment :Chart and Scientific Calculator are not allowed.

Conductometry (acid mixture)

Q.No.1: To determine the concentrations of acids in a mixture of HCl + CH_3COOH conductometrically using standard 0.5N NaOH.

Perform the experiment as follows:

- Pipette out 50 cc of the given acid mixture solution in to a beaker and titrate against standard 0.5N NaOH by adding 0.5 cc at a time till 15 cc and note the conductance of the solution.
- Determine the end point graphically by plotting a graph of conductance versus volume of NaOH.
- Calculate the strength of HCl & CH_3COOH in terms of normality and grams per dm^3.

B.Sc. Sem VI: Paper-II
Conductometry (Solubility)

Q.No. 2. : To determine the solubility of sparingly soluble salt ($BaSO_4$) conductometrically.

Perform the experiment as follows:

- Determine the cell constant of the given conductivity cell using 0.1N KCl.
- Determine the conductance of water.
- Determine the conductance of supernatant solution of $BaSO_4$ for at least 3 readings.
- Calculate solubility of $BaSO_4$ both in mol /litre and g / litre.

***Given**:* 1. Specific conductance of 0.1N KCl = 0.01288 S cm^{-1}.

2. Molar Conductance of $BaSO_4$ at infinite dilution, $\lambda^0{}_m$ = 288.6

B.Sc. Sem VI: Paper-II
Potentiometry (dissociation constant)

Q.No.3: To determine the dissociation constant K_a of a weak acid (CH_3COOH) potentiometrically by titrating against 0.2N NaOH using quinhydrone and calomel electrodes.

Perform the experiment as follows:

- Pipette out 25 cc of given acetic acid to 100cc beaker and add a pinch of quinhydrone
- Place the platinum and calomel electrodes in the solution and titrate against 0.2N NaOH by adding 1 cc at a time.
- Note down EMF after each addition.
- Determine the end point from the plot of EMF vs NaOH and calculate the $E_{cel}{}^{l}$ and K_a.

B.Sc. Sem VI: Paper-II

Refractometry (formula method)

Q.No.4: To determine the percentage composition of A and B liquids in unknown composition, 'C' by formula method, measuring their refractive indices using Abbe's Refractometer.

Perform the experiment as follows:

- Determine the density of liquids, A, B and C.
- Find out the RI for all the liquids. Calculate the percentage composition of A and B liquids in unknown composition, 'C' by formula.

B.Sc. Sem VI: Paper-II

Refractometry (graphical method)

Q.No.5: To determine the percentage composition of A and B liquids in unknown composition, 'C' by graphical method, measuring their refractive indices using Abbe's Refractometer.

Perform the experiment as follows:

- Determine the density of liquid, C.
- Find out the RI for all the liquids.
- Calculate the percentage composition of A and B liquids in unknown composition,'C' by graphically.

Given

Liquid	A	B	C	M_1	M_2	M_3	M_4
Percentage Composition	100	100	-	A = 40 B = 60	A = 50 B = 50	A = 60 B = 40	A = 70 B = 30
Density, g/cc	0.873	1.579		1.212	1.138	1.075	1.034

B.Sc. Sem VI: Paper-II

Potentiometry (Solubility)

Q.No.6 : To determine the solubility and solubility product of sparingly soluble silverchloride potentiometrically.**Perform the experiment as follows:**

- Construct the cell by taking 25 cc of given 0.01 N KCl solution with 2 drops $AgNO_3$ in one beaker and 10 cc 0.01N $AgNO_3$ in another beaker. Place silver electrodes in both the beakers.
- Connect the solutions internally with NH_4NO_3 / KNO_3 salt bridge and externally with the electrodes to potentiometer. Determine EMF of the cell.
- Repeat the same with 0.005, 0.0025 and 0.0125N $AgNO_3$.
- Calculate the solubility & solubility product of AgCl both in mol / dm^3 and g / dm^3

B.Sc. Sem VI: Paper-II

Potentiometry (Redox)

Q.No.7: To determine the redox potential of Fe^{3+} / Fe^{2+} using $FeSO_4.7H_2O$ solution by potentiometric titration against the standard solution of 0.1N $K_2Cr_2O_7$.

