

## **Experimental Chemistry**

#### QUALITATIVE ORGANIC ANALYSIS

### Experiment 1

#### **Detection of Elements**

#### Object

To detect the elements (N, S and X) present in organic compound by Lassaigne's test.

#### Principle

As organic compounds are covalent compounds, so they do not ionise. For testing ions, the elements present in organic compounds have to be converted into ionic compounds. For this, the organic compound is heated with sodium, so that the element present in organic compound is converted into sodium compound ie, ionic compound eg, with Na, nitrogen forms sodium cyanide, sulphur forms sodium sulphide and halogens gives sodium halides.

$$Na + C + N$$
 $Nitrogen \longrightarrow NaCN$ 
 $Sodium cyanide$ 
 $2Na + S$ 
 $Sulphur \longrightarrow Na_2S$ 
 $Sodium sulphide$ 
 $2Na - X_2 \longrightarrow 2NaX$ 
 $Halogens \longrightarrow Sodium halide$ 

#### Procedure

A small piece of freshly cut sodium is dried between folds of a filter paper. It is taken in a clean ignition tube and then covered with the organic substance. The tube is morthy heated in the flame filtrate which should be clear, colourless and transparent, is known as Sodium Extract (Lassaigne's extract) if filtrate is not clear, colourless and transparent, then repeat the process by using a large piece of sodium.

Sodium extract so obtained is used for the detection of elements (N, S and X) by following methods.

#### Detection of Nitrogen

(i) Treat 1 mL of sodium extract with 1 or 2 drops of freshly prepared saturated solution of FeSO<sub>4</sub> followed by 2 drops of NaOH. Boil the contents for 1-2 minutes. Cool it and add dil H<sub>2</sub>SO<sub>4</sub> drop by drop with shaking till a clear solution is obtained. Then, add 2 drops of FeCl<sub>3</sub>. (If hydrochloric acid is used in place of sulphuric acid, then addition of femic chloride is not required). Formation of a prussian blue colour or greenish blue ppt indicates the presence of nitrogen.

nitrogen.

FeSO<sub>4</sub> 
$$\div$$
 2NaOH  $\longrightarrow$ 

Ferrore sulphate

$$Fe(OII)_2 \downarrow \quad \div \text{Na}_2\text{SO}_4$$
Ferrore hydroxide

Fe(OII)<sub>2</sub> + 6NaCN  $\longrightarrow$ 
(From sodium extract)

$$\text{Na}_4[\text{Fe}(\text{CN})_6] + 2\text{NaOH}$$

$$3\text{Na}_4[\text{Fe}(\text{CN})_6] + 4\text{FeCI}_3 \longrightarrow$$

$$\text{Fe}_4[\text{Fe}(\text{CN})_5]_3 + 12\text{NaCl}$$
Ferric ferroryanide

test of nitrogen a red colour is obtained instead of green or blue due to formation of ferric sulphocyanide.

$$3NaCNS + FeCl_2 \longrightarrow Fe(CNS)_3 + 3NaGl$$
[Red)

#### Detection of Sulphur

(i) Add 2-3 mL of freshly prepared sodium nitroprusside (colourless) to 1 mL of sodium extract. Formation of violet or purple colour indicates the presence of sulphur.

Na<sub>4</sub>[Fe(CN)<sub>S</sub> · NOS] Sodium thionitroprusside (Violet colour)

(ii) Add acetic acid to acidified the sodium extract. Now a small quantity of lead acetate is added to this acidic solution. Formation of a black ppt indicates the presence of sulphur.

$$Na_2S + (CH_3COO)_2Pb \longrightarrow PbS \downarrow + 2CH_3COONa$$
  
Black ppt

#### Detection of Halogens

- (i) Boil 1 mL of sodium extract with 2 or 3 drops of cone HNO<sub>3</sub>, cool and then, add AgNO<sub>3</sub>. After adding AgNO<sub>3</sub> if
  - (a) White curdy ppt is formed which is soluble in NH<sub>4</sub>OH, then it indicates the presence of Cl<sup>-</sup> ion.

$$NaCl + AgNO_3 \longrightarrow AgCl \downarrow + NaNO_3$$

White opt

$$AgCI + 2NH_4OH \longrightarrow |Ag(NH_3)_2|CI + 2H_2O$$

(b) Pale yellow ppt is obtained which is partially soluble in NH<sub>4</sub>OH, then it indicates the presence of Br<sup>-</sup> ion.

$$\begin{array}{ccc} \text{NaBt} + \text{AgNO}_3 & \longrightarrow & \text{AgBr} \downarrow \\ & & \text{Pale yellow ppt} \end{array}$$

+ NaNO<sub>2</sub>

$$AgHr + 2NH_4OH \longrightarrow [Ag(NH_5)_2]Br + 2H_2O$$

(c) Yellow ppt is formed which is insoluble in NH<sub>4</sub>OH, then it indicates the presence of I lon.

Note: Dering the east for hillagens, sodium extract is boiled with New drops of conc HISO, in order to

decompose NaCN and Na2S, so that they may not interfere in this test.

$$NaCN + HNO_3 \longrightarrow NaNO_3 + HCN$$
  
 $Na_2S + 2HNO_3 \longrightarrow 2NaNO_3 + H_2S$ 

(ii) Acidify the sodium extract with dil HNO<sub>3</sub>, add 2 drops of chloroform or carbon tetrachloride and shake vigorously with chlorine water. If chloroform layer turns pale brown—Bromine is present but if turns violet, then Iodine is present (chloroform test or layer test).

$$\begin{array}{ccc} 2\text{NaBr} + \text{Cl}_2 & \longrightarrow & 2\text{NaCl} + \text{Br}_2 \\ \text{Br}_2 + \text{CHCl}_3 & \longrightarrow & \text{Pale-brown colour} \\ 2\text{NaI} + \text{Cl}_2 & \longrightarrow & 2\text{NaCl} + \text{l}_2 \\ \text{I}_2 + \text{CHCl}_3 & \longrightarrow & \text{Violet colour} \end{array}$$

### Experiment 2

#### Object

To detect the presence of balogens by Beilstein's test.

#### Procedure

Take copper wire and heat its one end in a non-luminous flame till it ceases to impart green colour to the flame. Dip the hot wire in the organic compound to be tested. Again heat, formation of a green or bluish green flame identifies the presence of halogens.

Several halogen free compounds such as pyridine, purines, urea, thiourea etc also impart green colour to the flame, so the test is not very reliable. However, production of no green or blue colour confirms the absence of halogen. Therefore, the test is a confirmative one to show the absence of halogens rather than to show its presence.

## **Experiment 3**

### Detection of Functional Groups

#### Object

To detect various functional groups present in organic compounds.

#### Tests for Carboxylic (--COOH) Group

- (i) Litmus paper test: Dip blue litmus paper in the aqueous solution or suspension of the compound. It turns red.
- (ii) Sodium bicarbonate test: In a test tube take a little quantity of the compared and then

add a saturated solution of sodium bicarbonate. Formation of brisk effervescence shows the presence of —COOH group.

COOH + NaHCO3

Benzoic acid

COONa + 
$$H_2O + CO_2$$
?

Sodium benzoate

(iii) **Bster formation**: Heat a small quantity of organic compound with ethyl alcohol and a little conc H<sub>2</sub>SO<sub>4</sub>. Cool the solution and pour in a tube containing water. A fruity smell, due to formation of an ester, indicates the presence of carboxylic group.

$$R = COOH + C_2H_3OH \xrightarrow{conv H_2SO_4}$$

$$RCOOC_2H_5 + H_2O$$
Refer
(fruity smell)

(iv) Fluorescence test: Heat gently a little amount of organic compound with resorcinol and a few drops of conc H<sub>2</sub>SO<sub>4</sub> in a hard glass test tube for about two minutes. Cool and pour the contents into a beaker containing NaOH diluted largely with water. Formation of intense green orange fluorescence indicates the presence of 1,2 or or the dicarboxylic groups.

nitrate. A red colour indicates the presence of alcoholic hydroxy group.

$$\begin{array}{c} \text{2ROH} \div (\text{NH}_4)_2 \text{Ce}(\text{NO}_3)_6 & \longrightarrow \\ \text{Ce}(\text{NO}_3)_4 \cdot (\text{ROH})_2 + 2\text{NH}_4 \text{NO}_3 \\ \text{Red colour} \end{array}$$

- (ii) Evolution of HCl and H<sub>2</sub>: In a dry test rube take some organic compound and add a small amount of anhyd. CaSO<sub>4</sub>. Filter the solution in another dry test tube and divide the filtrate into two parts.
  - (a) To first part, add 2 drops of acetyl chloride very carefully and expose a moist blue litmus paper at the mouth of the test tube. If the compound contains an alcohol group, then the litmus paper turns red.

$$CH_3COCI + ROH \longrightarrow CH_3COOR + HCI$$
b) To second part, add a small piece of dry

(b) To second part, add a small piece of dry sodium. Effervescence indicates the presence of alcoholic group.

$$2ROH + 2Na \longrightarrow 2RONa + H_2\uparrow$$

(iii) Xanthate test: To 1 mL of the conc aqueous solution of organic compound add pelletes of KOH. Heat and cool. Then, add 1 mL of ether followed by the addition of 2-3 drops of carbon disulphide. Formation of a yellow ppt indicates the presence of alcoholic group.

$$ROH + KOH \longrightarrow ROK + H_2O$$
 $ROK + CS_2 \longrightarrow RO - C < SK$ 
 $(Yellow ppt)$ 

Fifter the above solution and to 1 ml. of the filtrate add 0.5 ml. of ammonium molybdate solution and excess of dil HCl. Production of a red or blue colour confirms the presence of alcoholic group.

(iv) Lucas test: This test is used to distinguish between primary, secondary and tertiary

- (b) Formation of turbidity after 4-5 min shows the presence of secondary alcohols,
- (c) If solution remains clear, then primary alcohol is present.

#### Tests for Phenolic (Ph—OH) Group

(i) Ferric chloride test: To aqueous or alcoholic solution of compound, add few drops of ferric chloride (FeCl<sub>3</sub>). Formation of green, blue or violet colour shows the presence of phenol.

presence of phenoi.  

$$6C_6H_5OH + FeCl_3 \longrightarrow 3H^+ + [Fe(OC_5H_3)_6]^{3-} + 3HCl_{Volet}$$

(ii) Liebermann's nitroso reaction: Fuse a little amount of compound with a crystal of NaNO<sub>2</sub> in a test tube. Cool the mixture and add 1 mL conc H<sub>2</sub>SO<sub>4</sub>. A deep green to blue solution is formed which turns red when poured in a large excess of water. The red aqueous solution becomes again deep green or blue if made alkaline with NaOH. It shows the presence of phenol.

Note - Paraphenol in microscond to FeCl, test as unll as Liebermann's biliesa reactions

(iii) Dye test: Take a drop of aniline in a test tube and add 0.5 mL conc HCl solution, dilute it with water, cool in a freezing bath and add dilute NaNO<sub>2</sub> solution while shaking. In a second test tube take the organic compound and add excess of 2N NaOH solution. Now add the content of both the test tubes. Formation of a dye of orange or red colour indicates the presence of phenolic group.

(iv) Phthalein test: Heat a small amount of organic compound with double amount of phthalic anhydride and a drop of conc H<sub>2</sub>SO<sub>4</sub> for one minute. Cool and make it alkaline with dif NaOH. Pour a few drops of the alkaline liquid in 20 mL of water. Formation of characteristic colour shows the presence of phenol.

