B.Sc. Programme
(Chemistry Major)

Laboratory Manual

Gravimetric / Organic Analyses
&
Organic Preparations

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S. No.	**Object to be weighed**	**Weight in grams**
1	Empty silica crucible	
2	Silica crucible + Barium chloride crystals (with water of hydration)	
3	Silica crucible + Barium chloride after heating (without water of hydration)	

Calculation

Weight of empty crucible (w_1) = **a** g

Weight of crucible + barium chloride crystals (w_2) (with water of hydration) = **b** g

Weight of barium chloride crystals ($w_2 - w_1$) = **c** g

Weight of crucible + anhydrous barium chloride (w_3) = **d** g

Weight of anhydrous barium chloride ($w_3 - w_1$) = **e** g

Weight of water $[(w_2 - w_1) - (w_3 - w_1)]$ = **f** g

c g of barium chloride crystals contains **f** g of water

$\therefore$ 100 g of barium chloride crystals contains $\dfrac{\mathbf{f \times 100}}{\mathbf{c}}$ g of water

= **G** g

Percentage of water of hydration in the given barium chloride crystals = **G**

Ex. No. 1

Date :

Estimation of percentage of water of hydration in barium chloride crystals

Aim

To estimate the percentage of water of hydration in bariumchloride crystals.

Procedure

A clean dry silica crucible with lid is heated in an electrical incinerator for about 10 minutes. The crucible with the lid is removed and allowed to cool in a desiccator. After cooling the crucible is weighed with the lid accurately. About 2g of pure barium chloride crystals is placed in the crucible and weighed once again. Crucible with barium chloride crystals is heated for about 15 minutes and allowed to cool in a desiccator and weighed as before. Heating cooling and weighing are repeated to constant weight. From the weights of barium chloride crystals taken and anhydrous barium chloride obtained, percentage of water of hydration is calculated.

Result

The percentage of water of hydration in the given barium chloride crystals =

S. No.	**Object to be weighed**	**Weight in grams**	
		Crucible A	**Crucible B**
1	Empty sintered glass crucible		
2	Sintered glass crucible + barium chromate		
3	Barium chromate		
	Weight of barium chromate	**a**	**b**

Calculation

Molecular weight of barium chromate = 253.34

Atomic weight of barium = 137.34

Crucible A

253.34 g of barium chromate contains 137.34 g of Ba

a g of barium chromate contains $\frac{137.34 \times \mathbf{a}}{253.34}$

= **c** g of Ba

20 ml of the given barium solution contains **c** g of Ba

∴ 100 ml of the given barium solution contains $\frac{\mathbf{c} \times 100}{20}$

= **d** g of Ba

Crucible B

253.34 g of barium chromate contains 137.34 g of Ba

b g of barium chromate contains $\frac{137.34 \times \mathbf{b}}{253.34}$

= **e** g of Ba

20 ml of the given barium solution contains **e** g of Ba

∴ 100 ml of the given barium solution contains $\frac{\mathbf{e} \times 100}{20}$

= **f** g of Ba

Ex. No. 2

Date :

Estimation of barium as barium chromate

Aim

To estimate the weight of barium present in the whole of the given solution.

Procedure

The given solution of barium chloride is made upto 100ml in a standard flask with distilled water. 20 ml of this is pipetted out into a 400ml beaker provided with a watch glass and glass rod. The solution is diluted to 50ml and heated to boiling. About 5g of ammonium acetate crystals and 10ml of 1N acetic acid are added to the hot solution. 20 ml of 4% potassium chromate is then added drop by drop with constant stirring. The precipitate of barium chromate formed is digested over a steam bath and allowed to settle and tested for complete precipitation by adding a few more drops of potassium chromate to the supernatant liquid. The precipitate is filtered through a preheated and weighed sintered glass crucible using cold water as wash solution. The precipitate is dried in an electrical oven at 100°C, cooled and weighed. Heating, cooling and weighing are repeated to constant weight. A duplicate is also performed.

Result

The amount of barium present in the whole of the given solution is

A = **d** g ; B = **f** g

S. No.	Object to be weighed	Weight in grams	
		Crucible A	**Crucible B**
1	Empty silica crucible		
2	Silica crucible + barium sulphate		
3	Barium sulphate		
	Weight of barium sulphate	**a**	**b**

Calculation

Molecular weight of barium sulphate = 233.40

Atomic weight of barium = 137.34

Crucible A

233.40 g of barium sulphate contains 137.34 g of Ba

a g of barium sulphate contains $\frac{137.34 \times \mathbf{a}}{233.40}$

= **c** g of Ba

20 ml of the given barium solution contains **c** g of Ba

∴ 100 ml of the given barium solution contains $\frac{\mathbf{c} \times 100}{20}$

= **d** g of Ba

Crucible A

233.40 g of barium sulphate contains 137.34 g of Ba

b g of barium sulphate contains $\frac{137.34 \times \mathbf{b}}{233.40}$

= **e** g of Ba

20 ml of the given barium solution contains **e** g of Ba

∴ 100 ml of the given barium solution contains $\frac{\mathbf{e} \times 100}{20}$

= **f** g of Ba

Ex. No. 3

Date :

Estimation of barium as barium sulphate

Aim

To estimate the weight of barium present in the whole of the given solution.

Procedure

The given solution of barium chloride is made upto 100ml in a standard flask with distilled water. 20 ml of madeup solution is pipetted out into a clean 400ml beaker provided with a watch glass and a glass rod. The solution is diluted to 50ml with distilled water and acidified with 2ml of dilute hydrochloric acid and heated to boiling. To the hot solution added about 20ml of hot 5N H_2SO_4 very slowly with constant stirring. The precipitated barium sulphate is digested over a steam bath for half an hour and allowed to settle. Tested for complete precipitation by adding 2 drops of 5N sulphuric acid to the supernatant liquid in the beaker. Then the precipitate is filtered through whatmann No. 41 filter paper using hot water as wash solution. The filter paper along with the precipitate is incinerated in a preheated and weighed silica crucible. It is cooled and weighed. Heating, cooling and weighing are repeated to constant weight. A duplicate is also performed.