Perform the experiment as follows:

- Pipette out 25 cc of given ferrous sulfate solution and add 25 cc 4N H_2SO_4.
- Place the platinum and calomel electrodes and titrate against 0.1N $K_2Cr_2O_7$ solution by adding 1 cc at a time up to 30cc. Note the EMF at each addition.
- Determine the end point from the plot of $\Delta E/\Delta V$ vs volume of $K_2Cr_2O_7$ and half equivalence point from the plot of EMF vs volume of $K_2Cr_2O_7$.

Given: **$E^o_{calomel} = 0.2422$ V**

Appendix 1 :

LIST OF UNDERGRADUATE CHEMISTRY TEACHERS IN VARIOUS AFFILIATED COLLEGES:2017-18

Sl.No.	Name of the College	Name of Teacher	Ph. No.	E-Mail ID
1	1002: Karnatak Science College,Dharwad	M S Salunke	9448861528	
		S D Dummawad	9449188294	
		S M Tuwar	9449796557	
		V A Amminbhavi	9731574873	
		O Kotresh	9448838665	
		R G Kalkamkar	9902831760	
		Jayalaxmi K	9742079633	
		K S Katagi	9916248809	
		S P Masti	9980556298	
		Rajappa S K	9620626968	
2	1013 : Govt First Grade Arts/Comm/BBA College, Dharwad	Sangeeta Kulkarni	9916148463	
3	1015 : Govt First Grade Arts/Sci/Comm/ BBA College, Rajnagar, Hubli	R S Gurumath	8095483274	
4	1202: JSSArts/Commerce/ Science College, Dharwad	J G Baragi	9449190721	
		S B Mekali	9448148828	
		Venkatesh	9448777011	
		S S Kulkarni	9448629565	
		A.M. Shirahatti	9480555341	
		Mahesh R T	9740299477	
5	1204: BMHECS Kittel Science College ,Dharwad	B K Megalamani	9980067406	
		Stanley Peters	9141373549	
		Darla Lazarus	9343102306	
		A L Harihar	9379565950	
		Vimala G	9972205867	
6	1232: KLES S K Arts/Hsk Science Inst, Hubli	S N Sherewad	9886259857	
7	1234: Nehru Arts/Comm/Sci College,Hubli	Sardar Hussain	9481287238	
		M S Gulannavar	9738587562	
8	2201: KLES J T Arts/Sci/ Comm/BCA College,	S. M. Bangalore	9481527012	
		C Lingareddy	9448564349	

	Gadag-Betageri	S H Narasinavar	9448591348	
9	2205: KSS Arts/Sci/Comm College,Gadag	S K Vandakar	9886394167	
		C C Amattennavar	9945119572	
10	2222 : SKVP`S Arts/Sci/Comm College,Hole-Alur	P S Kanavi	9449121688	
		S B Sajjanar	9945555084	
11	2230: AVVPs S A Arts/ Sci/Comm College, Naregal	A J Handi	9741782405	
12	3015 : Govt First Grade Arts/ Sci/Comm/BBA College, Haveri	Navyashree M O	9686653068	
13	3204 : MASC Arts/Sci/Comm College, Haunsabhavi	Srinivasareddy	9880944919	
		Ravi S Naik	9482211085	
14	3205 : KLES GH Arts/Sci/ Commerce Degree College ,Haveri	Vasudev Nayak	9449419740	
		N M Gundannavar	9986167290	
		S V Madiwale	9448340336	
		K H Byadagi	9448916002	
		S S Patil	9964833297	
15	3212 : RTES Arts/Sci/Comm College ,Ranebennur	S R Pratap	9449972628	
		H Manjunath	9448564626	
		B I Koli	9448326501	
		R D Nayak	9845130354	
16	7017 : Govt First Grade Arts/ Comm/Sci/BBA College, Karwar	K K Kerwadikar	9449629599	
		Ullas N Shetti	9448818242	
		A Pragasam	9845648560	
		Vinayak M Naik	9448866454	
		Preeti Tallur	9945072665	
17	7201 : Gokhale Centenary Arts/Sci College, Ankola.	Imthyaz Ahmed Khan	9449369804	
		Sharada Airani	9448344973	
		J S Fernadies	9448223576	
18	7205: Anjuman Arts/ Sci/Comm College, Bhatkal	M K Shaikh	9886212692	
19	7210 : B N Degree College, Dandeli	F B Holi	9448722879	
		H H Adavi	9448611391	
		Shobha Sharma	9480561861	
		H Y Merwade	9448344996	
20	7214: SDM Arts/Comm/	P M Honnvar	9448153586	

