# +2 PRACTICALS

## I-ORGANIC QUALITATIVE ANALYSIS

| S.no | Experiment | Observation | Inference |
|---|---|---|---|
| | **Preliminary tests** | | |
| 1 | **Odour:**<br>Note the Odour of the organic compound. | (i) Fish odour<br>(ii) Bitter almond odour<br>(iii) Phenolic odour<br>(iv) Pleasant fruity odour | (i) May be an amine<br>(ii) May be benzaldehyde<br>(iii) May be phenol<br>(iv) May be an ester |
| 2 | **Test with litmus paper:**<br>Touch the Moist litmus paper with an organic compound. | (i) Blue litmus turns red<br>(ii) Red litmus turns blue<br>(iii) No colour change is noted | (i) May be a carboxylic acid or phenol<br>(ii) May be an amine<br>(iii) Absence of carboxylic acid , phenol and amine |
| 3 | **Action with sodium bicarbonate:**<br>Take 2 ml of saturated sodium bi carbonate solution in a test tube. Add 2 or 3 drops (or a pinch of solid) of an organic compound to it. | (i) Brisk effervescence<br>(ii) No brisk effervescence | (i) Presence of a carboxylic acid.<br>(ii) Absence of a carboxylic acid. |
| 4 | **Action with Borsche's reagent:**<br>Take a small amount of an organic compound in a test tube. Add 3 ml of Borsche's reagent, 1 ml of Conc HCl to it, then warm the mixture gently and cool it. | yellow or orange or red precipitate | Presence of an aldehyde or ketone |

| | | | |
|---|---|---|---|
| 5 | **Charring test:**<br>Take a small amount of an organic compound in a dry test tube. Add 2 ml of conc $H_2SO_4$ to it, and heat the mixture. | Charring takes place with smell of burnt sugar | Presence of carbohydrate |
| | **Tests for Aliphatic or Aromatic nature:** | | |
| 6 | **Ignition test:**<br>Take small amount of the organic compound in a Nickel spatula and burn it in Bunsen flame. | (i) Burn with sooty flame<br>(ii) Burns with non sooty flame | (i) Presence of an aromatic compound<br>(ii) Presence of an aliphatic compound |
| | **Tests for an unsaturation:** | | |
| 7 | **Test with bromine water:**<br>Take small amount of the organic compound in a test tube add 2 ml of distilled water to dissolve it. To this solution add few drops of bromine water and shake it well. | (i) Orange - yellow colour of bromine water is decolourised<br>(ii) No Decolourisation takes place<br>(iii) Decolourisation with formation of white precipitate. | (i) Substance is unsaturated.<br>(ii) Substance is saturated.<br>(iii) Presence of an aromatic amine or phenol. |
| 8 | **Test with $KMnO_4$ solution:**<br>Take small amount of the organic compound in a test tube add 2 ml of distilled water to dissolve it. To this solution add few drops of very dilute alkaline $KmnO_4$ solution and shake it well. | (i) Pink colour of $KmnO_4$ solution is decolourised<br>(ii) No Decolourisation takes place | (i) Substance is unsaturated.<br>(ii) Substance is unsaturated. |
| | **TEST FOR SELECTED ORGANIC FUNCTIONAL GROUPS** | | |
| | **Test For Phenol** | | |
| 9 | **Neutral $FeCl_3$ test:**<br>Take 1 ml of neutral ferric chloride solution is taken in a dry clean test tube. Add 2 or 3 drops (or a pinch of solid) oforganic compound to it. If no colouration occurs add 3 or 4 drops of alcohol. | (i) Violet colouration is seen<br>(ii) violet – blue colouration is seen<br>(iii) green colouration is seen | (i) Presence of phenol.<br>(ii) Presence of α–naphthol<br>(iii) Presence of β– naphthol |

| | | | |
|---|---|---|---|
| | TEST FOR CARBOXYLIC ACIDS | | |
| **10** | **Esterification reaction:**<br>Take 1 ml (or a pinch of solid) of an organic compoundin a clean test tube. Add 1 ml of ethyl alcohol and 4 to 5 drops of conc. sulphuric acid to it. Heat the reaction mixture strongly for about 5 minutes. Then pour the mixture into a beaker containing dil. Sodium carbonate solution and note the smell. | A Pleasant fruity odour is noted. | Presence of carboxylic group. |
| | **Test for aldehydes.** | | |
| **11** | **Tollen's reagent test:**<br>Take 2 ml of Tollen's reagent in a clean dry test tube. Add 3-4 drops of an organic compound (or 0.2 g of solid) to it, and warm the mixture on a water bath for about 5 minutes. | Shining silver mirror is formed. | Presence of an aldehyde |
| **12** | **Fehling's test:**<br>Take 1 ml each of Fehling's solution A and B are taken in a test tube. Add 4-5 drops of an organic compound (or 0.2g of solid) to it, and warm the mixture on a water bath for about 5 minutes. | Red precipitate is formed. | Presence of an aldehyde |
| | **Test for ketones** | | |
| **13** | **Legal's test:**<br>A small amount of the substance is taken in a test tube. 1 ml sodium nitro prusside solution is added. Then sodium hydroxide solution is added dropwise. | Red colouration. | Presence of a ketone. |
| | **Test for an amine.** | | |