Result

The amount of barium present in the whole of the given solution is

A = **d** g ; B = **f** g

S. No.	Object to be weighed	Weight in grams	
		Crucible A	Crucible B
1	Empty sintered glass crucible		
2	Sintered glass crucible + lead chromate		
3	Lead chromate		
	Weight of lead chromate	**a**	**b**

Calculation

Molecular weight of lead chromate = 323.19

Atomic weight of lead = 207.19

Crucible A

323.19 g of lead chromate contains 207.19 g of lead

a g of lead chromate contains $\frac{207.19 \times \mathbf{a}}{323.19}$

= **c** g of lead

20 ml of the given lead solution contains **c** g of lead

$\therefore$ 100 ml of the given lead solution contains $\frac{\mathbf{c} \times 100}{20}$

= **d** g of lead

Crucible B

323.19 g of lead chromate contains 207.19 g of lead

b g of lead chromate contains $\frac{207.19 \times \mathbf{b}}{323.19}$

= **e** g of lead

20 ml of the given lead solution contains **e** g of lead

$\therefore$ 100 ml of the given lead solution contains $\frac{\mathbf{e} \times 100}{20}$

= **f** g of lead

Ex. No. 4

Date :

Estimation of lead as lead chromate

Aim

To estimate the weight of lead present in the whole of the given solution.

Procedure

The given solution is made upto 100ml in a standard flask with distilled water. 20 ml of this solution is pipetted out into a clean 400ml beaker provided with a watch glass and glass rod. The solution is diluted to about 50ml and is heated to boiling. To the hot solution 10ml of 4% potassium chromate solution is added drop by drop with constant stirring. The precipitate is digested over a water bath for half an hour and allowed to settle. The precipitate is then filtered through a preheated and weighed sintered glass crucible using hot water as wash solution. The precipitate is dried in an electric oven at 120°C for 45 minutes, cooled and weighed. Heating, cooling and weighing are repeated to constant weight. A duplicate is performed.

Result

The amount of lead present in the whole of the given solution is

A = _____**d**_____ g ; B = _____**f**_____ g

S. No.	Object to be weighed	Weight in grams	
		Crucible A	**Crucible B**
1	Empty silica crucible		
2	Silica crucible + lead sulphate		
3	Lead sulphate		
	Weight of lead sulphate	**a**	**b**

Calculation

Molecular weight of lead sulphate = 303.25

Atomic weight of lead = 207.19

Crucible A

303.25 g of lead sulphate contains 207.19 g of lead

a g of lead sulphate contains $\frac{207.19 \times \mathbf{a}}{303.25}$

= **c** g of lead

20 ml of the given lead solution contains **c** g of lead

∴ 100 ml of the given lead solution contains $\frac{\mathbf{c} \times 100}{20}$

= **d** g of lead

Crucible B

303.25 g of lead sulphate contains 207.19 g of lead

b g of lead sulphate contains $\frac{207.19 \times \mathbf{b}}{303.25}$

= **e** g of lead

20 ml of the given lead solution contains **e** g of lead

∴ 100 ml of the given lead solution contains $\frac{\mathbf{e} \times 100}{20}$

= **f** g of lead

Ex. No. 5

Date :

Estimation of lead as lead sulphate

Aim

To estimate the weight of lead present in the whole of the given solution.

Procedure

The given solution is made upto 100ml in a standard flask using distilled water. 20 ml of this is pipetted out into a clean 400ml beaker provided with a watch glass and glass rod and is diluted to about 50ml. This solution is heated to boiling and to this hot solution 20ml of dilute sulphuric acid is added in drops with constant stirring and then 40ml of alcohol. The precipitate is allowed to settle and filtered through Whatman 41 filter paper using 10% dilute alcohol as wash solution. The precipitate is dried. After drying, the precipitate is carefully removed from the filter paper and kept in a safe place. The filter paper alone is then incinerated in a preheated and weighed silica crucible. The crucible is cooled, a drop of concentrated sulphuric acid is added and heated again. It is cooled in a desiccator and then weighed along with the precipitate already removed from the filter paper. A duplicate is performed.

Result

The amount of lead present in the whole of the given solution is

A = **d** g ; B = **f** g

S. No.	**Object to be weighed**	**Weight in grams**	
		Crucible A	**Crucible B**
1	Empty sintered glass crucible		
2	Sintered glass crucible + Calcium oxalate monohydrate		
3	Calcium oxalate monohydrate		
	Weight of calcium oxalate monohydrate	**a**	**b**

Calculation

Molecular weight of calcium oxalate monohydrate = 146.08

Atomic weight of calcium = 40.08

Crucible A

146.08 g of calcium oxalate monohydrate contains 40.08 g of Ca

a g of calcium oxalate monohydrate contains $\frac{40.08 \times \mathbf{a}}{146.08}$

= **c** g of Ca

20 ml of the given calcium solution contains **c** g of Ca

∴ 100 ml of the given calcium solution contains $\frac{\mathbf{c} \times 100}{20}$

= **d** g of Ca

Crucible B

146.08 g of calcium oxalate monohydrate contains 40.08 g of Ca

b g of calcium oxalate monohydrate contains $\frac{40.08 \times \mathbf{b}}{146.08}$

= **e** g of Ca

20 ml of the given calcium solution contains **e** g of Ca

∴ 100 ml of the given calcium solution contains $\frac{\mathbf{e} \times 100}{20}$

= **f** g of Ca

Ex. No. 6

Date :

Estimation of calcium as calcium oxalate monohydrate

Aim

To estimate the weight of calcium present in the whole of the given solution.

