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2-	To determine the hardness of the given sample by titrimetric method.	3-4	7.9.19	4.12.19	
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4-	To determine the chlorides of the given Sample by titrimetric method.	8-9	29.9.19	4.12.19	
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Date

Expt. No. 1 Page No. 1

Object :→

To determine the total Alkalinity of the given Sample by titrimetric method.

Requirements →

① Grassware & appratus :-

(i) conical flask
(ii) Beaker
(iii) Burette
(iv) Pipette
(v) Burette stand.

② chemical :-

0.02(N) H_2SO_4, Phenolphthalein, indicator methyl orange indicator.

Theory :-

Alkalinity is a physical parameter. It is due to presence carbonate, bicarbonate, OH^- ion in water bodies. It can be measured by titrimetric method. It is sum of Phenolphthalein alkalinity & methyl orange alkalinity.

Procedure →

(i) To the 50 ml sample 2 drop Phenolphthalein is added pink colouration. phenolphthlein alkalinity is present.

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Calculation

Table-1

S.N	Initial Burette reading	Final burette Reading	Average
1	50	1.2	
2	50	1.2	1.2
3	50	1.2	

Phenolphthalein alkalinity in mg/l of $CaCO_3$ =

Methyl orange alkalinity →

S.N	Vol. of Sample	Vol. of titrator	Mean
1	50	4.3	
2	50	4.4	4.3
3	50	4.4	

Where

A = Vol. of NaOH used with phenolphthalein indicator

B = Vol of NaOH required with methyl orange indicator

N = Normality of H_2SO_4

V = Vol. of sample

Date

Expt. No. 1 Page No. 2

(11) The sample is titrated with std. 0.02(N) H_2SO_4 using methyl orange indicator, end point changed yellow to orange.

Result →

Total alkalinity of the given sample is

Phenolphthalin alkalinity = 29 mg/lit

total alkalinity = 12 mg/lit

∴ CO_3^{-2} ion conc. in terms of equivalent /lit

$= \frac{12 \times 2}{60} = 0.8$ M eq/lit

alkalinity due to HCO_3^- = (M − 2P)

HCO_3^- ion conc in terms of M equt/lit

$= 200.08 \times \frac{1}{16}$

$= 3.28$ Mequ/lit

CO_3^{2-} & HCO_3 alkalinity in this sample is 0.8 M. equ/lit & 3.28 M equit/lit

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calculation

Phenopthaline alkalinity

$N_1V_1 = N_2V_2$

$= \frac{1}{50} \times 1.2 = N_2 \times 50$

$\Rightarrow N_2 = 1.2 \times \frac{1}{50}$

$= 0.00048$

strength of ca_2co_3

equivalit $= 0.00048 \times$ eq. of $caco_3$

$= 0.00048 \times 50$ gm/lit

$= 0.024 \times 1000$ mg/lit

$= 24$

$HCO_3^{\ominus}$ ionic conc.

$= 242 - (2 \times 24) \times \frac{1}{50} \times$ mg/lit

$= 200.08$ Mg/li

Total alkalinity

$N_1V_1 = N_2V_2$

$\Rightarrow \frac{1}{50} \times 4.3 = N_2V_2$

$\Rightarrow N_2 = 4.3 \times \frac{1}{50 \times 50}$

$= 0.0017 2$

strength of $caco_3$

$= 0.00172 \times 50$

$= 0.086 \times 1000$ mg/lit.

$= 86$ mg/lit

co_3^- ion in conc.

$= 20 \times \frac{30}{50}$ mg/lit

$= 12$ mg/lit

Date

Expt. No. 2 Page No. 3

Object →

To determine the hardness of the given sample by tetrimetric method.

Requirements →

(i) E.B.T indicator
(ii) 0.01 (M) EDTA
(iii) Standard $caco_3$ sol^n.
(iv) Buffer sol^n (7gm NH_4cl + 60ml conc NH_3)

Theory :-

The hardness of water can be determined by complexometric method. EDTA is used as complexometric agent. The ca^{2+} & mg^{2+} present in water is treated with EDTA sol^n using EBT indicator. EBT is a blue colour dye from a unstable complex with ca & mg ion in the hard water at pH of 9.10. The complex is wine red in colour.

$ca^{2+}/mg^{2+}/EBT \longrightarrow ca^{2+}/mg^{2+} - EBT$ (unstable complex)

As the sol^n is treated against EDTA. The free ca^{2+} & mg in water, from a stable metal ion EDTA complex

$ca^{2+}/mg^{2+} + EDTA \longrightarrow ca^{2+}/mg^{2+} - EBT$ (stable colour complex)

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Calculation

S.N	Initial Burette Reading	Final Burette reading	Average Volume
1			
2			
3			

Hardness mg/L of $CaCO_3$ = $\frac{\text{ml of 0.01m EDTA used} \times 1000}{\text{ml of sample}}$

Date

Expt. No. 2 Page No. 4

Once, the free metal ion are complexed the EDTA replaces ca & mg ion from the unstable indicator complex also in from stable complex with this result the indicator is blue in colour of the above. The end point will be blue in colour —

Procedure →

It is measured by titrimetric method 25ml of the titrate is taken in the conical flask then 1-2 ml ammonia buffer solⁿ is added to maintain the pH of the sample 10.0+1 & titrate with standard EDTA solⁿ by adding 2 drop E.B.T indicator. At the end point wine red colour is changed to Blue colour.

Result →

Total hardness of the given sample is

Teacher's Signature :

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Object →

To determine the ca & mg in the given sample by titrimetric method.

Requirement →

(I) E.B.T indicator
(II) 0.01 (M) EDTA
(III) Standard $CaCO_3$ solⁿ.
(IV) Buffer solⁿ (7 mg NH_4Cl . 60 mL conical NH_3)
(V) 1(N) NaOH
(VI) Murexide indicator.

Theory →

The hardness of water can be determined by complexometric method. EDTA is used as complexometric against. The Ca^{2+} & Mg^{2+} present in water is titrated with EDTA solⁿ using EBT indicator. EBT is blue colour dye from a unstable complex with ca & mg ion in the hard water at pH of 9-10. The complex is wine red in colour.

$Ca^{2+}/Mg^{2+} + EBT \longrightarrow Ca^{2+}/Mg^{2+} - EBT$ (unstable Complex)

As the solⁿ is titrated against EDTA. The free Ca^{2+} & Mg^{2+} in water, from a stable metal ion EDTA complex.

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calculation –

Table - 1 - Determination of total hardness

SN	Initial Burette reading	Final burette reading	Average Volume
1	0.0	1.6	
2	1.6	3.1	1.6
3	3.1	4.7	

Hardness, mg/L of $CaCO_3$ = $\frac{4.7 \times 1000}{25}$ = 188 mg/lt.

Table-2 - Determination of calcium hardness -

SN	Initial burette reading	Final burette reading	Average volume
1	0	1.2	
2	1.2	2.5	1.2
3	2.5	3.7	

calcium hardness of mg/L of $CaCO_3$ = $\frac{1.2 \text{ mol of titrant} \times 1000 \times 1.05}{\text{ml of sample}}$

calcium present in Sample (mg/L) = 50.4 × 0.4

magnesium hardness mg/L of $CaCO_3$ = 188 − 50.4 = 137.6

magnesium present sample (mg/L) = 33.57 × 0.244

Date

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Ca^{2+}/Mg^{2+} + EDTA → Ca^{2+}/Mg^{2+} – EBT (Stable colour complex)

Once, the free metal ion are complexed the EDTA replaces Ca^{2+} Mg^{2+} ion from the unstable indicator complex also in from stable complex with this result, the indicator is blue in colour of the above. The end point will be blue in colour.

Procedure →

It is measured by titrimetric method 25ml of the titrate is taken in the conical flask then 1-2 ml ammonia buffer solⁿ is added to maintain the pH of the sample 10.0 ± 1 & titrate with standard EDTA solⁿ by adding 2 drop E.B.T indicator. at the end point wine red colour is changed to blue colour.

Calcium is determine by the EDTA complexometric titration method using murexide indicator through the estimation of calcium hardness. 25ml sample is taken in a conical flask, 1 ml 1N NaOH is added the solⁿ is titrated against 0.01 (M) EDTA using a pinch of murexide indicator. At the end point pink colour is changed to purple.

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The amount of magnesium in the sample is the calculated on the basis of the two titre value:

Result →

The amount of calcium & magnesium in the given sample is 137.6 ppm & 33.57 ppm respectively.

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Object →

To determine the chlorides of the given sample by titrimetric method.

Requirements →

(i) N/100 $AgNO_3$ solution.
(ii) K_2CrO_4 Indicator
(iii) conc. H_2SO_4
(iv) NaOH

Theory →

$AgNO_3$ can form ppt quantitatively with halides & their thiocyanate from their solⁿ and these precipitation reaction forms the basis of volumetric estimation of these ions.

$$AgNO_3 + X^- = AgX + NO_3^- \quad \text{where } X = Cl^-, Br^-, I^-$$

On gradual addition of silver nitrate to a std ~~on~~ ~~gradual~~ solⁿ of Nacl, Agcl precipitation. After completion of Cl^- as Agcl excess of Ag^+ ion added combined with CrO_4^- ion present as indicator to give light brown colour ppt. of silver chromate. For higher conc of Cl^- of the initial stage Agcl ppt will occur first.

$$AgNO_3 + NaCl = AgCl + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 = Ag_2CrO_4 + 2KNO_3$$

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Calculation →

Sl No	Initial burette reading	Final burette reading	Average volume ml
1	50	1.7	
2	50	1.8	1.8
3	50	1.8	

chloride in mg/L = $\frac{ml \times N) of\ AgNO_3 \times 100 \times 35.45}{V}$

$= \frac{1.8 \times 0.02 \times 1000 \times 35.45}{50} = 25.524\ mg/L$

Where N - Normality of $AgNO_3$

V = Vol of water sample in cc

Date

Expt. No. 4 Page No. 9

Procedure →

chloride is measured by the mohr's titrimetric method. 50 ml of water sample is taken in a conical flask as titrant than 1 ml of $K_2Cr_2O_7$ is added as indicator. pH adjusted betn 7-10 by adding H_2SO_4 or NaOH. Then it is titrated with 0.02N $AgNO_3$ solution until the persistent of reddish brown colour.

