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KARAIKUDI – 630 003



## Directorate of Distance Education

**M.Sc. [Chemistry]**

**I - Semester**

**344 14**

# **PRACTICAL : ANALYTICAL CHEMISTRY**

**Author**

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# PRACTICAL : ANALYTICAL CHEMISTRY

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## Syllabi

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### **BLOCK-1: QUANTITATIVE ESTIMATION-1**

#### **Unit 1: Quantitative estimation of aniline**

Estimate the amount of aniline present in the whole of the given solution.

#### **Unit 2: Quantitative estimation of phenol**

Estimate the amount of phenol present in the whole of the given solution

#### **Unit 3: Quantitative estimation of ethylmethylketone**

Estimate the amount of ethylmethylketone present in the whole of the given solution

#### **Unit 4: Quantitative estimation of glucose**

Estimate the amount of glucose present in the whole of the given solution

### **BLOCK-2: SEMI-MICRO QUALITATIVE ANALYSIS-1**

#### **Unit 5: Semi-micro qualitative analysis of mixture-1**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 6: Semi-micro qualitative analysis of mixture-2**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 7: Semi-micro qualitative analysis of mixture-3**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

### **BLOCK-3: SEMI-MICRO QUALITATIVE ANALYSIS-2**

#### **Unit 8: Semi-micro qualitative analysis of mixture-4**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 9: Semi-micro qualitative analysis of mixture-5**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 10: Semi-micro qualitative analysis of mixture-6**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

### **BLOCK-4: Semi-micro qualitative analysis-3**

#### **Unit 11: Semi-micro qualitative analysis of mixture-7**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 12: Semi-micro qualitative analysis of mixture-8**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 13: Semi-micro qualitative analysis of mixture-9**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

#### **Unit 14: Semi-micro qualitative analysis of mixture-10**

Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

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## INTRODUCTION

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### NOTES

Analytical chemistry has been important since the early days of chemistry, providing methods for determining which elements and chemicals are present in the object in question. Significant contributions to analytical chemistry include the development of systematic elemental analysis by Justus von Liebig and systematized organic analysis based on the specific reactions of functional groups. Analytical chemistry has broad applications to forensics, medicine, science and engineering.

Fundamentally, the analytical chemistry studies and uses instruments and methods used to separate, identify, and quantify matter. In practice, separation, identification or quantification may constitute the entire analysis or be combined with another method(s). Separation isolates analytes. Qualitative analysis identifies analytes, while quantitative analysis determines the numerical amount or concentration. Typically, the qualitative methods use separations, such as precipitation, extraction and distillation. Identification may be based on differences in colour, odour, melting point, boiling point, radioactivity or reactivity. Quantitative analysis uses mass or volume changes to quantify amount. Analytical chemistry is also focused on improvements in experimental designs, chemometrics, and the creation of new measurement tools.

Characteristically, a qualitative analysis helps in determining the presence or absence of a particular compound, but not the mass or concentration.

This book, *Analytical Chemistry*, deals with the practical aspects of qualitative and quantitative analysis of chemical compounds.

## BLOCK - I

*Practical : Analytical  
Chemistry*

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### QUANTITATIVE ESTIMATION

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#### Instructions

When move to Chemistry lab for performing the experiment - various safety rules keep in mind.

Any one found infringing upon the safety rules will be given a notice for the main offense, will be rejected from the lab space for the second offense, and will be pulled back from the class (lab) for the third offense.

#### Rules (Chemistry Lab)

1. Utilize Common Sense to keep away from mishaps.
2. Pupil who jeopardizes other's security, or himself, will be expelled from the lab.
4. Every pupil wear safety Glasses.
5. Every pupil must come with shoes in the lab.
6. Students must wear lab coats.
7. Eatable are not allowed in the lab.
8. Prescribe experiments allowed to be performed.
9. No chemical compounds or hardware might be evacuated from the laboratory.
10. After the completion of lab please put the material on their define spaces.
11. If any pupil have medical issue please notify the lab instructor.
12. Just pure water can go down the sink.
13. Clear all expendable or broken crystal with water before disposing of it in the broken glass squander holder. Clean all ordinary dishes completely with water before returning.
14. If there should arise an occurrence of flame, tell the educator instantly. Check the warmth source. In the event that the smoke or fire is wild, clear the room quickly, pull the fire caution in the corridor, and call the fire division.

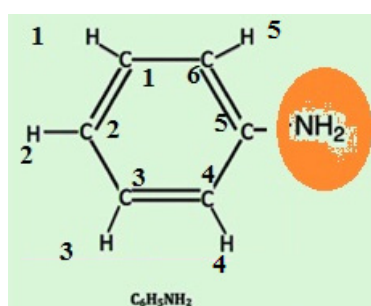
#### NOTES

## QUANTITATIVE ESTIMATION OF ANILINE

### NOTES

#### Aniline Introduction

**Anilines:** Anilines are the organic compounds in the class of group coming in organic chemistry which are likewise called as aminobenzene or phenylamine. These mixes are said to be harmful in nature and furthermore known to be one of the classes of sweet-smelling amines. These are utilized in a wide assortment of modern and are known to have every one of the qualities of a aromatic compound. The aniline compounds are said to have the formula  $C_6H_5NH_2$  where in the amino gathering attached to the Phenyl gathering.



Aniline generally found in yellowish color but some time colorless to light brown also. It has not good smell, it smells like an rotten fish. It is slightly soluble in water and it is flammable.

#### Physical Properties of aniline

- I. Boiling point of aniline is 184 degree Celsius and a melting point is -6 degree Celsius
- II. Aniline is slightly soluble in water and soluble in alcohol & ether.
- III. It felt darken when exposed to air and light.
- IV. It generally known as weak base
- V. It is toxic in nature for Human being (inhaled by air ).

#### Reactions

- I. **Oxidation Reaction-** Anilines in oxidation reaction produce and formed carbon -nitrogen bonds.
- II. **Basicity-** Anilines are weak bases. On reaction with strong acids, the compounds form ions of anilinium.
- III. **Acylation-** Anilines reacts in acylation reaction and immediately formed amides on reaction with carboxylic acids.

## Uses

- I. Anilines are used in the rubber industry ( processing of rubber )
- II. Anilines are used as a dyeing agent in industries.
- III. Anilines are used in the production of drugs like- Paracetamol (use in fever generally), acetaminophen.
- IV. Anilines are used in the production of pesticide and fungicides in the agricultural industry.

***Estimate the amount of aniline present in the given Solution.***

***There are two parts to this experiment***

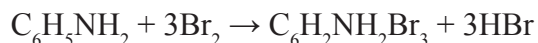
- Part 1: Find out the concentration of Sodium thiosulphate (Stand.)
- Part 2: Aniline Solution Estimation.

### ***Reagents Required***

- Solution having conc. (Sodium Thiosulfate) = 0.1N
- $\text{KBrO}_3/\text{KBr}$  Brominating Mixture having conc. = 0.1N
- Solution of Potassium Iodide 10 %
- Starch Solution

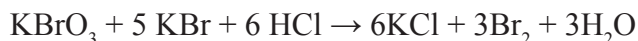
### ***Reason Behind the estimation of aniline $\text{C}_6\text{H}_5\text{NH}_2$***

- When the aniline react with bromine then the products are:



aniline having Mass (equi. Mass) - Molar Mass/6 = 15.5 gram equivalents

- When  $\text{KBrO}_3$  react with  $\text{KBr}$  in the presence of Conc.  $\text{HCl}$  then Bromine is the produce with  $\text{KCl}$  and water.



### ***Standardization of $\text{Na}_2\text{S}_2\text{O}_3$ (Sodium Thiosulphate)***

- 1.25 gm mass of potassium dichromate put into a flask (Capacity 250 ml)
- Shake this solution until the potassium dichromate dissolve in to the water (add water up the decent (min. level) level of solubility)
- Shake it well so that solution became uniform.
- 10 ml of this solution is pipeted into an Erlenmeyer Meyer flask, add about 3 ml of concentrated  $\text{HCl}$  (1M) followed by 5 ml of 10 %  $\text{KI}$  solution.
- Bromine which is the product of react with Sodium Thiusulfate (Solution taken in Bureete ).
- When pale yellow color in the solution (include the starch solution), titration process continue up the end point achieved in term of concordant qualities

## NOTES

## NOTES

### *Estimation of Aniline*

- First diluted the given solution (aniline solution).
- Aniline solution (volume 20ml) and brominating mixture (volume 40 ml) are pipetted into a stoppered 250 ml, Erlenmeyer flask (conical flask) and dilute it with (volume 20 ml) water and 5 ml of conc. HCl (strength may be used 1 M to 3 M).
- Close the Erlenmeyer flask and spin it so that solution mix properly.
- Cool the substance of the flask and open the flask painstakingly without losing any bromine liberated (Use an Erlenmeyer flask with a glass stopper pour some of the 10 % KI around the stopper and then open it carefully not to lose any bromine liberated, the KI will get sucked into the flask) and add 20 ml of 10% KI solution.
- Shake the flask for few seconds and then put it ideal up to few minutes.
- Expel the stopper wash down the contents using a little refined water and titrate the free iodine against sodium thiosulphate using starch as the indicator.
- When the solution became light yellow then put 1 ml starch solution.
- Titrate this solution until the starch iodine blue color diminish.
- Equal volume of sodium thiosulphate react with equal volume of bromine (excess bromine)
- Repeat this procedure for blank titration again.

### *Calculation*

- Sodium Thiosulfate Solution normality =  $N_1$
- Brominating Mixture (20 ml) is =  $V_1$  ml of Sodium Thiosulfate
- Brominating Mixture Volume used = 40 ml
- Brominating Mixture (40 ml) = double the volume of Sodium thiosulfate (=  $2V_1$ )
- (20 ml)Iodine solution and Brominating mixture (40 ml) =  $V_2$
- Sodium thiosulfate amount = aniline =  $(2V_1 - V_2)$ ml
- Aniline Normality =  $(2V_1 - V_2)$ mL x  $N_1/20 \times 10$
- Convert the normality with appropriate unit.

### Related Question:

1. What is the brown aniline before and color less aniline after distillation?
2. Write the process to remove aniline from reaction mixture?



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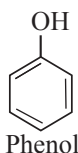
## QUANTITATIVE ESTIMATION OF PHENOL

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Practical : Analytical  
Chemistry

### Introduction of Phenol

Phenols are the chemical compound which have benzene ring bonded to a hydroxyl group. They are also known as carbolic acids. They show unique physical and chemical properties as compare to the other chemical Compounds.



### Properties of Phenol

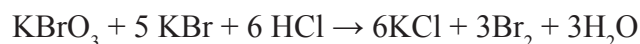
- I. Odor Sweet and tarry
- II. Density 1.07 g/cm<sup>3</sup>
- III. Melting point 40.5 °C (104.9 °F; 313.6 K)
- IV. Boiling point 181.7 °C (359.1 °F; 454.8 K)
- V. Solubility in water: 8.3 g/100 mL (20 °C)
- VI. Vapor pressure 0.4 mmHg (20 °C)[2]
- VII. Acidity (pKa) 9.95 (in water),
- VIII. 29.1 (in acetonitrile)
- IX. UV-vis (λ<sub>max</sub>) 270.75 nm
- X. Dipole moment 1.224 D

### (a) Estimate the Amount of Phenol Present in the Whole of the Given Solution

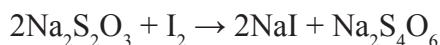
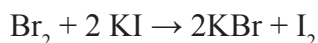
#### Principle

With brominating reagents (Bromination process) is used for determination of phenol in a solution.

**Note:** brominating reagents means- Mixture of Potassium Bromide + potassium Bromate and Concentrated HCl



The excess bromine is checked (React) with Potassium Iodide (KI) and the product of this reaction (Iodine) titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (Standard). The reaction is given below:



#### Reagents Required

1. Solution of Sodium Thiosulphate having Concentration =0.1N

### NOTES

2.  $\text{KBrO}_3/\text{KBr}$  Brominating Mixture having Concentration = 0.1N
3. Solution of Potassium Iodide (KI) 10%

## NOTES

### Procedure

1. Prepare brominating solution first and then prepare standard solution of phenol (0.4 gm phenol in two 250 ml water) in volumetric flask.
2. Perform blank titration (Brominating Solution)
3. **Titration with standard phenol solution:** In Pipette take  $25\text{ cm}^3$  standard phenol solution and take out in Conical flask ( $250\text{ cm}^3$ ) and add water (distilled only- $25\text{ cm}^3$  Volme) and Conc. HCL ( $5\text{ cm}^3$ ). Brominating mixture (taken in burette) and after this add the solution till the light yellow (pale yellow color achieved then add  $5\text{ cm}^3$  Potassium Iodide (KI) solution in it Liberated iodine is titrated against sodium thiosulphate solution using starch as an indicator. Repeat this process in similar manner. Record the observations in Table-I.
4. In next step we titrate with unknown phenol solution: In Pipette take  $25\text{ cm}^3$  unknown phenol solution and take out in Conical flask ( $250\text{ cm}^3$ ) and add distilled water ( $25\text{ cm}^3$ ) and Concentrated HCL ( $5\text{ cm}^3$ ). Brominating mixture (taken in burette) and after this add the solution till the light yellow (pale yellow color achieved then add  $5\text{ cm}^3$  Potassium Iodide (KI) solution in it Liberated iodine is titrated against sodium thiosulphate solution using starch as an indicator. Repeat this titration process in similar manner. Record the observations in Table-II

### Observation

Weighing Bottle mass =  $m_1 = \dots\dots\dots\text{g}$

Bottle mass + Phenol Solution =  $m_2 = \dots\dots\text{g}$

Mass of the bottle after shifting the Phenol =  $m_3 = \dots\dots\text{g}$

Now the Phenol Transfer mass =  $m_3 = m_2 - m_1 = \dots\dots\text{g}$

Phenol Molar mass ( $M_m$ ) =  $94\text{ gmol}^{-1}$

Table - I					
Standard Phenol Solution v a Sodium Thidsulphate Solution					
S.No	Volume of Phenol Soluntion (in $\text{cm}^3$ )	Volume of brominating Soluntion (in $\text{cm}^3$ )	Burette Reading		Volume ( $\text{Na}_2\text{S}_2\text{O}_3$ ) of Solution (in $\text{cm}^3$ ) Final – Initial
			Initial	Final	
1	25				
2	25				
3	25				

Table - II					
Unknown Phenol Solution v a Sodium Thiodsulphate Solution					
S.No	Volume of Unknown Phenol Solution (in cm <sup>3</sup> )	Volume of brominating Solution (in cm <sup>3</sup> )	Burette Reading		Volume (Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> ) of Solution (in cm <sup>3</sup> )
			Initial	Final	Final – Initial
1	25				
2	25				
3	25				