| | | | |
|---|---|---|---|
| 14 | **Dye test:**<br>Take A small amount of an organic substance in a clean test tube, add 2 ml of HCl to dissolve it. Add few crystals of $NaNO_2$, and cool the mixture in ice bath. Then add 2 ml of ice cold solution of β-naphtholin NaOH. | Scarlet red dye is obtained. | Presence of an aromatic primary amine |
| | **Test for diamide** | | |
| 15 | **Biuret test:**<br>Take A small amount of an organic compound in a test tube. Heat strongly and then allow to cool. Dissolve the residue with 2 ml of water. To this solution Add 1 ml of dilute copper sulphate solution and few drops of 10% NaOH solution drop by drop. | Violet colour is appeared. | presence of a diamide |
| | **Test for carbohydrates** | | |
| 16 | **Molisch's test:**<br>Take A small amount of an organic compound in a test tube. It is dissolved in 2 ml of water. Add 3-4 drops of alpha naphthol to it.Then add conc $H_2SO_4$ through the sides of test tube carefully. | Violet or purple ring is formed at the junction of the two liquids. | Presence of carbohydrate |
| 17 | **Osazone test:**<br>Take A small amount of an organic compound in a test tube. Add 1 ml of phenyl hydrazine solution and heat the mixture for about 5 minutes on a boiling water bath. | Yellow crystals are obtained | Presence of carbohydrate |

## Report:

The given organic compound contains /is

(i) Aromatic / aliphatic

(ii) Saturated / unsaturated

(iii) _____ functional group

## List of organic compounds for analysis:

1. Benzaldehyde
2. Cinnamaldehyde
3. Acetophenone
4. Benzophenone
5. Benzoic acid
6. Cinnamic acid
7. Urea
8. Glucose
9. Aniline
10. Salicylic acid

## REASONING

### 3. Action with sodium bicarbonate:

Carboxylic acids react with sodium bi carbonate and liberate $CO_2$. Evolution of carbon dioxide gives brisk effervescence.

$$2R\text{-}COOH + 2NaHCO_3 \longrightarrow 2R\text{-}COONa + CO_2 \uparrow + H_2O$$

### 4. Action with Borsches reagent:

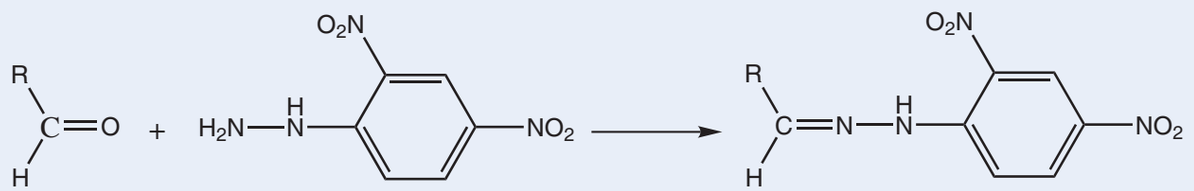
Borsches reagent is prepared by dissolving 2,4-dinitrophenylhydrazine in a solution containing methanol and little of conc sulphuric acid.

Aldehydes and ketones react with borsches reagent to form yellow, orange or red precipitate (dinitro phenylhydrazone)

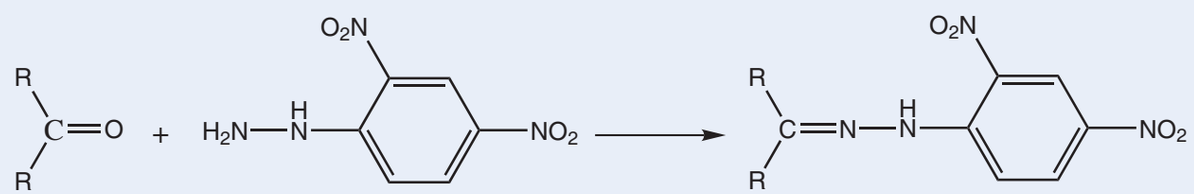
Aliphatic carbonyl compounds give deep yellow precipitate.

Aromatic carbonyl compounds give red precipitate.

2,4-dinitrophenyl hydrazine can be used to qualitatively detect the carbonyl group of an aldehyde or ketone. A positive result is indicated by the formation of an yellow or orange-red precipitate of 2,4-dinitrophenyl hydrazone.



Aldehyde + 2,4 dinitrophenylhydrazine → Aldehyde 2,4 dinitrophenylhydrazone (Yellow or orange)



Ketone + 2,4 dinitrophenylhydrazine → Ketone 2,4 dinitrophenylhydrazone (Yellow or orange or red)

### 5.Charring test:

When carbohydrates are treated with concentrated sulphuric acid, dehydration of carbohydrates results in charring.

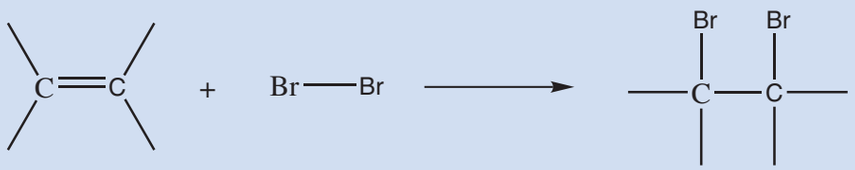
$$C_x(H_2O)_y \xrightarrow[\Delta]{H_2SO_4} x\,C + yH_2O$$

### 6. Ignition test

Aromatic compounds burn with a strong sooty yellow flame because of the high carbon–hydrogen ratio. Aliphatic compounds burn with non-sooty flame.