Result →

The amount of chloride in the given sample is 25.524 ppm.

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Object →

To determine the dissolve oxygen (DO) in the given sample by titrimetric method.

Requirements →

(I) $MnSO_4$ Solution.
(II) Alkli Iodide - Azide Solution
(III) conc. H_2SO_4
(IV) 0.025 (N) $Na_2S_2O_3$ solution
(V) Starch indicator
(VI) Formaldehyde.

Theory →

Dissolved oxygen is determined by azide modification winkler's method. The dissolved oxygen in water oxidises KI & equ. amount of Iodine is liberated. This liberated iodine is titrated against standard thiosulphate soln. however, since the dissolve oxygen in water in molecular state is carries such as manganese hydroxide is used.

The method involves introducing a conc. sol of $MnSO_4$, NaOH & KI azide reagent into the water sample. The white ppt of $Mn(OH)_2$ is oxidized by oxygen in the water sample to give a brown ppt of basic MnO. This is then titrated against standard thiosulphate soln using starch indicator.

Teacher's Signature :

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Calculation —

S.N	Initial burette reading	Final burette reading	Average volume (ml)
1	0	7.1	
2	7.1	14.2	7.1
3	14.2	21.3	

$$D.O \text{ in } mL/L = \frac{(A \times N) \text{ of the titrant} \times 1000 \times 50}{V_1 - V}$$

$$= \frac{7.1 \times 0.025 \times 1000 \times 8}{200 - 4}$$

$$= 7.2 \text{ mg/lit.}$$

where A = mL of titrant used

N = Normality of titrant

V_1 = vol of sample

V = vol. of mnso4 + alkali-iodide azide

Date

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Procedure:—

500ml of sample water is collected in BOD bottle, 2ml of each of manganese sulphate and alkaliodide azide reagent were added. Bottle is shaken for about 15 min & then allowed the ppt to settle down. Then 2ml conc. H_2SO_4 is added to dissolve the precipitate. 100ml of this solⁿ is titrated against 0.025N $Na_2S_2O_3$ solⁿ in present of starch indicator till blue colour appears.

Result:

The amount of D.O is the given sample is 7.2 mg/lit.

Teacher's Signature :

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S. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
1	Preparation of azo dye by using Aniline & vaniline compound & its spectophotometric determination				
2.	Preparation of azo dye compound by using Aniline & 1 napthol & its spectophotometric determination.				
3.	Preparation of azo dye compound by using Aniline and resorcinol and its spectrophotometric determination.				
4.	Preparation of azo dye compound by using Ampyron and vaniline and its spectrophotometric determination.				
5.	Preparation of Azo dye compound by using Ampyron and salicylic acid and its spectrophotometric determination.				
6.	To determine Biological dissolve oxygen in the given Sample by titrimetric method.				

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S. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
7.	to determine the chemical oxygen in the given sample by titrimetric method.				
8.	To determine the iron from the plant matics by spectrophotometric method.				

Date

Expt. No. 1 Page No.

Object:-

Preparation of azo dye by using Aniline and Vaniline compound and its spectrophotometer determination.

Requirement :-

Chemical Reagent:-

- Aromatic amine (Aniline)
- phenolic compound (Vaniline)
- NaOH
- conc. HCl
- $NaNO_2$
- Distilled water

Apparatus:-

→ Beaker.
→ Test tube
→ Measuring cylinder
→ Glass rod
→ dropper
→ china dish

Theory :-

Aromatic amine (Aniline) react in presence of $NaNO_2$ and HCl in 0-5° temperature to form diazonium salt.

And the same diazonium salt react with any phenolic compound (Vaniline) to form the coloure compound i.e azo dye compound.

Teacher's Signature

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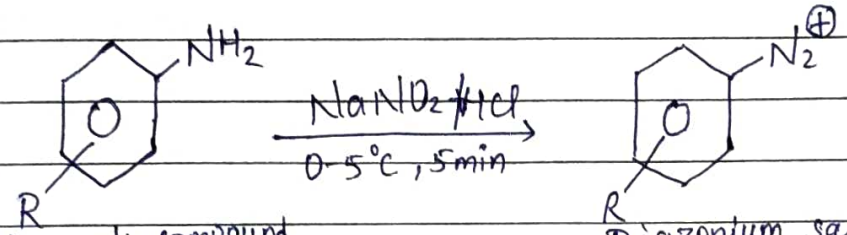
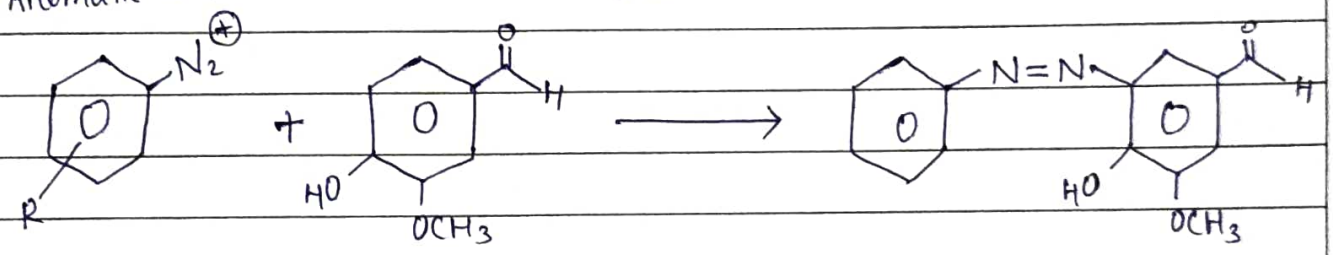
Expt. No. 1 Page No.

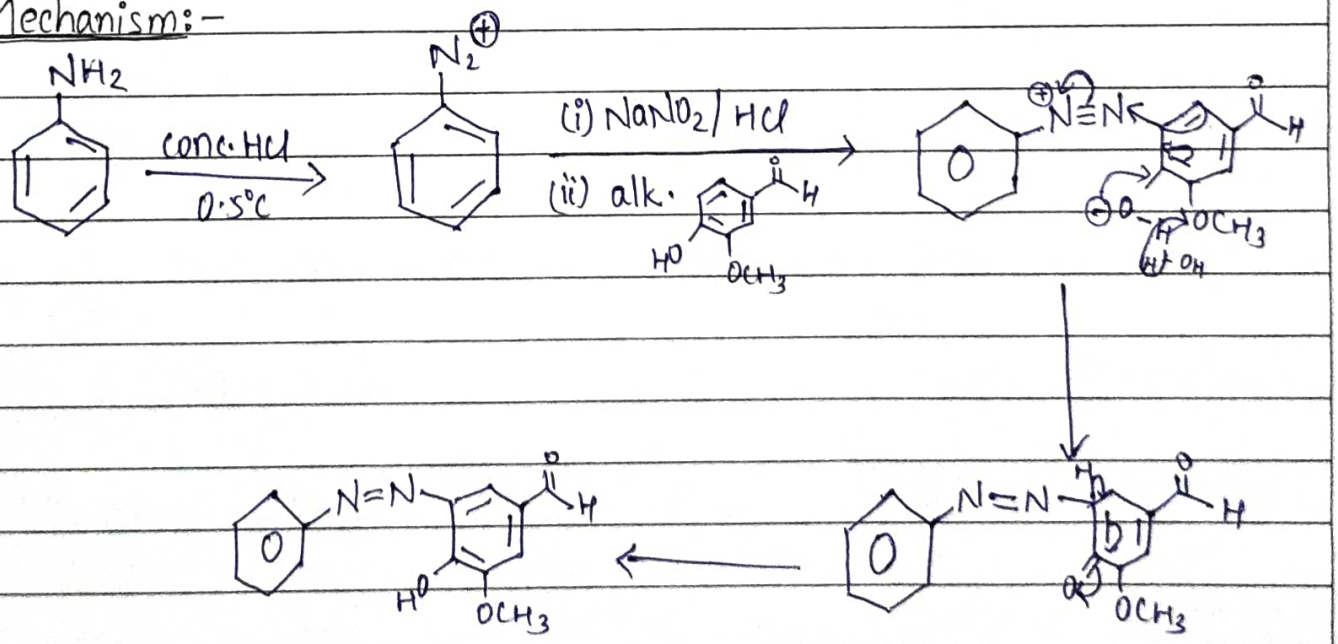


Aromatic compound Diazonium salt



Phenolic compound
(Vanilin)

Mechanism:-



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Procedure:-

(i) Taking 0.5 mg or 1g aromatic amine (Aniline) in a 50ml beaker, 5ml conc. HCl and the mixture kept in ice bath and stirred. Then 20% (10ml) $NaNO_2$ sol^n added to it and shaken.

(ii) Alkaline phenolic compound (vaniline) (1g or 0.5 mg) (phenolic compound + NaOH) was mixed in the beaker and stirred and kept the the. temp 0-5°C and colour dye observed.

(iii) The dye is evapourated to dryness in a water bath in a china dish untill crystalline shape was appeared.

(iv) Cool the china dish and weight was taken and the yield of the compound , colour melting point.

(v) prepare differente conc^n of dye in milimole i.e 10nm 5nm, 1m, 0.1nm, 0.01nm and the major the O.D (optical density) & setting the spectrophotometer corresponding to colour of sol^n.

