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Experiment - 1

Aim - Preparation of sodium trioxalate ferrate (III) $Na_3[Fe(C_2O_4)_3]$

Reaction - It is prepared by hydrated ferric oxide on reaction with sodium oxalate.

$$Fe_2O_3 + 6Na_2 \longrightarrow 2Na_3[Fe(C_2O_4)_3] + 3Na_2O$$

Reagents - Ferric chloride - 10 gms
Sodium hydroxide - 12 gms
Oxalic Acid - 12 gms

Apparatus - Beakers, watch glass, buckner funnel, keep.

Procedure - 1. Take 10 gms ferric chloride and dissolve it in 10-15 ml water.

2. Similarly in another beaker 7.5 gm sodium hydroxide dissolve it in 5ml water.
3 - Now add sodium hydroxide solution to ferric chloride solution slowly in small lots. In this way precipitate of ferric hydroxide (ferricox) is obtained.
4 - Filter this by buckner funnel and wash the precipitate thoroughly by water.

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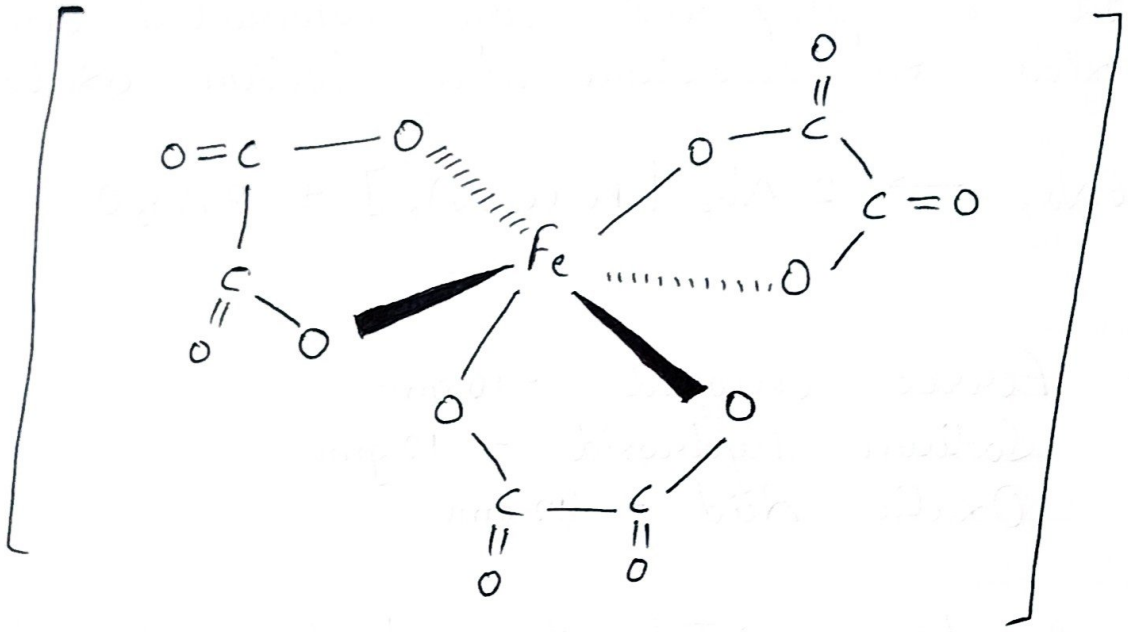


5 - In another beaker dissolve 12 gm oxalic acid in 50 ml water of then add 4 gm (NaOH) pallets so the solution of sodium oxalate is formed. Boil this solution.

6 - Now add to this solution above prepared ferric hydroxide (ferric oxide) solution slowly with constant stirring with glass rod a green colour solution is obtained.

7 - Filter the solution. Take the filtrate in porcelain disc and concentrate on water bath. The green crystals of ferric oxalate is formed.

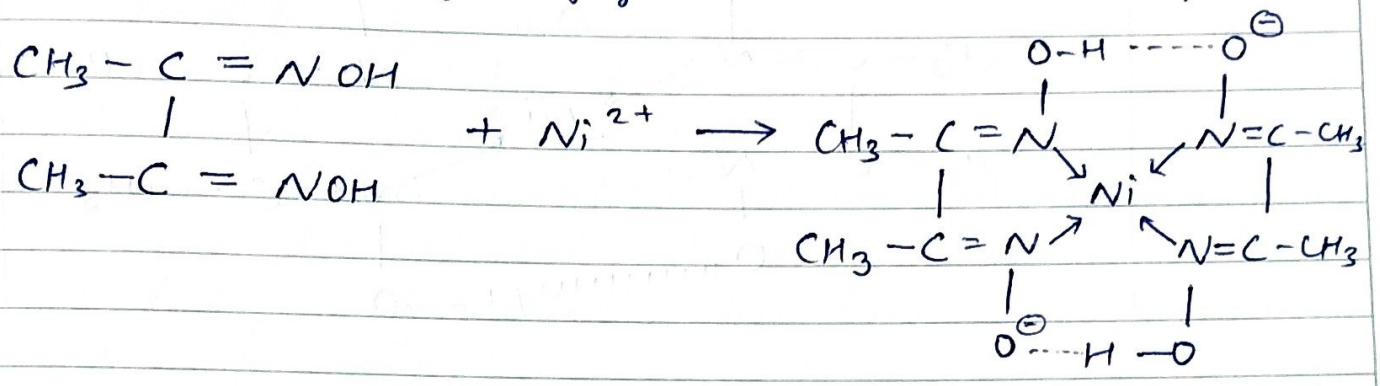
8 - The crystals is first washed, with cold water then with alcohol. Keep crystals in desicator for dyness.