Resoccinol

α-naphthol

β-naphthol

#### Tests for Aldehyde (—CHO) Group

Fluorescent green —

Green

Light green

(i) Tollen's reagent test: Take a little quantity of the compound in a test tube and add 2 mL of freshly prepared reagent. Shake, warm and allow the contents to stand for 2–3 minutes. Formation of silver mirror or a grey ppt indicates the presence of an aldebudic group.

$$\begin{array}{ccc} 2 \mathrm{Ag} (\mathrm{NH_3})_2 \mathrm{OH} + R \cdot \mathrm{CHO} & \longrightarrow & 2 \mathrm{Ag} \downarrow \\ & & \mathrm{Silver} & \mathrm{mirror} \\ & & \mathrm{or} & \mathrm{grey} & \mathrm{ppt} \\ & & & + R \mathrm{COONH}_4 \end{array}$$

 $+3NH_3 + H_2O$ 

(ii) Fehling's solution test: Take a mixture of equal amounts of Fehling's solution A and B, and a few drops of organic compound and boil the contents. Formation of a red ppt shows the presence of an aldehyde.

Note: Both the opene see use also given by reducing sugars:

(iii) Schiff's reagent test: Add 5-6 drops of organic compound to 2 mL of the reagent. Shake vigorously. After some time formation of a deep red or violet colour indicates the presence of an aldehydic group.

Schiff's reagent (Colourless)

(iv) Benedict's solution test: Boil the compound with 2–3 mL of Benedict' solution for few minutes. Appearance of a red-yellow ppi confirms the presence of aliphatic

(i) 2,4-dinitro phenyl hydrazine test: In a dry test tube add few drops of the organic compound (if liquid) or its alcoholic solution (if solid) to about 2 mL of the reagent and one drop of conc H<sub>2</sub>SO<sub>4</sub>. Shake vigorously, heat (if necessary) and allow to stand for about 5 minutes. A yellow or orange ppt separates out in case of a compound containing carbonyl group due to formation of respective hydrazones.

2 : 4 Dinitro phenyl hydrazine

$$>$$
C:=N·HN-NO<sub>2</sub> $\downarrow$  + H<sub>2</sub>O

Yellow or orange :ed coloured crystalline derivative of 2 : 4 dinitrophenyl hydrazine of carboayl compound

(ii) Sodium bisulphite test: Add a very small quantity of organic compound to 1 mL of saturated solution of sodium bisulphite and shake vigorously. Formation of white ppt shows the presence of carbonyl group.

Note: The first two tasts are given by iddelydes also. Therefore, to difficulties between aldelydes and kerones it should be known that kerone to not give only use with Tollon's reason. Schiff's schamo. Feblurg's solution and Bounday solution.

#### Tests for Primary Amine (--NH<sub>2</sub>) Group

(i) Carbylamine test: Boil a little quantity of the compound with 2 drops of chloroform and 2 mi. of alcoholic caustic potash. An intolerable offensive odour of carbylamine indicates the presence of primary amine.

$$R - NH_2 + CHCl_3 + 3KOH$$
  $\rightarrow$   $R \cdot NC \uparrow$  Carbylamine Carbylamine

 $+ 3KCI + 3H_2O$ 

(ii) Dye test: Dissolve about 0.2 g of the compound in dil HCl and cool. Now, add 10% aq NaNO<sub>2</sub> solution. Pour all this content into a beaker containing alkaline  $\beta$ -naphthol solution. Formation of a red or orange dye indicates the presence of aromatic primary amino group.

Benzene élazonium chlorida

ß-naphthoi

Phenyi azo-#-naphthol (Red dye)

(iii) Rimini test: To about 0.3 g of the compound taken in a test rube add 5 mi. of water, 1 mL acetone and few drops of sodium nitroprusside. Allow to stand for 2 min. Formation of a violet red colour shows the presence of aliphatic primary amine.

#### Test for Secondary Amine ( NH) Group

Libermann's nitroso test: To about  $0.2 \, \mathrm{g}$  of organic compound, add freshly prepared solution of nitrous acid and then add 1-2 drops each of phenol and conc  $\mathrm{H_2SO_4}$ . On heating a blue colouration which changes to red on dilution with water and black with aq alkali confirms the presence of secondary amine.

#### Test for Tertiory Amine (→ N) Group

Ohkuma test: Add about 5-6 drops of the citric acid-acetic anhydride reagent to the alcoholic solution of the compound, shake and heat in a boiling water bath. Development of a red or purple colour within 2-3 minutes, indicates the presence of terriary amine.

## Tests to Distinguish between Primary, Secondary and Tertiary Amines

- (i) Nitrous acid test: Prepare a solution of nitrous acid by adding ice cold dil HCl to a solution of 1% aq NaNO<sub>2</sub>. Add gradually this solution to 0.2 g of the organic compound in 10 mL dil HCl.
  - (a) Formation of brisk effervescence shows the presence of aliphatic primary amine.

$$R \longrightarrow NH_2 + HNO_2 \longrightarrow ROH + H_2O + N_2 \uparrow$$

(b) Formation of an oily dark coloured liquid indicates the presence of secondary amine.

$$R$$
 $R$ 
 $NH + HNO_2 \longrightarrow R$ 

Secondary arrine
 $R$ 
 $R$ 
 $N \cdot NO = H_2O$ 
 $R$ 

Nitrose compound (nily)

- (c) No reaction indicates the presence of aliphatic tertiary amine while production of green or brown colour indicates the presence of aromatic tertiary amines.
- (ii) Hinsberg's test: To about 0.2 g of the compound, add 1 mL of 5% NaOH and 3 mL pyridine. Shake well and add few drops of benzene sulphonyl chloride with continuous shaking.
  - (a) Formation of yellow colour indicates the presence of primary amine.
  - (b) Formation of orange colour shows the presence of secondary amine.
  - (c) Formation of a red or purple colour shows the presence of tertiary amines.

## PREPARATION OF SOME INORGANIC COMPOUNDS

## **Experiment 4**

#### Mohr's Salt

#### Object:

To prepare Mohr's salt or ferrous ammonium sulphate  $[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$ .

#### Principle

When solutions of ferrous sulphate and ammonium sulphate are mixed together, evaporated and cooled, very light green coloured crystals of ferrous ammonium sulphate are obtained.

$$(NH_4)_2SO_4 + FeSO_4 + 6H_2O \longrightarrow$$
  
 $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$   
Perrous anymonium sulphate

reason for adding dil H<sub>2</sub>SO<sub>4</sub> is to prevent the hydrolysis of ferrous sulphate *ie*, to prevent the conversion of ferrous sulphate into ferrous hydroxide [Fc(OH)<sub>2</sub>]. Now, add 13 g of ammonium sulphate solution in water to the above solution, concentrate and crystallise by cooling. Separate the crystals and dry between the folds of filter papers.

#### Result

Yield: 30 文

Appearance: Light green crystals.

## PREPARATION OF ORGANIC COMPOUNDS

### Experiment 5

#### Acetanilide

#### Object

To prepare acctanilide from aniline.

#### **Principle**

Amines containing —NH<sub>2</sub> and >NH groups respectively can be directly acctylated. Their reactive hydrogen atoms get replaced by the acetyl group (—COCH<sub>3</sub>) to give acetyl derivatives of the type  $RNH \cdot COCH_3$  and  $R_2N \cdot COCH_3$  respectively which may be regarded as mono and di-alkyl substituted acetamide.

$$R$$
— $NH_2 + (CH_3CO)_2O \longrightarrow RNH \cdot COCH_3 + CH_3COOH$ 

The mechanism of this reaction is as follows:

Acetic anhydride

$$\begin{array}{c} -H \stackrel{+}{\circlearrowleft} \\ & \stackrel{-}{\rightleftharpoons} \\ & \stackrel{-}{\rightleftharpoons}$$

#### (i) First method

#### Reagents

Aniline — 5 ml.

Acetic anhydride — 5 ml.

Acetic acid — 5 ml.

Zinc dust — 0.025 g

#### **Procedure**

Take 5 mL of aniline in a 150 mL conical flask and add 10 mL of a mixture of equal volumes of 5 mL acetic anhydride and 5 mL acetic acid followed by 0.025 g zinc. Now fit a reflux water condenser and boil the convents gently for about 12–15 minutes. Now pour the hot mixture in 200 mL of ice cold water with continuous stining. Acetanilide rapidly crystallises. Filter at the pump and wash with cold water.

Recrystallise 1 g of it from 60 mL dilute acetic acid. Filter at the pump and wash thoroughly with water and dry.

#### (ii) Second method

#### Reagents

Aniline -- 5 mL
Conc HCl -- 4.5 mL
Acetic anhydride -- 6.4 mL (6.95 g)

Sodium acetate — 8.5 g

#### Procedure

In a 400 mL beaker containing 250 mL of water, take 1 mL of conc HCl and 5 mL of aniline. Stir the solution and now to it add redistilled acetic aphydride (6.4 mL) and stir again and immediately.

pour it into a solution of sodium acetate (prepared by dissolving 8.5 g of crystallised  $\mathrm{CH_3COONa}$  in 25 mL of water). Piace the beaker in ire bath and stir vigorously. Colourless crystals of almost pure acetanilide separate out. Filter at pump and wash with cold water. Dry on a porous plate or filter paper in air and weigh.

On recrystallisation from about 250 mL of boiling water (containing 5 mL of methylated spirit), snow white leaflets are obtained.

#### Result

- (i) Weight of the crude acetanilide = ...,... g
- (ii) Weight of the recrystallised acetanilide =
- (iii) Yield : 5 g<sup>†</sup>
- (iv) MP: 114°C
- (v) Appearance: Snow white leaflets

#### Precautions

- (i) Zinc dust reduces the coloured impurities present in aniline and also heips to prevent oxidation of aniline during the reaction.
- A large amount of acetic anhydride and continued heating is however, avoided otherwise amounts of the diacetyl derivatives are formed.
- (iii) The solution of aniline in cone HCl should not be coloured. If it is so, then it should be heated with carbon for 5-10 minutes and filtered.

Note: State accidentified ride is quite costly, it is not used note a days, interpal glacial accidenced is used which depends upon the displacement of the resetsible equilibrium to the right by the rimoval of water on disafficient.

R NH<sub>2</sub>+CH<sub>3</sub>COOH -- R - NHEOCH<sub>3</sub>+H<sub>2</sub>O

It is introve but feasible to remove writer in with a way. That's why by laboratory a mission of aceta acid and acetic analydride is employed.

## Experiment 6

#### p-Nitro Acetanilide Object

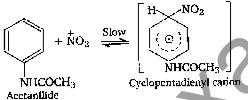
To prepare p-nitro a

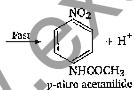
To prepare p-nitro acetanilide from

#### Principle

When acetanilide is treated with a mixture of conc  $\text{HNO}_3$  and cone  $\text{H}_2\text{SO}_4$ , it gives p-nitro acetanilide alongwith a little amount of o-isomer. In this process furning  $\text{HNO}_3$  in the presence of cone  $\text{H}_2\text{SO}_4$ , gives nitronium ion  $(\text{NO}_2^+)$  which attack on acetanilide to form p-nitro acetanilide through the cyclopentadienyl cation (intermediate) formation.

$$HNO_3 \div H_2SO_4 \longrightarrow NO_3^- + H_2O + HSO_4^-$$
  
Nitronium ion





$$\dot{H} + \dot{H}SO_4 \longrightarrow H_2SO_4$$

#### Reagents

Acctanitide — 10 g Gl.  $CH_3COOH$  — 10.4 mL Furning  $HNO_3$  — 4.4 mL Conc  $H_2SO_4$  — 20 mL Ico

#### Procedure

Dissolve 10.0 g of acetanilide in 10.4 mL of glacial acetic acid, taken in 100 mL beaker (warm

100 g of crushed ice, whereby the crude attroacetanilide is precipitated. The product is diluted with 100 mL of cold water and allowed to stand for 10 min, o-nitro acetanilide (a little amount formed) goes into the solution while  $\rho$ -isomer remains insoluble. Filter it at pump, wash thoroughly with cold water and drain well. Recrystallise 1 g of it from alcohol.