Conclusion:-

Azo dye compound gives the colour & having its yield & melting point is

Teacher's Signature

Date

Expt. No. 2 Page No.

Object:-

Preparation of azo dye by using Aniline and 1-napthol compound & its spectrophotometric determination.

Requirement:-

Chemical Reagent:-

- Aromatic amine (Aniline)
- phenolic compound (1-napthol)
- NaOH
- conc HCl
- $NaNO_2$
- Distilled water

Apparatus:-

→ Beaker
→ Test tube
→ Measuring cylinder
→ Glass rod
→ dropper
→ China dish

Theory:-

Aromatic amine (Aniline) react in presence of $NaNO_2$ and HCl in 0-5°c temperature to form diazonium salt.

And the same diazonium salt react with any phenolic compound (~~vaniline~~) to form the colour compound i.e azo dye compound.

Teacher's Signature

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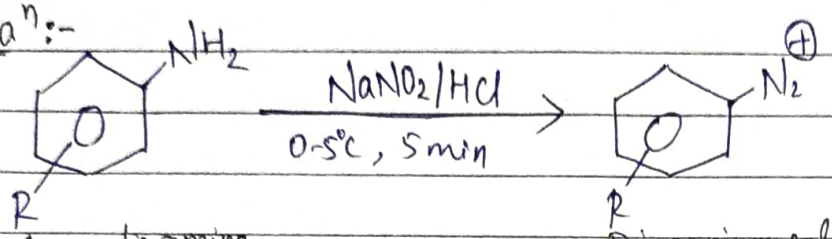
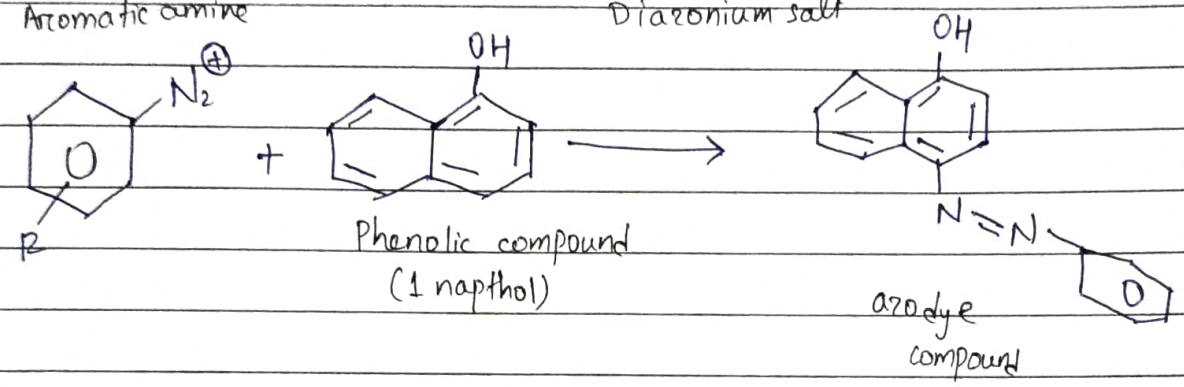
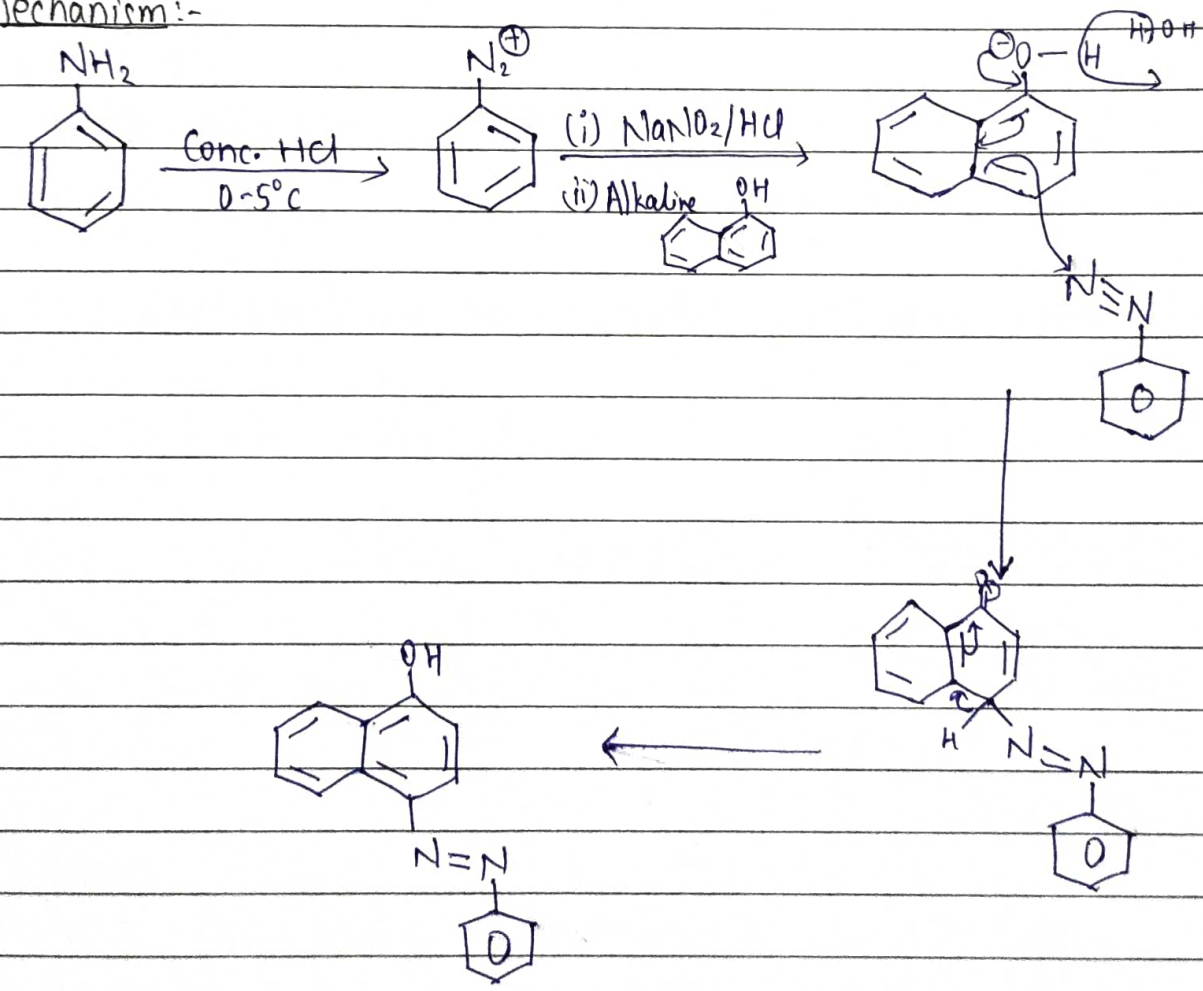
Reaⁿ:-





Mechanism:-



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Procedure:-

(i) Taking 0.5mg or 1g Aromatic amine (Aniline) in a 50ml beaker, 5ml conc. HCl was added & the mixture kept in ice bath & stirred.
Then 20% $NaNO_2$ (10ml) sol^n added to it & shaken.

(ii) Alkaline phenolic compound (1-napthol) [(0.5mg or 1g) phenolic compound + NaOH] was mixed in the beaker and Stirred kept the temp 0.5°c and orange colour dye is observed.

(iii) The dye is evapourated to dryness in a water bath in a china dish untill the crystalline shape was appeared.

(iv) Cool the china dish and weight was taken and the yield of the compound. , colour melting point.

(v) Prepare the different concentration of dye in millimole i.e 10nm, 1nm, 0.1nm, 0.01nm and major optical density and setting the sepectrophotometric corresponding to the colour of the sol^n.

Conclusion:-

1-napthol aniline dye compound having colour orange and yield is and melting point. and spectrophotometric value is

Teacher's Signature

Date

Expt. No. 3 Page No.

Object:-

Preparation of azo dye by using Aniline and resorcinol compound and its spectrophotometric determination.

Requirement:-

Chemical Reagent:-

- Aromatic amine (Aniline)
- phenolic compound (resorcinol)
- NaOH
- conc HCl
- $NaNO_2$
- Distilled water

Apparatus:-

→ Beaker
→ Test tube
→ Measuring cylinder
→ Glass rod
→ dropper
→ China dish

Theory:-

Aromatic amine (Aniline) react in presence of $NaNO_2$ and HCl in 0-5°c temperature to form diazonium salt.

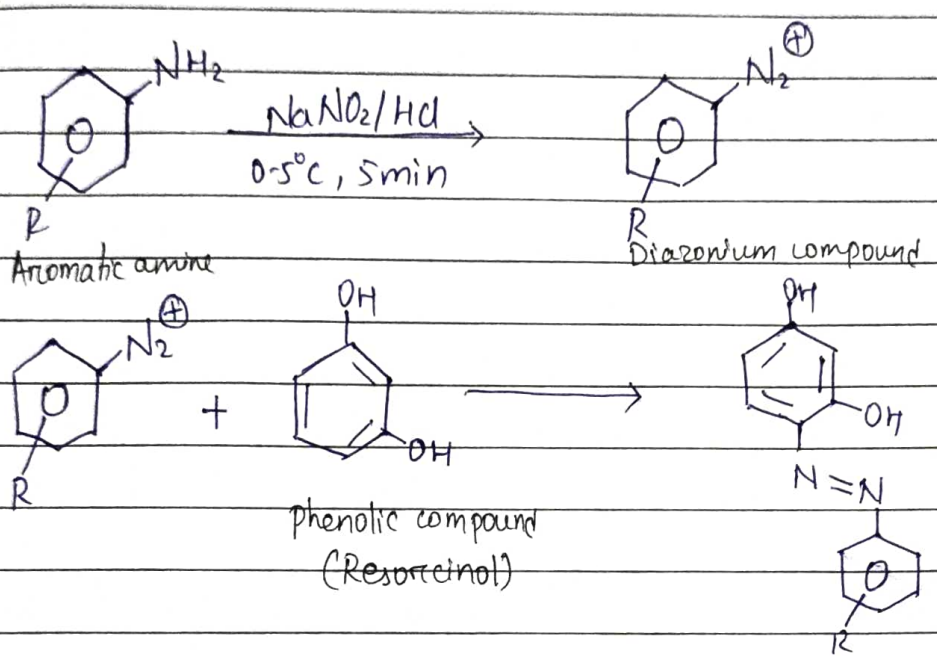
And the same diazonium salt react with any phenolic compound (resorcinol) to form the colour compound i.e azo dye compound.