## NOTES

### Calculation

Phenol weight in Solution (Standard Solution)	=m	=.....g
Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> Solution volume in blank from Table-I	=V	=.....cm <sup>3</sup>
Sodium Thiosulphate Volume which is used for Phenol Solution (from Table-I)	= V1	=.....cm <sup>3</sup>
Sodium Thiosulphate Volume which is used for Unknown Phenol Solution (from Table-II)	= V2	=.....cm <sup>3</sup>
Vcm <sup>3</sup> of Sodium Thiosulphate Tolution =25cm <sup>3</sup> Brominating Solution		
So 1cm <sup>3</sup> of Sodium Thiosulphate 25/V cm <sup>3</sup> Brominating Solution		
Therefore, V <sub>1</sub> cm <sup>3</sup> of sodium Thiosulphate = (25/V) * V <sub>1</sub> cm <sup>3</sup> Srominating Solution		
Brominating solution volume used 25cm <sup>3</sup> of phenol solution = (V-25)/V * V <sub>3</sub> cm <sup>3</sup>		
Similarly, volume of brominating solution used for 25 cm <sup>3</sup> of unknown solution= (V- ((25/V)*V <sub>2</sub> ) = V <sub>4</sub> cm <sup>3</sup>		
In same process the brominating solution used to for unknown phenol solution= (V-((25/V)*V <sub>2</sub> ) = V <sub>4</sub> cm <sup>3</sup>		
Using this relation (phenol solution mass (known sol.) / mass of phenol in standard solution) = (In unknown phenol solution Volume used of brominating solution / In standard phenol sol. Volume used of brominating solution)		
Unknown solution have the phenol (weight) = m(V <sub>4</sub> / V <sub>3</sub> ) =.....g		
Strength of Phenol in Unknown Solution = (strength of standard phenol solution * V <sub>4</sub> ) / V <sub>3</sub> = 4* m* (V <sub>4</sub> / V <sub>3</sub> ) =.....g dm <sup>-3</sup>		

## NOTES

### Result

The Weight of phenol in solution (unknown)= .....g

Phenol strength in Solution (unknown)= ..... g dm<sup>-3</sup>

Phenol purity will be found with the help of the given formula

$$= ((V-V_2) * M * M_m * 100) / M * Z * 2000$$

where,

V= Sodium Thiosulphate Volume used in blank experiment

V<sub>2</sub> = Sodium Thiosulphate Volume used in sample of phenol

M= Sodium Thiosulphate Molarity

M<sub>m</sub> = Phenol Molar Mass

m = Phenol Mass taken in g

Z = number of bromine atoms Substitute in phenol

### (b) Estimate the Amount of Hydroxyl Group By Acetylation Method

#### Principle

Acetylation Method used to estimate the Phenol or Hydroxy groups. In this experiment, determine the number of hydroxyl groups in a phenol.



In this Pyridine work as Solvent because it is neutral with reagents, it react with acids group and form salt formation and it also serves as a catalyst.



#### Procedure

- Step 1: First prepare the Acetylating reagent after this take two conical flask label then A and B and attached it with reflux Condenser (water for Condenser). Take 1g Phenol in to the conical flask A.
- Step 2: Add 10 cm<sup>3</sup> of acetylating reagent to the both flasks A and B.
- Step 3: Attach the flasks to the condensers and provide heat to flask A and B by with the help of hot water up to 30mins. Then remove both the flasks from water-bath and pour 20 cm<sup>3</sup> of distilled water, shaking the contents of each flask gently to ensure complete hydrolysis of the un-reacted acetic anhydride.
- Step 4: Cool each flask in cold water and wait for 10 min. Then titrate the each flask with (M) NaOH solution, using phenolphthalein or any indicator. A fine emulsion of phenyl acetate will form

when the contents of the flask A are diluted and ensure that all the free acetic acid is extracted by the sodium hydroxide solution.

Step 5: Repeat this process and put this observation in the Table 1 and Table 2.

## NOTES

### Observation

Weighing Bottle mass =  $m_1 = \dots\dots\dots$ g

Bottle mass + Phenol Solution =  $m_2 = \dots\dots$ g

Mass of the bottle after shifting the Phenol =  $m_3 = \dots\dots$ g

Now the Phenol Transfer mass =  $m_3 = m_2 - m_1 = \dots\dots$ g

Phenol Molar mass ( $M_m$ ) =  $94 \text{ gmol}^{-1}$

Table - I				
<i>Acetylating Reagent vs. Sodium Hydroxide Solution</i>				
S.No	Acetylating Reagent volume (in $\text{cm}^3$ )	Burette Reading		NaOH Solution Volume (in $\text{cm}^3$ ) Final -- Initial
		Initial	Final	
1	10			
2	10			
3	10			

NaOH volume used in neutralising  $10 \text{ cm}^3$  of Acetylating Reagent =  $V_1 = \dots\dots\dots \text{cm}^3$

Table - II				
<i>Acetylating Reagent vs. Sodium Hydroxide Solution</i>				
S.No	Sample + $10 \text{ cm}^3$ Acetylating Reagent (in $\text{cm}^3$ )	Burette Reading		NaOH Solution Volume (in $\text{cm}^3$ ) Final -- Initial
		Initial	Final	
1				
2				
3				

### Calculation

### NOTES

Sample mass	= m	=...g
Sodium Hydroxide Solution difference (from Table -1 & Table 2 = $V_1 - V_2$ cm <sup>3</sup> )	= V	=...cm <sup>3</sup>
1000 cm <sup>3</sup> M <sub>2</sub> NaOH=M <sub>2</sub> g mol. , NaOH=M <sub>2</sub> g mol. CH <sub>3</sub> COOH=M <sub>2</sub> g mol. OH, where M <sub>2</sub> =molarity of sodium hydroxide solution.		
(V <sub>1</sub> - V <sub>2</sub> ) cm <sup>3</sup> of M <sub>2</sub> NaOH = (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) / 1000 g. mol of OH Or (V <sub>1</sub> - V <sub>2</sub> ) cm <sup>3</sup> of M <sub>2</sub> NaOH = 17 * (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) / 100 g. of OH		
We know this is due to m sample (in g), hence 100gms of sample contain (% of the OH <sub>2</sub> ) group %OH = 17 * (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) * 100 / m * 100 0		
Hydroxy Group (OH) number can be calculated in a given sample (phenol) : 94 gm (1 g mol) of sample contain = 17 * (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) * 94 / m * 100 0 Since 16.03 g mass is due to the one OH group Therefore phenol contain =17 * (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) * 94 / m * 100 0 * 17 = (M <sub>2</sub> (V <sub>1</sub> - V <sub>2</sub> )) * 0. 94 / m OH groups		
<b>Result</b> In given sample % of Hydroxy Group (OH) = .....% Number of Hydroxy Group (OH) in Phenol = .....		

## QUANTITATIVE ESTIMATION OF ETHYL METHYL KETONE

### Ethyl Methyl Ketone Introduction

Ethyl Methyl Ketone is a federal hazardous air pollutant and was identified as a toxic air contaminant.

### Properties of Ethyl Methyl Ketone

I. Molecular Weight	72.10
II. Boiling Point	79.6 oC
III. Melting Point	-86.35 °C
IV. Flash Point	-6 °C (21 °F)
V. Vapor Pressure	90.6 mm Hg at 25 °C
VI. Vapor Density	2.41 (air = 1)
VII. Density/Specific Gravity	0.805 at -20/4 °C (water = 1)

VIII. Log/Octanol Partition Coefficient	0.29
IX. Henry's Law Constant	$1.05 \times 10^{-5} \text{ atm-m}^3/\text{mole}$
X. Conversion Factor	1 ppm = 2.95 mg/m <sup>3</sup>

Practical : Analytical  
Chemistry

Estimate the amount of Ethyl Methyl Ketone present in the whole of the given solution.

## NOTES

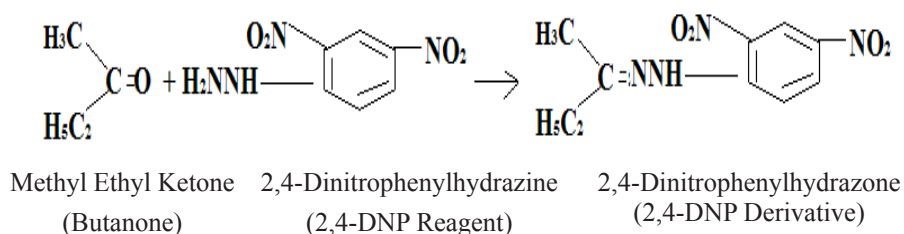
### Objectives

- Find out the amount of Ethyl Methyl Ketone present in given solution

### Principle

Ketone or aldehyde group have carbonyl chromophoric group (C=O) which absorb the in the ultraviolet region of EM spectrum and provide two band 195 nm and another 270-285 nm.

Generally Methyl Ethyl Ketone is determine by 2,4-dinitrophenylhydrazone derivative (2,4-DNP). In high conc. ketone react with 2,4-dinitrophenylhydrazine and produced 2,4-dinitrophenylhydrazone and separate out easily and gravimetrically measure.



If the conc. is less than  $10^{-3}\text{M}$ , then hydrazone remains in methanol and produce a red color on the addition of alkali which absorb nearly 480nm. Reagent hydrazine have color yellow. In spectro-photometric easily determine the ethylmethylketone.

### Requirements

Spectrophotometer
Cuvette (matched cuvette)
Flask (volume 100cm <sup>3</sup> )
Beaker (volume 100cm <sup>3</sup> )
Test tube Stoppered type (volume 20cm <sup>3</sup> )
Pipette (volume 10cm <sup>3</sup> )
Weighing Bottle

## NOTES

### Chemical List

Ethyl Methyl Ketone  
Methanol  
Hydrochloric acid  
Potassium hydroxide  
2,4-dinitrophenylhydrazone

### Solutions Provided

- (i) **2, 4-Dinitrophenylhydrazine (saturated)**: In Spectroscopic grade methanol dissolve the reagent in sufficient amount and prepare this solution.
- (ii) **Stock solution of methylethyl ketone**:  
In Spectroscopic grade methanol dissolve the ethylmethylketone (0.1g only) and make it up to volume 100cm<sup>3</sup> in similar fashion.
- (iii) **Potassium hydroxide**:  
In Spectroscopic grade methanol (Volume 100cm<sup>3</sup> )add the already prepare solution (10gm KOH + 20cm<sup>3</sup> distilled water)

### Procedure

#### Follow the steps which is given below

Step 1: Take the Stopered test tube and label it 1 to 5  
And pore the 1,2,3,4 and 5 cm<sup>3</sup> ethylmethylketone in same series

Step 2: Pore the Spectroscopic grade methanol 4,3,2,1 and 0 cm<sup>3</sup> in the same ref. of tube which are label 1 to 5, hence the the toytal volume in each test tube is 5 cm<sup>3</sup> now and it give the standard solution of 0.2, 0.4, 0.6, 0.8 and 1mg/cm<sup>3</sup>

Step 3: Take one more stopered test tube and label it sample and pore 5 cm<sup>3</sup> solution in it from unknown sample

Step 4: Take one more stopered test tube and label it Blank and pore 5 cm<sup>3</sup> methanol for preparing the blank solution.

Step 5: In each label standard, sample and blank test tubes pore 1cm<sup>3</sup> 2,4-DNP solution and also pore 1 drop of conc. HCL

Step 6: Stoppered test tube put in the warm (temp. more than 90 degree) water up to 5-7 mins. And then cool it.

Step 7: Pore 5ml of KOH solution in each test tube and mix it gentely

Step 8: With the help of spectrophotometer measure the absorbance of all the solution at 480nm against the reagent.

Step 9: Put this measure data in table 1 and draw the graph b/w absorbance vs. concentration and got a calibrated curve

Step 10: Find the conc. Of sample with the help of calibrated curve

Step 11: Find the conc. Of ethylmethylketone in given unkown sample using dilute factor and report value



## Observations and Calculations

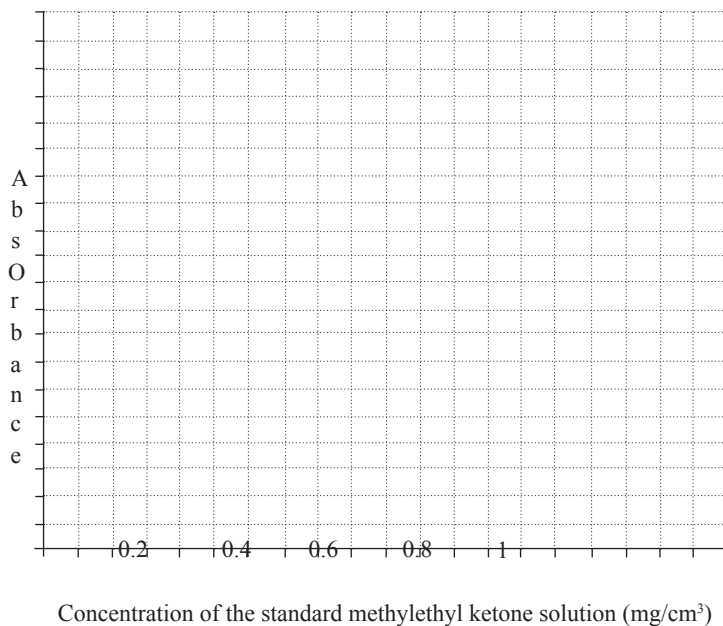
Practical : Analytical  
Chemistry

Table I: Absorbance values of the 2,4-Dinitrophenylhydrazine derivative of standard and sample solutions of methylethyl ketone

1	2	3	4	5	6	7	8
	Volume of the	Volume	Conc.	Volume of	Volume of	Volume	Absorbance
S. No.	standard /	of	of	KOH	Conc. HCl	of	at
	sample	methanol	standard	(cm <sup>3</sup> )	(Drops)	2,4-DNP	480 nm
	solution (cm <sup>3</sup> )	(cm <sup>3</sup> )	solution			solution	
						(cm <sup>3</sup> )	
1	1	4	0.1	5	1	1	
2	2	3	0.2	5	1	1	
3	3	2	0.3	5	1	1	
4	4	1	0.5	5	1	1	
5	5	0	1.0	5	1	1	
Sample	5	0	?	5	1	1	

## NOTES

### 2. Plotting the Calibration Curve



## NOTES

- Find the conc. of sample with the help of calibrated curve
- Plot the value of absorbance of the sample solution on the calibration curve obtained and determine the corresponding concentration value in mg.

Ethyl Methy Ketone Solution conc. = ..... mg/cm<sup>3</sup>

### Result

Ethyl Methy Ketone Solution conc. = .....×1000 mg/dm<sup>3</sup>  
= .....g/dm<sup>3</sup>

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## QUANTITATIVE ESTIMATION OF GLUCOSE

---

Estimate the amount of glucose present in the whole of the given solution

### Objective

To estimate the amount of glucose in the whole of the given solution.