#### Result

- (i) Weight of the crude p-nitroacetanilide =
- (ii) Weight of recrystallised p-nitroacetardlide = ..., g
- (jii) Yield : 8 g (iv) MP : 212°C
- (v) Appearance : Colourless crystals

#### **Precautions**

- (i) The temperature of the flask containing reaction mixture should be maintained below 10°C.
- (ii) To get good yield, reagents should be of pure quality.
- (iii) All the reagents are concentrated, so used yeary carefully.

### Experiment 7

### **Aniline Yellow**

#### Object

To prepare aniline yellow (p-amino azo benzene) from diazo amino benzene.

#### Principle

When diazo amino benzenc is heated with aniline and a little amount of aniline hydrochloride at about 40°C for a short time. It gives p-amino azo

$$\sim$$
 N--N--N--NH<sub>2</sub>

a amino azo henzene

The mechanism of the reaction is based on the equilibrium involving the diazo amino compound, phenyl diazoniam chloride and aniline.

The reaction takes place between the two latter compounds under weakly acidic conditions.

#### Reagents

Diazo amino benzenc ... 5 g
Aniline ... 14 mL
Aniline hydrochloride ... 2.5 g
Gl. acetic acid ... 15 mL

#### Procedure

In a 150 mL conical flask, dissolve 5 g of diazo amino benzene in 14 mL of aniline and to it add 2.5 g of finely powdered aniline hydrochloride, warm the contents with occasional shaking on a water bath at 40–45°C for about one hour. Now allow the mixture to stand at room temperature for next 14 min and add 15 mL of gl acetic acid which has been previously diluted with an equal volume of water (ie, 30 mL dil acetic acid). Shake the contents vigorously and allow again to stand for another 15 min. Filter the crude product at pump, wash with water and dry. Recrystallise 1 g from CCl<sub>4</sub> or dil alcohol.

#### Result

(i) Weight of crude aniline yellow = ...... g

- (ii) Weight of recrystallised aniline yellow = ......, g
- (iii) Yield: 3.5 g
- (iv) M.P.: 125°C
- (v) Colour: Yellow crystals.

#### **Precoutions**

- (i) After the addition of acetic acid to the reaction mixtuere, it should be shaken for sufficient time in order to convert excess of aniline in the form of its soluble acetate.
- (ii) To get good yield, reagent used should be of pure quality.

## Experiment 8

#### lodoform

#### Object

To prepare iodoform from acetone.

#### Principle

Acetone when treated with porassium iodide and sodium hypochorite (NaOCI), gives iodoform.

$$\begin{array}{c} \text{NaOCl} + \text{Kl} & \longrightarrow \text{NaOI} + \text{KCl} \\ \\ \text{H}_3\text{C} & \longrightarrow \\ \text{II}_3\text{C} & \longrightarrow \\ \\ \text{II}_3\text{C} & \longrightarrow \\ \\ \hline & & \longrightarrow \\ \\ \hline & & \\ \\ \text{NaOH} & \rightarrow \\ \\ \text{CH}_3\text{COONa} + \\ \\ \text{CHI}_3 & \longrightarrow \\ \\ \end{array}$$

#### Reagents

Acetone	$2.5 \mathrm{mL}$
KĮ	$7.5\mathrm{g}$
NaOCI (5%)	70–80 mL

#### Procedure

Take the solution of 7.5 g of potassium iodide (in 125 mL of water) in a 200 mL round bottom flask. Now add 2.5 mL acetone to it. Now stir the content and add slowly 5% NaOCI solution with frequent shaking, till the complete precipitation of iodoform (about 70-80 mL are required) occurs. Allow the contents to stand for 15 minutes and filter the product at pump, wash with cold water and recrystallise 1 g of it from alcohol.

#### Result

CTS asset to the second and a

(ii) Weight of recrystallised iodoform = ...... g

(iii) Yield : 4 g (iv) M.P. : 119°C

(v) Colour : Yellow crystals

#### Precautions

To get good yield reagents should be of pure quality preferably of B.D.H.

#### TITRIMETRIC EXERCISES

### Experiment 9

### Na<sub>2</sub>CO<sub>3</sub> vs HCl Titration

#### Object

To prepare  $\frac{N}{10} \mathrm{Na_2CO_3}$  standard solution and

find out the strength of the supplied  ${\rm Na}_2{\rm CO}_3$  solution using hydrochloric acid as an intermediate solution.

#### Principle

The titration of Na<sub>2</sub>CO<sub>3</sub> vs HCl is a neutralisation titration (acidimetry and alkalimetry) which involve the neutralisation of an acid with a base, eg. sodium carbonate is attacked by dil HCl in the following way

$$Na_2CO_3 + 2HCI \longrightarrow 2NaCI + H_2O + CO_2 \hat{1}$$

#### Indicator

Methyl orange (Dissolve 1 g of methyl orange in  $1\,\mathrm{L}$  water).

#### End Point

Appearance of light pink colour.

#### Intermediate Solution

Intermediate solution (HCl) can be prepared by diluting concentrated hydrochloric acid one

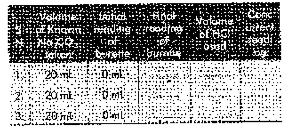
#### Procedure

Rinse the burette with intermediate HCl solution and fill with it. Remove the air bubbles, if there, by opening the stopcock. Now pipette out 20 ml, of this sodium carbonate solution in a conical flask and add 2-3 drops of methyl orange indicator. Then, gradually add HCl solution from the burette into the solution of conical flask with continuous shaking till a light pink colour just appears. Light pink colour will indicate the end-point. Repeat the process till concurrent readings are obtained.

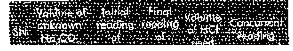
Now, wash the pipette with water and ringe it with supplied Na<sub>2</sub>CO<sub>3</sub> solution. Pipette out 20 mL of this solution in a clean conical flask. Repeat the titration, using the same HCl solution in the burette, as usual.

#### **Observations**

- (i) Weight of empty weighing tube  $(x) = \dots$  §
- (ii) Weight of weighing tube  $+ \text{Na}_2\text{CO}_3(y) = \dots g$
- (iii) Weight of sodium carbonate  $(Na_2OO_3)(x) = y x = \dots g$
- (iv) Volume of HCl used with 20 mL of known (prepared) Na  $_2\text{CO}_3$  solution



(v) Volume of HCl used with 20 mL of unknown (supplied) Na  $_2$ CO  $_3$  solution.



Weight of Na<sub>2</sub>CO<sub>3</sub> in 100 mJ, = ....... x 4  
= ....... g/L  
Normality of Na<sub>2</sub>CO<sub>3</sub> (prepared)  
= 
$$\frac{\text{Strength } (g/L)}{\text{Eq. wt. of oxalic acid}}$$
  
 $-\frac{......}{53}$  N

(ii) For the titration using standard Na<sub>2</sub>CO<sub>3</sub> solution

$$egin{aligned} N_1 V_1 &= N_2 V_2 \ Na_2 CO_3 & HCF \ (Known) \end{aligned}$$

$$N_2$$
 = ........ N  
r the titration using samplied

(iii) For the titration using supplied Na  $_2\mathrm{CO}_3$  solution

$$N_3 V_3 = N_4 V_4$$
  
 $N_{32} CO_3$  HCI [:  $N_4 = N_2$ ]  
 $N_5 = \dots N$ 

Strength of Na<sub>2</sub>CO<sub>3</sub> in  $g/L = N_3 \times Eq$ , wt. of Na<sub>2</sub>CO<sub>3</sub> ···..., g/L

#### Result

The strength of supplied sodium carbonate solution is ... g/l.

Note: (i) In welcontery and alkaliment, the chiese of indicators mainly depends apper the mature of the weld, and alkalies used. Methyl orange, plean holistolein are some of the important indicators used in it esettlements. (ii) As no indicator gives current results in the through of weak axide against weak bases, such attractors are to be grouded.

## Experiment 10

## Oxalic Acid vs KMnO<sub>4</sub> Titration

Object

To prepare N/30 oxalic acid standard solution and find out the strength of the supplied oxalic acid solution using potassium permanganate as an intermediate solution.

#### Principie

This is an example of Redox titrations, in which a reducing agent (as oxalic acid) is estimated by titrating it with a standard solution of oxidising agent (as KMnO<sub>4</sub>). Such reactions are

accompanied by the change in valency of ions. In these thrations oxidation and reduction takes place simultaneously ie, while one substance is being oxidised, the other one is being reduced.

$$\begin{array}{ccc} 2 \text{KMnO}_4 + 3 \text{H}_2 \text{SO}_4 & \longrightarrow & \text{K}_2 \text{SO}_4 + 2 \text{MnSO}_4 \\ \text{Oxidising} & & & \\ & & & + 3 \text{H}_2 \text{O} + 5 \text{[O]} \\ & & & & \\ & & & \text{COOII} \\ 5 & & & + 5 \text{[O]} & \longrightarrow & 5 \text{H}_2 \text{O} + 10 \text{CO}_2 \uparrow \\ & & & \\ & & & \text{COOH} \\ \text{Reducing agent} \end{array}$$

#### Indicator

The last drop of  $KMnO_4$  itself acts as an indicator (self indicator).

#### End Point

Appearance of light pink colour.

#### Intermediate Solution

Dissolve 0.2107 g of KMnO<sub>4</sub> (potassium permanganate) in 200 mL measuring flask with distilled water to prepare  $\frac{N}{30}$  solution of KMnO<sub>4</sub>.  $\frac{N}{30}$  solution of KMnO<sub>4</sub> is an intermediate solution.

#### Standard Solution

Dissolve 0.525 g of oxalic acid (AR) in a 250 mL measuring flask with distilled water to prepare a standard solution of oxalic acid (approx.  $\frac{N}{30}$ )

#### **Procedure**

Rinse the burette with intermediate solution of  $KMnO_4$  and fill the burette with it. If there is any air bubbles in burette, then

remove them by opening the stopcock. Rinse the pipette with oxalic acid solution and draw out 20 mL of it in a clean conical flask. Add one full test tube of dil H<sub>2</sub>SO<sub>2</sub> in it and heat the flask to 70°-80°C. Gradually add KMnO<sub>4</sub> solution from the burette into this warm solution with continuous shaking till a light pink colour just appears. Repeat this process until concurrent readings are obtained.

Now wash the pipette with water and rinse it with supplied (anknown) oxalic acid solution. Now pipette out 20 mL of this solution in a clean conical flask. Repeat the titration, using the same KMnO<sub>4</sub> solution in the burette, as usual.

#### **Observations**

- (i) Weight of empty weighing tube  $(x) = \dots$  2
- (ii) Weight of weighing tube + oxalic acid  $(H_2C_2O_4)(y) = \dots$  g
- (iii) Weight of oxalic acid  $(H_2G_2O_4)(z) = y - x = .......g$
- (iv) Volume of KMnO<sub>4</sub> used with 20 mL of known (prepared) uxalic acid solution

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20 mL 20 mL	to <b>以可能</b> 的tated	

(v) Volume of KMnO<sub>4</sub> used with 20 mL of unknown (supplied) oxalic acid solution.