Teacher's Signature

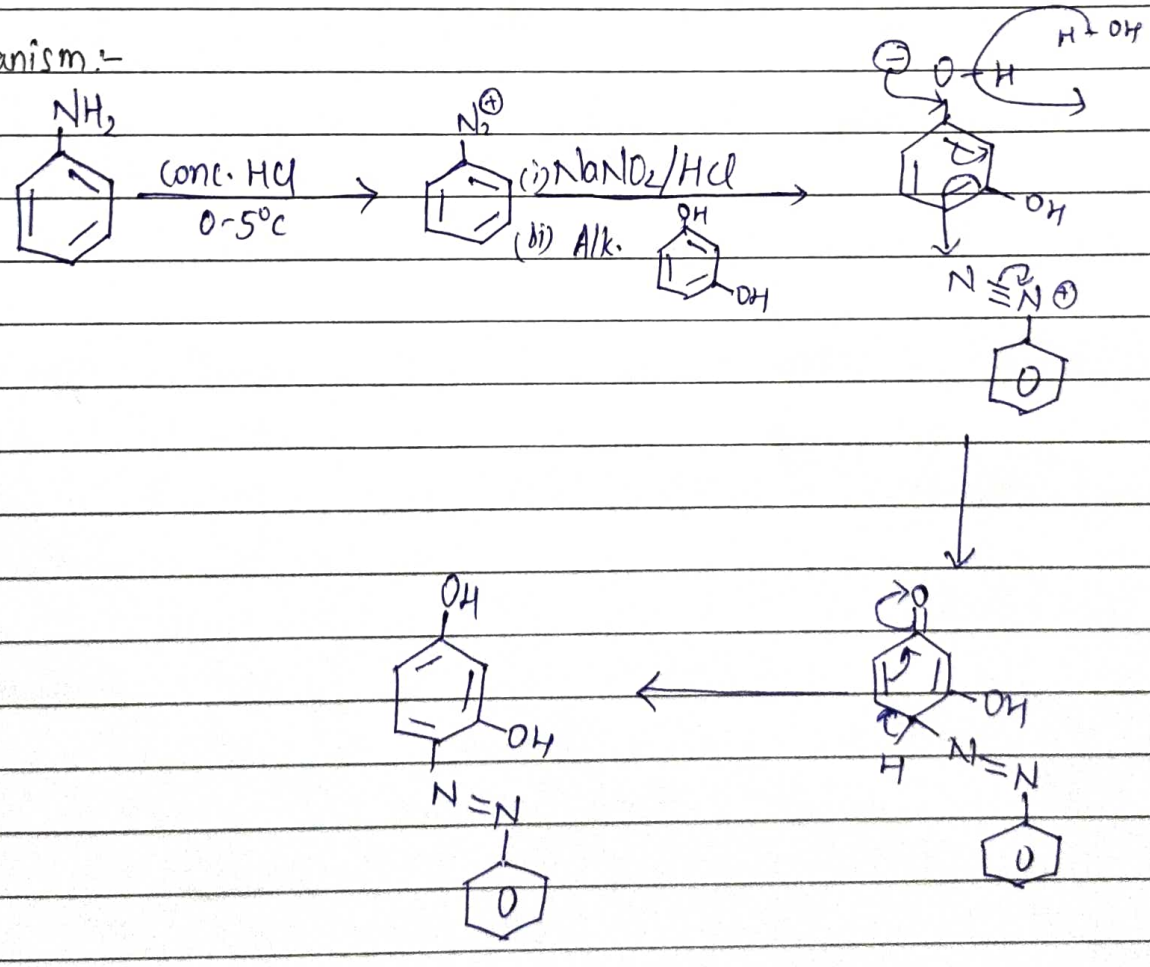
Date

Expt. No. Page No.

Rea^n:-



Mechanism:-



Teacher's Signature

Date

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Procedure :-

(i) Taking 0.5mg or 1g Aromatic amine (Aniline) in a 50ml beaker. 5ml conc. HCl added & the mixture kept & mixture is kept in ice bath & stirred.
Then 20% $NaNO_2$ (10ml) solution added to it and shaken.

(ii) Alkaline phenolic compound (Resorcinol) [2g or 2ml phenolic compound + NaOH] was mixed and stirred kept the temp 0-5°c and colour dye observed.

(iii) The dye is evapourated to dryness in a water bath in a china dish untill the crystalline shape was appeared.

(iv) Cool the china dish and weight was taken and the yield of compound , colour and melting point.

(v) Prepare different concⁿ of dye in millimole i.e 10nm 5nm, 1nm, 0.1nm, 0.01nm and the major the optical density & setting the spectrophotometric corresponding to colour of solution

Conclusion:-

Azo dye compound having colour and yield is and melting point

Teacher's Signature

Date

Expt. No. 4

Page No.

Object:-

Preparation of azo dye by using Ampyron and vaniline compound and its spectrophotometric determination.

Requirement:-

Chemical Reagent:-

- Aromatic amine
- Phenolic compound
- NaOH
- conc. HCl
- $NaNO_2$
- Distilled water

Apparatus:-

- Beaker
- Test tube
- Measuring cylinder
- Glass rod
- dropper
- Chia dish

Theory:-

Aromatic amine (Ampyron) react in presence of $NaNO_2$ and HCl in 0-5°c temp for 5 min to form diazonium salt.

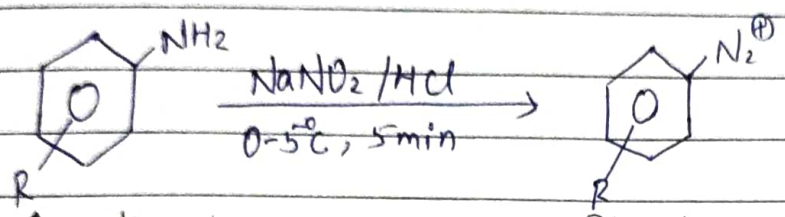
And then that diazonium salt is react with phenolic compound (vaniline) to form the colour compound i.e azo dye compound.

Teacher's Signature

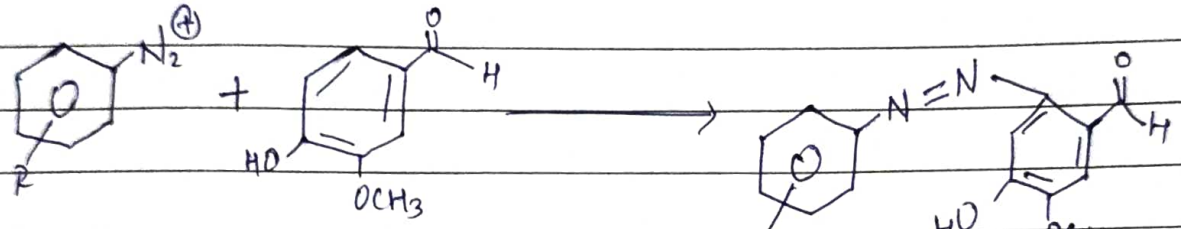
Date

Expt. No. Page No.