### 7.Test with bromine water:

In this test, the orange-red colour of bromine solution disappears when it is added to an unsaturated organic compound.

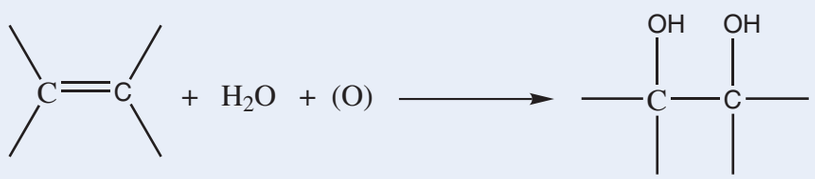



unsaturated compound — Orange-yellow — Colour less

### 8. Test with $KMnO_4$ (Baeyer's Test )

In this test, pink colour of $KMnO_4$ disappears, when alkaline $KMnO_4$ is added to an unsaturated hydrocarbon. The disappearance of pink colour may take place with or without the formation of brown precipitate of $MnO_2$.

$$2KMnO_4 + H_2O \longrightarrow 2KOH + 2MnO_2 + 3(O)$$




unsaturated compound — Colour less

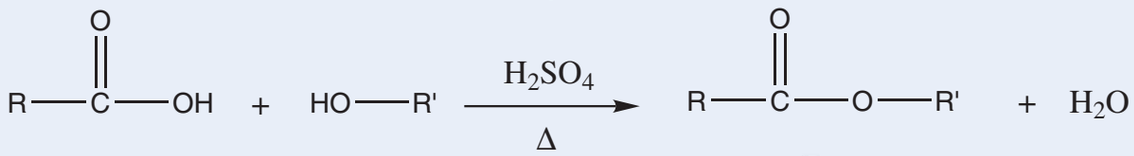
### 9. Neutral $FeCl_3$ test:

Phenol reacts with ferric ions to form violet coloured complex.

Aqueous solution Naphthols do not give any characteristic colour with neutral ferric chloride. But alcoholic solution of α and β naphtholsgiveblue-violet and green colouration respectively due to the formation of binaphthols.

## 10. Esterification test:

Alcohols react with carboxylic acids to form fruity smelling compounds called esters. This esterification is catalysed by an acid such as concentrated sulphuric acid.




Carboxylic acid — Alcohol — Ester (pleasent fruity odour)

## 11. Tollen's reagent test:

Aldehydes react with Tollen's reagent to form elemental silver, accumulated onto the inner surface of the test tube. Thus silver mirror is produced on the inner walls of the test tube.

$$\underset{\text{Aldehyde}}{\text{R-CHO}} + 2\underset{\text{Tollen'sreagent}}{\left[Ag(NH_3)_2\right]OH} \longrightarrow \underset{\substack{\text{Metallic silver}\\ \text{(Silver mirror)}}}{2Ag\downarrow} + R-COONH_4 + H_2O + 3NH_3$$

### Tollen's reagent preparation:

Tollen's reagent is ammoniacal silver nitrate. It is prepared as follows. About 1 g of silver nitrate crystals are dissolved in distilled water in a clean dry test tube. To this aqueous solution of silver nitrate, add 2 ml of dilute NaOH solution to it. A brown precipitate of silver oxide is formed. This precipitate is dissolved by adding dilute ammonia solution drop wise.

## 12. Fehling's Test

Fehling's solution A is an aqueous solution of copper sulphate.

Fehling's solution B is a clear solution of sodium potassium tartrate (Rochelle salt) and strong alkali (NaOH).

The Fehling's solution is obtained by mixing equal volumes of both Fehling's solution A and Fehling's solution B that has a deep blue colour. In Fehling's solution, copper (II) ions form a complex with tartrate ions in alkali. Aldehydes reduces the Cu(II) ions in the Fehling's solution to red precipitate of cuprous oxide(copper (I) oxide).

$$\underset{\text{Aldehyde}}{RCHO} + \underset{\substack{\downarrow\\ \text{Fehling's solution}}}{2Cu^{2+} + 5OH^-} \longrightarrow \underset{\substack{\text{(Cuprous oxide)}\\ \text{(Red colour)}}}{Cu_2O\downarrow} + RCOO^- + 3H_2O$$

Note: Benzaldehyde may not give this test as the reaction is very slow.

## 13. Sodium nitroprusside Test

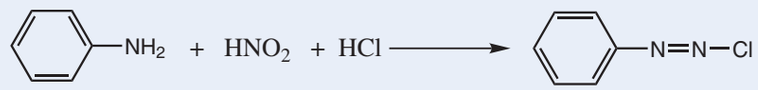
The anion of the ketone formed by a alkali reacts with nitroprusside ion to form a red coloured complex.this test is not given by aldehydes.