#### Calculations

(i) Weight of oxalic acid dissolved in 250 mL measuring flask  $\pm z = .........g$ Weight of oxalic acid in 1000 mL  $= \frac{.... \times 1000}{250} = .... g/L$ 

Normality of oxalic acid (prepared)

$$\frac{\dots}{63.04} \text{ N} \times 20 \text{ roL} = N_2 \times \dots$$

$$N_2 = \dots, N$$

(iii) For the titration using supplied oxalic acid solution

$$N_3V_3 \leftarrow N_4V_4$$
  
Oxalic acid KMnO<sub>1</sub> [ $v N_2 = N_2$ ]  
(unknown)

$$N_3 = \dots N$$

Strength of exalic acid in  $g/L = N_{ij} \times \mathbb{N}q$ , we, of exalic acid

$$=$$
 ,.....  $g/L$ 

#### Resul

The strength of supplied oxalic acid solution is ...... g/L

#### **Precoutions**

- The oxalic acid solution with dil H<sub>2</sub>SO<sub>4</sub> is heated to near about 70° – 80°C.
- (ii) Sulphuric acid should be in excess otherwise a brown ppt due to formation of MnO<sub>2</sub> will be formed.
- (iii) This ritration cannot be carried out in presence of acid like HNO<sub>3</sub> and HCl because HNO<sub>3</sub> itself is an oxidising agent, so it will interfere with the oxidising action of KMnO<sub>4</sub> and HCl reacts chemically with KMnO<sub>3</sub> solution.

### Experiment 11

FeSO<sub>4</sub>  $\cdot$  (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>  $\cdot$  6H<sub>2</sub>O (Mohr's salf) vs KMnO<sub>4</sub> Titration

#### Object

Prepare  $\frac{N}{30}$  ferrous ammonium sulphate (Mohr's salt) standard solution and find out the

salt) is ferrous sulphate, which is oxidised to ferric sulphate by acidified potassium permanganate as follows.

$$\begin{array}{c} 2\text{KMnO}_4 + 3\text{H}_2\text{SO}_4 & \longrightarrow \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 \\ & + 3\text{H}_2\text{O} + 5[\text{O}] \\ \\ [2\text{FeSO}_4 + \text{H}_2\text{SO}_4 + [\text{O}] & \longrightarrow \text{Fe}_2(\text{SO}_4)_3 \\ & + \text{H}_2\text{O}] \times 5 \\ \hline \\ 2\text{KMnO}_4 + 8\text{H}_2\text{SO}_4 + 10\text{FeSO}_4 & \longrightarrow \text{K}_2\text{SO}_4 \end{array}$$

 $+ 2 \text{MnSO}_4 + 5 \text{l/e}_2 (\text{SO}_4)_3 \div 8 \text{H}_2 \text{O}$ 

#### Indicator

The last drop of KMnO4 itself acts as an indicator (self indicator).

#### End Point

Appearance of light pink colour

#### Intermediate Solution

Dissolve 0.2107 g of KMnO<sub>4</sub> (potassium permanganate) in 200 mL measuring flask with distilled water to prepare  $\frac{N}{30}$  solution of KMnO<sub>4</sub>.

 $\frac{N}{20}$  solution of KMnO<sub>4</sub> is an intermediate solution.

#### Standard Solution

Dissolve 3.2666 g ferrous ammonium sulphate (Mohr's sait) (AR) in 250 mL meas Ling flask with distilled water to prepare a standard solution of nearly  $\frac{N}{30}$  ferrous ammonium sulphate.

#### **Procedure**

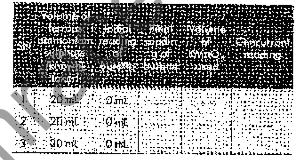
Rinse the burette with intermediate solution of KMnO<sub>4</sub> and fill with it. If hurette contains any air bubble, then remove it by opening the stopcock. Now, rinse the pipette with standard ferrous ammonium sulphate solution and draw out 20 mL of it in a clean conical flask. Add one small test tube of dil 11,50, and titrate with KMnO, taken in burette. In the beginning KMnO<sub>4</sub> should be added in drops with constant shaking. At the end point when all the ferrous salt has been exidised, the slight excess of KMnO<sub>4</sub> will make the solution light pink. The titration is repeated till concurrent readings are obtained.

Now wash the pipette with water and rinse it with supplied unknown ferrous ammonium sulphate solution and pipette out 20 mL of this solution in a clean conical flask. Repeat the tiration, using the same KMnO4 solution in the burette, as usual.

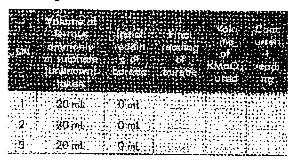
#### Observations

- (i) Weight of empty weighing tube  $(x) = \dots g$
- (ii) Weight of weighing tube ferrous ammonium sulphate  $(y) = \dots g$
- (iii) Weight of ferrous ammonium suiphate  $(\boldsymbol{z}) = \boldsymbol{\gamma} - \boldsymbol{x}$

(iv) Volume of KMnO4 used with 20 mL of known (prepared) ferrous ammonium sulphate



(v) Volume of KMnO<sub>4</sub> used with 20 mL of unknown (supplied) ferrous ammonium sulphate



#### Calculations

(i) Weight of ferrous ammonium sulphate dissolved in 250 mL measuring flask = z = ...gweight of ferrous ammonium sulphate in 1000 mL

$$= \frac{... \times 1000}{250}$$
$$= ... \times L$$

Normality of ferrous ammonium sulphate (prepared)

$$=\frac{\dots}{63.04}\,\mathrm{N}$$

(ii) For the titrations using standard ferrous ammonium sulphate solution

$$N_1V_1 = N_2V_2$$
  
Mohr's salt KMnO<sub>4</sub>  
(Known)

$$\frac{...}{63.04}$$
 N × 20 mL =  $N_2$  × ...

$$N_2 = ..., N$$

(iii) For the titration using supplied ferrous ammonium sulphate solution

$$N_3V_3 = N_4V_4$$

ferrous ammonium sulphate KMpO<sub>s</sub> (unknown)

$$[ \cdot N_4 = N_2]$$

$$N_2 = ... N$$

Strength of ferrous ammonium sulphate in  $g/L = N_3 \times Eq$ . wt. of ferrous ammonium sulphate

$$= ...g/L$$

#### Result

The strength of supplied Mohr's salt solution is . .  $\chi/L$ 

### Precautions

- (i) This titration is carried out at laboratory temperature.
- (ii) Sulphuric acid should be present in excess otherwise a brown ppt due to formation of MnO<sub>2</sub> will be formed.

#### QUALITATIVE INORGANIC ANALYSIS

#### Experiment 12

#### Principle

Acidic radicals or anions of first group, when treated with dil H<sub>2</sub>SO<sub>4</sub>, evolve gases with characteristic colour and smell. On the basis of action of these gases, anions are identified.

#### Tests for the anions of group I radicals

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## Reactions involved in the test of carbonate $(CO_3^2)$

(i) Na<sub>2</sub>CO<sub>3</sub> + 2HCl 
$$\longrightarrow$$
 2NaCl   
 + H<sub>2</sub>O + CO<sub>2</sub>↑ Colouriess, odouriess gas

(ii) 
$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_2 \lor Calcium carbonste (Wilky)$$

(iii) 
$$GaCO_3 + H_2O + CO_2(excess) \longrightarrow Ca(HCO_3)_2$$
  
Calcium bicarbonate (colourless)

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## Reactions involved in the test of sulphide $(S^2)$

(i) 
$$Na_2S + H_2SO_2 \longrightarrow H_2S + Na_2SO_4$$

(ii) 
$$(CH_3COO)_2Pb+H_2S \longrightarrow PbS \ _{Black}^{$\xi$} + 2CH_3COOH$$

Na<sub>4</sub>[Fe(CN)<sub>5</sub>NOS] Sodium thiositroprusside (violet)

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## Reactions involved in the test of nitrite (NO<sub>2</sub>)

(i) 
$$NaNO_2 + 11_2SO_4 \longrightarrow Na_2SO_4 + 2HNO_2$$
  
 $3HNO_2 \longrightarrow HNO_3 + 2NO \uparrow + i0_2O$   
 $2NO + O_2 \longrightarrow 2NO_2 \uparrow$   
(Brown)

(ii) 
$$2KI + 2NO_2 \longrightarrow 2KNO_2 + I_2$$
  
 $I_2 + starch \longrightarrow Blue colour$ 

## Experiment 13

### Analysis of Second Group Anions

#### Object

To identify the acidic radicals or anions of group second [ie, Gl], Br.,  $I^-$ ,  $NO_3^-$ ].

#### Principle

Acidic radicals or anions of second group give no response with dil acids but with conc acids, they evolve gases with characteristic colour and smell, thus can be identified by using conc acids.

#### Tests for the anions of group II radicals

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# Reactions involved in the test of chloride $\{CI^-\}$

- (i) NaCl =  $H_2SO_4 \longrightarrow NaHSO_4 + HCl \uparrow$ conc Pungent smelling gas
- (ii)  $HCl + NH_4OH \longrightarrow NH_4Cl \uparrow + H_2O$ White fumes
- (iji)  $2\text{NaCl} + \text{MnO}_2 + 3\text{H}_2\text{SO}_4 \longrightarrow 2\text{NaHSO}_4 + \text{MnSO}_4 + 2\text{H}_2\text{O} + \frac{\text{Cl}_2}{\text{Yellowish-green}}$
- (iv) Chromyl chloride test

$$4NaCl + K_2Cl_2O_7 - 3H_2SO_4 \longrightarrow 2CrO_2Cl_2$$

$$Chromyl chloride (orange yellow gas)$$

$$+ 2Na_2SO_4 + K_2SO_4 + 3H_2O$$

$$CrO_2Cl_2 + 4NaOH \longrightarrow Na_2CrO_4$$
Vallage

# Reactions involved in the test of bromide (Br")

- (i) NaBr +  $II_2SO_4 \longrightarrow NaHSO_4 + HET \hat{1}$
- 2)  $HBr + H_2SO_4(cone) \longrightarrow 2H_2O + SO_2 \uparrow + Br_2 \uparrow$ Reddish brown
- (ii)  $2NaBr + MnO_2 + 3H_2SO_4 + \rightarrow 2NaHSO_4 + MnSO_4 + 2H_2O + Br_2 \uparrow$ Reddish brown
- (iii) NaBr + AgNO<sub>3</sub>  $\longrightarrow$  AgBr  $\vee$  + NaNO<sub>3</sub> Pale yellow

AgBr + 
$$2NH_4OH \rightarrow [Ag(NH_3)_2]Br + 2H_2O$$
Diammine silver (I)
bromide

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$$\begin{array}{c} {\rm H_2SO_4 + 2HI \longrightarrow H_2 } \uparrow + {\rm SO_2 + 2H_2O} \\ {\rm Violet} \\ {\rm vapours} \end{array}$$

(ii)  $2\text{Na}I + \text{MnO}_2 + 3\text{H}_2\text{SO}_4 \longrightarrow \text{I}_2 \uparrow + 2\text{Na}\text{HSO}_4$ 

 $\vdash MnSO_4 + 2H_2O$ 

- (iii)  $l_2$  + starch  $\longrightarrow$  Blue colour
- (iv) Nal+  $AgNO_3 \longrightarrow AgI_{\psi} + NaNO_3$ yellow

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# Reactions involved in the test of nitrate $(NO_3)$

(i) 
$$NaNO_3 + H_2SO_4 - \longrightarrow NaHSO_4 + HNO_3$$
  
(conr.)

$$\begin{array}{ccc} 4 \text{HNO}_3 & \longrightarrow 4 \text{NO}_2 \uparrow + \text{O}_3 \uparrow + 2 \text{H}_2 \text{O} \\ \text{Brown} \end{array}$$

(ii) 
$$3 \text{ Cu} + 8 \text{HNO}_3 \longrightarrow 3 \text{Cu}(\text{NO}_3)_2 + 2 \text{NO} \uparrow$$

$$2NO + O_2 \longrightarrow 2NO_2 \uparrow$$

(iii) Ring test

$$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \longrightarrow 3Fe_2(SO_4)_g$$

$$\begin{array}{ccc} \operatorname{FeSO}_4 + \operatorname{NO} & \to \operatorname{FeSO}_4 \cdot \operatorname{NO} \\ & & \operatorname{Ferrous\ nitroso} \\ & & \operatorname{sulphate} \\ & & (\operatorname{Rtown\ ring}) \end{array}$$

## Experiment 14

# Analysis of Third Group Anions

#### **Object**

To identify the anions of group third  $[SO_4^2]_k$ 

### Principle

Third group radicals are identified on the basis of precipitate obtained.