Reaⁿ:-

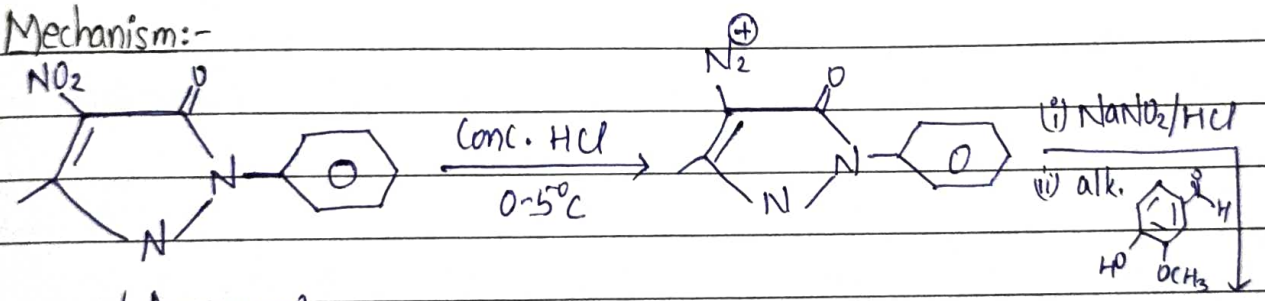


Aromatic amine Diazonium salt

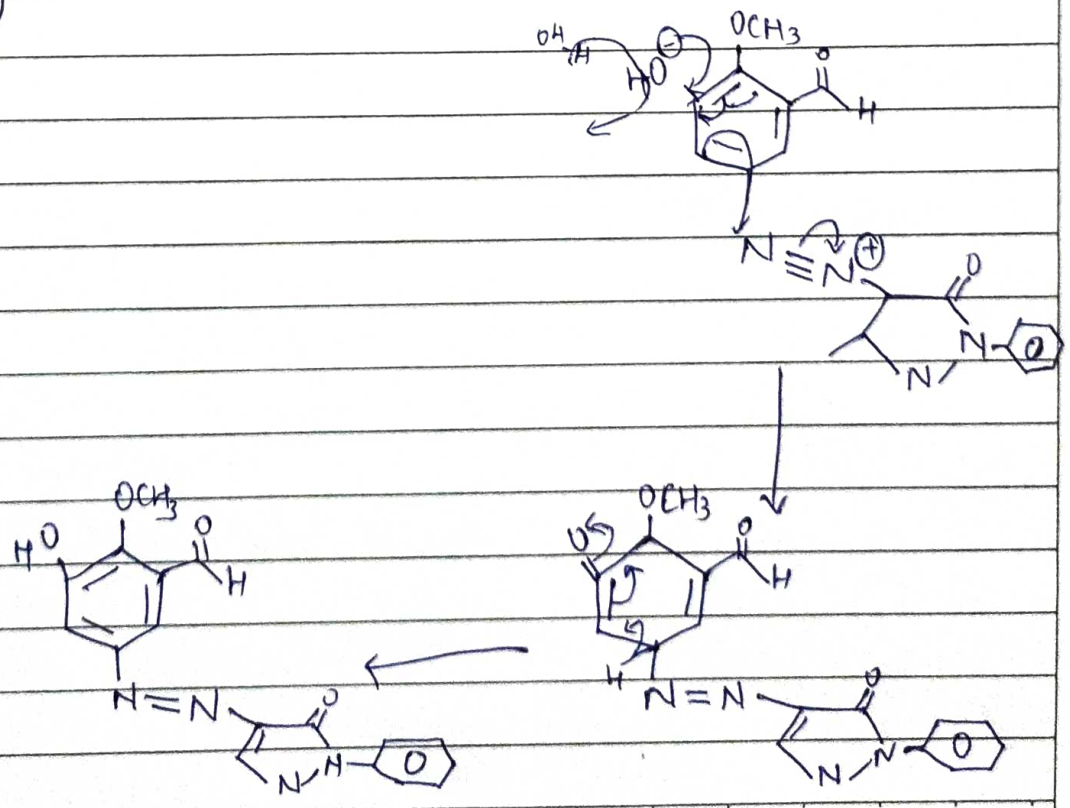


Phenolic compound
Vanitine

Mechanism:-



(Ampyron)



Teacher's Signature

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Procedure:-

(i) Taking 0.5mg or 1g Aromatic amine (Ampyrion) in a 50ml beaker, 5ml conc. HCl added & the mixture kept in ice bath & stirred.
Then 20% $NaNO_2$ (10m) sol^n added to it and shaken.

(ii) Alkaline phenolic compound (vanilin) [0.5mg or 1m) phenolic compound + NaOH] was mixed in the beaker and stirred kept the temp 0-5°c & colour dye observed.

(iii) The dye is evapourated to dryness in a water bath in a china dish untill the crystalline shape was appeared.

(iv) Cool the china dish and weight was taken and the yield of the compound , colour and melting point.

(v) prepare different concentration of dye. in millimole i.e 10nm, 5nm, 1mm, 0.1mm, 0.01mm and the major the optical density & setting the spectrophotometric corresponding to colour of solution.

Conclusion:-

Azo dye compound having colour yield & melting point.

Teacher's Signature

Date

Expt. No. 5 Page No.

Object:-

Preparation of azo dye by using Ampyron and salicylic acid compound and its spectrophotometric determination.

Requirement:-

Chemical Reagent:-

- Aromatic amine (Ampyron)
- Phenolic compound (Salicylic acid)
- NaOH
- conc. HCl
- $NaNO_2$
- Distilled water

Apparatus:-

→ Beaker
→ Test tube
→ Measuring cylinder
→ Glass rod
→ dropper
→ China dish

Theory:-

Aromatic amine (Ampyron) react in presence of $NaNO_2$ and HCl in 0-5°c temp for 5min to form diazonium salt And when diazonium salt is react with phenolic compound (salicylic acid) to form the colour compound i.e azo dye compound.

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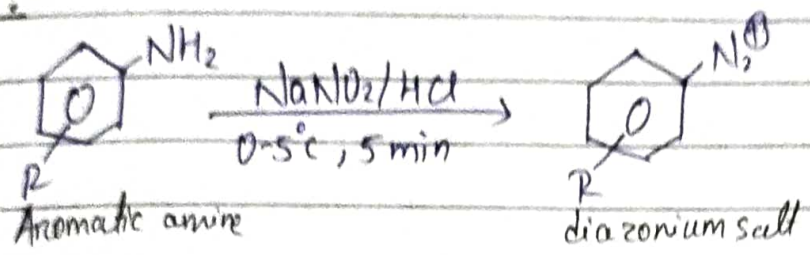
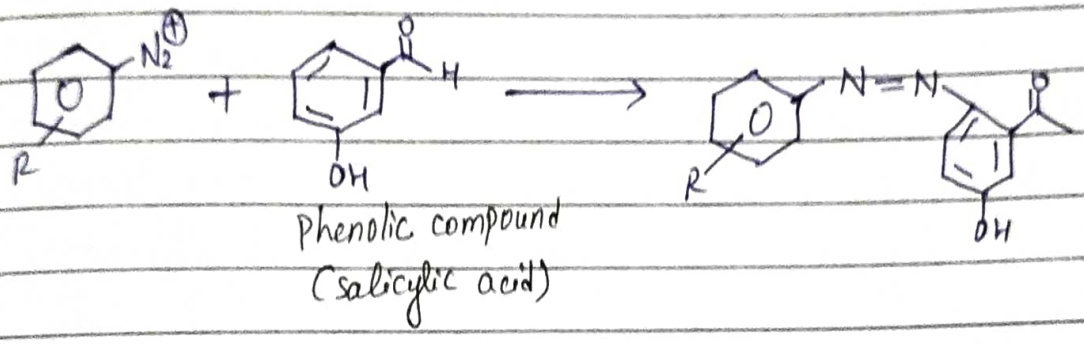
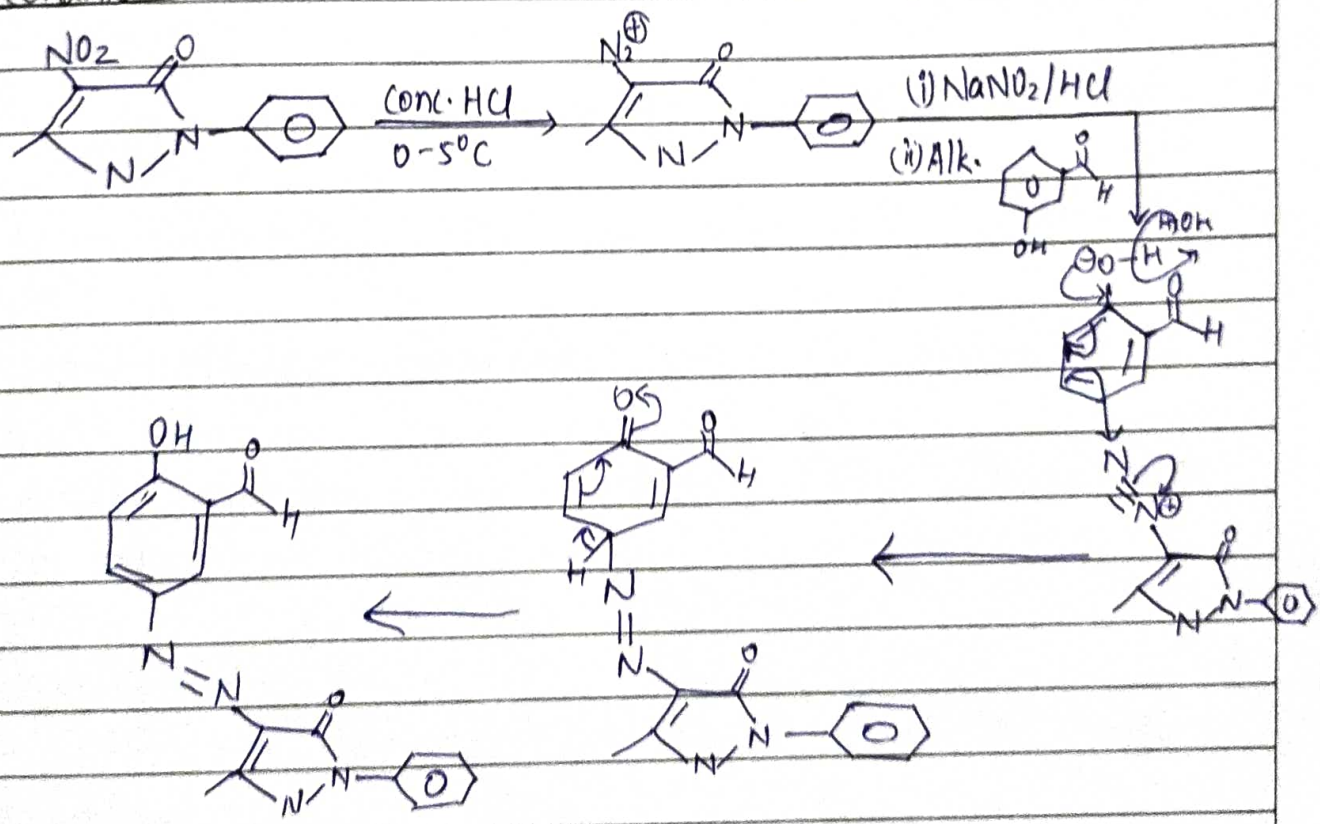
Reaⁿ:-





Mechanism:-



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Procedure:-

(i) Taking 0.5mg or 1g aromatic amine (Ampyron) in a 50ml beaker, 5ml conc. HCl added and the mixture kept in ice bath and stirred.
and then 20% $NaNO_2$ (10ml) sol^n added to it and shaken.