### Introduction

A famous monosaccharide is Glucose in biology. It is one of the real results of photosynthesis. The living cell utilizes it as a wellspring of vitality and metabolic transitional. The name “Gluc” originates from the Greek word “glykys”, which means “sweet”, plus the suffix “-ose” which indicates a sugar. Two stereoisomers of the aldohexose sugars are known as glucose, only one of which (D-glucose) is biologically active. This form (D-glucose) is regularly alluded to as dextrose monohydrate, or, particularly in the sustenance business, basically dextrose (from dextrorotatory glucose)

### Structure of Glucose: (D or L Designation)

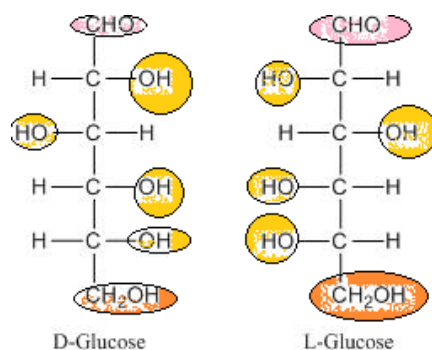
For the structure of carbohydrates the holding example of the hydrogen and hydroxyl bunches around every carbon molecule is vital. Carbon atom is called asymmetric carbon atom when a carbon atom which is bonded to four different groups. Glucose, with six carbon atoms, has four asymmetric carbon atoms. The pattern of the OH's and H's arrangements on these atoms is very important. Structural formulas for sugar molecules are often written in the vertical arrangement with the aldehyde or the ketone group at or near the top. When written in the specific way, the position of the OH on the last asymmetric carbon atom will tell us whether we are dealing with a “D” sugar or an “L” sugar. “D” stands for dextro and “L” stands for levo. If the OH is on the right, it is a “D” sugar, in this case D-glucose. If the OH is on the left, then it has been “L” sugars. When we see D's and L's in front of the names of carbohydrates, this is the logical reason for it.

## Fischer Projection Formula

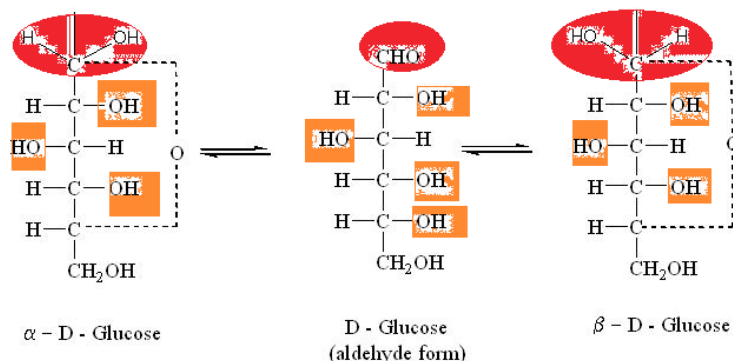
Hermann Emil Fischer in 1891 conceived the Fischer projection. It is a two-dimensional representation of a three-dimensional molecule by projection. Fischer projections were initially proposed for the delineation of sugars and utilized by scientific experts, especially in natural science and organic chemistry. The utilization of Fischer projections in non-carbohydrates is debilitated; accordingly illustrations are questionable when mistaken for different sorts of illustration.

In Fischer projection all bonds are depicted as horizontal or vertical lines and these entire horizontal bonds project toward the viewer, while vertical bonds project away from the viewer.

### Visualizing a Fischer Projection



Fischer Projection



## Haworth Projection Formula

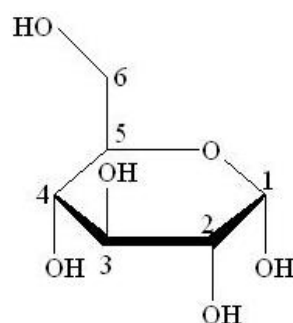
A Haworth projection is a typical method for representing to the cyclic structure of monosaccharide with a straightforward three-dimensional projection. The Haworth projection was named after the English Chemist Sir Walter N. Haworth.

## NOTES

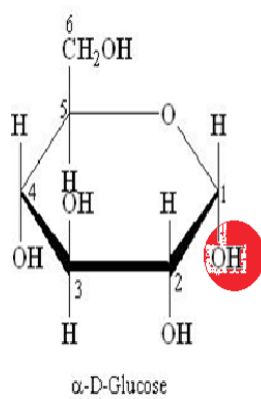
## NOTES

### A Haworth Projection has the Following Characteristics:

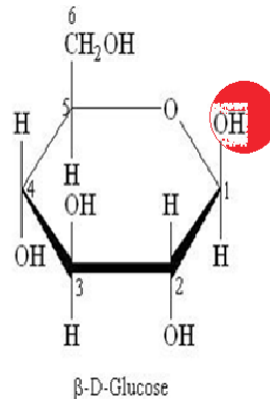
- Carbon is the implicit type of atom. In the example Fig (a), the atoms numbered from 1 to 6 are all carbon atoms. Carbon 1 is known as the **Anomeric Carbon**.
- Hydrogen atom on carbon are certain. In the model, atom 1 to 6 have additional hydrogen atom not delineated.
- A thicker line indicates atoms that are closer to the observer. In the example Fig (a), atoms 2 and 3 (and their corresponding OH groups) are the closest to the observer, atoms 1 and 4 are farther from the observer and finally the remaining atoms (5, etc.) are the farthest.



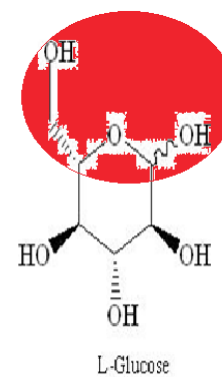
(a)



(b)



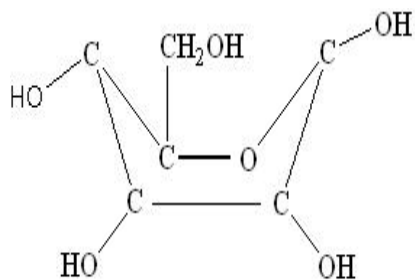
(c)



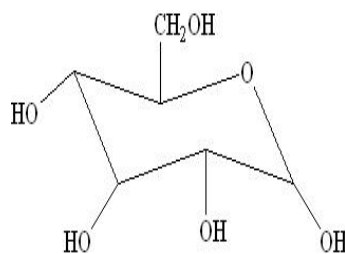
(d)

## Boat and Chair Forms

### (i) $\alpha$ -D-Glucose

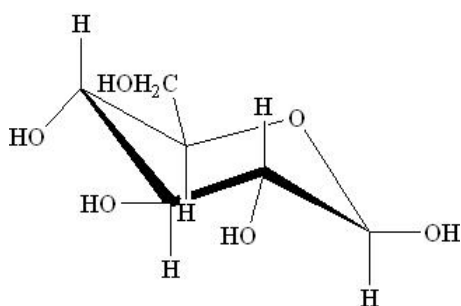


$\alpha$ -D-Glucose  
Boat

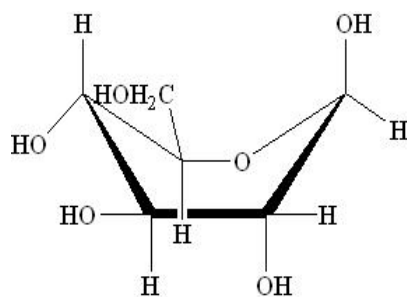


$\alpha$ -D-Glucose  
Chair

### (ii) $\beta$ -D-Glucose:



$\beta$ -D-Glucose  
Chair



$\beta$ -D-Glucose  
Boat

## NOTES

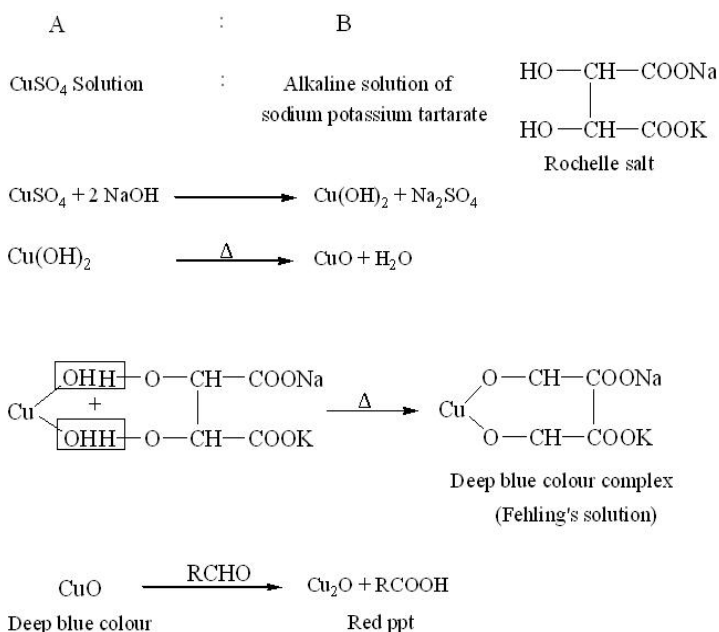
### Fehling's Solution

Fehling's solution test is used for the chemical testing of solutions to differentiate between water-soluble aldehyde and ketone functional groups, and as a test for monosaccharides. The test was developed by German Chemist **Hermann von Fehling** in 1849.

### Laboratory Preparation

First prepare the two separate solutions which are known as Fehling's A and Fehling's B. Fehling's A is an aqueous solution of copper sulphate pentahydrate crystals, but Fehling's B is a transparent solution of aqueous potassium sodium tartrate and it is a strong alkali. Equal amounts of these two solutions give the final Fehling's solution which is dark blue in color. Detailed reaction given below:

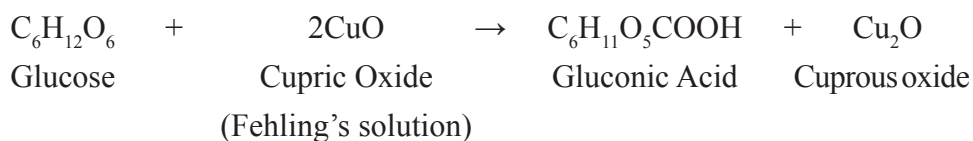
## NOTES



### Logics in Frame to Calculate/Determine the Glucose

A freshly prepared Fehling's solution is first standardized by titration against a standard solution of pure glucose. Then standardized Fehling's solution is then used to find out the amount of glucose in an unknown sample.

The Fehling's solution being a solution of cupric ions is blue in colour and at the end point changes to red color precipitate of cuprous oxide. As the supernatant liquid is blue and the precipitate is red in colour, there may be some difficulty in determination of end point accurately. Hence sometimes a methylene-blue indicator is employed for accurate determination of the end point.



### Estimation of Glucose in Lab

#### Standardisation of Fehling's Solution

Take 1.25 gm of glucose and dissolve it in the 250 ml water to make a standard solution. Take 20 ml of fehling's A and B each in a dry conical flask and shake it well. Pipette out 20 ml of this mixed solution in a conical flask and pore 20 ml water in it to dilute this solution. Warm the solution nearly 70 to 71 degree and take the standard solution of gulocose (freshly prepared) in a burette and pore it gently in the boiling solution of fehing's solution until the blue color diminish. Maintain this temperature for every addition of glucose solution. Repeat this process until the end point approached then add glucose solution drop bt drop till the end point achived.

### Procedure

- Step 1: Choose titrant and titrate for the test
- Step 2: Set the normality of the solution for test
- Step 3: Set the volume for titrate
- Step 4 : Start titration
- Step 5: When the blue color just start diminishing, select the indicator
- Step 6: Continue this process up the color change of solution and note the final points.
- Step 7: From the reading the normality of titrant calculated by putting this equation:

$$N_1 V_1 = N_2 V_2$$

After calculating the normality the amount of substance can be calculated.  
(Atomic Weight \* Normality \* Volume of given solution upto which it is made up)/ 1000

**Note:** Atomic weight of Glucose is =180.1559 g mol.<sup>-1</sup>

### Observations and Calculations

S.NO	Tirate Volume (in mL)	Reading in Burette		Tirant volume used (in mL)
		Initial Reading	Final Reading	
1				
2				
3				
4				
5				
6				
7				

Titrate Normality which we used in experiment, N1=.....N.

Titrate Volume used- V1 =.....mL.

Titrant Volume Used- V2 =.....mL.

Normality of tirrant N2=  $N_1 V_1 / V_2$  =.....N.

The quantity of substance in solution =

(Atomic Weight \* Normality \* Volume of given solution upto which it is made up)/ 1000 =.....g.

### Result

The weight of substance in the given solution =.....g.

### NOTES

NOTES

**EXPERIMENT-SEMI-MICRO  
QUALITATIVE ANALYSIS OF MIXTURES**

**Semi-Micro Qualitative Analysis of Mixture**

Aim: Analysis of mixtures containing two less familiar cations like W, Tl, Mo, Se, Te, Ce, Zr, Th, Ti, V, U and Li and two familiar cations like Pb, Cu, Bi, Cd, Mn, Ni, Co, Zn, Ca, Ba, Sr and Mg.

**Separation of Cations in to two Group- One which have very familiar cations and Group second have less familiar cations (familiar means common for us)**

***Ions to be Identified***

**Two Familiar Cations**

- (a) Ammonium ( $\text{NH}_4^+$ ), Silver ( $\text{Ag}^+$ ), Mercurous ( $\text{Hg}^+$ ), Mercuric ( $\text{Hg}^{2+}$ ), Lead ( $\text{Pb}^{2+}$ ), Bismuth ( $\text{Bi}^{3+}$ ), Cupric ( $\text{Cu}^{2+}$ ), Cadmium ( $\text{Cd}^{2+}$ ), Arsenic ( $\text{As}^{3+}$ ), Antimony ( $\text{Sb}^{3+}$ ), Stannous ( $\text{Sn}^{2+}$ ), Stannic ( $\text{Sn}^{4+}$ ), Aluminum ( $\text{Al}^{3+}$ ), Chromic ( $\text{Cr}^{3+}$ ), Ferrous ( $\text{Fe}^{2+}$ ), Ferric ( $\text{Fe}^{3+}$ ), Manganous ( $\text{Mn}^{2+}$ ), Cobalt ( $\text{Co}^{2+}$ ), Nickel ( $\text{Ni}^{2+}$ ), Zinc ( $\text{Zn}^{2+}$ ), Calcium ( $\text{Ca}^{2+}$ ), Strontium ( $\text{Sr}^{2+}$ ), Barium ( $\text{Ba}^{2+}$ ), Magnesium ( $\text{Mg}^{2+}$ ).