Result - yeild - 3 gm.

Experiment - 2

Aim - preparation of Ni - DMG Complex $[Ni(DMG)_2]$

Chemical reaction - when dimethyl glyoxime is added to nickel solution scarlet precipitate of nickel dimethyl glyoximate complex is formed.



OR

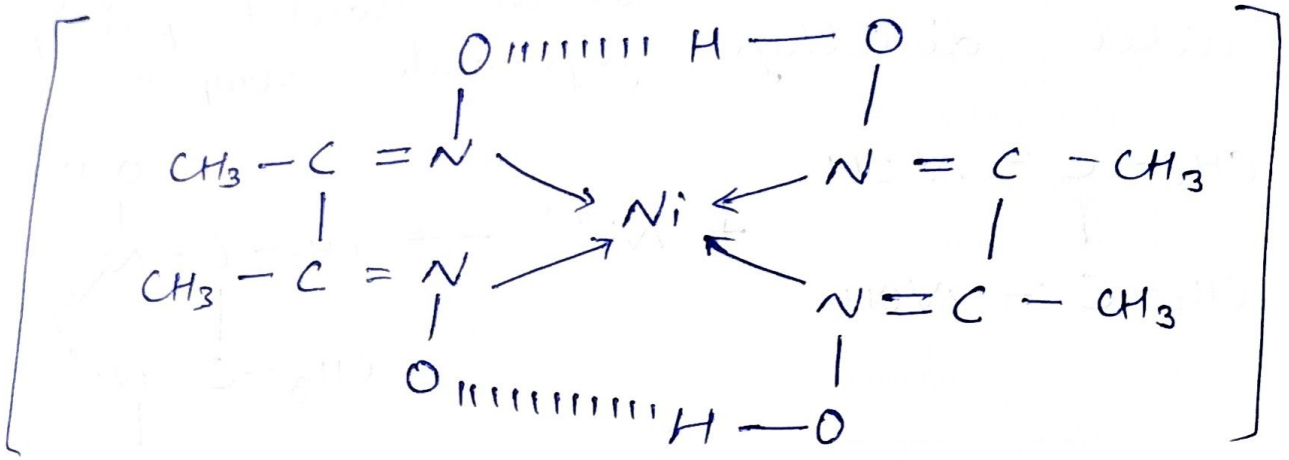
$$2H_2DMG + Ni^{2+} \longrightarrow Ni(DMG)_2 + 2H^+$$

Nickel dimethyl glyoximate complex (scarlet or red ppt)

Reagents - (i) Nickel chloride = 6.0 gms

(ii) Dimethyl glyoximin - 1.0 % (in alcohol)

(iii) Ammonia solution - 1:1



Procedure – 1. Nickel chloride (6 gm) dissolved in 100 -125 ml water in beaker (500 ml capacity)
2. The solution is warm up to 70 - 80°C
3. In hot solution add 40 - 50 ml 1% dimethyl glyoxime solution.
4. Now add drop by drop 1:1 ammonia solution & stir the solution by glass rod till sol^ gives permanent strong smell of ammonia scarlet (red) precipitate is formed.
5. The content of beaker is warm for 20 - 30 min on water bath.
6. Red precipitate settle down on bottom. In supernant liquid of beaker add few drops of dimethyl glyoxime reagent to confirm whether precipitation is complete. Keep beaker for half an hour.
7. Filter the solution by buckner funnel and wash the precipitate by cold water twice or thrice. Add atleast 5ml alcohol and dry the precipitate.

Result – 4.2 gm (yield)

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Experiment - 3

Aim - preparation of tetra - amine copper complex $[Cu(NH_3)_4]SO_4$

Chemical reaction - When ammonia liquid is added to copper sulphate solution it forms tetra amino cupric sulphate. To this by adding alcohol blue crystal of tetra-amine cupric sulphate is formed.

$$CuSO_4 + 4NH_3 + H_2O \rightarrow [Cu(NH_3)_4].H_2O \downarrow$$

Blue crystal

- If solution is not clear add 1-2 drops dil H_2SO_4

Reagents - (i) copper sulphate - 5gm
(ii) Ammonia liquour - 10ml
(iii) Alcohol - 40 ml

Apparatus - beaker, watchglass

Procedure -

1. Take 5 gm copper sulphate grind it & dissolve it in minimum amount of water.
2. To this add ammonia liquour solution slowly with constant stirring. Firstly blue precipitate of cupric hydroxide formed.

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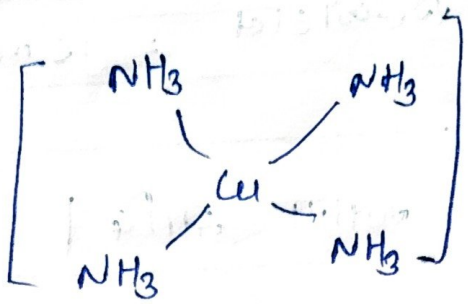
Symmetrical structure of complex -



$[Cu(NH_3)_4]^{+2}$ ion

3. Now add more ammonia liquour till blue precipitate dissolves, solution becomes blue & solution gives smell of ammonia.
4. Now to this blue solution add 40 ml alcohol slowly with constant stirring.
5. After that the solution of beaker is boiled (60 - 70 °C) on water bath for 10-15 minutes.
6. On cooling the content of beaker, needle type crystal of blue colour separates in beaker. Filter it and dried with filter paper and separate crystals.