Procedure

The given calcium solution is made upto 100ml in a standard flask, with distilled water. 20 ml of this is pipetted out into a clean 400ml beaker provided with a watch glass and glass rod. A drop of methyl organge is added to ensure the presence of acid. The solution is carefully neutralized with ammonium hydroxide and then 1ml of HCl is added. The solution is heated to boiling. To the hot solution about 20ml of hot saturated ammonium oxalate is added in drops with constant stirring and then ammonium hydroxide solution till it is alkaline. The precipitated calcium oxalate monohydrate is digested over a steam bath for half an hour and allowed to settle. The supernatant liquid is tested for complete precipitation. The precipitate is filtered through a preheated and weighed sintered glass crucible using 1% solution of ammonium hydroxide as wash solution. The precipitate is dried in an electric oven between 110°C and 120°C for 45 minutes cooled and weighed. Heating, cooling and weighing are repeated to constant weight. A duplicate is performed.

Result

The amount of calcium present in the whole of the given solution is

A = ____**d**____ g ; B = ____**f**____ g

S. No.	Object to be weighed	Weight in grams	
		Crucible A	Crucible B
1	Empty sintered glass crucible		
2	Sintered glass crucible + Nickel dimethylglyoximate		
3	Nickel dimethylglyoximate		
	Weight of Nickel dimethylglyoximate	**a**	**b**

Calculation

Molecular weight of Nickel dimethylglyoximate = 288.79

Atomic weight of Nickel = 58.71

Crucible A

288.79 g of Nickel dimethylglyoximate contains 58.71 g of Ni

a g of Nickel dimethylglyoximate contains $\frac{58.71}{288.79} \times \mathbf{a}$

= **c** g of Ni

20 ml of the given Nickel solution contains **c** g of Ni

∴100 ml of the given Nickel solution contains $\frac{\mathbf{c}}{20} \times 100$

= **d** g of Ni

Crucible B

288.79 g of Nickel dimethylglyoximate contains 58.71 g of Ni

b g of Nickel dimethylglyoximate contains $\frac{58.71}{288.79} \times \mathbf{b}$

= **e** g of Ni

20 ml of the given Nickel solution contains **e** g of Ni

∴100 ml of the given Nickel solution contains $\frac{\mathbf{e}}{20} \times 100$

= **f** g of Ni

Ex. No : 7

Date :

Estimation of Nickel as Nickel dimethylglyoximate

Aim

To estimate the amount of Nickel present in the whole of the given solution.

Procedure

The given solution is made upto 100ml in a standard flask using distilled water. 20 ml of this madeup solution is pipetted out into a clean 400ml beaker provided with a watch glass and glass rod. The solution is diluted to 50ml, 5ml of dilute hydrochloric acid is added and heated to boiling. To the hot solution 25ml of dimethylglyoxime is slowly added with constant stirring. To this solution required amount of ammonia solution is then added. The precipitate formed is digested over a steam bath and tested for complete precipitation. It is filtered through a preheated and weighed sintered glass crucible. The precipitate is washed with water containing ammonia solution and dried in an electric oven, cooled and weighed. Heating, cooling and weighing are repeated to constant weight. A duplicate is performed.

Result

The amount of Nickel present in the whole of the given solution is

A = **d** g ; B = **f** g

Determination of melting point of a solid

Aim

To determine the melting point of solid

Principle

A small amount of pure solid is finely powdered and taken in a capillary tube and heated in a bath containing concentrated sulphuric acid or liquid paraffin. The temperature of the bath when the compound melts is noted.

Apparatus

i. Thermometer (360°C)
ii. Capillary tubes
iii. Beaker (100ml)
iv. Stirrer
v. Stand

Procedure

A capillary tube of about 6 to 8 cm. length and about 1mm in diameter is taken and fused at one end by inserting into the edge of the flame. The solid whose melting point is to be determined is powdered well using a nickel spatula on a porcelain tile and then introduced into the capillary tube to get a column of the substance of length about 0.5 to 1cm. A 100ml beaker is filled three fourth with concentrated sulphuric acid (or) liquid paraffin. A glass strirrer is placed inside the bath. A 360°C thermometer is inserted into a one holed cork and clamped on an iron stand such that the bulb of the thermometer is well within sulphuric acid and is a little above the bottom of the beaker.

The capillary tube is carefully allowed to cling to the thermometer (by capillary action) so that the portion of the capillary tube with the substance is between the upper and lower ends of the bulb of the thermometer. The open end of the capillary tube must be above the surface of the liquid. The bath is gradually heated with constant stirring using the stirrer, without disturbing the capillary tube attached to the thermometer. The heating is regulated so that the temperature rises slowly. As the temperature rises, the solid in the capillary tube shrinks suddenly and melts. The temperature at which the solid just turns from opaque to transparent gives the melting point of the substance. The experiment is repeated to get concordant results.

Result

The melting point of the solid substance = °C

Determination of boiling point of a liquid

Aim

To determine the boiling point of a liquid

Principle

A small amount of the liquid is taken in a round bottomed flask with a side tube and fitted with a thermometer. The liquid in the round bottomed flask is heated slowly and the temperature at which the liquid boils is noted.

Apparatus

i. Round bottomed flask with a side tube
ii. Thermometer (110°C)
iii. Adapter
iv. Receiver
v. Stand
vi. Condenser

Procedure

The given liquid is taken to about one third of a round bottomed flask with side tube. To promote uniform heating, some porcelain pieces are introduced into it. The mouth of the flask is closed with mercury seal carrying the thermometer. The mercury seal is so arranged that the bottom of it is in level with the side tube of the round bottomed flask. The side tube of the apparatus is connected to a condenser. The condenser is attached to the adapter which in turn is introduced into a receiver. The flask is then slowly heated on a wire guaze. The temperature rises gradually and finally the liquid boils and the vapour escapes through the side tube and collects in the receiver in drops. The constant temperature at which the liquid distills steadily is the boiling point of the liquid. When two third of the liquid has been distilled away, heating is stopped. The apparatus is cooled. The experiment is repeated with the collected liquid to get concordant results.