$$CH_3COCH_3 \xrightarrow{^-OH} CH_3COCH_2^- + H_2O$$

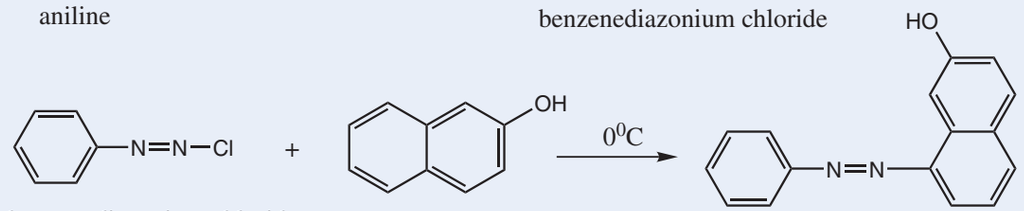
$$\underset{\text{sodium nitro prusside}}{[Fe(CN)_5\,NO]^{2-}} + CH_3COCH_2^- \longrightarrow \underset{\text{(Red coloured complex)}}{[Fe(CN)_5\,NO.CH_3COCH_2]^{3-}}$$

## 14. Azo-Dye Test

This test is given by aromatic primary amines. Aromatic primary amines react with nitrous acid to form diazonium salts. These diazonium salts undergo coupling reaction with β-naphthol to form orange coloured azo dye.



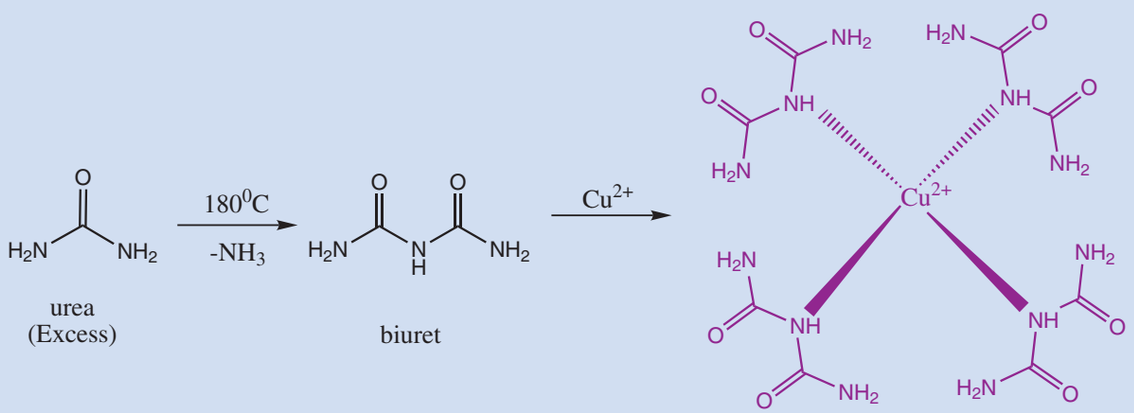

aniline — benzenediazonium chloride




benzenediazonium chloride + β-naphthol → Azo dye (Orange red )

## 15. Biuret test

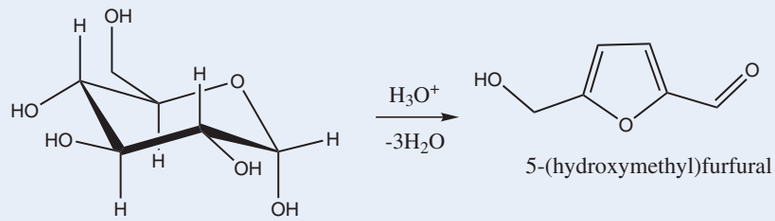
On strong heating Diamide (like urea) form biuret, which forms a copper complex with $Cu^{2+}$ ions from copper sulphate solution. This copper –biuret complex is deep violet coloured.



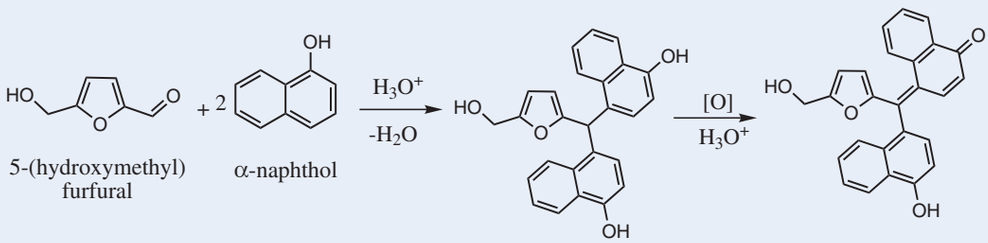

urea (Excess) → biuret → $[Cu(Biuret)_4]^{2+}$ complex (violet colour)

## 16. Molisch's test:

Disaccharides, and polysaccharidesare hydrolysed to Monosaccharides by strong mineral acids. Pentoses are then dehydrated to furfural, while hexoses are dehydrated to 5-hydroxymethylfurfural. These aldehydes formed will condense with two molecules of α-Naphthol to form a purple-coloured product, as shown below.




5-(hydroxymethyl)furfural



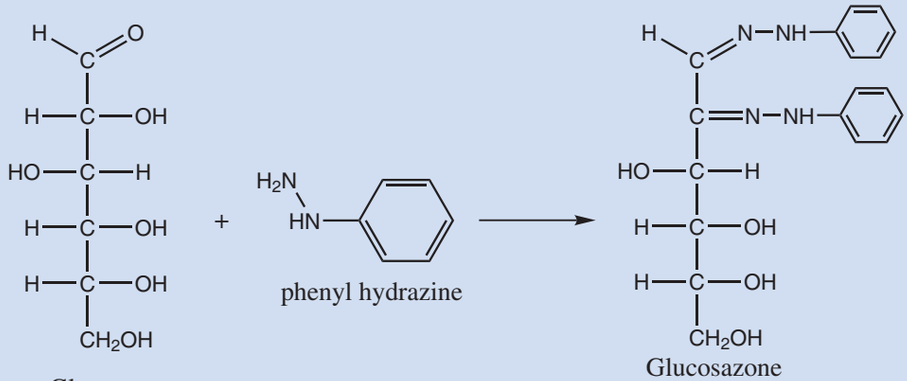

5-(hydroxymethyl) furfural

α-naphthol

A purple dye

## 17.Osazone test:

Phenyl hydrazine in acetic acid, when boiled with reducing sugars forms Osazone. The first two carbon atoms are involved in this reaction. The sugars that differ in their configuration on these carbon atoms give the same type of Osazone. Thus glucose, fructose and mannose give the same needle type yellow crystals.