(ii) Alkaline phenolic compound (Salicylic acid) [1g or 0.5mg phenolic compound + NaOH] was mixed in the beaker and stirred kept the temp 0-5°C and colour dye observed.

(iii) The dye is evaporated to dryness in water bath in china dish untill the crystalline shape was appeared.

(iv) Cool the china dish and weight was taken and the yield of the compound, colour and Melting point

(v) Prepare different concentrate of dye in milimole i.e 10nm, 5mm, 1mm, 0.1mm, 0.01mm and the major the optical density and setting the spectrophotometric corresponding to colour of solution.

Conclusion:-

Azo dye compound having colour yield and Melting point.

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Page No.

Object:-

To determine the Biological Dissolve oxygen (BOD) in the given sample by titrimetric method.

Requirement:-

(i) $MnSO_4$ sol^n
(ii) Alkali - iodide - Azide solution
(iii) Conc. H_2SO_4
(iv) 0.025 N $Na_2S_2O_3$
(v) starch indicator
(vi) $MgSO_4$ sol^n
(vii) $CaCl_2$ sol^n
(ix) $FeCl_3$ sol^n

Procedure:-

(i) First take 200ml sample was taken in BOD bottle. 1ml each of phosphate buffere, $MgSO_4$, $CaCl_2$ and $FeCl_3$ sol^n were added and mixed well.
(ii) Dissolved oxygen is determined by Azide modification of winkler's method.
(ii) The solution was kept in BOD incubatore fore 5 day at 20°c.
(iii) 200ml of sample watere is collected in BOD bottle. 2ml of each of manganese sulphate and alkali-iodide azide reagent were added.
(iv) Bottle was shaken fore about 15 min and allowed ppt to settle down.
(v) Then 2ml conc. H_2SO_4 was added to dissolve the precipitate.

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(vi) 100ml of this solution is titrated against 0.025N $Na_2S_2O_3$ solution in presence of starch soln are indicatore till blue colour appears.

Results:-

The amount of BOD in the given sample is ppm.

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Object:-

To determine the chemical oxygen (COD) in the given sample by titrimetric method.

Requirement :-

(i) Ag_2SO_4
(ii) conc. H_2SO_4
(iii) 0.025(N) $K_2Cr_2O_7$
(iv) 0.1(N) F.A.S solution
(v) Ferroin indicator

Procedure :-

(i) 20ml water sample was taken in COD flask fitted with condenser and then added 10ml 0.025N $K_2Cr_2O_7$ solution.
(ii) A pinch of $HgSO_4$, and Ag_2SO_4 and shaken mixture with the addition of 30ml conc. H_2SO_4.
(iii) The mixture was then reflux for about 2hr, cooled, diluted to 150ml with distilled water and titrated with 0.1N F.A.S sol^n using ferroin indicator untill the colour changed from bluish green to wine red.
(iv) The blank is tranferred and titrated in the same manner using distilled water instead of sample water.

Result :-

The amount of COD is the given sample is ppm.

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Object:-

To determine the iron from the plant matrix by spectrophotometric method.

Requirement:-

(i) Hydroxyl amine hydrochloride
(ii) Ammonium acetate buffere
(iii) phenonthroline solution
(iv) Iron

Procedure:-

Standard sol^n preepation:-~~of~~

→ First we took 2mg Iron powder or Iron wire in a beaker.
→ Add 20ml of 6N H_2SO_4 into the beaker and boil till the Ferrous powder is completly dissolve
→ The Add distilled water (mak up the volume up to 100ml) this is stock sol^n.
→ From this stock solution we prepare 100µg, 50µg, 20µg, 10µg and 1µg standard solutions i.e 10ml.

procedure ~~for~~ Iron determination:-

→ 50ml of sample ~~is~~ was taken in the flask.
→ 2ml conc. HCl & 1ml hydroxylamine hydrochloride sol^n is added
→ The mixture is then boiled reduce to half of volume & cool
→ Then add 10ml ammonium acetate buffer and 2ml phenonthroline sol^n.
→ Then the volume was made up 100 ml distilled water & intensity of red color is measured by spectrophotometer at 510nm.

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Conclusion:-

By preparing standard curve in the range of 1 to 4mg/L of iron, amount of iron in the plant matrics is calculated and the amount of iron present is ppm.

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Sl No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
(1)	Preparation of tetra-mmine cupric sulphate $[Cu(NH_3)_4SO_4] \cdot H_2O$	1-3	27-08-15	03-09-15	
2)	Preparation of sodium oxalate ferrate trihydrate	4-5	3-09-15	24-09-15	
(3)	Preparation of Prussian blue $K_4[Fe(CN)_6]_3$	6-6	8-09-15	24-09-15	
(4)	Preparation of cis and trans pottasium dioxalato diaquo chromate	7-9	10-9-15	24-09-15	
(5)	To estimate copper as cuprous thiocynate	9-12	18-09-15	17-10-15	

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Object ⇒

Preparation of tetraamine cupric sulphate $[Cu(NH_3)_4SO_4]\cdot H_2O$

Apparatus ⇒ (i) Round bottom flask
(ii) Water bath
(iii) Whatman filter paper.
(iv) Beaker, pipette, ice, glass rod etc.

Chemical Reagent ⇒ (i) $CuSO_4$ = 5g
(ii) liq. NH_3 = 10ml
(iii) EtOH = 30ml

Principle ⇒

It is prepared by adding ammonia to solution of $CuSO_4$, when cupric hydroxide is precipitated which dissolve in excess of NH_3 to give a deep blue soln.

The hydratated tetraammio cupric sulphate is than precipitated from the soln by adding EtOH, Since it is insoluble in alchol.

Reaction is :-

$$CuSO_4 + 4NH_3 + H_2O \longrightarrow [Cu(NH_3)_4]SO_4\cdot H_2O$$

tetra amine cupric sulphate (blue)

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Procedure :-

It is various step reaction or procedure are given in below :-

1) Take 5 gm of crystaline $CuSO_4 \cdot 5H_2O$ in a 250 ml beaker. Dissolve it in minium quantity of water adding a few drops of H_2SO_4 if necessary to clear up to soln.

2) Now add liq. NH_3 soln to the beaker with constant stirring from a dropping funnel untill the blue ppt of cupric hydroxide 1st formed completely dissolves to yeild a clear, deep blue soln and there should be a smell of ammonia in a beaker.

(3) Now add alchol (30 ml) dropwise from the dropping funnel to the beaker with constant stirring untill the blue colour is nearly discharged. Heat the beaker to the water bath for about 15-16 min after that stop heating and remove the beaker from the water bath and allow it to stand. Long needel shaped blue crystal of tetra ammine cuprate sulphate separate sulphate separate out filter and wash these crystal with few drops of alchol.

(4) Dry the crystal on a porous plate or

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in a desicator, weight the dry crystal and submit reading.

Result -

Yield - 4.25 gm.

Precaustion :- (i) Make sure you read the bottle level carefully and only we during an experiments.

ii) Don't touch anything that you are not authorized to touch.

iii) Wash every instrument before the experimen

iv) One must also no how to use the lab equipments properly and should ask the right way of usages.

3/9/15

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Object :-

Preparation of sodium oxilate ferrate trihydrate $Na_3[Fe(C_2O_4)_3] \cdot 3H_2O$.

Reagent :- (i) Ferric chloride = 10 gm.
(ii) NaOH = 11.5 g
(iii) Oxalic acid = 12 g.
(iv) ~~A~~ alchohal

Apparatus :- Chemical flask, Beaker, pippete, Burner, funnel, desicator.

Principle :- It is prepared by the action of sol^n oxalate on hydrated ~~fer~~ ferric oxide.

Procedure :- Dissolve ~~to~~ 10 gm of ferric chloride in 10 ml of water and dissolve 7.5 gm of NaOH in 5 ml water to the sol^n of ferric chloride added NaOH sol^n in small quantities resulting in the formation of ferric hydroxide. now the filter ppt of $Fe(OH)_3$ through Buchner funnel and wash saparat time with small quantities of hot water.

In the other beaker dissolve 12 g of oxalic acid in 50 ml of water to this ~~a~~ sol^n add the pellets NaOH, as resulting in the formation of sodium oxalate. Now add the waited hydrated ferric oxilate to the hot sodium oxalate sol^n with

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constant stirring by glass rod.
Filter the sodium and reagent the residue that wash the crystal first with ice or cold water and then with alchohol dry the crystal in the decicator.

Result:- Yeild = 6.005 gm

Precaustion:- (1) Before addition of $FeCl_3$ one should take care of that it must be anhydrous.

(2) The water must be heated middly but a higher flame otherwise it may cause damage of the filter paper during washing.

(3) The hydrated ferric oxilate must be added by contants stirring with glass rod.

24/9/15

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Object :-

Preparation of Prussion Blue
$K_4[Fe(CN)_6]_3$

Chemical Required :-

Anhydride ferric chloride = 2g.
potassium ferrocyanide = 4.5g

Procedure :-

dissolve 2g of anhydrous $FeCl_3$ in 3-4 ml of water. Also dissolve 4.5g of $K_4[Fe(CN)_6]_3$ in 20-25 ml of water. Mix up both the solution in a 100 ml beaker when a blue coloured precipitate of prussian blue is formed. Filter it and wash 2-3 times with water. Dry this blue paste in the folds of filter paper weight and determine the yeild

Yield = 6.88 gm

Precaution :- (1) Pestle & mortar must be dry otherwise a rean may setin.