Result - yeild - 2.9 gm.

Experiment -4

(a) Aim - preparation of cis potassium di-oxalato diaquachromate $K[Cr(C_2O_4)_2(H_2O)_2]$.

Chemical reaction - when potassium dichromate reacts with oxalic acid it form this compound.

$$K_2Cr_2O_7 + 7\begin{matrix}COOH\\|\\COOH\end{matrix} 2H_2O \rightarrow 2K[Cr(C_2O_4)_2(H_2O)_2]2H_2O + 6CO_2 + 3H_2O$$

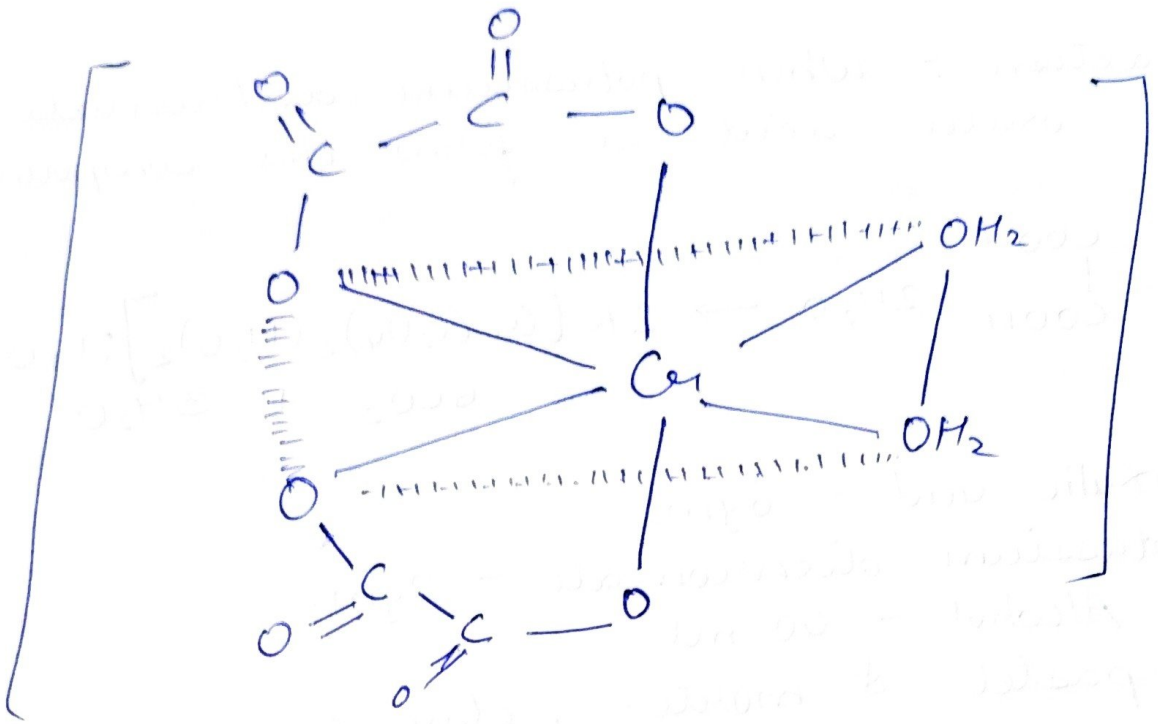
Reagents - oxalic acid - 6gms
potassium dichromate - 2gms
Alcohol - 40 ml

Apparatus - pastel & morter, clay cup

Procedure -

1. In a dry pastel take 6 gm oxalic acid and 2 gm potassium dichromate and grind this mixture in powder form by mortar.

2. Take this mixture in a procela is disc and wet by very small amount of water. This disc is covered with watch glass and heat on low flame of burner.



Cis form

3. On heating CO_2 is expled in the form of effervences and water vapours is also expeled. The mixture converts into dark coloured liquid.

4. In hot solution add alcohol and stir the solution with spatula, a solid substance is formed.

5. If solid substance is not formed, then decent the liquid and more add 20 ml alcohol and stir the solution well with steel spatula the complete substance becomes solid.

6. NaCl this substance is warm lightly with stirring. Till crystals or granular crystals are formed. These crystals are black in colour.

Result - yeild - 3gm

(b) Aim - preparation of trans potassium Di-oxalato diaquachromate (III)
$K[Cr(C_2O_4)_2(H_2O)_2]_2 H_2O$

Chemical reaction -

$$K_2Cr_2O_7 + 7 \begin{matrix} COOH \\ | \\ COOH \end{matrix} + 2H_2O \longrightarrow 2K[Cr(C_2O_4)_2(H_2O)_2] + 2H_2O + 6CO_2 + 3H_2O$$

Reagents - oxalic acid - 6gms
pot. dichromate - 2gms

Apparatus - beaker, watch glass

Procedure -

1. In a beaker (capacity 250 ml) dissolve 6gm oxalic acid in minimum about of water and boil.
2. Similarly take 2 gms potassium dichromate in boiling tube and dissolve it in minimum amount of water by boiling.
3. Add boiling tube solution slowly to beaker which contains oxalic acid solution.