Glucose

phenyl hydrazine

Glucosazone
(Yellow crystals)

# II-VOLUMETRIC ANALYSIS

## 1. Estimation of Ferrous Sulphate ($Fe^{2+}$)

### Aim :

To estimate the amount of ferrous sulphate dissolved in 750 ml of the given unknown solution volumetrically. For this you are given with a standard solution of ferrous ammonium sulphate (FAS) of normality 0.1102 N and potassium permanganate solution as link solution.

### Principle:

During these titrations, $Fe^{2+}$ ions (from ferrous salts) are oxidised to $MnO_4^-$ ions and $MnO_4^-$ ion (from $Mn^{2+}$) is reduced to $Mn^{2+}$ ion.

**Oxidation** : $5\ Fe^{2+} \longrightarrow 5\ Fe^{3+} + 5e^-$

**Reduction** : $\underset{\text{Pink}}{MnO_4^-} + 8H^+ + 5e^- \longrightarrow \underset{\text{colourless}}{Mn^{2+}} + 4H_2O$

**Overall reaction** : $5Fe^{2+} + MnO_4^- + 8H^+ \longrightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

**Short procedure:**

| S.no | Content | Titration-I | Titration-II |
|---|---|---|---|
| 1 | Burette solution | $KMnO_4$ | $KMnO_4$ |
| 2 | Pipette solution | 20 ml of standard FAS | 20 ml of unknown $FeSO_4$ |
| 3 | Acid to be added | 20ml of 2N $H_2SO_4$ (approx) | 20ml of 2N $H_2SO_4$ (approx) |
| 4 | Temperature | Lab temperature | Lab temperature |
| 5 | Indicator | Self-indicator ($KMnO_4$) | Self-indicator ($KMnO_4$) |
| 6 | End point | Appearance of permanent pale pink colour | Appearance of permanent pale pink colour |
| 7 | Equivalent weight of $FeSO_4$ = 278 | | |

### Procedure :

### Titration–I

(Link $KMnO_4$)Vs (Standard FAS)

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $KMnO_4$ solution up to the zero mark. Exactly 20 ml of standard FAS solution is pipetted out into the clean, washed conical flask. To this FAS solution, approximately 20ml of 2N sulphuric acid is added. This mixture is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading is noted, and the same procedure is repeated to get concordant values.

## Titration –I

(Link $KMnO_4$)Vs (Standard FAS)

| S.no | Volume of standard FAS (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

### Calculation :

Volume of $KMnO_4$ (link) solution ($V_1$) = ----------ml

Normality $KMnO_4$ (link) solution ($N_1$) =----------N

Volume of standard FAS solution ($V_2$) = 20 ml

Normality of standard FAS solution ($N_2$) = 0.1102 N

According to normality equation: $V_1 \times N_1 = V_2 \times N_2$

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

Normality of $KMnO_4$ (link) solution ($N_1$) = _____X_____ N

## Titration–II

(Unknown $FeSO_4$) Vs (Link $KMnO_4$)

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $KMnO_4$ solution up to the zero mark. Exactly 20 ml of unknown $FeSO_4$ solution is pipetted out into the clean, washed conical flask. To this $FeSO_4$ solution approximately 20ml of 2N sulphuric acid is added. This mixture is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –II

(Link $FeSO_4$)Vs (Unknown $FeSO_4$ solution)

| s.no | Volume of Unknown $FeSO_4$ (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

**Calculation :**

Volume of Unknown $FeSO_4$ solution $V_1$ = 20 ml
Normality of Unknown $FeSO_4$ solution $N_1$ = ? N

Volume of $KMnO_4$ (link) solution $V_2$ = ml

Normality $KMnO_4$ (link) solution $N_2$ = X N

According to normality equation: $V_1 \times N_1 = V_2 \times N_2$

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

$$N_1 = \frac{Y}{\_\_\_\_\_} \text{ N}$$

The normality of unknown $FeSO_4$ solution = _____ N

**Weight calculation:**

The amount of $FeSO_4$ dissolved in 1 lit of the solution = (Normality) x (equivalent weight)

The amount of $FeSO_4$ dissolved in 750 ml of the solution $= \frac{\text{Normality x equivalentweight x 750}}{1000}$

$$N_1 = \frac{Y \times 278 \times 3}{4}$$

= g

## Report :

The amount of $FeSO_4$ dissolved in 750 ml of the solution = g

## 2. Estimation of Ferrous Ammonium Sulphate (FAS)

**Aim :**

To estimate the amount of ferrous ammonium sulphate (FAS) dissolved in 1500 ml of the given unknown solution volumetrically. For this you are given with a standard solution of ferrous sulphate ( $FeSO_4$ )of normality 0.1024 N and potassium permanganate solution as link solution.