2) Heating should be very gently otherwise the rean may go out of control and cause explosion.

3) Since ethanol has to be added to the contents naked flame should kept away as far as possible

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Object :-

Preparation of cis and trans ~~potto~~ potassium dioxalato diaquo chromate $[K(Cr(C_2O_4))H_2O_2] \cdot 2H_2O$

Chemical Required :-

Oxalic acid = 6gm
Potassium dichromate ($K_2Cr_2O_7$) = 2gm
ethnol = 14 ml

Principle :-

It may be prepared by the reaction of potassium dichromate with oxalic acid.

$$K_2Cr_2O_7 + 7\begin{matrix}COOH\\|\\COOH\end{matrix} \cdot 2H_2O \rightarrow 2K[Cr(C_2O_4)_2(H_2O)_2]2H_2O + 6CO_2 + 13H_2O$$

Procedure :-

(i) Take a dry montal and put 6 gm of oxalic acid crystals and 2gm of $K_2Cr_2O_7$ crystals in it.

(ii) Gently grind the mixture with a pestle till the powders are quite intimately mixed.

(iii) Take a china dish (10 cm dia) and moistun it if with H_2O. Drain away the excess of water.

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(iv) Introduce the mixture of the center of the china dish in a compact heat.
(v) Covers the china dish with a clock glass.
(vi) that the contents in the china dish gently on a low flame.
(vii) After a few mixture, a vigorous reaⁿ will viet in accombanced with frothing due to evolution of CO_2 and H_2O val
(vii) Soon the reaⁿ will spreced through out the mixture which will become deeply coloured liquid.
(ix) Without waiting for the thick liq. ~~th~~ to cool, pour about 20ml ethanol over the liq.
(x) Using a matallic spatula, triturate the contents untill a solid is formed.
(xi) In case complete solidification is not achived decant the liq. and add it to another lot of 20ml of ethanol.
(xii) Warm and resume trituration untill the pdt is in the form of granular crystals.
(xiii) The crystals will look black in diffused day light and deep purple in artificial light
(xiv) Weight & record the ~~yeild~~ yield.

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Result :- 2.393 gm

Precaution :- Pestle and mortal must be dry otherwise a mean may set in.

(2) Heating should be very gently otherwise the mean may go out of control & cause explation.

(3) Since ethanol has to be added to the contents naked flame should kept away as far as possible

(4) It is safer to use hot plate instead of burn in this preparation.

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Object :-

To estimate copper ~~to~~ as cuprous thiocynate.

Requirement :-

Copper sulphate = 24.6344 gm.
Ammonium thiocynate = 10 gm.
Sulphurous acid = 20-30 ml

(i) Precipitating reagent → 10% ammonium.

ii) Thiocyanate solⁿ →
It is prepared by dissolving 10 g ammonium thiocyanate in (90 ml dis. water)

(iii) Washing solⁿ →
1% ammonium thiocyanate solⁿ is prepared by dissolving 1 g ammonium thiocynate solⁿ in 90 ml distilled water.

(iv) OR other is 20% alcohol →
It is prepared by diluting 20 ml alcohol in 80 ml distilled water

Principle :-

Cu^{2+} ion contant of a measured of solⁿ can be estimated by precipitating

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Calculation ⇒

Weight of empty crucible = 33.325 gm.

Weight of crucible + CuSCN = 33.725

33.325 + 0.4

Constant weight of CuSCN = (33.725 − 33.325)
= 0.4

CuSCN = Cu

121.64 g = 63.57 g

121.64 g CuSCN contain = 63.57 g of Cu.

0.4 g CuSCN contain = $\frac{63.57 \times 0.4}{121.64}$

= 0.20

25 ml solⁿ contain = 0.20 gm of Cu.

1000 ml solⁿ contain = $0.20 \times \frac{1000}{25}$

= 8 gm of Cu.

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quantitatively as CuSCN by with NH_4SCN solⁿ in sulphonous acid medium filtering through a glass crucible and weight the ppt after drying at (110 − 120°c).

$$2Cu^{2+} + HSO_3^- + H_2O \rightarrow 2Cu^+ + HSO_4^- + 2H^+$$

$$Cu^+ + NH_4SCN \rightarrow CuSCN + NH_4^+$$

Procedure :-

25 ml of the given Cu salt solⁿ was taken in 500 ml beaker. Then a few drop of conc. HCl was added to it and 25 ml con. H_2SO_4 was added to it was then dilute diluted with 100 ml distilled water and heated nearly to boiling and then 25 ml 10% ammonium thiocynate solⁿ was added to it after removal from and with constant stirring with a glass rod untill a ppt of CuSCN is complete ppt should be with whi and the mothers liquid should be colounles

The Beaker was kept on water bath for 30 min. complete ppt as chemical by adding carefully two droup of NH_4SCN allowed to be the super netant liquid and then allowed to stand for 2 hours.

The ppt was taken in

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Crusible and kept in air oven at 110-120°c for 30 min. cooled and weight. The process of heating & cooling and washing was repeated till constant wt was obtained.

Result → Provide sample soln = 8gm of Cu.

Precaution ⇒

(i) The solution should be kept slightly acidic.

(ii) Oxidising agents must be absent.

(iii) The presence of reducting agent in essential.

17/10/11

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Object :-
Preparation of Sodium trisoxalato Ferrate (III).

Principle :-
It is prepared by the action of Iron(III) hydroxide with oxalic acid and Sodium hydroxide.

$$FeCl_3 + 3NaOH \longrightarrow Fe(OH)_3 + 3NaCl$$
$$Fe(OH)_3 + 3H_2C_2O_4 + NaOH \longrightarrow Na_2[Fe(C_2O_4)] + 6H_2O$$

Apparatus required :-
Electronic weighing machine, beaker, spatula, china dish, watch glass, Glass rod, Buchner funnel, measuring cylinder, whatmann Filter paper, sand bath.

Chemical required :-
Ferric chloride (3.5 gm) anhydrous
Sodium hydroxide (4 gm)
Oxalic acid hydrated (4 gm)
Sodium oxalate hydrated (5.5 gm)
Ethyl alcohol.
Distilled water

Procedure :-
1. Preparation of Fresh iron(III) hydroxide $Fe(OH)_3$ Sol^n.
a. Take Ferric chloride (3.5 gm) anhydrous. Add slowly 50 ml distilled water to it using measuring cylinder with constant stirring with

Glass rod.

b- In another beaker, take sodium hydroxide (4gm) Add slowly 50 ml distilled water to it using measuring cylinder with constant stirring with rod.

c- Add sodium hydroxide (4gm), solution in ferric chloride solution in small proportion with constant stirring using glass rod. Ferric chloride reacts with sodium hydroxide to give brown ppt of ferric hydroxide.

d- Filter the brown ppt of ferric hydroxide through Buchner, funnel, wash the ppt with distilled water and transfer it into a watch glass.

2. In another beaker, take oxalic acid hydrated (4gm) and sodium oxalate hydrated (5.5gm). Add slowly 100ml distilled to it using measuring cylinder with constant stirring with glass rod to get a clear solution.

3. To the above solⁿ, Add freshly prepared brown ppt of Ferric hydroxide in small proportion with constant stirring with glass rod to dissolve the ppt.

4. Filter the above solution in a china dish using a funnel and filter paper. The insoluble impurities will left behind on the filter paper.

calculation :-

Weight of the petri dish (x) = 48.11 gm

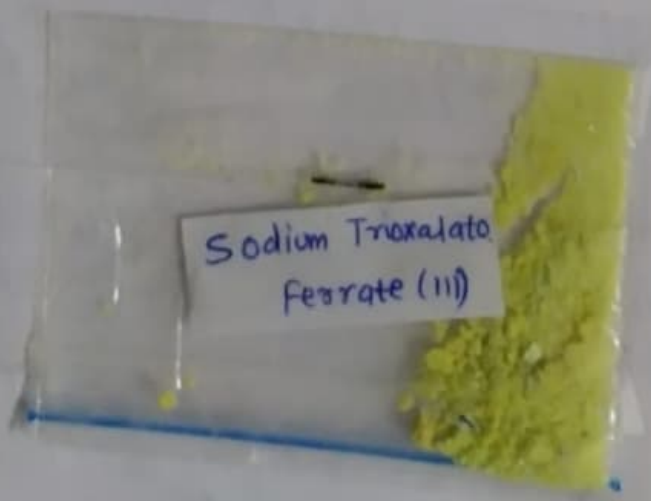
Weight of the petri dish with crystal (y) = 50.34 gm

Hence,

The weight of the crystal = (y − x)

[Sodium tris oxalato Ferrate (III)] = 50.34 gm − 48.11 gm

= 2.23 gm



5 - Keep the sol^n in china dish over the sand bath. Heat the sol^n to reduce their volume to the one half at their original volume with constant stirring to avoid crust formation on the wall at china dish

6 - Take out the concentrated solution on glass rod and blow air over it to check out the precipitation point. The formation of thick crust layer on the glass rod indicator that the crystalazation point has been reached.

7 - Place the china dish in the through containing cold water cover the china dish with black paper and allow to it stand undisturbed for some time to get green crystals.

8 - Decant the mother, liquar in the beaker and wash the crystal 3.4 times with small amount of Ethanol.

Result :-
The yield of sodium trioxalato. Ferrate (III) is 2.23 gm

Precaution :-
1. Before addition of $FeCl_3$, one should be take care of that it must be anhydrous.
2. Do not concentratate the solution too much.
3. Do not disturb the solution during crystallization.