**Two Less Familiar Cations**

- (b) Thallium ( $\text{Tl}^+$ ), Tungsten as tungstate ( $\text{WO}_4^{2-}$ ), Selenium as selenite ( $\text{SeO}_3^{2-}$ ) or selenate ( $\text{SeO}_4^{2-}$ ), Tellurium as tellurite ( $\text{TeO}_3^{2-}$ ) or tellurate ( $\text{TeO}_4^{2-}$ ), Molybdenum as molybdate ( $\text{MoO}_4^{2-}$ ), Cerium ( $\text{Ce}^{4+}$ ), Thorium ( $\text{Th}^{4+}$ ), Titanium ( $\text{Ti}^{4+}$ ), Zirconium as zirconyl ( $\text{ZrO}^{2+}$ ), Vanadium as metavanadate ( $\text{VO}_3^{2-}$ ), Uranium as uranyl ( $\text{UO}_2^{2+}$ ), Lithium ( $\text{Li}^+$ ).

Information: In the given solution (aq.) or mixture find the cations

Process:

On the basis of reagent type for detection of cations we divide the cations in to six groups.

Group	Reagent	Cations
Group 1	In which no common reagents	$\text{NH}_4^+$ , $\text{Mg}^{2+}$ , $\text{Li}^+$
Group 2	Dil. HCl (2M)	$\text{Hg}^+$ , $\text{Ag}^+$ , $\text{Pb}^{2+}$ , $\text{Tl}^+$ , $\text{WO}_4^{2-}$
Group 3	Dil. HCl + $\text{H}_2\text{S}$ gas	$\text{Hg}^{2+}$ , $\text{Pb}^{2+}$ , $\text{Bi}^{3+}$ , $\text{Cu}^{2+}$ , $\text{Cd}^{2+}$ $\text{As}^{3+}$ , $\text{Sb}^{3+}$ , $\text{Sn}^{2+}$ , $\text{Sn}^{4+}$ , $\text{SeO}_3^{2-}$ , $\text{TeO}_3^{2-}$ , $\text{TeO}_4^{2-}$ , $\text{MoO}_4^{2-}$
Group 4	$\text{NH}_4\text{Cl}$ + $\text{NH}_4\text{OH}$ Solutions (2M)	$\text{Al}^{3+}$ , $\text{Cr}^{3+}$ , $\text{Fe}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Ce}^{4+}$ , $\text{Th}^{4+}$ , $\text{Ti}^{4+}$ , $\text{ZrO}^{2+}$ , $\text{VO}_3^{2+}$ , $\text{UO}_2^{2+}$ .
Group 5	$\text{NH}_4\text{Cl}$ + $\text{NH}_4\text{OH}$ + $\text{H}_2\text{S}$ gas	$\text{Co}^{2+}$ , $\text{Ni}^{2+}$ , $\text{Mn}^{2+}$ , $\text{Zn}^{2+}$ .
Group 6	$\text{NH}_4\text{Cl}$ + $\text{NH}_4\text{OH}$ + $(\text{NH}_4)_2\text{CO}_3$	$\text{Ca}^{2+}$ , $\text{Sr}^{2+}$ , $\text{Ba}^{2+}$



**Procedure:** For find out the cations in a given mixture or solution, we Pour the solution/mixture in various test tube and perform various test on it.

**First we check for Group 6**

Test for Ammonium Cations:

For Ammonium ( $\text{NH}_4^+$ ) – For ammonium cation detection we perform four test:

Take the

**NOTES**

Test 1: Pour 0.5 cm <sup>3</sup> of NaOH solution (2M) in to 0.5 cm <sup>3</sup> of given solution in a test tube and heat gently. If a pungent and suffocating smell	Result and Reasons: It is the smell of Ammonium, it means Ammonium cations may be present Reason: $\text{NH}_4^+ + \text{OH}^- \rightarrow \text{NH}_3(\text{g}) + \text{H}_2\text{O}$
Test 2: Pour 0.5 cm <sup>3</sup> of NaOH solution (2M) in to 0.5 cm <sup>3</sup> of given solution in a test tube and heat gently. A rod dipped in Conc. HCl put near to the mouth of test tube If white fumes seen.	Result and Reasons: Ammonium may be present, ammonia react with HCl and produced copious white fumes of $\text{NH}_4\text{Cl}$
Test 3- Confirmatory test : 0.5 cm <sup>3</sup> of saturated sodium hydrogen tartrate solution in to the test tube after this Pour few drops of given solution, If white crystalline precipitate is formed.	Result and Reasons: Ammonium $\text{NH}_4^+$ confirmed Reason : $\text{NH}_4^+ + \text{H}_2\text{C}_4\text{H}_4\text{O}_6 \rightarrow \text{NH}_4\text{HC}_4\text{H}_4\text{O}_6 + \text{H}^+$ (white precipitate)
Test 4- Confirmatory test : 0.5 cm <sup>3</sup> of saturated sodium hexanitritocobaltate (III) solution in to the test tube after this Pour few drops of given solution, If yellow precipitate is formed.	Result and Reasons: Ammonium $\text{NH}_4^+$ confirmed, yellow precipitate of ammonium hexanitritocobaltate (III) Reason : $3\text{NH}_4^+ + [\text{Co}(\text{NO}_2)_6]^{3-} \rightarrow (\text{NH}_4)_3[\text{Co}(\text{NO}_2)_6]$ (yellow precipitate)

**Test for Lithium Cations**

For Lithium ( $\text{Li}^+$ ) – For Lithium cation detection we perform two test:

Test 1: Take 1 cm <sup>3</sup> of the solution, Pour $\text{NH}_3$ solution dropwise till smell persists then Pour 1 cm <sup>3</sup> of ammonium fluoride solution. If white crystalline precipitate is formed.	Result and Reasons: Lithium ( $\text{Li}^+$ ) confirmed Reason : $\text{LiF}$ (white precipitate)
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## NOTES

Test 2: Take 1 cm <sup>3</sup> of the solution, Pour NH <sub>3</sub> solution dropwise till smell persists then Pour 1 cm <sup>3</sup> of ammonium carbonate solution. If white crystalline precipitate is formed.	Result and Reasons: Lithium (Li <sup>+</sup> ) confirmed Reason : Li <sub>2</sub> CO <sub>3</sub> (white precipitate)
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### *Test for Magnesium Cations*

For Magnesium (Mg<sup>2+</sup>) – For magnesium cation detection we perform two test:

Test 1: Take 1 cm <sup>3</sup> of the solution, pour some solid NH <sub>4</sub> Cl, and then NH <sub>3</sub> solution dropwise till smell persists then Pour 1 cm <sup>3</sup> of saturated Na <sub>2</sub> HPO <sub>4</sub> solution and stir with a glass rod. If white crystalline precipitate is formed.	Result and Reasons: Magnesium (Mg <sup>2+</sup> ) confirmed Reason : Mg(NH <sub>4</sub> )PO <sub>4</sub> .6H <sub>2</sub> O (white precipitate)
Test 2: Take 1 cm <sup>3</sup> of the solution, Pour some solid NH <sub>4</sub> Cl, and then NH <sub>3</sub> solution dropwise till smell persists then Pour 1 cm <sup>3</sup> of oxine reagent (C <sub>9</sub> H <sub>6</sub> NO) and boiled it If yellow crystalline precipitate is formed.	Result and Reasons: Magnesium (Mg <sup>2+</sup> ) confirmed Reason : Mg(C <sub>9</sub> H <sub>6</sub> NO) <sub>2</sub> .4H <sub>2</sub> O (Yellow precipitate)

## Group 2

### Centrifugation

#### Process to determine the group two ions.

Pour a few drop of dilute HCL(2M) in to 5 cm<sup>3</sup> cold of the given mixture or solution. If a precipitate forms then continue pouring the acid and shake till no further precipitate take place.

After centrifuging transfer centrifugate in to a small beaker for evaporation, carry out the precipitation process in the beaker and after this transfer the precipitate in to the centrifuge tube.

Result: White precipitate – indicate the presence of one or more cations of -Hg<sup>+</sup>, Ag<sup>+</sup>, Pb<sup>2+</sup>, Tl<sup>+</sup>, WO<sub>4</sub><sup>2-</sup> (Group two ions present in the solution)

#### Procedure

##### Step 1:

Wash the residue with 2 cm<sup>3</sup> of HCl(dil.) after it with water nearly 2 cm<sup>3</sup>, then boil the residue in the centrifuge tube with 2 cm<sup>3</sup> of water. Decant the centrifugate into a test tube. Repeat extraction with 2 cm<sup>3</sup> boiling water and combine the centrifugates.

## NOTES

<p><b>Result1</b></p> <p>If white residue form then may be <math>Hg^+</math>, <math>Ag^+</math> (<math>Hg_2Cl_2</math> or <math>AgCl</math>) present or if Yellow then <math>WO_4^{2-}</math> (<math>H_2WO_4</math>) may be present</p> <p>Boil residue with (5 cm<sup>3</sup>) <math>NH_3</math> dilute solution and centrifuge .</p> <p>Result Step 1 : If Black precipitate forms (due to <math>Hg(NH_2)Cl</math> and <math>Hg</math>)</p> <p>Then dissolve these residue with (2 cm<sup>3</sup>) aqua regia, then cool it and perform these two test</p> <p><b>Test 1:</b> Pour (1 cm<sup>3</sup>) of solution then pour <math>NaOH</math> solution drop by drop and shake it well, Black precipitate of <math>Hg_2O</math> is formed</p> <p><b>Test 2:</b> Pour (1 cm<sup>3</sup>) of solution then pour <math>K_2CrO_4</math> solution and heat it. If Brown precipitate of <math>Hg_{2Cr}O_4</math> is formed</p> <p><b>Presence of <math>Hg^+</math> confirmed</b></p> <p>Result Step 2 :</p> <p>The solution may contain <math>Ag^+</math>, <math>WO_4^{2-}</math> (<math>AgNH_3</math> or <math>H_2WO_4</math>)</p> <p>Pour Dilute <math>HCl</math> drop wise and shake it well till the faint precipitate forms , which re-dissolve by pouring dilute <math>NH_3</math> solution , Pour <math>KI</math> solution drop wise and shake it well till the precipitate</p> <p>If yellow Precipitate Form (May be <math>Ag</math> is present)</p> <p><b>Test 1:</b> Pour sodium thiosulphate of solution if the precipitate dissolve then <math>Ag^+</math></p> <p><b>Presence of <math>Ag^+</math> confirmed</b> (May be <math>H_2WO_4</math> Present)</p> <p>Centrifugate- Tungstic acid <math>H_2WO_4</math> solution,</p> <p><b>Test 1:</b> Reduce the volume by evaporating and cool it, then Pour (0.5 cm<sup>3</sup>) of solution in test tube and then pour ferrous sulphate. If Brown precipitate of ferrous tungstate is formed , then add (1 cm<sup>3</sup>) dilute <math>HCl</math> and boil then precipitate turns white then yellow due to tungstic acid.</p> <p><b>Presence of W confirmed</b></p>	<p><b>Centrifugates:</b> it May contain <math>Pb^{2+}</math>, <math>Tl^+</math>, which may be provide crystal on cooling, Pour 1 cm<sup>3</sup> of Conc. <math>H_2SO_4</math> and evaporate till fumes are seen, then cool it and dilute it with water (5 cm<sup>3</sup>) if Precipitate is formed, centrifuge.</p> <p><b>Result2 :</b></p> <p>If white precipitate formed</p> <p>It may contain <math>Pb^{2+}</math>, <math>Tl^+</math></p> <p>For <math>Pb^{2+}</math> following test and process we have to follow</p> <p>For <math>Pb^{2+}</math> :</p> <p>Dissolve the precipitate in ammonium acetate solution and following test to be perform</p> <p><b>Test 1:</b> In 1 cm<sup>3</sup> solution pour acetic acid and <math>K_2CrO_4</math> solution, If Yellow precipitate formed (<math>PbCrO_4</math>)</p> <p><b>Presence of <math>Pb^{2+}</math> Confirmed</b></p> <p><b>Test 2:</b> In 1 cm<sup>3</sup> solution pour <math>KI</math> and Boil it then cool it , If Yellow precipitate formed (<math>PbI_2</math>) which dissolve on heating and reprecipitate on cooling</p> <p><b>Presence of <math>Pb^{2+}</math> Confirmed</b></p> <p>For <math>Tl^+</math> following test and process we have to follow</p> <p>For <math>Tl^+</math> :</p> <p>Pour the dilute <math>NH_3</math> Solution and boil of excess <math>NH_3</math>. Cool it and then perform the following tests:</p> <p><b>Test 1:</b> In 1 cm<sup>3</sup> solution pour acetic acid and <math>K_2CrO_4</math> solution, If Yellow precipitate formed (<math>Tl_2CrO_4</math>)</p> <p><b>Presence of <math>Tl^+</math> Confirmed</b></p> <p><b>Flame Test 2:</b> Evaporate the 2 cm<sup>3</sup> solution to dryness in china dish, Cool it and mix the residue in Conc. <math>HCl</math>. A speck of the mixture taken at the tip of nickel spatula and introduced in to Bunsen flame. An intense Green color appear.</p> <p><b>Presence of <math>Tl^+</math> Confirmed</b></p>
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### Group 3

#### Centrifugation

#### NOTES

##### Process to determine the group three ions.

Pour a few drop of dilute  $H_2O_2$  and dilute the solution with twice of its volume with water, Heat it nearly boiling point and saturate it with  $H_2S$  gas, Shake it well if precipitate is formed then pass the  $H_2S$  gas till no more precipitate formed

**Result:** Black precipitate – indicate the presence of one or more cations of  $Hg^{2+}$ ,  $Pb^{2+}$ ,  $Bi^{3+}$ ,  $Cu^{2+}$  (Group three ions present in the solution)

Yellow precipitate – indicate the presence of one or more cations of  $Cd^{2+}$ ,  $As^{3+}$ ,  $Sn^{2+}$ ,  $Sn^{4+}$ ,  $Se$ , (Group three ions present in the solution)

Orange precipitate – indicate the presence of one or more cations of  $Sb^{3+}$  (Group three ions present in the solution)

Brown precipitate – indicate the presence of one or more cations of  $Sn^{2+}$ ,  $Te$ ,  $Mo^{6+}$

(Group three ions present in the solution)

Wash the residue with 2 cm<sup>3</sup> ammonium chloride solution and saturated with  $H_2S$  then centrifugate. Transfer the residue in to small beaker and 5 cm<sup>3</sup> of ammonium sulphide solution then heat it nearly 60 degree and maintain this temperature up 5 minutes, if precipitate formed , cool it and centrifuge.

##### Result

If Black Precipitate : them may be  $HgS$ ,  $PbS$ ,  $Bi_2S_3$  or  $CuS$  present

If yellow Precipitate : them may be  $CdS$  present

Step 1: Wash the residue with water (2cm<sup>3</sup>) and centrifuged, transfer the residue in small beaker and pour 10cm<sup>3</sup> of dilute nitric acid and boil it up 5 minutes, If solid material form then cool it and centrifuge.