**Principle:**

**Oxidation** : $5\ Fe^{2+} \longrightarrow 5\ Fe^{3+} + 5e^-$

**Reduction** : $5Fe^{2+} + MnO_4^- + 8H^+ \longrightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

**Overall reaction** : $5Fe^{2+} + MnO_4^- + 8H^+ \longrightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

**Short procedure:**

| s.no | Content | Titration-I | Titration-II |
|---|---|---|---|
| 1 | Burette solution | $KMnO_4$ | $KMnO_4$ |
| 2 | Pipette solution | 20 ml of standard $FeSO_4$ | 20 ml of unknown FAS |
| 3 | Acid to be added | 20ml of 2N $H_2SO_4$(approx) | 20ml of 2N $H_2SO_4$ (approx) |
| 4 | Temperature | Lab temperature | Lab temperature |
| 5 | Indicator | Self-indicator ( $KMnO_4$ ) | Self-indicator ( $KMnO_4$ ) |
| 6 | End point | Appearance of permanent pale pink colour | Appearance of permanent pale pink colour |
| 7 | Equivalent weight of FAS = 392 | | |

**Procedure :**

**Titration–I**

(Link $KMnO_4$)Vs (Standard $FeSO_4$ )

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $FeSO_4$ solution up to the zero mark. Exactly 20 ml of standard $FeSO_4$ solution is pipetted out into the clean, washed conical flask. To this solution, approximately 20ml of 2N sulphuric acid is added. This mixture is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading are noted, the same procedure is repeated to get concordant values.

## Titration –I

(Link $KMnO_4$)Vs (Standard $FeSO_4$)

| s.no | Volume of standard $FeSO_4$ (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

## Calculation :

Volume of $KMnO_4$ (link) solution $V_1$ = ml

Normality $KMnO_4$ (link) solution $N_1$ = ?N

Volume of standard $FeSO_4$ solution $V_2$ = 20 ml

Normality of standard $FeSO_4$ solution $N_2$ = 0.1024 N

## According to normality equation:

According to normality equation: $V_1 \times N_1 = V_2 \times N_2$

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

Normality of $KMnO_4$ (link) solution $(N_1)$ = _____X_____ N

## Titration–II

(Unknown FAS) Vs (Link $KMnO_4$)

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $KMnO_4$ solution up to the zero mark. Exactly 20 ml of unknown FAS solution is pipetted out into the clean, washed conical flask. To this FAS solution approximately 20ml of 2N sulphuric acid is added. This mixture is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –II

(Link $KMnO_4$)Vs (Unknown FAS)

| s.no | Volume of Unknown FAS (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

## Calculation :

Volume of Unknown FAS solution $V_1$ = 20ml

Normality of Unknown FAS solution $N_1$ = ? N

Volume of $KMnO_4$ (link) solution $V_2$ = ml

Normality $KMnO_4$ (link) solution $N_2$ = N

According to normality equation: $V_1 \times N_1 = V_2 \times N_2$

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

$$N_1 = \frac{Y}{\quad\quad} N$$

The normality of unknown FAS solution = $\frac{Y}{\quad\quad}$ N

## Weight calculation:

The amount of FAS dissolved in 1 lit of the solution = (Normality) x (equivalent weight)

The amount of FAS dissolved in 1500 ml of the solution $= \frac{\text{Normality} \times \text{equivalentweight} \times 1500}{1000}$

$$= \frac{Y \times 392 \times 1500}{1000}$$

$$= \quad g$$

## Report :

The amount of FAS dissolved in 1500 ml of the solution = g

## 3. Estimation of oxalic acid

### Aim :

To estimate the amount of oxalic acid dissolved in 500 ml of the given solution volumetrically. For this you are given with a standard solution of ferrous ammonium sulphate (FAS) of normality 0.1 N and potassium permanganate solution as link solution.

### Principle:

During these titrations, oxalic acid is oxidized to $CO_2$ and $MnO_4^-$ ions (from $KMnO_4$) is reduced to $Mn^{2+}$ ion.

**Oxidation** : $\underset{\text{Pink}}{MnO_4^-} + 8H^+ + 5e^- \longrightarrow \underset{\text{colourless}}{Mn^{2+}} + 4H_2O$

**Reduction** : $\underset{\text{Pink}}{MnO_4^-} + 8H^+ + 5e^- \longrightarrow \underset{\text{colourless}}{Mn^{2+}} + 4H_2O$

**Overall reaction** : $5(COOH)_2 + 2MnO_4^- + 6H^+ \longrightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$

Since one mole oxalic acid releases 2 moles of electrons, the equivalent weight of oxalic acid $= \frac{106}{2} = 63$ (oxalic acid is dihydrated)

### Short procedure:

| s.no | Content | Titration-I | Titration-II |
|---|---|---|---|
| 1 | Burette solution | $KMnO_4$ | $KMnO_4$ |
| 2 | Pipette solution | 20 ml of standard FAS | 20 ml of unknown oxalic acid |
| 3 | Acid to be added | 20ml of 2N $H_2SO_4$(approx) | 20ml of 2N $H_2SO_4$ (approx) |
| 4 | Temperature | Lab temperature | 60 – 70 $^0$C |
| 5 | Indicator | Self-indicator ( $KMnO_4$ ) | Self-indicator ($KMnO_4$) |
| 6 | End point | Appearance of permanent pale pink colour | Appearance of permanent pale pink colour |
| 7 | Equivalent weight of oxalic acid = 63 | | |