Result

The boiling point of the given liquid = °C

ANALYSIS OF ORGANIC COMPOUNDS

S.No	Experiment	Observation	Inference
	Preliminary Reactions		
1	Colour and appearance	a. Colourless solid b. Coloured solid c. Colourless liquid d. Coloured liquid e. Yellow coloured liquid or solid	a. Presence of acid, carbohydrate, amide or anilide b. Presence of polyhydric phenol like resorcinol c. Presence of simple phenol, aldehyde or ketone d. Presence of amine or phenol e. Presence of nitro compound
2	The odour of the substance is observed	a. Fishy odour b. Fruity odour c. Pleasant odour d. Phenolic or carbolic odour e. Bitter almond smell f. No characteristic odour	a. Presence of amine b. Presence of ester c. Presence of ketone d. Presence of phenol e. Presence of aldehyde Absence of amine, ester, ketone, phenol and aldehyde

3	*Solubility Test*		
i.	Solubility in Water	a. Soluble in cold water b. Soluble in hot water c. Insoluble	a. Presence of carbohydrate or urea b. Presence of aromatic acid or phenol c. Absence of aromatic acid, carbohydrate, phenol and urea
	Note: If the substance is soluble in water, solubility in dilute hydrochloric acid test is not done		
ii.	Solubility in dilute hydrochloric acid	a. Soluble b. Insoluble	a. Presence of amine b. Absence of amine
4	*Test with litmus paper* Substance in water is tested with litmus paper	a. Blue litmus turns red b. Red litmus turns blue c. Neutral to litmus	a. Presence of acid or phenol b. Presence of amine c. Absence of acid, phenol and amine
5	**Test for aromatic / aliphatic compound**		
i.	Ignition test A little of the substance is burnt in a nickel spatula	a. The substance burns with a luminous sooty flame b. Substance burns with non-sooty flame or blue flame	a. Presence of aromatic compound b. Presence of aliphatic compound

ii.	Nitration test A mixture of conc. nitric acid and conc. sulphuric acid is prepared. This is added to a little of the substance, heated in a water bath and poured into a beaker containing water	a. Yellow or brown solution / precipitate is obtained b. A colourless solution is obtained	a. Presence of aromatic compound b. Presence aliphatic compound
6	**Test for Saturation / Unsaturation**		
i.	A little of the Substance is treated with bromine water and shaken well	a. Decolourisation takes place without evolution of HBr b. Decolourisation takes place with the formation of precipitate c. No decolourisation takes place	a. Presence of unsaturated compound b. Presence of saturated but easily brominated compound like amine or phenol c. Presence of saturated compound
ii.	A little of the substance is treated with a dilute solution of potassium permanganate	a. Decolourisation takes place b. Slow decolourisation takes place c. No decolourisation takes place	a. Presence of unsaturated compound b. Easily oxidisable saturated compounds like aldehyde, ketone, phenol or amine c. Presence of saturated compound

7	**Test for elements** **Preparation of sodium fusion extract (Lassaigne's extract)** A dry piece of sodium metal is heated in a dry fusion tube till it melts, cooled and a little of the substance is added. It is then heated first gently and then strongly till the bottom of the fusion tube becomes red hot and plunged into 10ml of distilled water taken in a china dish, stirred well, concentrated the solution to 5 ml and filtered (the filtrate should be colourless). The filtrate is known as sodium fusion extract or Lassaigne's extract. The following tests are performed with the filtrate.		
i	*Test for Nitrogen* To one portion of the extract, a strong solution of ferrous sulphate is added. The solution is boiled and acidified with dilute HCl	a. A green or blue colour solution is got b. No green or blue colour solution is got	a. Presence of nitrogen b. Absence of nitrogen
ii	*Test for halogens* To another portion of the extract, dilute HNO_3 is added, boiled, cooled well and then $AgNO_3$ solution is added	a. Curdy white precipitate completely soluble in NH_4OH is got b. Pale yellow precipitate sparingly soluble in NH_4OH is got c. Deep yellow precipitate insoluble in NH_4OH is got	a. Presence of chlorine b. Presence of bromine c. Presence of iodine

iii	*Test for sulphur* To another portion of the extract, sodium nitroprusside solution is added	a. Violet colour solution is got b. No violet colour solution is got	a. Presence of sulphur b. Absence of sulphur
8	*Action with H_2SO_4* A little amount of the substance is treated with a little of conc. H_2SO_4 and warmed slowly	a. Substance gets charred on warming with the smell of burnt sugar. b. No characteristic reaction is observed	a. Presence of carbohydrate b. Absence of carbohydrate
9	*Action of NaOH*		
i	Substance is treated with NaOH solution and acidified with conc.HCl	a. Substance dissolves in NaOH and reappears on acidification b Substance does not dissolve in NaOH in cold	a. Presence of acid or phenol b. Absence of acid or phenol

ii	Substance is heated with NaOH solution	a. NH_3 gas is evolved and a white precipitate is got on acidification b. NH_3 gas is evolved and no white precipitate is got on acidification c. Oily drops are seen on the sides of the test tube d. No characteristic reaction	a. Presence of aromatic amide b. Presence of aliphatic amide c. Presence of anilide d. Absence of aromatic and aliphatic amides and anilide
10	*Sodium bicarbonate test*		
	A little of the substance in water is treated with saturated solution of sodium bicarbonate	a. Brisk effervescence is noted b. No brisk effervescence is noted	a. Presence of carboxylic acid b. Absence of carboxylic acid
11	*Neutral ferric chloride test*		
	Substance is treated with a drop of neutral ferric chloride and mixed well with water.	a. A green colour solution is got b. Violet colour is got c. Puff coloured precipitate is got d. No characteristic reaction	a. Presence of amine b. Presence of phenol c. Presence of acid d. Absence of amine, phenol and acid