(a) For Black Residue: It means  $HgS$ .

Dissolve it with boiling 2cm<sup>3</sup> aqua regia and perform the following test

**Centrifugate:** Pour conc.  $HCl$  drop-wise for make it acidic only , warm it. If white or pale yellow precipitate formed then sulphur may be there and if Flocculant precipitate formed then centrifuge it, if not then discard the solution.

If **yellow** Residue appears then may be  $Se$  or  $As_2S_3$  may be present then go to Step 1(a)

If **Brown** Residue appears then may be  $Te$  or  $MoS_3$  may be present then go to Step 1(b).

If not then go to Step 1(c)

Step 1(a)(i): Dissolve the residue in conc.  $HCl$  (5cm<sup>3</sup>) and pour a pinch of potassium chlorate ( $KClO_3$ ).

Transfer the solution in to small beaker concentrate by evaporateing of water bath. Cool it and pour  $NH_3$  in this after it pour  $MgNO_3$  solution in it and wait for 5 minutes if precipitate appears(**White**) due to  $MgNH_4AsO_4 \cdot 6H_2O$

## NOTES

<p><b>Test1</b> : In 0.5 cm<sup>3</sup> solution pour NaOH drop-wise and shake it well, if brownish red precipitate change in to yellow (HgO)</p> <p><b>Test2</b> : In 0.5 cm<sup>3</sup> solution pour Na<sub>2</sub>CO<sub>3</sub> drop-wise and shake it well, and KI solution drop wise, shake it well if red precipitate formed (HgI<sub>2</sub>)</p> <p><b>Presence of Hg<sup>2+</sup> confirmed</b> if this is not then further move on Step 1: Centrifugate it: Step 2: after centrifugate it may contain Pb, Bi, Cu, Cd. Then pour dilute H<sub>2</sub>SO<sub>4</sub> till the precipitate formed (i) If White Precipitate Formed Dissolve it ammonium acetate mixture and perform the following test</p> <p><b>Test 1</b> : In 1 cm<sup>3</sup> solution pour acetic acid and K<sub>2</sub>CrO<sub>4</sub> solution yellow precipitate formed it means Pb CrO<sub>4</sub> is formed</p> <p><b>Test 2</b> : In 1 cm<sup>3</sup> solution pour KI solution and boil it first then cool it if a yellow precipitate formed which dissolve on heating and reappear after cooling (golden spangles)</p> <p><b>Presence of Pb(II) confirmed</b> If Not then again centrifugate then may be Cu, Cd or Bi present, Pour Conc. NH<sub>3</sub> solution drop wise shake it well if residue remains If white Residue is there then perform these two test for confirm Bi presence</p> <p><b>Test1</b> : In residue pour conc. H<sub>2</sub>O<sub>2</sub> solution, If yellow brown precipitate formed it means Bi ions is there.</p>	<p>Shake the residue and perform two test on it</p> <p>Test 1: Pour AgNO<sub>3</sub> solution in it contain few drops of acetic acid . A red precipitate of silver arsenate, Ag<sub>3</sub>AsO<sub>4</sub> is formed.</p> <p>Test 2: Pour 0.5 cm<sup>3</sup> of conc. HNO<sub>3</sub> and excess of ammonium molybdate solution. A yellow precipitate formed (due to ammonium arsenomolybdate)</p> <p><b>Presence of As confirmed</b> Step 1(a) (ii): Centrifugate: may contain Se, Te or Mo as chloride. Transfer it in to a small beaker and boil off NH<sub>3</sub> then pour 2 cm<sup>3</sup> conc. HCl and evaporate to half of its volume on a water bath ., then add 2 cm<sup>3</sup> of saturated solution of Sodium sulphite (Na<sub>2</sub>SO<sub>3</sub>) solution. If Red precipitate is formed , centrifuge Then perform the following process: Dissolve the residue by heating with 1 cm<sup>3</sup> HNO<sub>3</sub> and neutralize it pouring some solution of ammonium carbonate then perform two test on it</p> <p>Test 1: Pour BaCl<sub>2</sub> in the above solution, A white precipitate formed (BaSeO<sub>3</sub>)</p> <p>Test 2: Pour CuSO<sub>4</sub> in the solution , A bluish precipitate formed (CuSeO<sub>3</sub>)</p> <p><b>Presence of Se(IV) confirmed</b> Step 1(b)(i) : Dilute the solution with equal volume of water , pour 1 cm<sup>3</sup> of KI solution and a drop of sodium sulphite (Na<sub>2</sub>SO<sub>3</sub>) solution, If precipitate formed (Black ) then move in the process given below else move on step 1 (b) (ii) Process: Dissolve the black residue in Cold dilute HCl (2 cm<sup>3</sup>) and perform two test</p> <p>Test 1: Pour ammonium carbonate solution drop wise then pour BaCl<sub>2</sub> solution, if white Precipitate formed it means Te present</p> <p>Test 2: pour KI solution in it. If red colour is produced (due to [TeI<sub>6</sub>]<sup>2-</sup> it means Te present</p>
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**NOTES**

<p><b>Test2</b> : Dissolve the residue in 1 cm<sup>3</sup> dilute HNO<sub>3</sub> solution and pour KI solution in drop wise shake it well, Black precipitate appears due to BiI<sub>3</sub></p> <p><b>Presence of Bi(III) confirmed</b> if this is not then further move on Next step 1 , Centrifugate it: after centrifugate it may contain [Cu(NH<sub>3</sub>)<sub>4</sub>]<sup>3+</sup> or [Cd(NH<sub>3</sub>)<sub>4</sub>]<sup>3+</sup>. If deep blue color , Cu may be present . pour dilute acetic acid and shake it well till a clear solution is obtained and divide this solution in two part</p> <p>IN Fisrt Part: Perform the two test after separating the solution in two parts again</p> <p>Test 1: Pour Potassium ferrocyanide solution in it if reddish brown precipitate appears (due to copper ferrocyanide )</p> <p>Test 2: Pour potassium thiocyanate solution in it if black precipitate appears turning slowly in white (2Cu(SCN)<sub>2</sub> --à 2Cu(SCN) + (SCN)<sub>2</sub>)</p> <p><b>Presence of Cu(II) confirmed</b> IN Second Part: Perform the two test after separating the solution in two parts again</p> <p>Test 1: Pour NaOH solution in it if white precipitate appears (due to Cd(OH)<sub>2</sub> )</p> <p>Test 2: Pass the H<sub>2</sub>S gas in the solution if yellow precipitate appears (due to CdS)</p> <p><b>Presence of Cd(II) confirmed</b></p>	<p><b>Presence of Te(IV) confirmed</b> step 1 (b) (ii): Pour the 1 cm<sup>3</sup> of Conc. HCl and boil the solution to remove SO<sub>2</sub> then perform the two test to confirm the cations</p> <p>Test 1: Pour the potassium ferrocyanide in the above solution if reddish brown precipitate formed it means Mo present</p> <p>Test2: Pour two drops of ferrous sulphate in the above solution. A blue color is produced , It means Mo present</p> <p><b>Presence of Mo(IV) confirmed</b> <b>Step 1 (C) :</b> Centrifugate: May contain Sb or Sn as chloride, Boil to remove the H<sub>2</sub>S from the solution. Then cool the solution and take two step</p> <p>Step 1 c-(i)</p> <p>Step 1 c-(ii)</p> <p>Step 1 c-(i)</p> <p>Take the 2 cm<sup>3</sup> above solution and pour pinch of iron filling in it, then boil it to reduce Sn (IV) to Sn ( II). Cool it and centrifuge. And perform the two test</p> <p>Test 1: take the above solution nearly 1 cm<sup>3</sup> and pour two drops of HgCl<sub>2</sub> in it. If silky white precipitate form ( Hg<sub>2</sub>Cl<sub>2</sub>) it means Sn is confirmed</p> <p>Test 2: take the above solution nearly 0.5 cm<sup>3</sup> and pour few drops of NaOH solution in it. If white precipitate form ( Sn(OH)<sub>2</sub>) which is soluble in excess of NaOH, it means Sn is confirmed</p> <p><b>Presence of Sn confirmed</b> <b>Step 1 c-(ii):</b> Test: take the above solution nearly 1 cm<sup>3</sup> in a test tube then pour NH<sub>3</sub> in to it and then add a pinch of Solid oxalic acid in it and boil it , Pass the H<sub>2</sub>S from the solution , An Ornage precipitate is formed (Sb<sub>2</sub>S<sub>3</sub>)</p> <p><b>Presence of Sb(II) confirmed</b></p>
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#### Group 4: Centrifugation

##### Process to determine the group four ions.

Residue is washed with 2% ammonium nitrite solution and dissolve in the minimum quantity of dilute HCl, Prepare a mixture containing 2 cm<sup>3</sup> of 30% H<sub>2</sub>O<sub>2</sub> solution and a fresh pellet of NaOH dissolved in 2 cm<sup>3</sup> of water . then pour Weakly acidic test in to it and boil it for 5 minute. Centrifuge. (precipitate may appears which dissolve by boiling with dilute HCl or No precipitate)

If **White** Residue appears it means TiO<sub>2</sub> xH<sub>2</sub>O or ZnO<sub>2</sub> xH<sub>2</sub>O or MnO<sub>2</sub> xH<sub>2</sub>O

Or

If **Brown** Residue appears Fe(OH)<sub>3</sub>

Or

If **Yellow** Residue appears (CeO<sub>3</sub> xH<sub>2</sub>O)

Else No precipitate then move step X

- (i) **Test For Confirmation of Fe:** Pour drop of KCNS in 0.5 cm<sup>3</sup> solution, If Red color appears which indicate FeCNS

##### Presence of Fe(III) Confirmed

- (ii) **Test For Confirmation of Ti and Zr:** Pour drop of H<sub>2</sub>O<sub>2</sub> in 1cm<sup>3</sup> solution, If orange color appears which indicate **Ti** or If white precipitate it means Zr present

##### Presence of Ti (IV) confirmed or Presence of Zr (IV) confirmed

- (iii) **Test For Confirmation of Ce:** In solution nearly 1 cm<sup>3</sup> pour few drops of saturate oxalic acid if white precipitate formed that indicate Th or Ce, Centrifuge and discard supernatant. Boil residue for 5 minute in saturate ammonium Oxalate solution. If there is residue centrifuge it Boil residue with 2 cm<sup>3</sup> of NaOH solution. A yellow precipitate form (Ce(OH)<sub>4</sub>) that confirm the presence of Ce

##### Presence of Ce(IV) Confirmed

- (iv) **Test For Confirmation of Th:** In solution nearly 1 cm<sup>3</sup> pour few drops of saturate oxalic acid if white precipitate formed that indicate Th or Ce, Centrifuge and discard supernatant. Boil residue for 5 minute in saturate ammonium Oxalate solution. If there is residue centrifuge it Centrifugate it: Acidity 1 cm<sup>3</sup> of the solution with dilute HCl, Thorium Oxalate Precipitate formed that confirm the presence of Th.

#### NOTES



## NOTES

### Presence of Th(IV) Confirmed

(v) **Test For Confirmation of Mn** : In solution nearly 1 cm<sup>3</sup> pour few drops conc. H<sub>2</sub>SO<sub>4</sub> and evaporate till fumes. Cool, dilute and add two drops of conc. HNO<sub>3</sub> and a pinch of PbO<sub>2</sub>. Boil and allow to stand. Purple supernatant (MnO<sub>4</sub><sup>-</sup>) Indicates Mn

### Presence of Mn Confirmed

**Step X: Centrifugate: It may contain CrO<sub>4</sub><sup>2-</sup>, U<sub>2</sub>O<sub>7</sub><sup>2-</sup>**. Transfer to a small beaker and acidify with dilute 2 cm<sup>3</sup> of conc. HNO<sub>3</sub> and dilute with 5 cm<sup>3</sup> of water. Boil and concentrate to half volume. Pour 2 cm<sup>3</sup> of lead nitrate in it and pinch of ammonium acetate. Boil it and centrifuge.

If yellow Residue appears (PbCrO<sub>4</sub>) else go for centrifugate

**Test For Confirmation of Cr and V** : Dissolve the above yellow precipitate in 5 cm<sup>3</sup> of dilute HNO<sub>3</sub> and heat it and transfer it in test tube and cool it, then pour 5 cm<sup>3</sup> of amyl alcohol and two drops of H<sub>2</sub>O<sub>2</sub> (conc.). Close the tube mouth and mix it well if a blue layer comes indicate CrO<sub>5</sub>

It means **Presence of Cr confirmed**

If red Color layer appears it indicate VO<sub>2</sub><sup>3+</sup>

It means **Presence of V confirmed**

When we go for centrifugate: may contain Al<sup>3+</sup> or UO<sub>2</sub><sup>2+</sup> and excess of Pb. Pass H<sub>2</sub>S in it so all PbS appears, centrifuge again, wash the precipitate with water and combine the centrifugates. Boil the solution to remove all H<sub>2</sub>S and then put NH<sub>3</sub> in it, then add 5 cm<sup>3</sup> of ammonium carbonate solution in it, cool it and centrifuge it

If white Precipitate appears Then test for Aluminum or it may contain (UO<sub>2</sub>(CO<sub>3</sub>)<sub>3</sub>)<sup>4-</sup>

### *Test for Confirmation of Al*

Dissolve the residue in 1 cm<sup>3</sup> dilute HCl. Pour NaOH solution drop wise shake it well, White precipitate appears and which dissolve in excess NaOH.

It indicate Al presence

### Presence of Al confirmed

**Test for Confirmation of U:** Centrifugate it, Evaporate to small volume, acidify it with acetic acid and perform the test

In test pour NaOH in it if yellow precipitate appears (Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub>) it means presence of U



## Presence of U (VI) Confirmed

### Group 5

#### Centrifugation

#### Process to determine the group five ions.

Residue may contain CoS (black) NiS (black) or ZnS (grey). Wash the precipitate with 2 cm<sup>3</sup> of water containing little NH<sub>3</sub>, NH<sub>4</sub>Cl and H<sub>2</sub>S. Centrifuge and reject washing. Transfer the precipitate in to small beaker and add 5 cm<sup>3</sup> of dilute HCl followed by equal amount of water , Stir well and wait for 5 minute if there is residue or centrifuge it

**If precipitate appears:** Black : CoS , NiS . Dissolve it in 4 cm<sup>3</sup> of aqua regia and transfer to a china dish. Evaporate carefully by heating over a wire gauge to dryness, residue appears

For blue Residue:

It indicate Co<sup>2+</sup>

If it dissolve in 2 cm<sup>3</sup> water and pink solution appears it indicate Co<sup>2+</sup>

Test for Co:

In 1 cm<sup>3</sup> above solution pour NaOH drop wise and shake it well, and warm it gently. A blue precipitate appears (Co(OH)NO<sub>3</sub>) which change in pink (Co(OH)<sub>2</sub>

**Presence of Co<sup>2+</sup> confirmed**

For yellow Residue:

It indicate Ni<sup>2+</sup>

If it dissolve in 2 cm<sup>3</sup> water. Green color indicate Ni<sup>2+</sup>

Test for Ni:

In 1 cm<sup>3</sup> above solution pour NaOH drop wise and shake it well, and warm it gently. A green precipitate appears (Ni(OH)<sub>2</sub>)

**Presence of Ni<sup>2+</sup> confirmed**

If residue not formed then centrifuge it.