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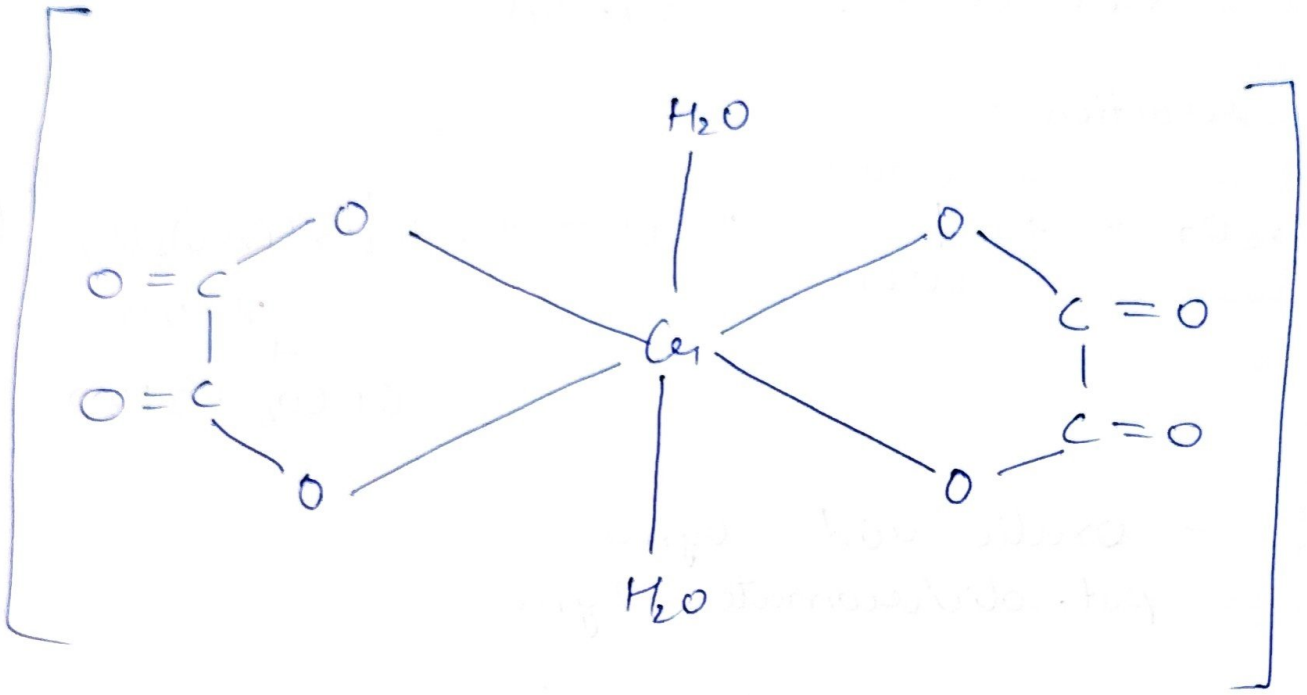


Trans form

4. Reaction takes place fast so cover the beaker with watch glass mix both solution completely. The solution of beaker becomes dark in colour.

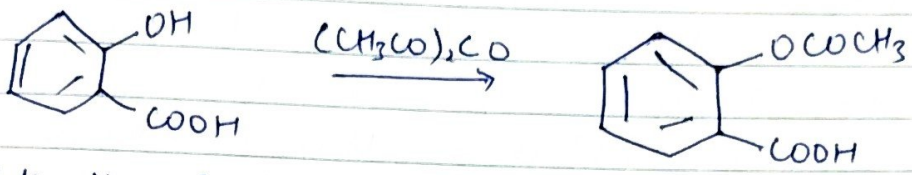
5. Transfer this solution to procela in disc and boil till it becomes on third. Now cool this solution to room temperature. After some time crystal formation takes place in china cup. Crystal washed with cold water and finally wash with alcohol. Pink colour crystal are formed.

Result - yield - 3.2 gm

Experiment -5

Aim - preparation of aspirin from salicylic acid.

Reaction - Acetylation of salicylic acid with acetic anhydride it form aspirin (acetylsalicylic acid)



Salicylic Acid → Aspirin

Chemical required -

(i) Salicylic acid - 5 gm
(ii) Acetic anhydride - 7 ml
(iii) Conc. H_2SO_4 - 0.5 ml

Apparatus - Round bottom flask, keep, beakers

Procedure -

In a 100 ml flask take 5 gm salicylic acid 7 ml acetic anhydride & 0.5 ml conc. H_2SO_4 Shake the mixture, warm the reaction mixture with constant stirring on a water for

about 15 minutes at temperature 50° - 60°C cool this mixture and then pour it in 100 ml cold water taken in a beaker with stirring. Filter the solution and solid compound separate. Now dissolve solid substance into 50 % alcohol. Keep at room temperature wash the ppt and recrystallise from acetic acid needle type crystals obtained.

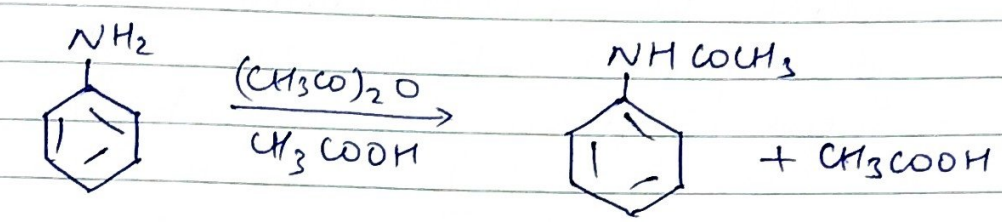
Melting point - 136° - 137°C

Result - Aspirin is colourless, needle type crystals.
Yeild - 3gms

Experiment - 6

Aim - preparation of acetanilide from aniline

Chemical reaction - acetylation of aniline with acetic anhydride form acetanilide.