12	*Borshe's reagent test*		
	To the substance in alcohol, Borshe's reagent and 1 ml of conc. HCl are added and heated in a water bath	a. Red orange precipitate is got b. No red orange precipitate is got	a. Presence of aldehyde or ketone b. Absence of aldehyde and ketone
13	*Tollen's test*		
	To the substance in alcohol, Tollen's reagent is added	a. A black precipitate or silver mirror is get b. No black precipitate or silver mirror	a. Presence of aldehyde, polyhydric phenol or reducing sugar b. Absence of aldehyde, polyhydric phenol and reducing sugar
14	Fehling's solution test		
	One ml of Fehling solution A is mixed with 1 ml of Fehling solution B. The mixture is added to a little of the substance dissolved in alcohol, shaken well and heated in a boiling water bath	a. A red brown precipitate is formed b. No red brown precipitate	a. Presence of aldehyde, polyhydric phenol or reducing sugar b. Absence of aldehyde, polyhydric phenol or reducing sugar

PRELIMINARY REPORT

The given organic substance is aliphatic / aromatic, saturated / unsaturated, containing the characteristic element ----------- / no characteristic element. The expected functional group is -----------------.

	TESTS FOR FUNCTIONAL GROUPS		
	Tests for compounds which does not contain nitrogen		
I	**Tests for carboxylic acids**		
i	*Phenolphthalein test*		
	One ml of dilute NaOH is taken in a test tube and a drop of phenolphthalein indicator is added to it. The solution turns to pink colour. To this solution a small amount of the substance is added and shaken well.	Pink colour is disappeared	Presence of acid
ii	*Esterification reaction*		
	A little of the substance is shaken with a few drops of alcohol and two drops of conc. H_2SO_4. The mixture is heated for 15 minutes in a water bath and the content is poured into a dilute solution of sodium carbonate contained in 100 ml beaker.	A fruity odour is noted	Presence of acid is confirmed

iii	*Phthalein fusion test(Fluorescein test) for dicarboxylic acid*		
	A little of the substance is heated with resorcinol and conc.H_2SO_4, cooled and an excess of water is added. The solution is added to sodium hydroxide solution contained in a beaker	a. Yellowish green fluorescein is produced b. No yellowish green fluorescein is produced	a. Presence of dicarboxylic acid c. Absence of dicarboxylic acid
	Derivative for acid		
	Anilide or para toluidide derivative The substance is mixed with an excess of aniline or para toluidine in a dry test tube and hearted for 30 mintues in a water bath It is then poured into a beaker containing dil. HCl and cooled to get the crystalline solid anilide or para toluidide.The solid derivative is filtered and dried		
II.	**Tests for phenol**		
i	*Libermann's test*		
	To a small amount of the substance, 5 drops of H_2SO_4 are added followed by a few crystals of $NaNO_2$ and heated. The contents are poured into a little of water taken in a beaker. To this solution, a dilute sodium hydroxide solution is added in slight excess	Red colour forms which turns blue or green on adding NaOH	Presence of phenol is confirmed

ii	*Phthalein fusion test*		
	A little of the substance is mixed with a little of puthalic anhydride in a dry test tube. To this mixture 2 drops of conc. H_2SO_4 is added, heated and poured into a 100ml beaker containing water. To this solution, a dilute sodium hydroxide solution is added in slight excess	a. Pink colour solution is got b. Yellowish green fluorescein is got	a. Presence of monohydric phenol b. Presence of polyhydric phenol
	Derivative for phenol		
i	***Bromo derivative*** A small amount of the substance is shaken well with saturated bromine water to give a crystalline soild of bromo derivative . It is poured into water , filtered and dried.		
ii	***Benzoyl derivative*** A small amount of the substance is mixed with dilute NaOH. Benzoyl chloride is added little by little with vigorous shaking. NaOH solution is again added and shaken well until the smell of benzoyl choride disappears . It is poured into water and the solid obtained is filtered and dried.		
III	**Test for carbohydrates**		
	Molisch's test A little of the substance in water is mixed with a few drops of an alcoholic solution of α-napthol. To the mixture conc.H_2SO_4 is added along the sides of the test tube.	A violet ring is formed at the bottom of the test tube	Presence of carbohydrate is confirmed

Derivative for carbolydrate			
	Osazone derivative for reducing sugar One part of reducing sugar, 2 parts of phenylhydrazine hydrochloride and 3 parts of sodium acetate are taken in a test tube and dissolved in minimum amount of water and heated in a water bath with repeated shaking. The yellow precipitate of osazone obtained is filtered and dried.		
	Osazone derivative for non-reducing sugar One part of non-reducing sugar is warmed with dil. HCl. After warming, 2 parts of phenlhydrazine hydrochloride and 3 parts of sodium acetate are added and heated in a water bath with repeated shaking. The yellow precipitate of osazone obtained is filtered and dried.		
IV	**Tests for aldehydes and ketones**		
i	*Sodium bisulphite test*		
	Sodiumbisulphite solution is added to a little of the substance and shaken well	White precipitate is got	Presence of aldehyde or ketone
ii	*Phenylhydrazine test*		
	A little of the substance is dissolved in alcohol and to this solution phenyl hydrazine hydrochloride and sodium acetate are added and heated in a water bath	White precipitate is got	Presence of aldehyde or ketone

iii	*Semicarbazide test*		
	A little of the substance is dissolved in alcohol and to this solution semicarbazide hydrochloride and sodium acetate are added and heated in a water bath	White precipitate is got	Presence of aldehyde or ketone
Iv	A little of the substance in alcohol is shaken with a little of meta dinitrobenzene and then added a small amount of dilute sodium hydroxide solution	Violet colour solution is got	Presence of ketone is confirmed
V	A little of the substance in water is mixed a few drops of freshly prepared solution of sodium nitroprusside followed by 4 drops of 10% solution of NaOH	Wine red colour is formed	Presence of ketone is confirmed
	Derivative of aldehydes and ketones		
a	***Semicarbazone derivative***		
	A small amount of the substance in dilute alcohol is mixed with a solution of semicarbazide hydrochloride and a few crystals of sodium acetate. The mixture is heated in a water bath and then cooled to give a crystalline soild semicarbazone derivative which is filtered and dried.		