**Centrifugate:** May Contain Mn<sup>2+</sup> Or Zn<sup>2+</sup>. Boil it up to half volume to remove all H<sub>2</sub>S removes from it, then add 2 cm<sup>3</sup> of NaOH solution in it and two drops of conc. H<sub>2</sub>O<sub>2</sub>. Boil it for 2 minutes if there is residue or not

If not then go for again centrifuge or precipitate is there it means dark brown residue us there the go for Mn test

**Test for Confirmation of Mn:** if precipitate dissolve in boiling with dilute sulphuric acid, Pink color appears indicate Mn

So in Test: in above solution add NaOH drop wise and shake it well and warm in gently, a white precipitate Mn(OH)<sub>2</sub>. The precipitate became brown when expose in air.

#### Presence of Mn(II) Confirmed

If no precipitate in above case then we go first centrifugate, then perform the test for Zn.

#### NOTES

**NOTES**

**Test for Confirmation of Zn**

Pour the dilute  $H_2SO_4$  in  $1\text{ cm}^3$  of above solution drop wise, and shake it well, A white precipitate appears ( $Zn(OH)_2$ ) and dissolve in excess of  $H_2SO_4$  due to formation of  $ZnSO_4$

**Presence of Zn(II) confirmed**

**Group 6:**

**Centrifugation**

**Process to determine the group Six ions.**

Transfer the solution in small beaker and dissolve in  $5\text{ cm}^3$  of dilute acetic acid by heating on water bath. When completely dissolve the pour some potassium chromate ( $K_2CrO_4$ ) solution drop wise while stirring the hot solution, till faint yellow color persists in the supernatant indicating excess of the reagent. If precipitate forms, then boil it and cool it and centrifuge.

**If Residue is Yellow ( $BaCrO_4$ ), then prepare a flame test.**

**Flame test for Ba(II):** Dissolve the residue by heating with  $1\text{ cm}^3$  of conc. HCl and transfer in to china dish and evaporate to dryness, take this dry residue at tip of charred match stick and introduce in to the colorless Bunsen flame near the base, the flame became apple green it indicate Ba

**Presence of Ba (II) confirmed**

If not then then centrifugate it. Transfer the solution in to the beaker. Pour  $2\text{ cm}^3$  of saturated ammonium sulphate solution and  $0.5\text{ cm}^3$  of sodium thiosulphate solution. Boil it and reduce it to its half volume. If precipitate form then cool it if not then again go for centrifuge.

White Precipitate form	If no precipitate form the go for centrifugate Centrifugate: may contain Ca, Transfer the solution in to the beaker and make it alkaline by adding some amount of $NH_3$ solution and shake it well, then pour $2\text{ cm}^3$ of ammonium oxalate solution. A white precipitate of $CaC_2O_4$
<b>Flame test for Sr(II):</b> Dissolve the residue by heating with $1\text{ cm}^3$ of conc. HCl and transfer in to china dish and evaporate to dryness, take this dry residue at tip of charred match stick and introduce in to the colorless Bunsen flame near the base, the flame became crimson, it indicate Sr <b>Presence of Sr (II) confirmed</b>	Centrifuge and discard the centrifugate. Dissolve the residue by heating with $1\text{ cm}^3$ of conc. HCl and transfer in to china dish, evaporate to dryness <b>Flame test for Ca(II):</b> take this dry residue at tip of charred match stick and introduce in to the colorless Bunsen flame near the base, the flame became brick red, it indicate Ca <b>Presence of Ca (II) confirmed</b>

## **Description About Elements**

### **Silver (Ag)**

Unadulterated silver is almost white, glossy, delicate, exceptionally pliable, pliant, it is an incredible channel of warmth and power. It's anything but a synthetically dynamic metal, however it is assaulted by nitric corrosive (forming the nitrate) and by hot concentrated sulfuric corrosive. It has the most elevated electrical conductivity all things considered, yet its more prominent expense has kept it from being broadly utilized for electrical purposes.

### **Mercurous (Hg)**

It is a silver white toxic metallic component with a mirror-like appearance. It is a fluid at room temperature and utilized in thermometers, gauges, vapor lights, and batteries and in synthetic pesticides. The Physical and Chemical Properties are the attributes of a substance, similar to Mercury, which recognizes it from some other substance.

### **Lead (Pb)**

Lead is a somewhat blue white radiant metal. It is delicate, profoundly pliable, flexible, and a moderately poor transmitter of power. It is extremely impervious to erosion yet discolors upon presentation to air. Lead isotopes are the final results of every one of the three arrangement of normally happening radioactive components.

### **Bismuth (Bi)**

Bismuth is a white, crystalline, weak metal with a pinkish tinge. Bismuth is the most diamagnetic everything being equal, and the warm conductivity is lower than any metal aside from mercury. It has a high electrical opposition, and has the most noteworthy Hall impact of any metal (that is, the best increment in electrical obstruction when put in an attractive field). Bismuth is steady to oxygen and water yet disintegrates in concentrated nitric acid. All bismuth salts shape insoluble mixes when put into water.

### **Copper (Cu)**

Copper is a chemical element with symbol Cu (from Latin: cuprum) and atomic number 29. It is a delicate, moldable, and bendable metal with high warm and electrical conductivity. A newly uncovered surface of unadulterated copper has a pinkish-orange shading. Copper is utilized as a channel of warmth and power, as a building material, and as a constituent of different metal combinations, for example, sterling silver utilized in adornments, cupronickel used to make marine equipment and coins, and constantan utilized in strain checks and thermocouples for temperature estimation. Copper is one of only a handful couple of metals that can happen in nature in a specifically usable metallic shape (local metals). This prompted early human use in a few areas, from c. 8000 BC. A huge number of years after the fact, it was the primary metal to be purified from sulfide minerals.

## **NOTES**

## NOTES

### **Cadmium (Cd)**

Cadmium is a radiant, silver-white, bendable, entirely flexible metal. Its surface has a somewhat blue tinge and the metal is sufficiently delicate to be cut with a blade, yet it discolors in air. It is solvent in acids however not in soluble bases. It is comparable in numerous regards to zinc yet it frames progressively complex mixes. Around three-fourths of cadmium is utilized in Ni-Cd batteries, a large portion of the staying one-fourth is utilized for the most part for colors, coatings and plating, and as stabilizers for plastics. Cadmium has been utilized especially to electroplate steel where a film of cadmium just 0.05 mm thick will give finish security against the ocean. Cadmium can ingest neutrons, so it is utilized as a boundary to control atomic splitting.

### **Arsenic (As)**

Arsenic shows up in three allotropic structures: yellow, dark and dim; the steady shape is a silver-dim, weak crystalline strong. It discolors quickly in air, and at high temperatures copies shaping a white billow of arsenic trioxide. Arsenic is an individual from gathering Va of the occasional table, which consolidates promptly with numerous components. The metallic frame is weak, tharnishes and when warmed it quickly oxidizes to arsenic trioxide, which has a garlic scent. The non metallic frame is less responsive yet will break down when warmed with solid oxidizing acids and antacids. Arsenic mixes are utilized in making unique sorts of glass, as a wood additive and, recently, in the semiconductor gallium arsenade, which can change over electric flow to laser light.

### **Antimony (Sb)**

Antimony is a semimetallic synthetic component which can exist in two structures: the metallic shape is brilliant, shiny, hard and weak; the non metallic frame is a dim powder. Antimony is a poor conduit of warmth and power, it is steady in dry air and isn't assaulted by weaken acids or antacids. Antimony and a portion of its composites develop cooling. Antimony has been known since antiquated occasions. It is once in a while discovered free in nature.

### **Tin (Sn)**

Tin is a delicate, malleable, gleaming white metal. Tin isn't actually oxidized and opposes erosion since it is secured by an oxide film. Tin opposes erosion from refined ocean and delicate faucet water, and can be assaulted by solid acids, soluble bases and corrosive salts. Tin is utilized in for can covering: tin-plated steel compartments are broadly utilized for sustenance safeguarding. Tin composites are utilized from numerous points of view: as patch for joining channels or electric circuits, pewter, ringer metal, babbitt metal and dental amalgams. The niobium-tin compound is utilized for superconducting magnets, tin oxide is utilized for earthenware production

and in gas sensors (as it ingests a gas its electrical conductivity increments and this can be checked). Tin thwart was previously a typical wrapping material for nourishments and medications, now supplanted by the utilization of aluminum thwart.

### **Aluminum (Al)**

It is a shimmering white, flexible metallic component, the most plenteous in the world's outside layer however discovered just in mix, primarily in bauxite. Having great conductive and warm properties, it is utilized to frame some hard, light, consumption safe compounds. The Physical and Chemical Properties are the qualities of a substance, similar to Aluminum, which recognizes it from some other substance

### **Chromium (Cr)**

Chromium is a brilliant, fragile, hard metal. Its shading is silver-dark and it tends to be profoundly cleaned. It doesn't discolor in air, when warmed it borns and structures the green chromic oxide. Chromium is insecure in oxygen, it instantly delivers a thin oxide layer that is impermeable to oxygen and ensures the metal underneath. Chromium principle utilizes are in compounds, for example, treated steel, in chrome plating and in metal earthenware production. Chromium plating was once broadly used to give steel a cleaned shimmering mirror covering. Chromium is utilized in metallurgy to bestow consumption opposition and a glossy complete; as colors and paints, its salts shading glass an emerald green and it is utilized to create engineered rubies

### **Iron (Fe)**

Iron is a shiny, pliable, moldable, silver-dim metal. It is known to exist in four particular crystalline structures. Iron rusts in soggy air, yet not in dry air. It breaks up promptly in weaken acids. Iron is synthetically dynamic and structures two noteworthy arrangement of concoction intensifies, the bivalent iron (II), or ferrous, mixes and the trivalent iron (III), or ferric, compounds. Iron is the most utilized of the considerable number of metals, including 95 % of all the metal tonnage delivered around the world. On account of the mix of minimal effort and high quality it is crucial. Its applications go from sustenance compartments to family vehicles, from screddrivers to clothes washers, from load boats to paper staples. Steel is the best known combination of iron.

### **Manganese (Mn)**

Manganese is a pinkish-dim, synthetically dynamic component. It is a hard metal and is extremely weak. It is difficult to dissolve, yet effectively oxidized. Manganese is receptive when unadulterated, and as a powder it will consume in oxygen, it responds with water (it rusts like iron) and disintegrates in weaken acids. Manganese is fundamental to iron and steel

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generation. At present steel making accounts 85% to 90% of the aggregate interest, the majority of the aggregate interest. Manganese is a key segment of minimal effort hardened steel details and certain generally utilized aluminum composites.

### **Cobalt (Co)**

Cobalt is a hard ferromagnetic, silver-white, hard, shiny, weak component. Like iron, it tends to be charged. It is like iron and nickel in its physical properties. The component is dynamic artificially, framing numerous mixes. Cobalt is steady in air and unaffected by water, yet is gradually assaulted by weaken acids. Cobalt is utilized in many composites (superalloys for parts in gas turbine aircraft motors, consumption safe combinations, fast steels, established carbides), in magents and attractive chronicle media, as impetuses for the oil and concoction ventures, as drying specialists for paints and inks. Cobalt blue is a vital piece of craftsmen's palette and is utilized by make specialists in porcelain, earthenware, recolored glass, tiles and veneer adornments.

### **Nickel (Ni)**

Nickel is brilliant white. hard, pliant, and flexible metal. It is of the iron gathering and it goes up against a high clean. It is a genuinely decent conveyor of warmth and power. In its commonplace mixes nickel is bivalent, despite the fact that it accept different valences. It likewise frames various complex mixes. Most nickel mixes are blue or green. Nickel disintegrates gradually in weaken acids be that as it may, similar to press, winds up inactive when treated with nitric corrosive. Finely isolated nickel adsorbs hydrogen. The significant utilization of nickel is in the planning of combinations. Nickel amalgams are portrayed by quality, malleability, and protection from erosion and warmth. Around 65 % of the nickel devoured in the Western World is utilized to make treated steel, whose arrangement can fluctuate yet is ordinarily press with around 18% chromium and 8% nickel. 12 % of all the nickel devoured goes into super amalgams.

### **Zinc (Zn)**

Zinc is a radiant somewhat blue white metal. It is weak and crystalline at normal temperatures, yet it winds up bendable and pliant when warmed somewhere in the range of 110°C and 150°C. It is a genuinely receptive metal that will consolidate with oxygen and other non-metals, and will respond with weaken acids to discharge hydrogen. It is utilized mainly to electrify press, over half of metallic zinc goes into arousing steel, but at the same time is vital in the arrangement of certain amalgams. It is utilized for the negative plates in some electric batteries and for material and drains in building development.

### **Calcium (Ca)**

The chemical element Calcium (Ca), atomic number 20, is the fifth element and the third most abundant metal in the earth's crust. The metal is trimorphic, harder than sodium, yet milder than aluminum. As well as beryllium and aluminum, and not at all like the antacid metals, it doesn't cause skin-consumes. It is less synthetically receptive than basic metals and than the other soluble earth metals.

Calcium particles explained in water frame stores in channels and boilers and when the water is hard, that is, the point at which it contains excessively calcium or magnesium. This can be kept away from with the water conditioners. In the business, metallic calcium is isolated from the liquefied calcium chloride by electrolysis. This is acquired by treatment of carbonated minerals with chlorhydric corrosive, or like a sub result of the carbonates Solvay process. In contact with air, calcium builds up an oxide and nitride covering, which shields it from further consumption. It consumes noticeable all around at a high temperature to create nitride. The monetarily delivered metal responds effortlessly with water and acids and it produces hydrogen which contains amazing measures of smelling salts and hydrocarbides as debasements.

### **Strontium (Sr)**

Strontium is a delicate, silver-yellow, basic earth metal. It has three allotropic crystalline structures and in its physical and concoction properties it is like calcium and barium. Strontium responds energetically with water and rapidly discolors in air, so it must be put away out of contact with air and water. Because of its outrageous reactivity to air, this component in every case normally happens joined with different components and mixes. Finely powdered strontium metal will touch off immediately in air to deliver both strontium oxide and strontium nitride.