Tests for group III radicals

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# Reactions involved in the test of sulphate ( $SO_4^{2-}$ )

(i) 
$$Na_2SO_4 + BaCI_2 \longrightarrow BaSO_4 \downarrow + 2NaCl$$
  
White

(ii) 
$$(CH_3COO)_2Pb + Na_2SO_4 + \rightarrow PbSO_4\downarrow$$
 white  $+ 2CH_3COONa$ 

## Experiment-15

## Analysis of Basic Radicals by Dry Tests

### Object

To identify basic radicals or cations in a mixture

#### Flame Test

**Principle**: This test is based upon the fact that in a state of high ionisation of chloride some of the cations impart characteristic colour to the flame, as the cation absorbs energy from the flame and transmit the same as light of characteristic colour.

Procedure: Make a loop at the tip of platinum wire round the point of pencil and clean it by conc HCl. Now take some of the paste of substance (substance + 2-3 drops conc HCl) on it and introduce it into the edge of a non-luminous bunsen flame. Observe the colour of flame

#### Observation

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#### Borax Bead Test

Principle: Borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>3</sub>O) on strong heating, first loses its water of crystallisation and then shrinks to form a transparent glassy bead of sodium metaborate (NaBO<sub>2</sub>) and boric anhydride ( $B_2O_3$ ).

$$Na_2B_4O_7 \cdot 10H_2O \longrightarrow Na_2B_2O_7 - 10H_2O$$

$$Na_{-}R.O_{+} \longrightarrow 2NaBO_{2} + B_{2}O_{3}$$

$$\begin{aligned} \text{CuO} + \text{B}_2\text{O}_3 & \longrightarrow \text{Cu}(\text{BO}_2)_2 \\ \text{CuSO}_4 & \div \text{B}_2\text{O}_3 & \longrightarrow \text{Cu}(\text{BO}_2)_2 + \text{SO}_3 & \uparrow \\ & \text{(Green when bott blue when cold)} \end{aligned}$$

#### (b) Reducing flame

$$2Cu(BO_2)_2 + C \longleftrightarrow 2CuBO_2 - B_2O_3 + CO \uparrow$$
Colourless

$$2\text{CuBO}_2 + \text{C} \longrightarrow 2\text{Cu} \downarrow + \text{B}_2\text{O}_3 \div \text{CO} \uparrow \\ \text{8cd}$$

Procedure: Make a loop at the tip of the wire, heat in the flame and dip it into borax powder. Heat if strongly when borax loses its water of crystallisation, swells up and is finally converted into a transparent glassy bead. Now this bead is touched with mixture under examination. Heat the bead strongly in non-luminous flame and then in turninous flame.

#### Observation

Njekel (NR)	-
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increifie) Yellow Bottle green CLaste (ct)   Digit sheeps area   Colombes or red	

## **Experiment 16**

### Analysis of Basic Radicals by Wet Tests

#### Object

To identify basic radicals or cation by wet tests.

#### Tests for the cations of various groups

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	KI solution:	<b>40</b>	con istned

### Reactions involved in the test of lead (Pb<sup>21</sup>)

(i) 
$$Pb(NO_3)_2 + 2HCl \longrightarrow PbCl_{2^{\prime\prime}} + 2HNO_3$$

(b) 
$$PbCl_2 + K_2CrO_4 \longrightarrow PbCrO_4 + 2KCl_{Yellow}$$

(c) 
$$PbCl_2 + 2KI \longrightarrow Pbl_2 + 2KCl$$
  
Yellow

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## Reactions involved in the test of copper $(Cv^{2-})$

(i) 
$$\operatorname{GuCl}_2 + \operatorname{II}_2 S \longrightarrow \operatorname{GuS} \downarrow + 2\operatorname{HCI}_{\operatorname{Black ppr}} + 2\operatorname{HCI}_{\operatorname{Black ppr}}$$

(ii) 
$$3\text{CuS} + 8\text{HNO}_3 \longrightarrow 3\text{Cu(NO}_3)_2 + 2\text{NO}\uparrow + 3\text{S}\downarrow + 4\text{H}_2\text{O}$$

$$Cu(NO_3)_2 + 4NH_4OH \longrightarrow$$

 $\begin{array}{l} [\mathrm{Cu}(\mathrm{NH_3})_4](\mathrm{NO_3})_2 & + 4\mathrm{H_2O} \\ \mathrm{Tetransmise\ copper\ (II)\ nitrate} \\ & (\mathrm{Blue\ colour)} \end{array}$ 

(iii) 
$$[Cu(NH_3)_4](NO_3)_2 + 4CH_3COOH \longrightarrow Cu(NO_3)_2 + 4CH_3COONH_4 2Cu(NO_3)_2 + K_4[Fe(CN)_6] \longrightarrow$$

Cti<sub>2</sub>[Fe(CN)<sub>6</sub>] ↓ + 4KNO<sub>3</sub> Copper hexacyano ferrate (ft) (Chocolate red)

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## Reactions involved in the test of iron (Fe 3+) and aluminium (Al<sup>3+</sup>)

#### For iron

(i) 
$$2 \text{FeCl}_2 + 8 \text{HNO}_3 \longrightarrow 2 \text{Fe(NO}_3)_3 + 4 \text{HCl} + 2 \text{NO}_2 \uparrow + 2 \text{H}_2 \text{O}_3 \uparrow$$

(ii) 
$$FeGl_3 + 3NH_4OII \longrightarrow Fe(OH)_3^{\frac{1}{2}}$$
Brown

(iii) 
$$Fe(OH)_3 \div 3HCl \longrightarrow FeCl_3 + 3H_2O$$

(iv) (a) 
$$\operatorname{FeCl}_3 + \operatorname{3NH}_4\operatorname{CNS} \longrightarrow \operatorname{Fe}(\operatorname{CNS})_3 + \operatorname{3NH}_4\operatorname{Cl}$$
  
Fermic thiocyanate  
(Blood red colour)

(b)  $4 \text{FeCl}_3 + 3 \text{K}_4 [\text{Fe}(\text{CN})_6] \longrightarrow$  $\begin{array}{l} Fe_4 \lceil Fe(CN)_6 \rceil_3 \downarrow + 12 KGl \\ \text{Ferric ferroryande} \end{array}$ (Deep blue)

#### For aluminium

(i) 
$$AlCl_3 + 3NH_4OH \longrightarrow Al(OH)_3 \downarrow$$
White

 $\div \, 3N H_{\Delta} C I$ 

 $\rightarrow$  Al(OH)<sub>3</sub> $\circ$ (iii) NaAlO<sub>2</sub> + NH<sub>4</sub>Cl + H<sub>2</sub>O<sub>2</sub>  $\pm$  NaCl  $\pm$  NH<sub>3</sub> 等的使的现在 海科 afficient bug Albeid for in ក្ខាត្តបក្ខ 🗼 🚓 🕍 m di MCI 670 NAC COME To one parti-Sportet red (V group (Ni<sup>44</sup>) odd Nift, CM parti Economical iji picasi നേർ ർണ്ടിന്റ glyswine To second Grey while IV group Dit V ponfusies.

### Reactions involved in the test of nickel $(Ni^{2+})$ and zinc $(Zn^{2+})$

#### For nickel

(i) 
$$NiCl_2 + H_2S \longrightarrow NiS \downarrow + 2HCl$$

(ii) 
$$3NiS + 2HNO_3 + 6HCl \longrightarrow 3NiCl_2 + 2NO\uparrow$$
Aqua-regia

$$+3$$$$$$$$$$$+411{}_{2}$$$$$

+ 2NH<sub>4</sub>OH + NiCl<sub>2</sub>

Nickel dimethyl glyoxime (Red)

 $+ 2NH_4Cl + 2H_2O$ 

(iii) 
$$Na_2ZnO_2 + H_2S \longrightarrow ZnS \downarrow + 2NaOH$$
  
White

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		countried. Vylite pot of	₩ g(cwp)(Ca <sup>2a</sup> )
	fort let port		continued.

## Reactions involved in the test of barium (8a<sup>2+</sup>) and calcium (Ca<sup>2+</sup>)

#### For barium

(i) 
$$BaCl_2 + (NH_4)_2CO_3 \longrightarrow BaCO_3 \downarrow + 2NH_4Cl$$
  
White

(ii) 
$$BaCO_3 + 2CH_3COOH \longrightarrow$$

(iii) 
$$(CH_3COO)_2Ba + K_2CrO_4 \longrightarrow BaCrO_4 \downarrow$$
  
Yeliov

+ 3CH<sub>3</sub>COOK

#### For calcium

(I) 
$$CaCl_2 + (NH_4)_2CO_3 \longrightarrow CaCO_3 \downarrow + 2NH_4Cl$$
  
White

(ii) CaCO<sub>3</sub> + 3CH<sub>3</sub>COOH 
$$\longrightarrow$$
 (CH<sub>3</sub>COO)<sub>2</sub>C<sub>3</sub> + H<sub>2</sub>O + CO<sub>2</sub>↑

(iii) 
$$(CH_3COO)_2C_0 + (NH_4)_2C_2O_4 \longrightarrow CaC_4O_4 \downarrow$$
 White  $+ 3CH_3COONH_4$ 

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## Reactions involved in the test of magnesium (Mg $^{2+}$ )

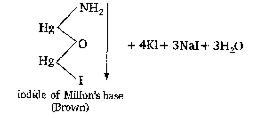
$$\begin{array}{c} \operatorname{Mg(NO_3)_2} + \operatorname{Na_3HPO_4} + \operatorname{NH_4OH} & \longrightarrow \\ \operatorname{Mg(NH_4)PO_4} \downarrow & + 2\operatorname{NaNO_3} + \operatorname{H_2O} \\ \operatorname{Magnesium ammonium} \\ \operatorname{phosphate} \\ (\operatorname{White}) \end{array}$$

<b>25. Constant</b>	
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## Reactions involved in the test of ammonium ion $(NH_A^+)$

(i) 
$$NH_4CI + NaOH \longrightarrow VaCI + H_2O + NH_3\uparrow$$

$$\begin{array}{ccc} \text{(iii)} & 2K_2HgI_1 & \pm 3NaOH \pm NH_4OH & --- \\ & \text{Potassium} & \text{intraction inclide} \\ & \text{(Nessler's reagent)} \end{array}$$



## Practical Based Questions

- Phenolphthalein is an indicator for acid-base titration, it exists as
  - (a) benzenoid form in acid and quinonoid form in basic solution
  - (b) quinonoid form in acid and benzenoid form in basic solution
  - (c) quinomoid form in both
  - (d) benzenoid form in both
- 2. Which statement is correct?
  - (a) Fc<sup>2-</sup> gives brown colour with ammonium thiocyanate
  - (b) Fe<sup>24</sup> gives blue precipitate with potassium fecticyanide
  - (c) Fe<sup>3+</sup> gives brown colour with potassium ferticyanide
  - (d) Fe<sup>3+</sup> gives red colour with porassium ferroryanide
- Four samples of acids and bases are taken for an experiment
  - (1) 100 mJ, of 1 M NaOH and 100 mL of 1 M HCl
  - (2) 100 mL of 2 M KOH and 100 mL of 1 M H-SO.
  - (3) 100 mL of 1 M CH<sub>2</sub>COOH and 100 mL of 1 M NaOH
  - (4) 100 mL of 0.5 M KOH and 100 mL of 0.5 M HNO.