b	***Phenylhydrazone derivative***		
	A small amount of the substance is mixed with an equal amount of phenylhydrazine hydrochloride and boiled for 5 minutes. Then 1ml of alcohol is added followed by the addition of water drop by drop to get a crystalline phenylhydrazone derivative which is filtered and dried.		
c	***Borsche's derivative (2,4-dinitrophenylhydrazone derivative)***		
	A small amount of the substance in alcohol is mixed with 2 ml of Borsche's reagent and 1ml of conc.HCl. The mixture is heated in a water bath and the crystalline red orange precipitate formed is filtered and dried		
	Tests for compounds containing nitrogen		
V	*Test for amine* *Dyetest* A small amount of the substance is dissolved in dilute hydrochloric acid, cooled and to this a cold solution of $NaNO_2$ is added slowly with constant stirring. This mixture is added to β-naphthol in NaOH solution which is also previously cooled	Red orange dye is got	Presence of primary amine is confirmed

	Derivatives of amine		
a	***Bromo derivative***		
	A small amount of the substance is shaken well with a saturated solution of bromine water and poured into a beaker containing water. The precipitate obtained is filtered and dried.		
b	***Benzoyl derivative***		
	A small amount of the substance is mixed with 1 ml of 1% NaOH solution . Benzoyl chloride is added drop by drop with vigorous shaking. Sodium hydroxide solution is again added and shaken well until the smell of benzoyl chloride disappears. It is then poured into water and the solid derivative thus obtained is filtered and dried.		
VI	*Test for aromatic amide*		
	Hydrolysis A little of the substance is boiled with a strong solution of NaOH. If ammonia gas is evolved, the solution is cooled and acidified with conc. HCl	a. Ammonia gas is evolved b. No ammonia gas is evolved c. A white precipitate is formed d. No white precipitate	a. Presence of amide b. Absence of amide c. Presence of aromatic amide d. Absence of aromatic amide
	Derivative of aromatic amide		
	Acid derivative		
	About one gram of the substance is mixed with strong solution of NaOH, heated, cooled and acidified with conc. HCl. The solid derivative obtained is filtered and dried.		

VII	*Test for aliphatic amide*		
i	*Biuret test*		
	A little of the substance is heated in a dry test tube till no more NH_3 gas is evolved. The sublimate thus obtained is cooled and dissolved in 10% NaOH and then 1 or 2 drops of very dilute $CuSO_4$ solution is added	A violet colour is formed	Presence of aliphatic diamide is confirmed
	Derivative of aliphatic diamide		
i	***Nitrate derivative***		
	A saturated solution of the substance in water is mixed with conc.HNO_3 and stirred well. The solid nitrate of diamide formed is filtered and dried.		
ii	***Oxalate derivative***		
	A saturated solution of the substance in water is mixed with a saturated solution of oxalic acid and stirred well. The soild oxalate of diamide obtained is filtered and dried.		
VII	*Test for anilide*		
	Dye test A small amount of the substance is boiled with Conc. HCl and cooled and diluted with water. To this solution, cold solution of $NaNO_2$ is added slowly with constant stirring. This mixture is then added to β-naphthol in NaOH which is also previously cooled.	Red orange dye is got	Presence of anilide

	Derivative of anilide ***Bromo derivative***
	A small amount of the substance in glacial acetic acid is shaken well with a solution of bromine in acetic acid . It is then poured into cold water to get a crystalline solid bromo derivative. It is filtered and dried.

Report

The given organic compound is

(i) Aromatic (or) Aliphatic ---

(ii) Saturated (or) unsaturated ---

(iii) Characteristic element present --

(iv) Functional group present --

(v) Derivative --

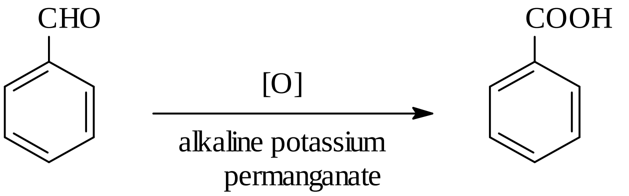
OXIDATION

Preparation of benzoic acid from benzaldehyde

Chemicals Required

i.	Benzaldehyde	2.5ml
ii.	Sodium carbonate	2.5g
iii.	Potassium permanganate	3g

Reaction



Benzoic acid

Procedure

About 2.5g of sodium carbonate is dissolved in about 20ml of water in a round bottom flask. Benzaldehyde (2.5ml) and a few porcelain bits are then added to it. The flask is fitted with a water condenser and heated over the wire gauze. When the mixture begins to boil, a saturated solution of 3g of potassium permanganate is added to the flask from the top of the condenser till the solution in the flask remains pink in colour. The contents are refluxed for one hour and then cooled. It is then transferred to a beaker and solid sodium metabisulphite is added to the beaker followed by dilute sulphuric acid till all the product of manganese dioxide dissolves and the solution becomes acidic. The precipitate of benzoic acid is filtered and dried. A portion of the sample is recrystallised from hot water.

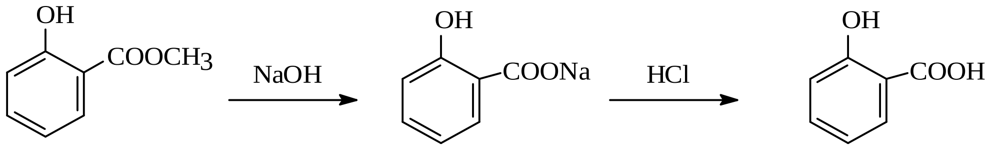
HYDROLYSIS

Preparation of salicyclic acid from methyl salicylate

Chemicals Required

i.	Methyl salicylate	2.5ml
ii.	Sodium hydroxide	15ml

Reaction



Methyl Salicylate Sodium Salicylate Salicylic acid

Procedure

Methyl salicylate (2.5ml), 15ml of sodium hydroxide and a few porcelain bits are taken in a round bottom flask. The flask is fitted with water condenser and the contents are boiled till the oily drops are not seen. The contents are cooled and transferred into a beaker. Concentrated hydrochloric acid is added to the solution in small quantities with vigorous stirring till the solution is acidic. Salicylic acid is precipitated and the contents are cooled well. The precipitate is filtered and washed with water. A portion of the sample is recrystallised from hot water.