### **Barium (Ba)**

Barium is a gleaming white metal that can be found in the earth, where it exists normally. It happens joined with different synthetic concoctions, for example, sulfur, carbon or oxygen. It is light and its thickness is a large portion of that of iron. Barium oxidizes in air, responds vigorously with water to shape the hydroxide, freeing hydrogen. Barium responds with all the non-metals, framing frequently poisoning mixes. Barium is frequently utilized in barium-nickel amalgams for start plug terminals and in vacuum tubes as drying and oxygen-evacuating operator. It is additionally utilized in fluorescent lights: debased barium sulfide phosphoresces after presentation to the light.

### **Magnesium (Mg)**

Magnesium is synthetically dynamic, it replaces hydrogen in bubbling water and an extraordinary number of metals can be created by thermic decrease

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of its salts and oxidized structures with magnesium. It consolidates with most non-metals and pretty much every corrosive. Magnesium responds just marginally or not in the slightest degree with the greater part of the antacids and numerous natural substances, similar to hydrocarbons, aldehydes, alcohols, phenols, amines, esters and the majority of the oils. Utilized as an impetus, magnesium advances natural responses of buildup, decrease, expansion and dehalogenization. It was utilized for quite a while for orchestrating unique and complex natural segments by the notable Grignard response. The principle elements of the compounds are: aluminum, manganese, zircon, zinc, uncommon earth metals and thorium.

### **Thallium (Tl)**

When freshly exposed to air, thallium exhibits a metallic lustre, but soon develops a pale blue dim tinge, looking like lead in appearance. A substantial oxide develops on thallium whenever left in air, and within the sight of water the hydroxide is framed. The metal is delicate and moldable. It very well may be cut with a knife. Thallium is utilized for making low-softening point extraordinary glass for exceptionally intelligent focal points. Thallium salts are utilized as reagents in compound research. Thallium sulfate is as yet sold in creating nations where it is still allowed as a pesticide, albeit prohibited in Western nations

### **Tungsten (W)**

Tungsten is a radiant and gleaming white metal. The mass metal opposes assault by oxygen, acids and soluble bases. Tungsten has the most astounding liquefying purpose of any metal.

Tungsten is utilized in fibers in glowing lights, it is likewise utilized in electric contacts and bend welding terminals. Tungsten is utilized in composites, for example, steel, to which it gives incredible quality. Bond carbide is the most essential use for tungsten

### **Selenium (Se)**

Selenium is a non metallic substance component. In synthetic action and physical properties it looks like sulfur and tellurium. Selenium appears in various allotropic structures: the most mainstream are a red nebulous powder, a red crystalline material, and a dark crystalline metallike frame called metallic selenium. This last shape conducts power preferred in the light over in obscurity and is utilized in photocells. Selenium consumes in air and is unaffected by water, yet disintegrates in concentrated nitric corrosive and alkalis. Selenium has great photovoltaic and photoconductive properties, and it is utilized broadly in gadgets, for example, photocells, light meters and sun based cells. The second biggest utilization of selenium is in the glass business: selenium is utilized to expel shading from glass, to give a red shading to glasses and veneers.



### **Tellurium (Te)**

Tellurium is a semimetallic, glistening, crystalline, fragile, silver-white component. It is normally accessible as a dull dim powder, it has the properties both of the metals and the non metals. Tellurium frames numerous mixes comparing to those of sulfur and selenium. At the point when consumed in air tellurium has a greenish-blue fire and structures tellurium dioxide accordingly. Tellurium is uneffected by water or hydrochloric corrosive, yet breaks up in nitric corrosive. Tellurium is regularly utilized as an added substance to steel and usually alloyed to aluminum, copper, lead or tin. Tellurium is added to prompt enhance its sturdiness, strength and resistance to consumption. It tends to be utilized for cast press, earthenware production, impacting tops, sun oriented boards, chalcogenide glasses.

### **Molybdenum (Mo)**

The metal is brilliant white, hard change metal, yet is gentler and more pliable than tungsten. Scheele found it in 1778. It was frequently mistaken for graphite and lead mineral. It has a high flexible modulus, and just tungsten and tantalum, of the more promptly accessible metals, have higher dissolving focuses. Molybdenum has one of the most noteworthy dissolving purposes of every unadulterated component. Molybdenum is assaulted gradually by acids. Molybdenum is a significant alloying specialist, as it adds to the hardenability and strength of extinguished and tempered steels. It additionally enhances the quality of steel at high temperatures. Molybdenum is utilized in compounds, cathodes and impetuses.

### **Cerium (Ce)**

Cerium is a moldable, delicate, malleable, press dim metal, somewhat harder than lead. It is extremely responsive: it discolors promptly noticeable all around, it oxidizes gradually in cool water and quickly in high temp water. It breaks down in acids. It can consume when warmed or scratched with a knife. The metal is utilized as a center for the carbon terminals of circular segment lights, for radiant mantles for gas lighting. Cerium is utilized in aluminum and iron combinations, in tempered steel as a precipitation solidifying operator, to make perpetual magnets. Cerium oxide is a piece of the impetus of exhaust systems used to tidy up fumes vehicles

### **Thorium (Th)**

Whenever unadulterated, thorium is a shimmering white metal which is air-stable and holds its shine for a while. At the point when polluted with the oxide, thorium gradually discolors in air, getting to be dim lastly dark. Thorium oxide has a dissolving purpose of 3300°C, the most noteworthy everything being equal. Just a couple of components, for example, tungsten, and a couple of mixes, for example, tantalum carbide, have higher dissolving focuses. Thorium is gradually assaulted by water, yet does not break down

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promptly in most normal acids, with the exception of hydrochloric. Powdered thorium metal is regularly pyrophoric and ought to be painstakingly taken care of. At the point when warmed in air, thorium turnings touch off and copy splendidly with a white light. Thorium was found by Jöns Jacob Berzelius, a Swedish scientist, in 1828. He found it in an example of a mineral that was given to him by the Reverend Has Morten Thrane Esmark, who speculated that it contained an obscure substance. Esmark's mineral is currently known as thorite ( $\text{ThSiO}_4$ ). Thorium is named for Thor, the Scandinavian divine force of war. It is found in thorite and thorianite in New England (USA) and other sites. Thorium is a wellspring of atomic power. There is likely progressively undiscovered vitality accessible for use from thorium in the minerals of the world's outside layer than from joined uranium and petroleum product sources. A great part of the inside warmth the earth has been credited to thorium and uranium.

### **Titanium (Ti)**

Titanium chemical behaviour indicates numerous similitudes with that of silica and zirconium, as a component having a place with the main change gathering. Its science in watery arrangement, particularly in the lower oxidation states, has a few similitudes with that of chrome and vanadium. Titanium is a change metal light with a white-brilliant metallic shading. It is solid, glossy, consumption safe. Unadulterated titanium isn't solvent in water yet is dissolvable in concentrated acids. This metal structures an inactive however defensive oxide covering (prompting consumption opposition) when presented to raised temperatures in air yet at room temperatures it opposes discoloring. The principle oxidation state is 4+, in spite of the fact that the states 3+ and 2+ are additionally known, however are less steady. This component consumes noticeable all around when it's warmed up to acquire the dioxide,  $\text{TiO}_2$ , and when it is joined with incandescent lamp. It lessens the water vapor to frame the dioxide and hydrogen, and it responds comparatively with hot concentrated acids, in spite of the fact that it shapes trichloride with chlorhydric corrosive.

### **Zirconium (Zr)**

Zirconium is an extremely solid, pliant, malleable, brilliant silver-dim metal. Its substance and physical properties are like those of titanium. Zirconium is amazingly impervious to warmth and consumption. Zirconium is lighter than steel and its hardness is like copper. When it is finely partitioned, the metal can precipitously light in air, particularly at high temperatures. Zirconium powder is dark and is viewed as exceptionally unsafe fire danger. Zirconium does not break up in acids and soluble bases. Zirconium is utilized in amalgams, for example, zircaloy, which is utilized in atomic applications since it doesn't promptly ingest neutrons. Likewise utilized in exhaust systems, percussion

tops and heater blocks. Baddeleyite and polluted zirconium (zirconia) are utilized in lab cauldrons.

### **Vanadium (V)**

Vanadium is an uncommon, delicate, bendable dim white component found consolidated in specific minerals and utilized for the most part to deliver certain compounds. Vanadium opposes consumption because of a defensive film of oxide at first glance. Regular oxidation conditions of vanadium incorporate +2, +3, +4 and +5.

A large portion of the vanadium (about 80%) delivered is utilized as ferrovanadium or as a steel added substance. Blended with aluminum in titanium amalgams is utilized in fly motors and rapid air-casings, and steel composites are utilized in axles, crankshafts, gears and other basic segments. Vanadium compounds are likewise utilized in atomic reactors since vanadium has low neutron-adsorption capacities and it doesn't distort in crawling under high temperatures.

### **Uranium (U)**

Uranium is a hard, thick, pliant, bendable, silver-white, radioactive metal. Uranium metal has high thickness. At the point when finely separated, it can respond with chilly water. In air it is covered by uranium oxide, discoloring quickly. It is assaulted by steam and acids. Uranium can shape solids arrangements and intermetallic mixes with huge numbers of the metals.

Uranium picked up significance with the improvement of pragmatic employments of atomic vitality. Exhausted uranium is utilized as shielding to secure tanks, and furthermore in projectiles and rockets. The main nuclear bomb utilized in fighting was a uranium bomb. This bomb sufficiently contained of the uranium-235 isotope to begin a runaway chain response which in a small amount of a second made a substantial number of the uranium molecules experience parting, there by discharging a fireball of vitality.

### **Lithium (Li)**

Lithium is the first of the soluble bases in the intermittent table. In nature it's discovered like a blend of the isotopes  $Li6$  and  $Li7$ . It's the lightest strong metal, it's delicate, gleaming white, with a low dissolving point and receptive. Between the most noteworthy properties of lithium we locate its high explicit warmth (calorific limit), the immense temperature interim in the fluid state, high termic conductivity, low thickness and low thickness. Metallic lithium is dissolvable in short chain aliphatic amines, as etilamine. It's insoluble in hydrocarbons.

Lithium partakes in a colossal number of responses, with natural reactants and additionally with inorganic reactants. It responds with oxygen to shape monoxide and peroxide. It's the main basic metal that responds

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with nitrogen at surrounding temperature to create a dark nitride. It responds effectively with hydrogen at relatively 500°C (930°F) to shape lithium hydride. Metallic lithium's response with water is to a great degree vivacious. Lithium responds straightforwardly with the carbon to create the carburets. It ties effectively with incandescent lamp and structures halogens with light discharge.

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## APPENDIX-I

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### Reagents Used in Qualitative Analysis

#### Reagents -General

- I. HCl
- II.  $\text{NH}_3$
- III.  $(\text{NH}_4)_2\text{CO}_3$
- IV. NaOH
- V.  $\text{K}_2\text{CrO}_4$
- VI.  $\text{Na}_2\text{S}_2\text{O}_3$
- VII. KI
- VIII.  $\text{FeSO}_4$
- IX.  $\text{SnCl}_2$
- X. HOAc
- XI.  $\text{Na}_2\text{CO}_3$
- XII.  $\text{MgNO}_3$
- XIII.  $\text{AgNO}_3$
- XIV.  $\text{CuSO}_4$
- XV.  $\text{BaCl}_2$
- XVI.  $\text{Pb}(\text{NO}_3)_2$
- XVII. Ammonium Fluoride
- XVIII. Ammonium Polysulphide
- XIX. Ammonium Acetate
- XX. Ammonium Molybdate
- XXI. Potassium Ferrocyanide
- XXII. Mercuric Chloride

#### Reagents-Concentrated

- I. HCl
- II.  $\text{H}_2\text{SO}_4$
- III.  $\text{HNO}_3$
- IV.  $\text{NH}_3$
- V.  $\text{H}_2\text{O}_2$
- VI. HOAc

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### Reagents–Saturated

- I. Tartaric Acid
- II.  $\text{CaSO}_4$  Solution
- III.  $\text{Na}_2\text{HPO}_4$  Solution,
- IV. Oxine Reagent (Mg)

### Reagents –Solid

- I.  $\text{NH}_4\text{Cl}$
- II.  $\text{KNO}_2$
- III.  $\text{KClO}_3$
- IV.  $\text{PbO}_2$
- V. Sodium Sulphite
- VI. Iron Filings
- VII. Oxalic Acid
- VIII. Ammonium Nitrite
- IX. Ammonium Acetate
- X. Ammonium(or Potassium Thiocyanate
- XI. Sodium Bismuthate

If you want to go for Spot Test then Reagents are:

- I. Nessler's Reagent (for Ammonium)
- II. Potassium Nitrite (Hg)
- III.  $\text{K}_2\text{CrO}_4$  (Ag)
- IV.  $\text{SnCl}_2$  (W, Mo)
- V. Ammonium Sulphide (Pb)
- VI. KI (Ti)
- VII. Potassium Thiocyanate (Hg, Mo, Co)
- VIII. Thiourea (Bi, Se)
- IX. Tartaric Acid (Cu)
- X. Cadion-2B (Cd)
- XI.  $\text{AgNO}_3$  (As)
- XII. Hypophosphorus Acid (Te)
- XIII. Bismuth Nitrate (Sn)
- XIV. Rhodamine B (Sb)
- XV. Catechol (Ti)
- XVI. Alizarin-S (Zr, Al)

- XVII. Anthranilic Acid (Ce)
- XVIII. Potassium Ferrocyanide (Th, U)
- XIX.  $\text{CuSO}_4$  (V)
- XX. Diphenylcarbazide (Cr)
- XXI. Dimethylglyoxime (Ni)
- XXII. Sodium Bismuthate (Mn)
- XXIII. Mercuric-Ammonium Thiocyanate (Zn)
- XXIV. Sodium Rhodizonate (Ba, Sr)
- XXV. Picrolonic Acid (Ca)
- XXVI. Magneson Reagent (Mg)
- XXVII. Ferric Periodate Reagent (Li)