Now for each sample eathalpy of neutralisation is calculated. Now the result shows that

- (a) enchalpy of neutralisation calculated in each case is found same
- (b) in case (1) and (4), the value of enthalpy of neutralisation is same
- (c) in case (l), (2) and (4) the value of enthalpy calculated is same
- (d) the value of enthalpy calculated is different for each sample
- 4. Which of the following will not give Lassaigne's

- During the preparation of acetanilide from aniline a small amount of zinc is added to the reaction mixture because
  - (a) zinc induces the precipitation
  - (b) zinc prevents the reduction of antime during the reaction
  - (c) zinc reduces the coloured impurities in the aniline and also prevents its oxidation during the reaction
  - (d) zinc form a white crystalline complex with and inc
- 7. An aqueous solution of colourless metal sulphate M gives a white precipitate with NH<sub>4</sub>OH. This was soluble in excess of NH<sub>4</sub>OH. On passing H<sub>2</sub>S through this solution a white ppt is formed. The metal M in the salt is
  - (a) Ca
- (b) Ba
- (c) Al.
- (d) Zn
- In the titration of oxalic acid vs potassium permanganate, potassium permanganate acts as
  - (a) external indicator (b) self indicator
  - (c) reductant
- (d) both (5) and (c)
- 9. In the preparation of p-niaro acetamilide from aniline, nitration is not done by nitrating muxture (a mixture of cond H<sub>2</sub>SO<sub>4</sub> and cond HNO<sub>3</sub>) because
  - (a) on nitration it gives o nitro acetanilide
  - (b) it gives a mixture of a- and p-mirro amiline
  - (c)  $-NH_2$  group gers oxidised
  - (d) it forms a mixture of o- and p-mixton acctanilide
- 10. In the reaction of KMnO<sub>4</sub> with an oxalate in acidic medium, MnO<sub>4</sub> is reduced to Mn<sup>2+</sup> and C<sub>2</sub>O<sub>4</sub><sup>2+</sup> is oxidised to GO<sub>2</sub>. Hence, 50 mL of 0.04 M KMnO<sub>4</sub> is equivalent to
  - (a) 100 mL of 0.1 M  $\rm H_2C_2O_4$
  - (b) 50 mL of 0.2 M H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>

- (c) in cold water egg albumin mix well whereas yolk get separated
- (d) cold water is purest form of water
- 12. Which of the following compounds cannot used in preparation of iodoform?
  - (a) CH<sub>3</sub>CHO
- (b) CH<sub>3</sub>COCH<sub>3</sub>
- (c) HCHO
- (d) 2-propanol
- In Lassaigne's test, the organic compound is fused with a piece of sodium metal in order to
  - (a) increase the ionisation of the compound
  - (b) decrease the melting point of the compound
  - (c) convert the ionic compound into a mixture of covalent compounds
  - (d) convert the covalent compound into a mixture of ionic compounds
- 14. In the kinetic study of reaction of iodide ion with hydrogen peroxide, a known volume of sodium thiosulphate solution is added to
  - (a) oxidise indide ion to fodine
  - (b) reduce iodine to iodide ion.
  - (c) form a soluble blue complex
  - (d) induce the reaction rate
- 15. A white sodium salt dissolves readily in water to give a solution which is neutral to litmus. When silver nitrate solution is added to the solution, a white precipitate is obtained which does not dissolve in dil HNO<sub>2</sub>. The anion could be
  - (a)  $CO_3^{2-}$
- (b) Ci<sup>-</sup>
- (c)  $SO_4^{2-}$
- (d)  $S^{2}$
- 16. In organic analysis, the reagent 2, 4 dinitro phenyl hydraxine is used for the detection of which of the following functional groups?
  - (a) Alcoltol
- (b) Acid
- (c) Aldehyde
- (d) Amines
- 17. Which of the following pairs has heat of neutralisation equal to 13.7 kcals?
  - (a) HCL NH<sub>2</sub>OH
- (b) HNO<sub>3</sub>, KOH
- (c) NaOH, CH<sub>2</sub>COOH (d) H<sub>2</sub>SO<sub>4</sub>, NH<sub>4</sub>OH
- 18. For preparing 250 mL of N/20 solution of Mohr's cast, the amount of Mohr's salt needed is
  - (a) 9.8 g
- (b) 4.9 g
- (c) 19.6 g
- (d) 3.2 g
- 19. 0.5 g mixture of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and KMnO<sub>4</sub> was treated with excess of KI in acidic medium. I<sub>2</sub> liberated required 100 cm<sup>3</sup> of 0.15 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution for titration. The percentage amount of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in the mixture is
  - (a) 85.36%
- (b) 14**.64**%
- (c) 58.63%
- (d) 26.14%

- An unknown inorganic compound (A) gave the following reactions
  - (1) The compound (A) on heating gave a residue, oxygen and an exide of nitrogen.
  - (2) An aqueous solution of compound (A), on addition of tap water gave a turbidity which is insoluble in nitric acid,
  - (3) The turbidity dissolved in NH<sub>4</sub>OH solution. Thus, the compound (A) is
  - (a) NaCl
- (b) AgCl (d) AgNO<sub>3</sub>
- (c) NaNO,
- An organic compound gave positive iodoform and Tollen's tests. The organic compound is
  - (a) CH<sub>3</sub>CH<sub>2</sub>OII
- (b) CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CHO
- (c) CH<sub>3</sub>CHQ\_
- (d) CH<sub>2</sub>COCH<sub>3</sub>
- One desires to prepare a positively charged sol of silver fodide. This can be achieved by
  - (a) adding a little AgNO<sub>3</sub> solution to KI solution in slight excess
  - (b) adding a little KI solution to AgNO<sub>3</sub> solution in slight excess
  - (c) mixing equal volumes of equimolar solutions of AgNO<sub>3</sub> and KI
  - (d) None of the above
- 23. During the test for halogens, sodium extract is heated with conc HNO<sub>2</sub>. The reason for this is
  - (a) to decompose NaCN and Na<sub>2</sub>S
  - (b) Na<sub>2</sub>S is soluble in HNO<sub>3</sub>
  - (c) NaCN is soluble in HNO<sub>3</sub>
  - (d) silver halides are insoluble in HNO<sub>3</sub>.
- 24. Which of the following will oxidise 126 g of oxalic acid ( $H_2C_2O_4 \cdot 2H_2O$ ) in acidic medium?
  - (a) 1/3 mole of K<sub>2</sub>Cr<sub>2</sub>O<sub>y</sub>.
  - **(b)** 2 mole of  $K_2C_{L_2}O_{\gamma}$
  - (c) 1/3 mole of KMnO,
  - (d) 5/2 mole of KMnO<sub>a</sub>
- 25. A laboratory reasont imparts green colours to the flame. On heating with solid K<sub>2</sub>Cr<sub>1</sub>O<sub>7</sub> and conc H<sub>2</sub>SO<sub>4</sub> it evolves a grange red gas. Identify the reasont.
  - (a) CaCl<sub>2</sub>
- (b) BaCl<sub>2</sub>
- (c) CuCl<sub>2</sub>
- (d) None of these
- 26. Which reagent can be used to identify nickel ion?
  - (a) Resorcinol.
  - (b) Dimethyl glyoxime
  - (c) Diphenyl benzidine
  - (d) Potassium ferrocyanide
- 27. The titration of Mohr's salt vs KMnO<sub>4</sub> is an

- KMnO<sub>4</sub> oxidises only ferrous salt to the ferric salt (no effect on other ions), but we can not use ferrous sulphate in place of Mohr's salt because
- (a) it is less stable than Mohr's sait
- (b) in air it oxidised to ferric sulphate
- (c) in air it loses water of crystallisation
- (d) All of the above
- 28. 3.92 g of ferrous ammonium sulphate (Mohr's sait) react completely with 50 mL  $\frac{N}{10}$  KMnO<sub>4</sub> solution. The percentage purity of the sample is
  - (a) 50
- (b) 76.4
- (c) 80
- (1) 39.2
- **29.** A compound liberates  $CO_2$  with NaHCO<sub>2</sub> and also gives colour with neutral FeCl<sub>3</sub> solution. The compound can be

- 30. Positive Beilstein's test for halogens shows that
  - (a) a halogen is definitely present
  - (b) a halogen may be present
  - (c) a halogon is absent
  - (d) None of the above
- 31. Which of the following statement is wrong about antiline yellow?
  - (a) It is carcinogenic
  - (b) It is also called p-amino azobenzene
  - (c) It is an acid dye
  - (d) It is also called 4-phenyl azoamline
- 32. Leveling balb is used during experiment to study kinetics of the dissociation of hydrogen peroxide

- (b) in acidic solution, dichromate ions are converted to chromate ions
- (c)  $(NH_4)_2Ci_2O_7$  on heating undergo exothermic decomposition to give  $Ci_2O_3$
- (d) Potassium dichromate is used as a citrant for estimation of Fe<sup>2-</sup> jours
- 34. Which of the following reagents can be used to distinguish between sodium earbonate and sodium sulphite?
  - (a) Lime water
  - (b) Baryts water
  - (c) Acidified K<sub>2</sub>Ct<sub>2</sub>O<sub>7</sub> solution
  - (d) H<sub>2</sub>SO<sub>4</sub> solution
- 35. An aqueous solution of 6.3 g oxalic acid dihydrate is made up to 250 mL. The volume of 0.1 N NaOH required to completely neutralize 10 mL of this solution is
  - (a) 40 mL
- (b) 20 mL
- (c) 10 mL
- (d) 4 mL
- 36. In case of weak acid and strong base, the heat of neutralisation is less than 13.7 keal because some part of heat is utilized in
  - (a) dissociation of base
  - (b) association of base
  - (c) dissociation of acid
  - (d) association of acid
- The methods used for the preparation of lyophilic and lyophobic sols are respectively
  - (a) oxidation and reduction
  - (b) dissolution in water and peptisation
  - (c) peptisation and oxidation
  - (d) All of the above
- 38. Which of the following substances is not used in the preparation of Mohr's salt?
  - (a) Ferrous sulphate (b) Ammonium sulphate
  - (c)\*Dil. sulphuric atid (d) All are used
- 39. The gas liberated on heating a mixture of two salts with NaOH, give a reddish brown precipitate with an alkaline solution of K<sub>2</sub>Hgl<sub>2</sub>. The aqueous solution of the mixture on treatment with BaCl<sub>2</sub>

- (c)  $NH_4^+$ ,  $Fe^{2+}$ ,  $SO_4^{2+}$ ,  $Cl^-$
- (d)  $NII_4^+, Ca^{2+}, SO_4^{2+}, CI^-$
- 40. An organic compound does not reduce Tollen's reagent and Febling's solution but gives red colour with ceric arimonium nitrate, then the organic compound is
  - (a) an alcohol
- (b) an aldehyde
- (c) a phenol
- (d) a ketone
- 41. In the reaction  $2H_2O_2 \longrightarrow \stackrel{\Gamma}{\longrightarrow} 2H_2O + O_2$ , the rate of reaction
  - (a) decreases as cone of I for increases
  - (b) increases as cond of I ion increases
  - (c) increases in the presence of UV light
  - (d) both (b) and (c)
- **42.** A solution of one mole of copper sulphate is prepared at infinite dilution. It is expressed as follows

$$CuSO_4(s) + (aq) \longrightarrow CuSO_4(aq)$$

On adding some solvent to this solution, the enthalpy of solution

- (a) increases
- (b) decreases
- (c) remains the same
- (d) may be increased or decreased
- 43. If Cl<sub>2</sub> gas is passed into aqueous solution of KI containing some CCl<sub>4</sub> and the mixture is shaken, then
  - (a) upper layer become violet
  - (b) lower layer becomes violet
  - (c) homogeneous violet layer is formed
  - (d) None of the above
- 44. 1.5 g pyrolusite ore is treated with 50 mL of N-oxalic acid and dil H<sub>2</sub>SO<sub>2</sub>. The remaining acid is transferred to 250 mL measuring flask, 25 mL of this solution requires 30 mL of N/10 KMnO<sub>2</sub> for litration. The percentage of MnO<sub>2</sub> in the sample of pyrolusite is
  - (a) 58%
- (b) 65%
- (c) 45%
- (d) 35%
- 45. Experiment to study kinetics of the dissociation of hydrogen peroxide must be performed by group of two or three so that
  - (a) when one is recording data other should be swirling flask at constant rate
  - (b) experiment can be performed by one student only as outcomes are independent on rate of mixing of mixture 1 and 3