### Procedure :

### Titration–I

(Link $KMnO_4$)Vs (Standard FAS )

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $KMnO_4$ solution up to the zero mark. Exactly 20 ml of standard FAS solution is pipetted out into the clean, washed conical flask. To this FAS solution, approximately 20ml of 2N sulphuric acid is added. This mixture is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –I

(Link $KMnO_4$ )Vs (Standard FAS solution)

| s.no | Volume of standard FAS (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$ ) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

## Calculation :

Volume of $KMnO_4$ (link) solution $V_1$ = ml

Normality $KMnO_4$ (link) solution $N_1$ = ? N

Volume of standard FAS solution $V_2$ = 20 ml

Normality of standard FAS solution $N_2$ = 0.1 N

According to normality equation:

$$V_1 \times N_1 = V_2 \times N_2$$

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

Normality $KMnO_4$ (link) solution $N_1$ = _____ N

## Titration–II

(Unknown oxalic acid ) Vs (Link $KMnO_4$ )

Burette is washed with water, rinsed with $KMnO_4$ solution and filled with same $KMnO_4$ solution up to the zero mark. Exactly 20 ml of unknown oxalic acid solution is pipetted out into the clean, washed conical flask. To this oxalic acid solution approximately 20ml of 2N sulphuric acid is added. This mixture is heated to 60 – $70^0$C using Bunsen burner and that hot solution is titrated against $KMnO_4$ Link solution from the burette. $KMnO_4$ is added drop wise till the appearance of permanent pale pink colour. Burette reading are noted, the same procedure is repeated to get concordant values.

## Titration –II

(Link $KMnO_4$)Vs (Unknown oxalic acid)

| s.no | Volume of Unknown oxalic acid (ml) | Burette readings | | Concordant value (Volume of $KMnO_4$) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

### Calculation :

Volume of Unknown oxalic acid solution $V_1$ = 20 ml

Normality of Unknown oxalic acid solution $N_1$ = ? N

Volume of $KMnO_4$ (link) solution $V_2$ = ml

Normality $KMnO_4$ (link) solution $N_2$ = N

According to normality equation:

$$V_1 \times N_1 = V_2 \times N_2$$

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

Normality of Unknown oxalic acid solution $N_1$ = $\frac{Y}{\quad\quad}$ N

### Weight calculation:

The amount of oxalic acid dissolved in 1 lit of the solution =(Normality) x (equivalent weight)

The amount of oxalic acid dissolved in 500 ml of the solution $= \frac{Y \times 63 \times 500}{1000}$

$$= \frac{\quad \text{x } 63 \text{ x } 500}{1000}$$

$$= \quad \text{g}$$

### Report :

The amount of oxalic acid dissolved in 500 ml of given the solution = g

## 4. Estimation of sodium hydroxide

### Aim :

To estimate the amount of sodium hydroxide dissolved in 250 ml of the given unknown solution volumetrically. For this you are given with a standard solution of sodium carbonate solution of normality 0.0948 N and hydrochloric acid solution as link solution.

### Principle:

Neutralization of Sodium carbonate by HCl is given below. To indicate the end point, methyl orange is used as an indicator.

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

Neutralization of Sodium hydroxide by HCl is given below. To indicate the end point, phenolphthalein is used as an indicator.

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

### Short procedure:

| s.no | Content | Titration-I | Titration-II |
|---|---|---|---|
| 1 | Burette solution | HCl ( link solution) | HCl ( link solution) |
| 2 | Pipette solution | 20 ml of standard $Na_2CO_3$ solution | 20 ml of unknown NaOH solution |
| 4 | Temperature | Lab temperature | Lab temperature |
| 5 | Indicator | Methyl orange | Phenolphthalein |
| 6 | End point | Colour change from straw yellow to pale pink | Disappearance of pink colour |
| 7 | Equivalent weight of NaOH = 40 | | |

### Procedure :

### Titration–I

(Link HCl )Vs (standard $Na_2CO_3$)

Burette is washed with water, rinsed with HCl solution and filled with same HCl solution up to the zero mark. Exactly 20 ml of standard $Na_2CO_3$solution is pipetted out into the clean, washed conical flask. To This solution 2 to 3 drops of methyl orange indicator is added and titrated against HCl link solution from the burette. HCl is added drop wise till the colour change from straw yellow to pale pink. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –I

(Link HCl )Vs (standard $Na_2CO_3$)

| s.no | Volume of standard $Na_2CO_3$ (ml) | Burette readings | | Concordant value (Volume of HCl) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

## Calculation :

Volume of HCl (link) solution $V_1$ = ml

Normality HCl (link) solution $N_1$ = ? N

Volume of standard $Na_2CO_3$ solution $V_2$ = 20 ml

Normality of standard $Na_2CO_3$ solution $N_2$ = 0.0948 N

## According to normality equation:

According to normality equation: $V_1 \times N_1 = V_2 \times N_2$

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

Normality of HCl (link) solution $(N_1)$ = _____X_____ N

## Titration–II

(Unknown NaOH ) Vs (Link HCl)