NITRATION

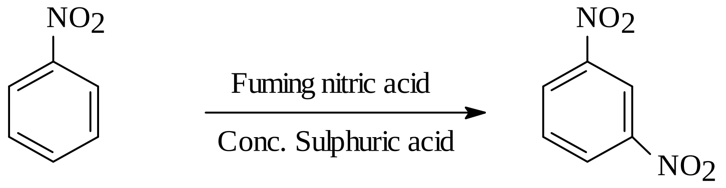
Preparation of m-dinitrobenzene from nitrobenzene

Chemicals Required

i.	Nitrobenzene	5.0ml
ii.	Fuming nitric acid	6ml
iii.	Concentrated sulphuric acid	3.5ml.

Reaction

m-dinitrobenzene is prepared by the nitration of nitrobenzene



Nitrobenzene m-Dinitrobenzene

Nitration is effected by fuming nitric acid in the presence of concentrated sulphuric acid.

Procedure

Fuming nitric acid is (6 ml) taken in a round bottom flask and 7ml of concentrated sulphuric acid is added little by little carefully. The flask is cooled during addition. Nitrobenzene (5ml) is then added in small quantitites to the nitrating mixture. Shaken well and heated for about 45 minutes, by immersing in a boiling water bath. The contents of the flask are then poured into water contained in a beaker. The mixture is stirred well. m-Dinitrobenzene separated as solid, is filtered off at the pump, washed with water and dried. A portion of the sample is recrystallised from alcohol.

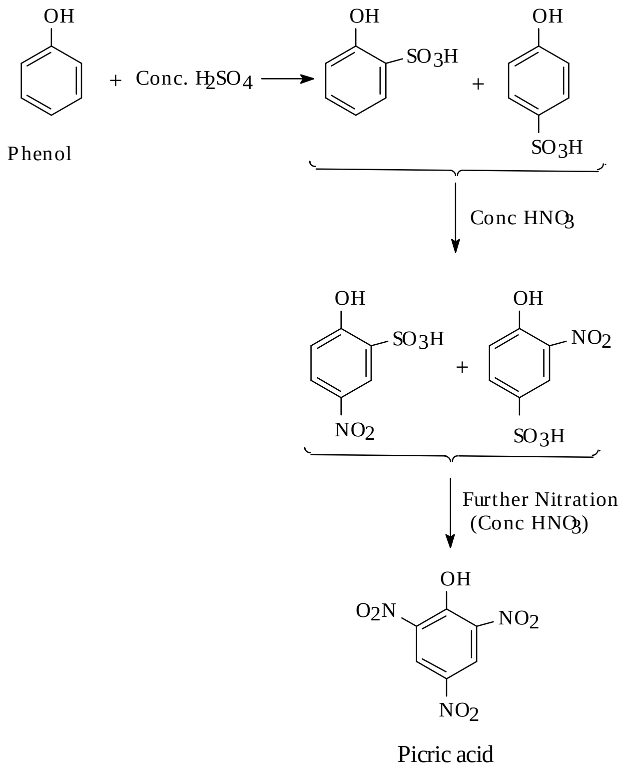
NITRATION

Preparation of picric acid from phenol

Chemicals Required

i.	Phenol	2.5ml
ii.	Concentrated nitric acid	7.5ml
iii.	Concentrated sulphuric acid	7.5ml

Reaction



Procedure

Phenol (2.5 ml) is taken in a dry china dish and 7.5ml of concentrated sulphuric acid is added to it. The mixture is homogenized by heating on a boiling water bath. It is cooled well and taken in a round bottom flask and 7.5ml of concentrated nitric acid is added in drops carefully with constant stirring. Reddish brown fumes are evolved during addition. When the reaction subsides, a few porcelain bits are added and the round bottom flask is fitted with an air condenser. The contents are heated in a boiling water bath for about two hours and then cooled. A portion of picric acid is recrystallised from hot water.

BROMINATION

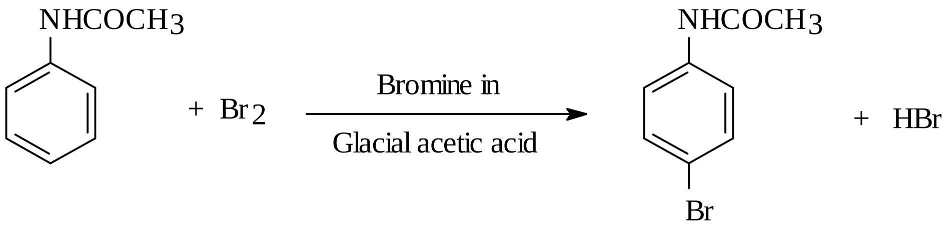
Preparation of parabromoacetanilide from acetanilide

Chemicals Required

i.	Acetanilide	2g
ii.	Glacial acetic acid	7ml
iii.	Bromine in glacial acetic acid	5ml.

Reaction

When acetanilide is treated with bromine in glacial acetic acid and diluted with water, p-bromo acetanilide is produced.



Acetanilide p-bromoacetanilide

Procedure

Acetanilide (2g) is dissolved in 7ml of glacial acetic acid in a conical flask fitted with a cork. The solution of bromine in glacial acetic acid is added in small quantities from a burette with vigorous shaking. After the addition of bromine in glacial acetic acid, the mixture is left as such for 15 minutes and about 100ml of water is added to precipitate p-bromoacetanilide. It is then filtered off at a pump, washed with water and dried. A portion of the sample is recrystallised from dilute alcohol.