Aniline

Acetanilide

Reagent required -
Aniline - 5 ml
Acetic anhydride - 5 ml
Glacial acetic acid - 5 ml
Ice

Apparatus - 100 ml capacity round bottom flask air conditioner (condenser), 250 ml capacity beaker.

Procedure -

In 100 ml, capacity round bottom flask take 5ml aniline, 5ml acetic anhydride and 5ml glacial acetic acid. Boil this mixture slowly for about 15-20 min by using air condenser.

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Take ice cold water and few pieces of ice in beaker and pour this mixture in it. A white solid substance separates out. Filter it and wash white substance with water. Now dissolve this solid substance is boiling acetic acid solution (10 ml acetic acid + 90m water) or in minimum amount of boiling wa Cool on room temperature a white shining crystals obtained. Filter it and dry it by pressing them between the filter paper.

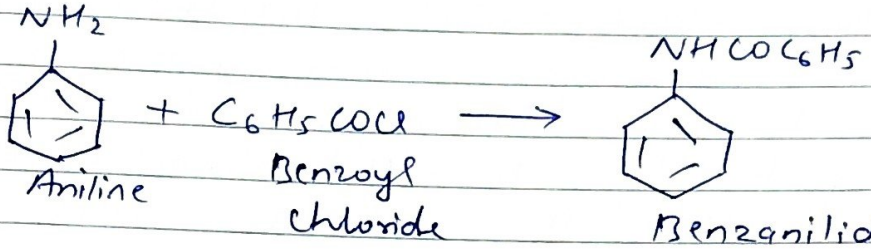
Physical state – white shining crystal
yield – about 5gm

Melting point – 114 °C

Experiment -7

Aim - preparation of benzanilide from aniline.

Chemical reaction - Benzoylation of aniline take place with benzyl chloride.



Reagents - Aniline - 5ml
Benzoyl chloride - 7ml
10% NaOH sol° - 50 ml

Apparatus - 250 ml capacity conical flask, beaker, keep.

Procedure - In a 250 ml capacity conical flask take 5ml aniline, 50 ml 10% NaOH solution and add 7ml (benzoyl chloride all at the same time. Cork the flask and shake it vigorously for about 15 min. In this process flask is hot, cool it & shake again. When the reaction is complete (indicated by the disappearance of pungent smell of benzoyl chloride). Add about 100 ml cold water to the flask it forms white solid substance.

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Filter it by buchner funnel. Wash ppt with cold water. Dry the product of ppt. Recrystallised it by hot alcohol or methylated sprit White crystalline substance obtained.

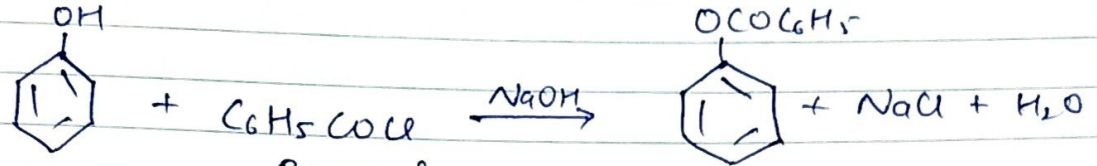
Physical state - white crystalline solid substance
yield - 8 gm

Melting point - 163 °c

Experiment - 8

Aim - preparation of phenyl benzoate from phenol.

Reaction -



Phenol — Benzoyl chloride — Phenyl benzoate

Reagents -
Phenol - 2gm
Benzoyl chloride - 4ml
10% NaOH solution - 30ml

Apparatus - Flask, beaker, keep

Procedure -

In 250 ml capacity flask take 2 gm phenol add to it 30 ml 10% NaOH solution slowly. Now add 4ml benzoyl chloride slowly with well shaking. Cork the flask. the reaction mixture shake vigorously for half an hour. The reaction is complete indicated by the disappearance of pungent smell of benzoyl chloride. white compound is formed. Cool the flask

Filter the compound wash the compound (ppt) with cold water. Dry it by pressing with filter paper. Recrystallised it with rectified sprit or methylated sprit.

Physical state - colourless crystal
yield - 3.7 gm

Melting point - 69 - 70 °C

Experiment - 9

Aim - preparation of iodoform from ethanol.

Chemical reaction - compounds having $CH_3CH(OH)$ or CH_3CO group reacts with alkali and iodine form iodoform.

$$CH_3CH_2OH + I_2 \longrightarrow CH_3CHO + 2HI$$
$$CH_3CHO + 3I_2 \longrightarrow CH_3CHO + 3HI$$

$$2CH_3CHO + Na_2CO_3 + H_2O \longrightarrow 2CHI_3 + 2HCOONa + CO_2$$

Reagents -
Ethyl alcohol - 5ml
Sodium carbonate - 10 gm
Iodine - 10 gm

Apparatus - flask capacity 250 ml, water bath, buckner funnel.

Procedure -

In a 250 ml capacity flask add 5ml ethyl alcohol, 10 gm sodium carbonate & 50-60 ml water. The mixture is heated 70-80°C on water bath. Take 10 gm iodine crush & form powder. Now in this flask add slowly powdered iodine. Again heat this flask 70-80°C

on water bath till violet colour of iodine does not disappear add 3-4 ml NaOH solution cool the solution, yellow crystal of iodo form is obtained. Filter it wash it with cold water and dry it with the help of filter paper. Recrystalline it with minimum amount of ethanol.