	Science College , Honnavar	Sanjeev Nayak	9242280591	
21	7217 : KCS Dr A V Baliga Arts/Sci College, Kumta	G T Kuchinad	9448526475	
		V M Pai	9480474458	
		S N Shetty	9448996383	
		N K Nayak	9448996212	
		Revati Nayak	9845836075	
22	7224: MM Arts/Science C ollege, Sirsi	A K Kini	9448965040	
		Ganesh Hegde	9242368889	
23	7227 : Mahasatee Arts/ Comm/Science College:Ulga - Karwar	C S Naik	9448679050	
24	MPES's S D M Arts, Science, Commerce & B.B.A. College , Honnavar.	No Permanent Staff: Attn. to Principal		
25	M G C Arts & Commerce and G H D Science College , Siddapur.	No Permanent Staff: Attn. to Principal		
26	SET Siddharth Degree College Harakali Shirali	No Permanent Staff: Attn. to Principal		
27	Beena Vaidya Commerce/Science College, Murdeshwar	No Permanent Staff: Attn. to Principal		
28	B R Tambakad First Grade Arts, Commerce and Science College, Hirekerur.	No Permanent Staff: Attn. to Principal		
29	Ballary Rudrappa Education Trust's Arts,Commerce and Science College Motebennur	No Permanent Staff: Attn. to Principal		
30	B.C.N Arts, Commerce & Science College, Laxmeshwar	No Permanent Staff: Attn. to Principal		
31	Vidya Arts/Science College, Gadag	No Permanent Staff: Attn. to Principal		
32	Govt. First Grade College, Hulakoti	No Permanent Staff: Attn. to Principal		
33	Govt. First Grade College, Mundaragi	No Permanent Staff: Attn. to Principal		
34	Govt. First Grade College, Gajendragad	No Permanent Staff: Attn. to Principal		
35	Govt. First Grade College,	No Permanent		

	Naragund	Staff: Attn. to Principal		
36	Govt. First Grade College, Navalgund	No Permanent Staff: Attn. to Principal		
37	Govt. First Grade College, Ranebennur	No Permanent Staff: Attn. to Principal		
38	Govt. First Grade College, Sirsi	No Permanent Staff: Attn. to Principal		
39	Govt. First Grade College, Haliyal	No Permanent Staff: Attn. to Principal		
40	Govt. First Grade College, Kumta	No Permanent Staff: Attn. to Principal		
41	Govt. First Grade College, Ankola	No Permanent Staff: Attn. to Principal		
42	Govt. First Grade College, Honnavar	No Permanent Staff: Attn. to Principal		
43	Govt. First Grade College, Bhatkal	No Permanent Staff: Attn. to Principal		

Appendix 2:

Chemistry Teachers' Association of Karnatak University's Affiliated Colleges: CHEM – FORUM

LIST OF CONVENTIONS HELD

Sl. No.	Conventions held	Year
1	Established in P.C. Jabin Science College, Hubli	1991-92
2	01st Annual convention, B.N. College, Dandeli	1991-92
3	02nd Annual convention, J. S. College, Dharwad	1992-93
4	03rd Annual convention, R.L.S. Science College, Belagavi	1993-94
5	04th Annual convention, K.C. P. Science College, Vijayapur	1994-95
6	05th Annual convention, H.S.K. Science Institute, Hubli	1995-96
7	06th Annual convention, BLDE's College, Jamakhandi	1996-97
8	07th Annual convention, KRC College, Bailhongal	1997-98
9	08th Annual convention, Basaveshwar College, Bagalkot	1998-99
10	09th Annual convention, Dr. A. V. Baliga College, Kumta	1999-2000
11	10th Annual convention, P.C. Jabin Science College, Hubli	2000-01
12	11th Annual convention, S.J. M. College, Ilkal	2001-02
13	12th Annual convention, B.N. College, Dandeli	2002-03
14	13th Annual convention, J.S. S. College, Gokak	2003-04
15	14th Annual convention, J. T. College, Gadag	2004-05
16	15th Annual convention, Karnatak Science College, Dharwad	2005-06
17	16th Annual convention, G. S. S. College, Belagavi	2006-07
18	17th Annual convention, R.T.E.S College, Ranebennur	2007-08
19	18th Annual convention, B.K. College, Belagavi	2008-09
20	19th Annual convention, S.D. M. College, Honnavar	2009-10
21	20th Annual convention, Kittel Science College, Dharwad	2010-11
22	21st Annual convention, S.A. College, Naregal	2010-12
23	22nd Annual convention, G.H. College, Haveri	2012-13
24	23rd Annual convention, G.C. College, Ankola	2013-14
25	24th Annual convention, Karnatak University, Dharwad	2017-18
26	25th Annual convention, Karnatak Science College, Dharwad	2018-19 (scheduled)