BROMINATION

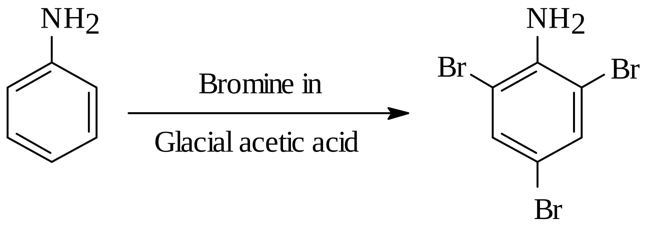
Preparation of symmetrical tribromoaniline from aniline

Chemicals Required

i.	Aniline	4ml
ii.	Glacial acetic acid	7ml
iii.	Bromine in glacial acetic acid	

Reaction

When Aniline is treated with bromine in glacial acetic acid and diluted with water, symmetrical tribromoaniline is produced.



Aniline 2,4,6-tribromoaniline

Procedure

Aniline (4ml) is dissolved in 7ml of glacial acetic acid in a conical flask fitted with a cork. The solution of bromine in glacial acetic acid is added in small quantities from a burette with vigorous shaking until the colour of the solution becomes yellow. After the addition of bromine in glacial acetic acid, the mixture is left as such for 15 minutes and about 100ml of water is added to precipitate symmetrical tribromoaniline. It is then filtered off at a pump, washed with water and dried. A portion of the sample is recrystallised from dilute alcohol.

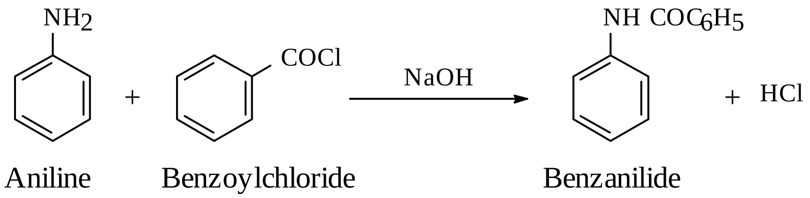
BENZOYLATION

Preparation of benzanilide from aniline

Chemicals Required

i.	Aniline	5ml
ii.	Benzoyl chloride	5ml
iii.	10% sodium hydroxide	50ml

Reaction



Procedure

Aniline (5ml) is taken in a conical flask fitted with a cork. About 5ml of sodium hydroxide and 0.5ml of benzoyl chloride are added to the flask. The flask is corked well and the contents are shaken vigorously. After cooling, again 5ml of sodium hydroxide and 0.5ml benzoylchloride are added and shaken well. The process is repeated till all the benzoyl chloride is added. If the mixture smells that of benzoylchloride, some more sodium hydroxide must be added. The white precipitate of benzanilide formed is filtered and dried. A portion of the sample is recrystallised from alcohol.

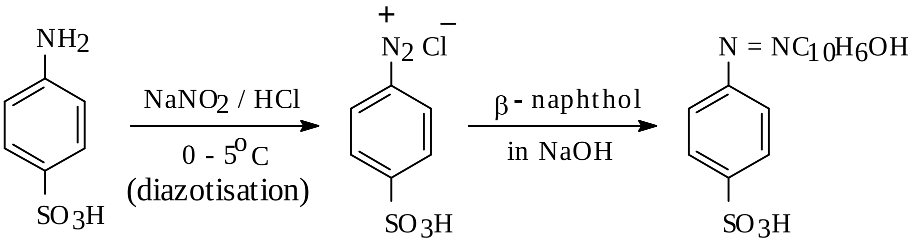
DIAZOTISATION

Preparation of beta-naphthol dye from para sulphanilic acid

Chemicals Required

i.	p-Sulphanilic acid	2g
ii.	Sodium carbonate	1g
iii.	Sodium nitrite	1g
iv.	Concentrated hydrochloric acid	5ml
v.	β-Naphthol	1g
vi.	Sodium hydroxide	10ml

Reaction



p - sulphanilic acid

β- Naphthol dye
(Red orange)

Procedure

Sodium carbonate is dissolved in water and taken in a 250ml beaker. p-Sulphanilic acid is added to sodium carbonate solution and warmed to get a clear solution and then precooled sodium nitrite solution is added and the contents are cooled in a freezing mixture. When the temperature is 5°C, cold concentrated hydrochloric acid is added in drops, taking care that the temperature is not raising above 5°C. This diazotised solution is added to β-naphthol in sodium hydroxide solution with constant stirring. A red orange dye is obtained.

PREPARATION OF REAGENTS

1. Phenolphthalein

5g of phenolphthalein is dissolved in 500ml of alcohol. Then 500ml of water is added with constant stirring. Any precipitate formed is filtered off and the clear filtrate is used.

2. Methyl Orange

0.5g of the free acid or the sodium salt is dissolved in a litre of cold water. Any precipitate formed is filtered off and the clear filterate is used.

3. Borsche's reagent

1 gram of 2.4 dinitrophenylhydrazine is refluxed with 100ml of methyl alcohol till dissolved.

4. Fehling's Solution A

6.9g of copper sulphate crystals are dissolved in 40ml of water and diluted to 100ml.

5. Fehling's Solution B

15g of Sodium hydroxide and 36g Sodium potassium tartrate (Rochelle Salt) are dissolved separately in 30ml of water each, mixed and diluted to 100ml after cooling. Equal volume of A & B are mixed before use.

6. Molisch's reagent

10g of β-naphthol is dissolved in 100ml of rectified spirit.

7. Neutral ferric chloride

About 1g of ferric chloride is dissolved in 100ml water. Sodium carbonate solution is added little by little to the above solution till the slight turbidity persists even after shaking. The precipitate is filtered off and the filterate is used as neutral ferric chloride solution.

8. $KMnO_4$ solution

1gm of potassium permanganate solid is dissolved in 100ml of distilled water.

9. Tollen's reagent

About one ml of silver nitrate solution is taken in a test tube. Two drops of NaOH is added and then dil. NH_4OH is added drop by drop until the precipitate is just redissolved.