- (c) for safety purpose
- (d) None of the above
- 46. In III group precipitation, NH<sub>4</sub>Cl is added before adding NII<sub>4</sub>OH to
  - (a) decrease cone of OHT
  - (b) prevent interference of  $PO_4^3$
  - (c) increase cone of CIT
  - (d) increase conc of O! ☐ ion
- 47. The enthalpy of neutralisation of a weak acid in 1 M solution with a strong base is -56.1 kJ mol<sup>-1</sup>. If the enthalpy of ionisation of acid is 1.5 kJ mol<sup>-1</sup> and enthalpy of neutralisation of the strong acid with a strong base is -57.3 kJ eq <sup>1</sup>. What is the percentage ionisation of the weak acid in molar solution (assume the acid is monobasic)
  - (a) 25
- (b) 20
- (c) 15
- 01 (b)
- 48. During the preparation of acetamilide from author, the excess of acetic anhydride and prolonged heating should be avoided because
  - (a) it is oxidisced
  - (b) it is reduced
  - (c) diacetyl derivative is formed
  - (d) Iriaceryl derivative is formed
- 49. At 298 K, the heat of solution of CuSO<sub>4</sub>(s) is -91.21 kJ mol<sup>-1</sup> and that of CuSO<sub>4</sub>·5H<sub>2</sub>O(s) is -20.18 kJ mol<sup>-1</sup>. The heat of hydration of CuSO<sub>4</sub>(s), ie, ΔH for the reaction

$$CuSO_{\alpha}(s) + SH_{2}O(t) - \rightarrow CuSO_{4} - SH_{2}O(s)$$
is

- (a)  $-111.39 \text{ kJ mol}^{-1}$  (b)  $-71.03 \text{ kJ mol}^{-1}$
- (c)  $-105.02 \text{ kJ mol}^{-1}$  (d)  $-75.05 \text{ kJ mol}^{-1}$
- 50. The gas liberated on treating a mixture of two salts with dil H<sub>2</sub>SO<sub>4</sub> turns lime water milky and turbidity disappears with the passage of excess of gas. The aqueous solution of mixture gives white crystalline ppt with NaCl solution. The filtrate gives a black precipitate, when H<sub>2</sub>S is passed into it. The aqueous solution of mixture on heating gives reddish brown gas and when treated with ammonium hydroxide and excess of disodium hydrogen phosphate gives a white crystalline precipitate. The mixture contains
  - (a)  $CO_3^{2+}$ ,  $Pb^{2+}$ ,  $NO_3^{2+}$ ,  $Mg^{2+}$
  - (b)  $CO_3^{2+}$ ,  $Pb^{3+}$ ,  $NO_4^{2+}$ ,  $Ca^{2+}$
  - (e)  $CO_3^{2+}$ ,  $Pb^{2+}$ , CP,  $Mz^{2+}$
  - (d)  $CO_2^{2+}$ ,  $Pb^{2+}$ ,  $CI_1$ ,  $Ca^{3+}$

## ANSWERS

		5 ( )	# CsN	5. (b)	<b>6.</b> (e)	<b>7.</b> (d)	<b>8.</b> (b)	9. (c)	<b>10.</b> (c)
<b>1.</b> (a)	<b>2</b> . (b)	3. (c)	<b>4.</b> (a)	2. (2)	• •		16 (1)	<b>19.</b> (b)	<b>20.</b> (d)
49 744	<b>12</b> , (c)	<b>13</b> . (d)	14. (ნ)	15. (b)	<b>16.</b> (c)	<b>17.</b> (b)	18. (b)	III. (D)	
<b>11</b> . (b)	TE (A)			515 (b)	<b>26.</b> (b)	<b>27.</b> (d)	28. (a)	29. (ն)	<b>30.</b> (b)
. <b>21</b> , (u)	<b>22.</b> (b)	23. (a)	<b>24.</b> (a)	<b>25.</b> (b)	20. (0)			AB 2-3	<b>40</b> . (a)
	44 /41	<b>33.</b> (b)	<b>34</b> , (c)	<b>35</b> . (a)	<b>36</b> . (c)	37. (b)	<b>38.</b> (d)	<b>39</b> . (c)	40. (2)
<b>31.</b> (0)	<b>32.</b> (5)	Jun (D)		• •	40. 7-3	<b>47.</b> (5)	48. (c)	49. (=)	<b>50.</b> (a)
<b>41</b> . (d)	<b>42.</b> (c)	43. (ზ)	<b>44.</b> (a)	<b>45.</b> (a)	<b>46</b> . (a)	42.13	777 (0)	(-)	•

## HINTS & SOLUTIONS

 Phenolphthalcin is colourless in acid solution (benzenoid form) and pink in alkali (basic) solution (quinomoid form).

colouriess (acid medium) benzeroùl form red (alkaline medium) quinenoid form

The blue precipitate of Fe<sup>2+</sup> ions with potassium ferricyonide is due to the formation of Tumbull's n on blue K Fc[Fe(CN)<sub>6</sub>]

3. As in cases (1), (2) and (4), the acids and bases taken are strong and the anthalpy of neutralisation of all strong acids with strong

- reaction. So, it is added to the reaction mixture during the preparation of acetanilide from aniline.
- 7. All the given metals form white ppt with NH<sub>2</sub>OH but only ppt of Zn is soluble in excess of NH<sub>2</sub>OH and on passing H<sub>2</sub>S it gives white ppt of ZnS, so the metal is Zn and reactions takes place as follows

To Hows
$$Zn^{2+} + 2NH_4OH \longrightarrow Zn(OH)_2 + 2NH_4^{\dagger}$$

$$Zn(OH)_2 + 2NH_4OH \longrightarrow (NH_4)_2ZnO_2 + 2H_2O$$

$$Solvible$$

$$(NH_4)_2ZnO_2 + H_2S \longrightarrow ZnS \downarrow + 2NH_4OH$$
white ppt

8. In the titration of oxalic acid vs KMnO<sub>4</sub>, KMnO<sub>4</sub> acts as a oxidant as well as a self indicator.  $2\text{KMnO}_4 + 3\text{H}_2\text{SO}_4 \longrightarrow \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 \\ + 3\text{H}_2\text{O} + 5[\text{O}] \\ 5 \mid + 5[\text{O}] \longrightarrow 5\text{H}_2\text{O} + 10\text{CO}_2 \uparrow$ 

- acetanilide which is then nitrated to give p-nitro acetanilide as a major product.

  O. Formulator mass of
- 10. Equivalent mass of moist mass molar mass

- 11. During the formation of egg albumin sol, hot water is not used because in hot water precipitation of egg albumin takes place whereas in cold water formation of precipitate does not ассце,
- Formaldehyde cannor produce iodoform, as only those compound which contains either  $CH_3$ —CH— group or  $CH_3$ —CH— group on

reaction with todine and sodium hydroxide (alkah) yield iodoform.

13. In Lassaigne's test, the organic compound is fused with a piece of Na metal to convert covalent compounds into a mixture of ionic compounds such as

$$Na + C + N \longrightarrow NaCN 
2Na + S \longrightarrow Na_2S 
Na - Y \longrightarrow Na_2S$$

 $Na = X \longrightarrow NaX$ (where, X = Cl, Br, I)

14. In the given experiment following reaction occurs  $H_2O_2 + 2\Gamma + 2H^{+} \longrightarrow 2H_2O + I_2$ 

todine liberated in this reaction reacts with sodium thiosulphate solution and is reduced to iodide ions.

$$I_2 + 2S_2O_3^2 \xrightarrow{\text{Fast}} S_4O_6^2 \xrightarrow{\text{Past}} 2T$$
.

15. NaCl is a salt of strong acid and strong base, hence on dissolution will give neutral solution. As white ppt is obtained by the addition of AgNO $_3$  to the solution of Na salt, it can be of AgCl. Futher AgCl is also insoluble in HNO. Hence, the anion is Cl<sup>+</sup>.

16. The reagent 2 4 dinitro phenyl hydrazine is used for the detection of carbonyl group, ie, aldehyde and ketone groups. With carbonyl group, this reagent gives red or yellow ppt due to the formation of respective hydrazones.

$$R$$
  $C = D + H_2 N \cdot NH - NC_2 NC_2$   $NC_2$ 

2, 4-dinitro phenyl hydrazine

$$R'$$
 C-N·NH-NO<sub>2</sub> -NO<sub>2</sub>  $\downarrow$  + H<sub>2</sub>O

Yellow or arrage red

- 17. Heat of neutralisation of strong acid and strong base is always 13.7 kcal.
- 18. The ionic equation for exidation of Mohr's salt is  $Fe^{21} \longrightarrow Fe^{3-} + e^{-}$

Now, Eq. of Mohr's salt =  $\frac{392}{1}$  = 392

Strength  $\simeq$  Normality  $\times$  Eq. mass  $=\frac{1}{20}\times392=19.6\,\mathrm{g/L}$ 

Thus, for preparing 250 mL of N/20 Mohr's salt solution, Molir's sait needed

$$=\frac{19.6}{1000}\times250=4.9\,\mathrm{g}$$

19. Let the amount of the  $\hat{\mathbf{K}}_{p}$ Cr<sub>2</sub>O<sub>7</sub> in the mixture be  $\mathbf{x}$ 

then, amount of KMnO $_4$  will be (0.5 - x)  ${
m g}$  $\left(\frac{x}{49} + \frac{0.5 - x}{31.6}\right) - \frac{100 \times 0.15}{1000}$ 

where, 49 is Eq. wt. of  $K_2Cr_2O_7$  and 31.6 is Eq. wi. of KMnO 4.

On solving, we get x = 0.0732 gpercentage of  $K_2 G_2 O_7 = \frac{0.0732 \times 100}{0.5} = 14.64\%$ 

- 20. The Solution of compound 'A' gives turbidity with tap water (which contains Cillions) and the turbidity (due to formation of AgCl) is insoluble in nitric acid but soluble in NH4OH. Hence, the compound 'A' is AgNO 3. The reactions are as
  - (1)  $2AgNO_3 \xrightarrow{A} 2Ag + 2NO_2 \uparrow + O_2 \uparrow$ (2)  $AgNO_3 + Cl^{\dagger}_{(from tap water)} = \rightarrow$   $AgCl = NO_3$   $AgCl = NO_3$

- (3)  $AgCl + 2NH_4OH \longrightarrow \{Ag(NH_3)_2\}Cl + 2H_2O$
- 21. The compound which contains —CHO group, gives positive Tollon's test and the compound with CH<sub>3</sub>—C— group gives positive iodoform test.