Physical state - Iodoform - solid yellow coloured crystal

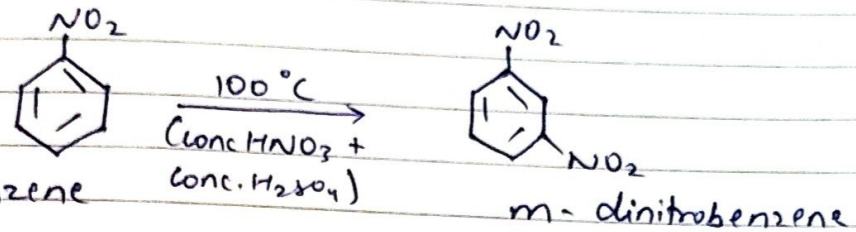
yeild - 5gm

Melting point - 120 - 122 °C

Experiment - 10

Aim - preparation of m-dinitrobenzene from nitrobenzene

Reaction - By the nitration of nitrobenzene from conc. HNO_3 and conc. H_2SO_4 at 100°C obtained m-dinitrobenzene.



nitrobenzene → m-dinitrobenzene

Reagents -
(i) Nitrobenzene - 5ml
(ii) Conc. HNO_3 - 15ml
(iii) Conc H_2SO_4 - 15ml
(iv) Alcohol

Apparatus - 100 ml capacity round bottom flask, reflux condenser, 25ml beaker, Keep etc.

Procedure -

In a 100 ml capacity round bottom flask add 15ml conc. HNO_3 add 15 ml conc. H_2SO_4 slowly with constant stirring. The flask is hot cool under tap. Now add

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5ml nitrobenzene slowly with shaking. Now add 3-4 small pieces of porcelain is added to conical flask and attach reflux condenser and heat the flask on water bath upto one hour. At this stage few drops of reaction mixture powred into cold water & if a hard pale yellow solids separates then the heating is discontinued. The flask is hot cool under tap. Now add 5ml nitrobenzene slowly with shaking. The reaction mixture is cooled and the contents powred with vigorous stirring into about 250 ml water containing few pieces of ice when m-dinitrobenzene separates as a hard yellow solid filter it in a buckner funnel. wash with water and dry by pressing with filter paper. Recrystallise from rectified spirit when pale yellow crystal of m-dinitrobenzene are obtained.

Physical state - crystalline solid of light pale yellow colour

yield - 3.1 gm

Melting point - 90°C

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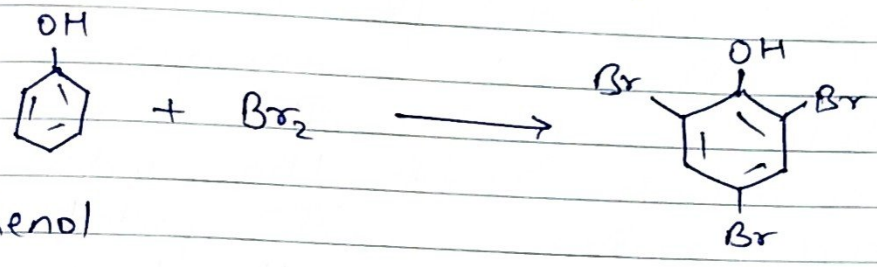
Experiment -11

Aim - preparation of 2,4,6 tribromophenol from phenol.

Reaction - When phenol reacts with bromine it forms 2:4:6 bromophenol.



Phenol

Reagents -
(i) Phenol - 5 gm
(ii) Bromine - 18 ml
(iii) Ice

Apparatus - Flask, keep, beaker etc.

Procedure -

In a flask take 5 gm phenol dissolve it in minimum amount of water. Cool it in ice cold water. Now add to it 18 ml bromine drop by drop with separating funnel with constant shaking. A precipitate is formed. Add entire amount of bromine. Keep it for sometime and filter it and wash with cold water. Dry it with filter paper.

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Solid substance is crystallised with alcohol (rectified spirit), colourless needle type crystals are obtained.

Physical state - solid needle type colourless crystal

yield - 12 gm

melting point - 93 °C

Experiment -12

Aim - To study the effect of a solute (e.g. NaCl) on the critical solution temperature of two partially miscible liquid (e.g. phenol-water system) and to determine the conc^n of the solute in the given phenol-water system.

Apparatus - Boiling tube, a thermometer, stirrer, 500ml beaker to be used as water bath, conical flasks, graduated pipette.

Chemicals - phenol, distilled water, NaCl.

Theory - phenol-water system has an upper critical solution temperature. If the composition (weight percentage) is plotted against temperature at which the two liquid become miscible, the curve shows a maximum. The temperature corresponding to the maximum of curve is known as critical solution temperature of phenol water system. The presence of impurity effect the co-solute point if the impurity is soluble in one of the liquid the value of co-solute point increases and if the impurity is soluble in both the liquids

the value of co-solute phenol water system point decreases, in addition of NaCl decreases.

Procedure -

Step I - To determine CST of phenol-water system

a - Nine clean of dry test tubes are taken and marked as 1, 2, 3 .... 7, 8, 9. The tube have phenol & water having the following composition.

1- phenol (9 gm) water (1 gm)
2 - phenol (8 gm) water (2 gm)
3 - phenol (7 gm) water (3 gm)
4 - phenol (6 gm) water (4 gm)
5 - phenol (5 gm) water (5 gm)
6 - phenol (4 gm) water (6 gm)
7 - phenol (3 gm) water (7 gm)
8 - phenol (2 gm) water (8 gm)
9 - phenol (1 gm) water (9 gm)

This gives concentration of phenol from 90 % to 10 %.

b - When the turbidity starts disappearing stop the heating & stir the solution continuously. Note the temperature (t_1) at which the turbidity completely disappear.