Burette is washed with water, rinsed with HCl solution and filled with same HCl solution up to the zero mark. Exactly 20 ml of unknown NaOH solution is pipetted out into the clean, washed conical flask. To This solution 2 to 3 drops of phenolphthalein indicator is added and titrated against HCl link solution from the burette. HCl is added drop wise till the pink colour disappears completely. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –II

## (Link HCl )Vs (Unknown NaOH solution)

| s.n | Volume of Unknown NaOH (ml) | Burette readings | | Concordant value (Volume of HCl) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

### Calculation :

Volume of Unknown NaOH solution $V_1 = 20$ ml

Normality of Unknown NaOH solution $N_1 = ?$ N

Volume of HCl (link) solution $V_2 =$ ml

Normality HCl (link) solution $N_2 =$ N

According to normality equation:

$$V_1 \times N_1 = V_2 \times N_2$$

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

Normality of Unknown HCl solution $N_1 = \frac{Y}{\quad} N$

### Weight calculation:

The amount of NaOH dissolved in 1 lit of the solution = (Normality) x (equivalent weight)

The amount of NaOH dissolved in 250 ml of the solution $= \frac{\text{Normality x equivalentweight x 250}}{1000}$

$$= \frac{Y \times 40 \times 250}{1000}$$

$$= \frac{\text{x 40 x 250}}{1000} \text{ g}$$

### Report :

The amount of NaOH dissolved in 750 ml of the solution = g

## 5. Estimation of oxalic acid

### Aim :

To estimate the amount of oxalic acid dissolved in 1250 ml of the given unknown solution volumetrically. For this you are given with a standard solution of HCl solution of normality 0.1010 N and sodium hydroxide solution as link solution.

### Principle:

Neutralization of Sodium hydroxide by HCl is given below. To indicate the end point, phenolphthalein is used as an indicator.

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

Neutralization of Sodium hydroxide by oxalic acid is given below. To indicate the end point, phenolphthalein is used as an indicator.

$$2NaOH + \underset{\text{Oxalic acid}}{(COOH)_2} \longrightarrow \underset{\text{Sodium oxalate}}{(COONa)_2} + 2H_2O$$

### Short procedure:

| s.no | Content | Titration-I | Titration-II |
|---|---|---|---|
| 1 | Burette solution | HCl (standard solution) | Oxalic acid ( unknown solution) |
| 2 | Pipette solution | 20 ml of NaOH link solution | 20 ml of NaOH link solution |
| 4 | Temperature | Lab temperature | Lab temperature |
| 5 | Indicator | Phenolphthalein | Phenolphthalein |
| 6 | End point | Disappearance of pink colour | Disappearance of pink colour |
| 7 | Equivalent weight of oxalic acid = 63 | | |

### Procedure :

### Titration–I

(standard HCl )Vs (link NaOH)

Burette is washed with water, rinsed with HCl solution and filled with same HCl solution up to the zero mark. Exactly 20 ml of NaOH is pipetted out into the clean, washed conical flask. To This solution 2 to 3 drops of phenolphthalein indicator is added and titrated against HCl solution from the burette. HCl is added drop wise till the pink colour disappears completely. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –I

(standard HCl )Vs **(link** NaOH)

| s.no | Volume of NaOH(ml) | Burette readings | | Concordant value (Volume of std HCl) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

### Calculation :

Volume of NaOH(link) solution $V_1 = 20$ ml

Normality NaOH(link) solution $N_1 = ?$ N

Volume of standard HCl solution $V_2 =$ ml

Normality of standard HCl solution $N_2 = 0.1010$ N

### According to normality equation:

$$V_1 \times N_1 = V_2 \times N_2$$

$$N_1 = \frac{\times\ 0.1010}{20} =$$

Normality NaOH (link) solution $N_1 =$ _____X_____ N

## Titration–II

(Unknown oxalic acid ) Vs (Link NaOH)

Burette is washed with water, rinsed with oxalic acid solution and filled with same oxalic acid solution up to the zero mark. Exactly 20 ml of NaOH solution is pipetted out into the clean, washed conical flask. To This solution 2 to 3 drops of phenolphthalein indicator is added and titrated against oxalic acid solution from the burette. oxalic acid is added drop wise till the pink colour disappears completely. Burette reading is noted and the same procedure is repeated to get concordant values.

## Titration –II

(Link NaOH )Vs (Unknown oxalic acid solution)

| s.no | Volume of NaOH link (ml) | Burette readings | | Concordant value (Volume of oxalic acid) (ml) |
|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

### Calculation :

Volume of Unknown oxalic acid solution $V_1$ = ml

Normality of Unknown oxalic acid solution $N_1$ = ? N

Volume of NaOH solution $V_2$ = 20 ml

Normality NaOH solution $N_2$ = N

According to normality equation:

$$V_1 \times N_1 = V_2 \times N_2$$

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

Normality of Unknown oxalic acid solution $N_1 = \frac{Y}{\_\_\_\_\_} N$

### Weight calculation:

The amount of oxalic acid dissolved in 1 lit of the solution = (Normality) x (equivalent weight)

The amount of oxalic acid dissolved in 1250 ml of the solution $= \frac{\text{Normality x equivalentweight x 1250}}{1000}$

$$= \frac{Y \times 63 \times 1250}{1000}$$

$$= \frac{\text{x 63 x 1250}}{1000}$$

$$= \text{g}$$

### Report :

The amount of oxalic acid dissolved in 1250 ml of the solution = g