Object :-

To prepare Ni - DMG Complex

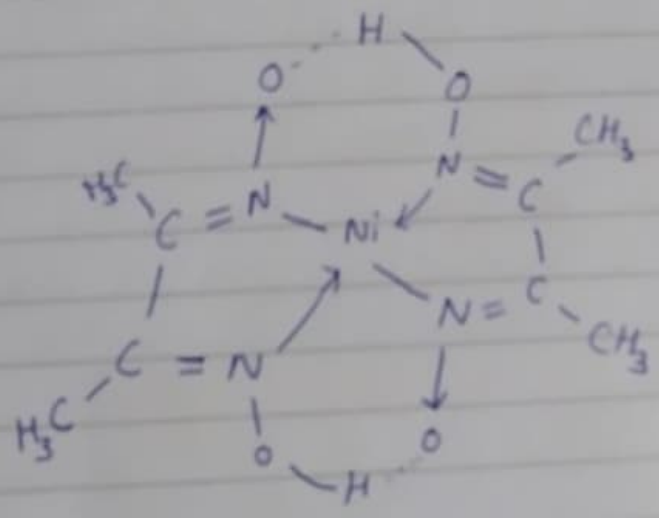
Priciple :-

It is prepared by the reaction of Nickel Sulphate Solution with alcoholic dimethyl Glyoxime Solution and then adding a slight excess of NH_4OH (1:1 NH_3 Solution)

$$NiSO_4(NH_4)_2SO_4 . 6H_2O \longrightarrow NiSO_4 + (NH_4)_2SO_4 + 6H_2O$$

$$NiSO_4 + 2HDMG + 2NH_4OH \longrightarrow [Ni(DMG)_2] + (NH_4)_2SO_4 + 2H_2O$$

Structure of Ni - DMG (Nickel dimethyl glyoxime) complex



Apparatus required :-

Watch glass, Beaker, rod, Measuring cylinder, funnel, Burner, Filter paper, tripod stand, pipette, Air oven, weighing Balance, Dessiccator.

calculation :-

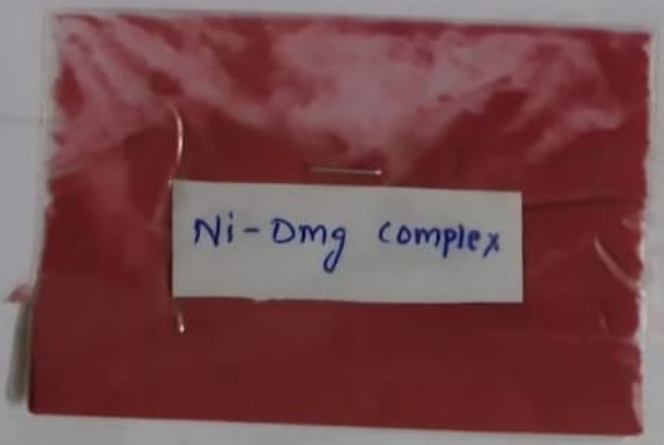
weight of the crucible (x) = 48.60 gm

weight of the crucible with precipitated (y) = 48.60 gm

Hence, weight of the precipitate = (y − x)

= 48.60 − 43.70

= 4.90 gm



- Chemicals required :-
Nickel Sulphate ($NiSO_4.6H_2O$), Alcohol (C_2H_5OH), Dimethyl glyoxime (DMG), NH_4OH or (1:1 NH_3 Solution, Dilute HCl Distilled water.

- Procedure :-
1. weigh 2gm of Nickel sulphate and transfer it to a 500ml beaker and dissolve it to minimum quantity of water.
2 - Add 2-3 ml of dilute HCl slowly with constant stirring
3 - Now, heat this solution to gentle boiling and add approximatly 30 ml of alcoholic Dimethylglyoxime reagent with constant stirring.
4 - Immediatly add 1:1 NH_3 Solution or NH_4OH Solution drop wise with stirring until precipitate occurs and add extra 10-15 drops of NH_3 Solution for complete precipitation.
5 - Scarlet red precipitate of Ni-DMG can be seen filter the precipitate and wash with water.
6 - After washing with water wash it with alcohol. and dry in air or in air oven.
7 - After drying weigh the sample.

Result :-
The yeild of the sample or Ni-DMG complex is 4.90gm

Precaution :-
1. keep away the DMG from heat, avoid eye contact with chemicals.
2. The apparatus should be raised and clean properly.

Object :-
Preparation of tetra ammine cupric sulphate complex.

Theory :-
Hydrated tetra ammine cupric sulphate is obtained by addition of liquid ammonia in a solution of copper sulphate followed by addition of ethyl alcohol in a solution.

$$CuSO_4 + 4NH_3 + H_2O \longrightarrow [Cu(NH_3)_4]\,SO_4 \cdot H_2O$$

Apparatus required :-
Electronic weighing machine, Beaker (500ml, 250ml), Filtration apparatus, conical flask, funnel, desiccator, Glass rod, Tongs, Tripod stand, watch glass and water bath.

Chemical required :-
copper sulphate (2.5 grams), Liquid ammonia (5ml) Ethyl alcohol (7.5 ml)

Procedure :-
The four steps involve during the formation of hydrated tetra ammine cupric sulphate complex given as :-

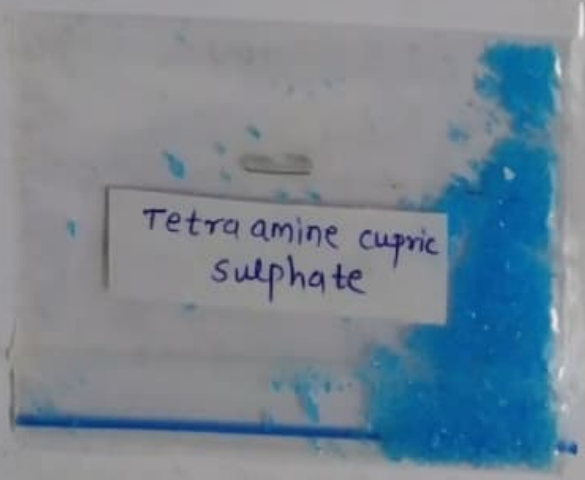
1. Firstly dissolve 2.5 gm of crystalline $CuSO_4\ 5H_2O$ in minimum amount of H_2O in 250 ml beaker. Now add few drop of concentrated H_2SO_4 to clear up the solution if necessary.

Calculation :-

weight at the crucible (x) =

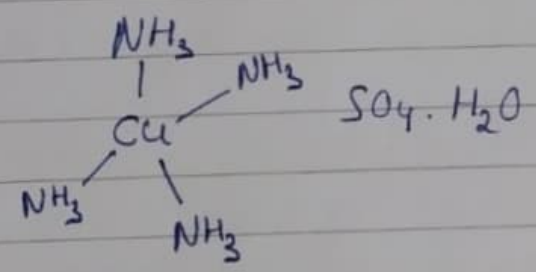
weight at the crucible with complex (y) =

Hence, weight at the complex = (y - x)



2. Add liquor ammonia to copper sulphate solution from dropping funnel with constant stirring until the blue precipitate formed is dissolved to give a deep solution.
3. Now, add 20ml of ethyl alcohol slowly with constant stirring to the deep blue solution. And allow it to stand so slow evaporation which gives long needle shaped brilliant dark blue - violet crystal of tetra ammine cupric sulphate complex.
4. Filter and wash it with ethyl alcohol and dry in a dessiccator.

Structure of tetra ammine cupric complex.



Result :-
The yield of tetra ammine cupric sulphate is gram.

Precautions :-
1. Add ammonia solution dropwise with constant stirring
2. weight the crystal correctly.
3. Apparatus should be clean and rinsed.

Object:-
Preparation of Prussian blue $Fe_4[Fe(CN)_6]_3$

Theory:-
The complex iron hexacyana Ferrate is known as Prussian blue.
It is obtained by oxidation of iron, Fe to Fe^{3+}, followed by reaction with potassium Ferrocyanide.

Reaction:-
$$Fe + H_2SO_4 \longrightarrow FeSO_4 + H_2$$
$$2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O$$
$$Fe(SO_4)_3 + 2K_2[Fe(CN)_6]_3 \longrightarrow Fe_4[Fe(CN)_6]_3 + 5K_2SO_4$$
$$Fe \longrightarrow Fe_4[Fe(CN)_6]_3$$
Molecular weight = 55.85 gram 859.25 gram

Apparatus required:-
Beaker, Measuring cylinder, Buchner flask, Filter flask, filter paper, Glass rod.

Chemicals required:-

Iron fillings, pottassium Ferro cyanide concentrated Nitric acid (HNO_3), Dilute Sulpuric acid (H_2SO_4) Ethanol.

Calculation :-

weight at the crucible (x) = 6.5.82 gram
weight at the crucible with prussian blue complex (y) = 72.79 gram

Hence, weight at prussian blue complex = (y-x)
=(72.79-65.82)
= 6.97 gram

Procedure :-

1. Take 0.5 gram at iron Fillings in a beaker and add 10ml dilute Sulfuric acid and heat it.
2. The reaction becomes vigorous then remove the burner and cool the solution.
3. when the reaction subsides, decant the supernatant liquid in another beaker and now, add 10ml at dilute sulfuric acid. Repeat the procedure till whole iron fillings are dissolved.
4. Combine the solution at ferrous sulphate and heat it, Now add 0.5 ml of concentrated HNO_3 and boil to oxidize iron to ferric state.
5. The solution becomes yellow, continue boiling for 5 mint to expel fumes at NO_2 gas and nitric acid.
6. Now, add the saturated solution at 4.5 gram at potassium Ferrocyanide, heat the solution until it aquires green colour
7. keep the green coloured product in air for 2 hours it changes to blue colour (Prussian blue)
8. Filter the product wash with a little alcohol, dry and weight it.

Result :-

The yield of prussian blue is

Precaution :-

1. The smell at nitric acid is extremely harmful So, wear the mask
2. Concentrated acid should be handled carefully.