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Observation Table

(a) phenol - water system -

S. No	Mass of H_2O (gm)	Mass of Phenol (gm)	% Phenol	Temp. of disappearance of Turbidity (°C)	Temp. of reappearance of Turbidity (°C)	Average (°C)
1	1 gm	9 gm	90 %	35	19	27
2	2 gm	8 gm	80 %	63	15	39
3	3 gm	7 gm	70 %	66	36	51
4	4 gm	6 gm	60 %	76	44	60
5	5 gm	5 gm	50 %	79	43	61
6	6 gm	4 gm	40 %	65	63	64
7	7 gm	3 gm	30 %	60	62	61
8	8 gm	2 gm	20 %	57	53	55
9	9 gm	1 gm	10 %	42	27	34

c- Now, the water bath is slowly cooled and note the temperature (t_2) at which the turbidity starts reappearing.

d- Now, plot a graph taking average temperature $\left[\frac{t_1 + t_2}{2} = t\right]$ at y-axis & percentage of phenol at x-axis.

Observation - Phenol water system of CST = 64°C

Step -II · To determine CST of phenol salt solution

a- Nine clean & dry boiling test tubes (numbered 1 to 9) are taken. In these 1, 2, 3, 4, 5, 6, 7, 8, 9 gms of phenol of 9, 8, 7, 6, 5, 4, 3, 2, 1 gms of distilled water respectively are taken. This gives phenol of 10% - 90% conc^n.

b- Now add 0.1 gm of NaCl to each boiling tube

c- Close each of the boiling tube with a cork having two holes, through one a stirrer and through other a thermometer (least count 0.1°C) are attached.

(8) phenol - water + 0.1 g NaCl

S. No.	Mass of H_2O (gm)	Mass of Phenol (gm)	% phenol	Temp. of disappearance of Turbidity	Temp. of reappearance of Turbidity	Average (°C)
1	1 gm	9 gm	90 %	50	30	40
2	2 gm	8 gm	80 %	80	20	50
3	3 gm	7 gm	70 %	90	30	60
4	4 gm	6 gm	60 %	95	35	65
5	5 gm	5 gm	50 %	97	37	67
6	6 gm	4 gm	40 %	80	60	70
7	7 gm	3 gm	30 %	75	59	67
8	8 gm	2 gm	20 %	65	55	60
9	9 gm	1 gm	10 %	60	20	40

d- Now, keep the boiling tube in an air jacket and heat slowly with continuous stirring on a water bath. Note the temperature (t_1°C) when turbidity disappears. Now, cool the water bath slowly & note the temperature when the turbidity reappears.

e- plot a graph taking average temperature $\left[\frac{t_1 + t_2}{2} = t\right]$ at y-axis & percentage of phenol at x-axis.

Observation - CST of phenol + salt solution - 70°C

Result - Critical solution temperature (co-solute temperature) of phenol water -system on mixing -NaCl 70°C [T_2]
- critical solution temperature of phenol -water system - 64°C [T_1]

- Rise in critical solution temperature o
mixing NaCl - $T_2 - T_1$
- 70 - 64 °C
- 6°C

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Experiment -13

Aim - Determine the distribution weffiient of Iodine in organic and aqueous medium (at room temperature).

Apparatus - Beaker, conical flask, water bath, pipette, burette, burette stand, bottle, funnel

Chemical required - Iodine crystals
Organic solvent CCl_4
$Na_2S_2O_3$ = 0.5, 0.01N
10% of KI
Starch as indicator

Procedure - (i) Add iodine in 150 ml of CCl_4 solution. It is prepared saturated solution.

Saturated CCl_4	50ml	40ml	30ml	20ml
CCl_4	0ml	10ml	20ml	30ml
H_2O	200ml	200ml	200ml	200ml
	250ml	250ml	250ml	250ml
	SET-I	SET-II	SET-III	SET-IV

(ii) Organic layer - 5ml + 50ml (10% of KI) + starch titrate against $Na_2S_2O_3$ (0.01m)

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SET - I (aq.) $N_1V_1 = N_2V_2$

$C_{aq} \times 50 = 0.01 \times 0.8$

$C_{aq} = 0.00016$

(or) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.05 \times 1.2$

$C_{or} = 0.012$

$$K = \frac{C_{or}}{C_{aq}} = \frac{0.012}{0.00015} = 75$$

SET - II (aq) $N_1V_1 = N_2V_2$

$C_{aq} \times 50 = 0.01 \times 0.2$

$C_{aq} = 10^{-5} \times 4$

Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.05 \times 0.$

$C_{or} = 0.005$

$$K = \frac{0.005}{4 \times 10^{-5}} = 150$$

SET - III (aq.) $N_1V_1 = N_2V_2$

$C_{aq} \times 50 = 0.01 \times 0.4$

$C_{aq} = 8 \times 10^{-5}$

Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.05 \times 3.1$

$C_{or} = 0.031$

$$K = \frac{0.031}{8 \times 10^{-5}} = 387$$

SET - IV (aq) $N_1V_1 = N_2V_2$

$C_{aq} \times 50 = 0.01 \times 0.3$

$C_{aq} = 6 \times 10^{-5}$

(or) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.05 \times 8.4$

$C_{or} = 0.084$

$$K = \frac{0.084}{6 \times 10^{-5}} = 1400$$

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Aqueous layer - 50ml + 5ml (10% of KI) + starch titrate against $Na_2S_2O_3$ (0.5m)

(iii) Shake well for 20 minutes
(iv) Place bottle in water bath for 20 min.
(v) Till organic and aqueous layer separated well organic layer in bottom and aqueous layer in upper portion.
(vi) Separate both layers with glass funnels
(vii) Disappearance of blue colour indicate the end point of titration.