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## APPENDIX - II

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### NOTES

#### Preparation of Reagents

- I. **Acetic Acid (Dilute)** - Dilute 114 cm<sup>3</sup> of glacial acetic acid add in to 1 dm<sup>3</sup> with distilled water.
- II. **Ammonium Carbonate Solution** – Dissolve 96 g Ammonium Carbonate in distilled water and dilute to add into 1 dm<sup>3</sup> in water.
- III. **Ammonium Fluoride Solution** - Dissolve 1 g NH<sub>4</sub>F in 250 cm<sup>3</sup> of distilled water.
- IV. **Ammonium Molybdate Solution** – Dissolve 11 g crystalline amm. molybdate in a mixture of 15 cm<sup>3</sup> conc. NH<sub>3</sub> and 10 cm<sup>3</sup> water. Add 30 g amm. nitrate and after complete dissolution and dilute to 250 cm<sup>3</sup> with distilled water.
- V. **Ammonium Polysulphide Solution** – Dilute 140 cm<sup>3</sup> of conc. NH<sub>3</sub> solution with distilled water to 1 dm<sup>3</sup> and divide into two equal portions. Saturate one portion with H<sub>2</sub>S gas (use fume cupboard!). Then add the second portion and mix. Add 32 g sulphur and heat gently until dissolved to give a yellow solution.
- VI. **Ammonia Solution (Dilute)** - Dilute 140 cm<sup>3</sup> of concentrated NH<sub>3</sub> solution to 1 dm<sup>3</sup> with distilled water.
- VII. **Copper Sulphate Solution** - Dissolve 15 g CuSO<sub>4</sub>.5H<sub>2</sub>O in to 250 cm<sup>3</sup> of distilled water.
- VIII. **Ferrous Sulphate Solution** - Add 5 drops of conc. H<sub>2</sub>SO<sub>4</sub> in 250 cm<sup>3</sup> of distilled water and dissolve 35 g FeSO<sub>4</sub>.7H<sub>2</sub>O in it.
- IX. **Lead Nitrate Solution** - Dissolve 20 g PbNO<sub>3</sub> in to 250 cm<sup>3</sup> of distilled water.
- X. **Potassium Ferrocyanide Solution** - Dissolve 2.5 g K<sub>4</sub>Fe(CN)<sub>6</sub>.3H<sub>2</sub>O in 250 cm<sup>3</sup> of distilled water.
- XI. **Potassium Chromate Solution** - Dissolve 20 g K<sub>2</sub>CrO<sub>4</sub> in 1 dm<sup>3</sup> of distilled water
- XII. **Potassium Iodide Solution** - Dissolve 8 g KI in 250 cm<sup>3</sup> of distilled water.
- XIII. **Silver Nitrate Solution** - Dissolve 5 g AgNO<sub>3</sub> in 250 cm<sup>3</sup> of distilled water. (Dark bottle).
- XIV. **Sodium Carbonate Solution** – Dissolve 13 g anhydrous Na<sub>2</sub>CO<sub>3</sub> in 250 cm<sup>3</sup> of distilled water.
- XV. **Sodium Hydroxide Solution** – Dissolve 20 g NaOH pellets in 250 cm<sup>3</sup> of distilled water.



XVI. **Sodium Thiosulphate Solution** - Dissolve 6 g  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in 250  $\text{cm}^3$  of distilled water.

XVII. **Stannous Chloride Solution** – Add 25  $\text{cm}^3$  of conc. HCl to 25  $\text{cm}^3$  of distilled water, mix and cool. Dissolve 14 g  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  and dilute to 250  $\text{cm}^3$ . Place a granule of tin metal at the bottom of the bottle to prevent oxidation.

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**Spot Testing of Some Cations for the Above Respective Solutions**

**Spot Test for Hg**

Put a drop of faintly acid in respective test solution on put it on reaction paper add some ( $\text{KNO}_2$ ) if black spot appears it means Hg (I) is present.

Put a drop of the respective test solution on a spot plate. Add a drop of Ammonium Thiocyanate solution and a drop of cobalt acetate solution. If blue color appears it means Hg (II) is present.

**Spot Test for Pb**

Put a drop of the respective solution on a drop reaction paper and add a drop of Ammonium Sulphide solution. If black spot appears it means PbS is present.

**Spot Test for Cd**

Put a drop of Cadion- 2B reagent on reaction paper. Place a drop from the third part over it, followed by a drop of KOH solution. If pink spot surrounded or covered by blue ring appears it means Cd is present.

**Spot Test for Sb**

Put a drop of the respective solution on a spot plate and add some crystals of Sodium Nitrite ( $\text{NaNO}_2$ ). Then add a drop of Rhodamine-B reagent. If blue color appears it means Sb is present.

So maximum time we require reagents for spot testing. Reagents which can be used for spot testing (Cations given in lab manual) and their methods of preparation is given below:

- I. **Alizarin-S Reagent** – Mix 100 mg of Alizarin-S in 100  $\text{cm}^3$  pure water.
- II. **Ammonium or Potassium Thiocyanate Solution** – Mix 100 mg of the salt in 0.5  $\text{cm}^3$  of acetone.
- III. **Mercuric-Ammonium Thiocyanate** – Mix solid Ammonium Thiocyanate and Mercuric Chloride in 1  $\text{cm}^3$  water.
- IV. **Rhodamine B Reagent** - Mix 10 mg of Rhodamine B in 100  $\text{cm}^3$  distilled water.
- V. **Sodium Rhodizonate** - Mix solid Sodium Rhodizonate in 1  $\text{cm}^3$  water
- VI. **Tartaric Acid Solution** – Mix 15 g Tartaric Acid in 100  $\text{cm}^3$  distilled water.

- VII. **Thiourea Solution** - In 50 cm<sup>3</sup> pure water, pour 43 cm<sup>3</sup> concentrated HCl. Allow to cool and mix it in 5 g Thiourea. Dilute to 100 cm<sup>3</sup> with pure water.
- VIII. **Picrolonic Acid Reagent** - Mix well 200 mg of Picrolonic Acid with 1 cm<sup>3</sup> water and use the clear supernatant.
- IX. **Anthranilic Acid Reagent** – Mix 5 g of Anthranilic Acid in 100 cm<sup>3</sup> Ethanol.
- X. **Catechol Solution** - Mix 10 g of Catechol in 100 cm<sup>3</sup> distilled water.
- XI. **Hypophosphorus Acid** – Add 20 cm<sup>3</sup> of HPO<sub>2</sub> into 80 cm<sup>3</sup> of pure water and mix it well.

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## APPENDIX-IV

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### NOTES

#### Instructions and Safety Rules

1. Make certain to peruse all fire caution and wellbeing signs and adhere to the guidelines in case of a mishap or crisis.
2. Guarantee you are completely mindful of your facility's/building's clearing strategies.
3. Ensure you know where your lab's wellbeing hardware—including medical aid kit(s), fire doublers, eye wash stations, and security showers—is found and how to legitimately utilize it.
4. Open flares ought to never be utilized in the research facility except if you have consent from a qualified expert or lab in-charge.
5. Ensure you know about where your lab's ways out and fire alerts are found.
6. A zone of 36" distance across must be kept clear consistently around all fire sprinkler heads.
7. On the off chance that there is a fire penetrate, make sure to switch off all electrical gears and close all compartments.
8. Continuously work in legitimately ventilated zones.
9. Try not to bite gum, drink, or eat while working in the lab.
10. Lab dish sets ought to never be used as sustenance or refreshment holders.
11. Each time you use crystal, make sure to check it for chips and splits. Request/inform your lab in-charge of any harmed dish sets so it very well may be legitimately discarded.
12. Never use lab gear that you are not affirmed or prepared by your administrator to work.
13. In the event that an instrument or bit of gear falls flat amid use, or is not working appropriately, report the issue to an expert immediately. Never endeavor to fix a gear issue alone.
14. On the off chance that you are the last individual to leave the lab, try to bolt every one of the entry ways and check and fix or close all start sources.
15. Never leave a progressing test unattended.
16. Never lift any dish sets, arrangements, or different sorts of device above eye level.
17. Never smell or taste synthetics.
18. Do not pipette by mouth.
19. Ensure you generally pursue the best possible strategies for arranging lab squander.
20. Report all wounds, mishaps, and split hardware or glass immediately, regardless of whether the episode appears to be little or irrelevant.

21. In the event that you have been harmed, immediately call out and be active or boisterous till you get help.
22. In case of a compound sprinkling into your eye(s) or on your skin, instantly flush the influenced area(s) with running water for no less than 20 minutes.
23. In the event that you see any risky conditions in the lab, let your lab in-charge know at the earliest.
24. Continuously tie back hair that is jaw length or more.
25. Ensure that free attire or dangling gems is anchored, or abstain from wearing it in any case.
26. Never wear open-toed shoes in the lab. Footwear ought to cover the foot totally.
27. Never wear shorts or skirts in the lab.
28. When working with Bunsen burners, lit supports, matches, and so on, acrylic nails are not permitted.
29. Try not to enable any dissolvable to come into contact with your skin.
30. All synthetic substances ought to be unmistakably named with the name of the substance, its focus, the date it was received, its expiry and the name of the individual lab in-charge of it.
31. Before expelling any of the substances from a compound container, read the name twice.
32. Never take a greater number of synthetic substances from a jug than you requirement for your work.
33. Try not to return unused synthetics to their unique holder.
34. Synthetics or different materials ought to never be removed from the research center.
35. Synthetic concoctions ought to never be blended in sink channels.
36. Combustible and unpredictable synthetic substances should just be utilized in a smoke hood.
37. In the event that a synthetic spill happens, tidy it up immediately.
38. Guarantee that all concoction squander is discarded legitimately.
39. Never pour synthetic concoctions that have been utilized once again into the stock compartment.
40. Never tap flagons that are under vacuum.
41. Synthetic substances ought to never be blended, estimated, or warmed before your face.
42. Water ought not to be filled concentrated corrosive. Rather, empty corrosive gradually into water while mixing always. By and large, blending corrosive with water is exothermic.

## **NOTES**

**NOTES**

**Viva Question Answer**

- Q.1. Differentiate between equivalence & endpoint in titrations?
- Q.2. Why is front door of the balance closed during weighing?
- Q.3. What is the maximum weight that can be weighed in a chemical balance?
- Q.4. What is the weight of a rider?
- Q.5. What is meant by equivalent weight of a base?
- Q.6. What is the principle of volumetric analysis?
- Q.7. What is titration?
- Q.8. What is basicity of an acid?
- Q.9. What is end point
- Q.10. Why a titration flask should not be rinsed?
- Q.11. What are primary and secondary standard substances?
- Q.12. Burette and pipette must be rinsed with the solution with which they are filled, why?
- Q.13. It is customary to read lower meniscus in case of colorless and transparent solutions and upper meniscus in case of highly colored solutions, why?
- Q.14. What is a normal solution?
- Q.15. Why the last drop of solution must not be blown out of a pipette?
- Q.16. What is meant by the term 'Concordant Readings'?
- Q.17. What is acidimetric and alkalimeter?
- Q.18. What do you mean by 1.0 M solution?
- Q.19. Can one take oxalic acid solution in the burette and sodium hydroxide solution in the titration flask? What are the limitations of doing so if any?
- Q.20. Which indicator is used in the titration of sodium carbonate against hydrochloric acid and what is the color change at the end point?
- Q.21. What is the difference between an end point and an equivalence point?
- Q.22. Will you read upper or lower meniscus of  $\text{KMnO}_4$  solution in a burette?
- Q.23. What is the indicator used in the titration of sodium carbonate against hydrochloric acid?
- Q.24. What is basicity of an acid?

- Q.25. What is the relation between equivalent mass of acid and its molecular mass?
- Q.26. What is acidity of a base?
- Q.27. What is the relation between equivalent mass of a base and its molecular mass?
- Q.28. What is qualitative analysis?
- Q.29. What is a radical?
- Q.30. What are acidic and basic radicals?
- Q.31. What type of bond is present in an inorganic salt?
- Q.32. Why do inorganic salts ionize when dissolved in water?
- Q.33. Give examples of some coloured basic radicals.
- Q.34. What is the colour of iron salts?
- Q.35. Name any iron salt which is light green.
- Q.36. What is the color of nickel salts?
- Q.37. What is the color of manganese salts?
- Q.38. Name the basic radicals which are absent, if the given salt is white.

Questions with Answers

- Q.39. Why 'Soda Extract' is prepared in the identification of acidic radicals.  
Ans. To avoid the interference of basic radicals, soda extract is prepared for identification of acidic radicals.
- Q.40. Which burner is commonly used in laboratory?  
Ans. Bunsen Burner is commonly used in laboratory.
- Q.41. What is lime water?  
Ans. It is a solution obtained by dissolving lime in water and then filtering the solution.
- Q.42. Which gas turns lime water milky?  
Ans.  $\text{CO}_2$  turns lime water milky.
- Q.43. What is the compound formed when  $\text{CO}_2$  or  $\text{SO}_2$  is passed in lime water and the solution becomes milky?  
Ans.  $\text{CaCO}_3$  (Calcium Carbonate)

Or

$\text{CaSO}_3$  (Calcium Sulphite)

- Q.44. Why the milky colour disappears on passing excess of  $\text{CO}_2$  gas in lime water?  
Ans. Due to formation of soluble colourless calcium bicarbonate.



**NOTES**

## NOTES

Q.45. Name some interfering radicals?

Ans. Oxalate ( $C_2O_4^{--}$ ), Borate ( $BO_3^{---}$ ),  $F^-$  (Fluoride), Phosphate ( $PO_4^{---}$ )  
These are called interfering radicals because they interfere in the systematic analysis of basic radicals after second group.

Q.46. How can you identify aliphatic and aromatic compounds?

Ans. Aliphatic compound burns with non-smoky flame while aromatic compound burns with black and smoky flame.

Q.47. Why aromatic compounds burn with black and smoky flame?

Ans. Because percentage of carbon is more in aromatic compounds.

Q.48. How can you test nitrogen in sodium extract?

Ans. Mix Sodium extract + Small amount of freshly prepared Ferrous Sulphate solution. Then dull green precipitate of Ferrous hydroxide is obtained. Boil it and add dilute  $H_2SO_4$ . Then Prussian blue or green colour confirms the presence of Nitrogen. Prussian blue colour is due to formation of Ferro Ferricyanide  $Fe_4[Fe(CN)_6]_3$ .

Q.49. How can you test sulphur in sodium extract?

Ans. Sodium extract + Sodium Nitroprusside gives violet or purple colour due to formation of Sodium Thio Nitroprusside.

Q.50. What is the formula of Sodium Nitroprusside and Sodium Thio Nitroprusside?

Ans.  $Na_2[Fe(CN)_5NO]$  Sodium Nitroprusside  
 $Na_4[Fe(CN)_5NOS]$ . Sodium Thionitroprusside

Q.51. What is the test of Ketonic group?

Ans. Take an organic compound and add freshly prepared Sodium Nitroprusside solution and excess of NaOH solution. Then a wine red colour indicates the presence of Ketonic group.

Q.52. How can you test Ketonic group with Iodoform test?

Ans. Take some organic compound, add NaOH solution and excess of Iodine solution, heat and cool the solution, a yellow precipitate of Iodoform confirms the presence of Ketonic group.

Q.53. How can you test Carbohydrate?

Ans. Take aqueous solution of compound, add Molisch's reagent ( $\alpha$ -Naphthol dissolved in Ethanol) and shake the mixture, Add 1-2ml of concentrated  $H_2SO_4$  with the side of test tube. Then a red-violet ring is formed at the junction of the two liquids which confirms the presence of Carbohydrate.