Appendix 3:

MODEL LABORATORY TABLES

MODERN CHEMISTRY LABORATORY TABLE (INORGANIC / ORGANIC)

Dimension of the Inorganic and Organic Laboratory Table

1. Width: 54 Inches : for double working table (face to face with middle rack).
2. Width: 30Inches : for single working table (table position will be adjacent to the wall with single rack).
3. Height : 36 Inches
4. Length: Depending on the space available.

Rack dimension to keep reagent bottles:

5. Height: 23 Inches : face to face with middle rack.
6. Width: 09Inches : face to face with middle rack.
7. Height: 23 Inches : one side working - single rack.
8. Width: 05Inches : one side working - single rack.

Dimension of the Physical Laboratory Table

9. Width: 54 Inches : for double working table
10. Height : 36 Inches
11. Length: Depending on the space available.

Note: Surface of the Table is preferably wood; Alternative to wood would be Ceramic Tiles / toughen glass with 19 mm thickness (non breakable, heat and chemical resistant). Do not use ordinary table glass.

ECONOMY CHEMISTRY LABORATORY TABLE (INORGANIC / ORGANIC)

PHYSICAL CHEMISTRY LABORATORY

KARNATAK COLLEGE, DHARWAD

Quality Education for all Illuminating at centenary celebration(1917-2017)

ABOUT CHEM-FORUM

Dr. S. M. Tuwar
President

Chem-Forum is a volunteer academic body that has all the undergraduate Chemistry teachers of Karnatak University's affiliated Colleges. It is established as a platform to discuss the academic matters of chemistry. The necessity of such forum for Chemistry Teachers was discussed in the meeting held on **25th Aug 1991** at P. C. Jabin Science College, Hubli with a galaxy of 45 Chemistry teachers from 19 different constituent and affiliated colleges of Karnatak University and launched immediately on the same day.

The main aims and objectives of Chem-Forum are as follows:

1. To bring together all the Chemistry teachers of different Affiliated and Constituent Colleges of Karnatak University under one banner by conducting annual conventions to be hosted by various Colleges at least once in a year
2. To provide a platform to express the views of chemistry teachers in the matters related to:
    - Syllabi of undergraduate classes.
    - Methodology of teaching and evaluation.
    - Organizing activities to assist and encourage academic excellence among Chemistry teachers, leading to M.Phil and Ph.D degrees.
    - Encourage innovative methods to inspire and bring out the talents of students.
    - Honor the retired teachers of that academic year for their services rendered.
    - Discuss the interdisciplinary academic matters progressed in the recent years.

All such discussions have to be taken in the annual conventions.

Earlier, it was in the jurisdiction of 07 districts, *viz.,* Dharwad, Gadag, Haveri, Uttar Kannada, Belagavi, Vijaypur, and Bagalkoti. Now, it is confined to first 04 districts. The rest are with the Rani Channamma University, Belagavi.

As on today, the Chem – Forum has conducted 24 annual conventions at various colleges and played a key role in revision of Syllabi during 1999 for Non-Semester course, 2005-06 for Semester structure and revision of Semester Syllabus during 2009-10. During 2009-10, three consecutive meetings were held one at Kittel Science College, and other at Karnatak Science College, and JSS College, Dharwad to finalize the Syllabus. Recently the forum actively has involved in revising the existing syllabus during 2011-12, and now releasing Lab Manual.

In all the walks of its activity, the forum extended a fullest support to Karnatak University for all its academic endeavors to incorporate in the UG Syllabus. Now, it is at the threshold of celebration of Silver Jubilee Annual Convention of Chem – Forum at Karnatak Science College, Dharwad on the occasion of Centenary Celebration of Karnatak College (1917 - 2017).