Observation Table -

	Aq. layer (0.01m)	readings	organic layer (0.05m)	readings
SET - I	55ml	0.8ml	25ml	1.2ml
SET - II	55ml	0.2ml	25ml	0.6ml
SET - III	55ml	0.4ml	25ml	3.1ml
SET - IV	55ml	0.3ml	25ml	5.4ml

Result - The distribution coefficient of Iodine in organic (CCl_4) & aqueous layer is -

for SET I - 75
SET II - 150
SET III - 387
SET IV - 1400

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Experiment -14

Aim - Study the distribution of benzoic acid between benzene & water.

Apparatus - pipette, beaker, burette stand, conical flask, funnel, bottle, water bath.

Chemical required - Benzoic acid
Benzene
NaOH (0.1N & 0.01N)
Phenopthalin as indicator

Theory - When the solute associate in one of the phases, say in benzene, the distribution coefficient is given by -

$$K = \sqrt[n]{\frac{C_{or}}{C_{aq.}}} = \frac{C_{or}^{1/n}}{C_{aq.}}$$

Where Cor & Caq. are the concentration of the acid in benzene & aq. layer, n is the number of molecules of acid which associate to form a complex molecule. The value of n can be determined by hit & trial method giving different values to n say 1, 2, 3, etc so as to obtain constant value of K.

Alternatively n can be determined in the following way.

Taking logarithm of above expression

$$\log C_{aq} = \frac{1}{n} \log C_{or} - \log k$$

A plot of log C_{aq} (Cordinate) against log C_{or} must be a straight line, the slope of which will be $\frac{1}{n}$ & the intercept on the ordinate axis $\log k$.

Procedure –

(i) Weight out about 1, 2, 3, 4 gm benzoic acid & transfer this amount to different stoppered bottles numbered as 1, 2, 3, 4 to each bottle add about 25 ml of pure benzene & about 25 ml of distilled water.

(ii) Stopper the bottles tightly & shake them vigorously for half an hours from time to time & allow them to find stand in a water bath at room temperature.

(III) The contents will separate in two layers. The lower layer will be the aqueous layer & upper one is benzene layer.

(IV) Remove 5ml of benzene layer from 1st bottle & delivered it into a conical flask containing about 10 ml distiled water. Titrate it with 0.1N NaOH using phenophthalin or indicator.

(V) During the titration the contents should be shaken vigorously in order ensure rapid & complete extraction of benzoic acid from benzene, make duplicate & triplicate titration.

(VI) Similarly titrate the benzene layer of other bottles.

(VII) By means of a separating funnel remove the lower aqueous layer of each bottle into separate dry conical flask.

(VIII) Titrate 10 ml of the aqueous layer of each bottle with 0.01N NaOH in the usual way.

SET I (Caq) $N_1V_1 = N_2V_2$

$C_{aq} \times 10 = 0.1 \times 03.7$

$C_{aq} = 0.037$

(Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.1 \times 32.1$

$C_{or} = 0.642$

$$k = \frac{C_{or}}{C_{aq}} = \frac{0.642}{0.037} = 17.3$$

SET - II (Caq) $N_1V_1 = N_2V_2$

$C_{aq} \times 10 = 0.1 \times 7.5$

$C_{aq} = 0.075$

(Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.1 \times 35.1$

$C_{or} = 0.702$

$$k = \frac{C_{or}}{C_{aq}} = \frac{0.702}{0.075} = 9.36$$

SET - III (Caq) $N_1V_1 = N_2V_2$

$C_{aq} \times 10 = 0.1 \times 9.7$

$C_{aq} = 0.097$

(Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.1 \times 35.4$

$C_{or} = 0.708$

$$k = \frac{0.708}{0.097} = 7.29$$

SET - IV (Caq) $N_1V_1 = N_2V_2$

$C_{aq} \times 10 = 0.1 \times 11.5$

$C_{aq} = 0.115$

(Cor) $N_1V_1 = N_2V_2$

$C_{or} \times 5 = 0.1 \times 36.7$

$C_{or} = 0.734$

$$k = \frac{0.734}{0.115} = 6.38$$

Observation Table -

	Bottle No.	Vol. of Benzene layer	Vol. of 0.1N NaOH
1	1	5ml	32.1 ml
2	2	5ml	35.1 ml
3	3	5ml	35.4 ml
4	4	5ml	36.7 ml

- Analysis of Aqueous layer

	Bottle No.	Vol. of Aq. layer	Vol. of 0.1N NaOH
1	1	10ml	3.7 ml
2	2	10ml	7.5ml
3	3	10ml	9.7ml
4	4	10ml	11.5ml

Bottle No.	Cor.	Caq.	$\frac{Cor}{Caq.}$
1	0.642	0.037	17.3
2	0.702	0.075	9.36
3	0.708	0.095	7.29
4	0.734	0.115	6.38

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Also plot the log C_{aq} values coordinate against log Cor. & from the straight line so obtained determine the value of n & K.

Result -

This ratio C_{or}/C_{aq} varies with conc indicating thereby the acid has different molecular state in the two phases on the other hand, the ratio C_{or}/C_{aq} is practically constant for different concentration. This shows that benzoic acid undergoes associatio in benzene with the formation of dimer i.e two molecules of acid combine to form an aggregate molecule. Verify this value obtained from the graph plotted as abo