Thus, the structure of the compound should be

**22.** KI + AgNO<sub>3</sub> (slight excess)  $\longrightarrow$  AgI + KNO<sub>3</sub>  $AgNO_3 \longrightarrow Ag^+ + NO_3^ AgI(i) + Ag^+ \longrightarrow [AgI]Ag^-$ silver include sol

23. During the test for halogens, sodium extract is heated with conc. HNO<sub>3</sub> to decompose sodium cyanide or sodium sulphide present in it, so that they do not interfere in the test for halogens.

$$NaCN + HNO_3 \longrightarrow NaNO_3 + HCN$$
  
 $Na_2S + ZHNO_3 \longrightarrow ZNaNO_3 + H_2S$ 

24. 126 g of oxalic acid = 1 mol of  $H_2C_2O_4$  $2M_1O_4^2 + 5C_2O_4^{2-} + 16H^+ \longrightarrow 2M_1^{2+}$ 

$$+1000_2 + 8H_2O$$

1 male of exalate ion require = 2/5 male KMnO<sub>4</sub>  $Cr_2O_7^{2-} + 3C_2O_4^{2-} + 14H^+ \longrightarrow 2Cr^{3+}$ 

$$+6CO_2 + 7H_2O$$

1 mole of oxalate ion require = 1/3 mole

$$K_2C_2O_7$$

25. Ba<sup>2+</sup> ion imparts green colour to the flame and Cl<sup>-</sup> ion forms chromyl chloride (which is orange red in colour) when treated with K<sub>2</sub>Cl<sub>2</sub>O<sub>7</sub> and cone H<sub>2</sub>SO<sub>4</sub>. Thus, the reagent is

26. Nickel salt reacts with dimethyl glyoxime in presence of NH<sub>4</sub>OH to give scarlet red ppt of nickel dimethyl glyoxime.

$$CH_4$$
— $C$ = $NOH$   
2 +  $NiCl_2$  +  $2NH_4OH$   $\longrightarrow$ 

$$\begin{array}{c|c}
CH_{2} - C = N & O \\
\downarrow & \uparrow & \uparrow \\
CH_{3} - C = N & N = C - CH_{3} \\
\downarrow & \downarrow & \downarrow \\
O & OH
\end{array}$$

Nickel dimethylglyoxime (scartet red ppt)

$$+2NH_4Cl + 2H_2O$$

27. Due to all of the given reasons ferrous sulphate

29. The compound liberates CO<sub>2</sub> with NaHCO<sub>3</sub>, so It contains —COOH group and it also give colour with neutral FeCl<sub>3</sub> solution, so it also contains a —OH group directly attached to the benzene ring (ie, phenol). Hence, the structure of the compound is

30. A positive Bellstein's test for halogens does not always indicate the presence of halogen since some halogen free compounds viz urea, thiourea, amides etc also responds this test. The reason being the fact that these halogen free compounds form cuprous cyanide which is volatile and decomposes to copper which burns with green flame.

Aniline yellow (p-amino azobenzene or 4 phenyi azoaniline)

It is a carcinogenic compound and a basic dye.

- 32. The main purpose of using leveling bulb is to assure that pressure within the reaction vessel is same as that in the room.
- Acidic solution favours dichromate, whereas alkaline solution favours chromate.
- 34. On treatment with dilute HCl, Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>SO<sub>3</sub> produce CO<sub>2</sub> and SO<sub>2</sub> respectively. SO<sub>2</sub> turns an acidified K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution green whereas CO<sub>2</sub> does not change the colour of the solution.

$$\begin{array}{lll} \text{Na}_2\text{CO}_3 + 2\text{HCl} & \longrightarrow & 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2\text{ }\hat{\text{1}} \\ \text{Na}_2\text{SO}_3 + 2\text{HCl} & \longrightarrow & 2\text{NaCl} + \text{H}_2\text{O} + \text{SO}_2\text{ }^{\uparrow} \\ \text{K}_2\text{Cr}_2\text{O}_7 & + \text{H}_2\text{SO}_4 + 3\text{SO}_2 & \longrightarrow & \text{K}_2\text{SO}_4 \\ \text{orange yellow} & + \text{Gr}_2(\text{SO}_4)_3 + \text{H}_2\text{O} \\ & & \text{green} \end{array}$$

- Lyophilic sols are prepared by merely dissolving substances like gelatin, starch in water while lyophobic sols are prepared by special methods such as peptisation, oxidation, reduction etc.
- 38. Ferrous ammonium sulphate (Mohr's salt) is prepared by dissolving ferrous sulphate in dil  $H_2SO_4$  and then by adding ammonium sulphate.

$$(NH_4)_2SO_4 + FeSO_4 + 6H_2O \xrightarrow{Dii. U_2SO_4}$$

FeSO  $_4$ · (NH $_4$ ) $_2$ SO  $_4$ · 6H $_2$ O Ferrous armmonium sulphate (Mohr's salt)

39. (i) The mixture on beating with NaOH liberate a gas which gives red ppt with alkaline K<sub>2</sub>HgI<sub>4</sub>, so the gas is NH<sub>3</sub> and mixture contains NH<sub>4</sub> ion.

(ii) The aqueous solution of mixture gives white ppr with  $BaCl_2$ , so it contains  $SO_4^2$  ions.

$$SO_4^{2-} + BaCl_2 \longrightarrow BaSO_4 + 2Cl^{-}$$

(jii) Mixture on heating with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and conc. H<sub>2</sub>SO<sub>4</sub> gives red varours (CrO<sub>2</sub>Cl<sub>2</sub>), so contains Cl<sup>+</sup> ions.

(iv) Aqueous solution of mixture gives blue colour with K<sub>3</sub>[Fe(CN)<sub>6</sub>], so mixture contains Fe<sup>2+</sup>

ions. 
$$3 {
m Fe}^{2+} + 2 {
m K}_3 {
m [Fe(CN)}_6] \longrightarrow {
m Fe}_8 {
m [Fe(CN)}_6]_7 + 6 {
m K}^6$$
 Blue

 Alcohols gives red colour with ceric ammonium nitrate so, the compound is alcohol

$$2R \longrightarrow OH + (NH_4)_2Ce(NO_3)_5 \longrightarrow Ceric ammonium nitrate  $Ce(NO_3)_4(ROH)_2 + 2NH_4NO_3$  Red colour$$

- The rate of the dissociation of hydrogen peroxide with U ion increases by increasing the conc of U ion and also in presence of UV light.
- 49 The enthalox change when one mole of a solute is

dilution, is called enthalpy of solution and further addition of solvent does not affect it.

43. 
$$2KI + CI_2 \longrightarrow 2KCI + I_2$$
  
 $I_2 + CCI_4 \longrightarrow \text{violet colour}$ 

Note: The excess of  $Gl_2$  should be avoided otherwise the layer may become colouriess due to conversion of  $I_2$  to  $HiO_3$ 

$$l_2 + 5Cl_2 + 6H_2O \longrightarrow 2HIO_3 + 10HCL$$

44. Normality equation

 $N_1V_3$  .  $N_2V_4$  dill exalte acid setterion . KMnO $_4$  solution

$$N_1 \times 25 = \frac{N}{10} \times 30$$

250 ml. of dil oxalic acid =  $\frac{N}{10} \times \frac{30}{25} \times 250$ 

$$= 300 \text{ mL}$$
 of  $\frac{\text{N}}{10} \text{ KMnO}$ .

= 30 mL of N KMnO $_4$  solution = 30 mL of N-ovalic acid

Thus, the amount of acid used with pyrolusite = 50 - 30 = 20 mL of N-oxalic acid

$$MnO_2 + H_2SO_4 \longrightarrow MnSO_2 + H_2O \div [O]$$

COOH  

$$\begin{array}{c} 27 \\ -200 \\ -200 \end{array}$$
  $\begin{array}{c} 260_{2}^{2} + 3H_{2}O \\ -25 \end{array}$ 

63 g oxalic acid = 
$$\frac{87}{2}$$
 g MnO<sub>2</sub> = 8g O<sub>2</sub>

So, the amount of exalte acid used =  $20 \times \frac{63}{1000}$ 

= 1.26 g 
$$\odot$$
 63 g oxalic acid =  $\frac{87}{2}$  g MnO  $_2$ 

$$\therefore$$
 1.26 g oxalic acid =  $\frac{287 \times 1.26}{2 \times 63}$  = 0.87 g

: The amount of MnO  $_2$  in 1.5 g pyrolusite = 0.87 g

. Amount of MnO  $_2$  in 100 g pyrolusite  $= \frac{0.87}{1.5} \times 100 = 58\%$ 

- **45.** Because it is necessary to record data and swirl the flask (at constant rate) simultaneously.
- 46. In III group precipitation, NTLCl is added before adding NH4OII to decrease the cond of OHT, as NH4Cl and NH4OH dissociates as follows

$$NH_2CI \rightleftharpoons NH_1^1 + CI$$

$$NH_4OH \implies NH_4^+ - OH^-$$
Common ion

Due to presence of common ion, cone of OHT decreases and for III group precipitation lesser cone of OHT is required.

 The enthalpy of ionisation of weak acid is given by

$$\Delta H_{\rm ion~[HA]} = \Delta H_{\rm N~(weak~and/succes have)}$$

 $\sim \Delta H_{
m N}$  (311000) acid/smars base).

$$= -56.1 - (-57.3) = 1.2 \text{ kJ mol}^{-1}$$

 $\Delta H_{\text{(ionisation)}} = 1.5 \text{ kJ mol}^{-1}$ 

Hence, percentage ionisation in 1M solution  $\pm \frac{(3.5-1.2)}{1.5} \times 100 = 20$ 

**48.** During acetylation of aniline, the excess of acetic anhalyride and prolonged heating results in the formation of diacetyl derivative.

$$C_cH_s$$
— $NH_2 + (CH_sCO)_2O \longrightarrow C_cH_s$ — $NH$ — $COCH_3$   
Anlline

`+ сн<sub>з</sub>соои

$$C_6H_5NH$$
— $COCH_3+(CH_2CO)_3O$  Prolonged beating

 $C_0H_0NH(COCH_3)_2 + CH_3COOH$ Diapetyl derivative

- 49. (i)  $CuSO_{\Delta}(s) + (mq) \longrightarrow CuSO_{+} \cdot 5H_{2}O(mq);$   $\Delta H_{1} = -91.21 \text{ kJ mol}^{-1}$ 
  - (ii)  $CuSO_4 5H_2O(s) + (aq) \rightarrow CuSO_4 5H_2O(aq);$  $\Delta H_2 = -20.18 \text{ kJ mol}^{-1}$

Eq. (i) involves two steps :

(iii) 
$$GuSO_4(s) + 5H_2O \longrightarrow GuSO_4 \cdot 5H_2O(s);$$
  
 $\Delta H_3 =$ 

(iv)  $CuSO_4 \cdot 5H_2O(s) + (aq) \rightarrow CuSO_4 \cdot 5H_2O(aq)$ ;

 $\lambda H_2 = -20.18 \, \mathrm{kJ/mol}$ 

We find (iii) 
$$+$$
 (iv)  $=$  (i)  

$$- [xH_2O + (aq) \longrightarrow (aq)]$$

$$\Delta H_3 + \Delta H_2 = \Delta H_1$$

thus, for

CuSO<sub>4</sub>(s) + 5H<sub>2</sub>O 
$$\longrightarrow$$
 CuSO<sub>4</sub> · 5H<sub>2</sub>O(s)  
 $\Delta H_3$  -  $\Delta H_1$  -  $\Delta H_2$   
- 91,21 - (-20.18)  
= -71.03 kJ mol <sup>1</sup>

50. (i) The mixture with dil H<sub>2</sub>SO<sub>4</sub> liberated a gas which turns lime water milky and turbidity disappears with the passage of excess of gas, so the gas is CO<sub>2</sub> and maxture contains CO<sub>3</sub><sup>2</sup> ion.

$$\begin{array}{ccc} \text{CO}_2 \div \text{Ca}(\text{OH})_2 & \longrightarrow & \text{CaCO}_3 + \text{H}_2\text{O} \\ \text{Gas} & & \text{Whice} \\ \text{CaCO}_3 + \text{H}_2\text{O} + \frac{\text{CO}_2}{\text{Excess}} & \longrightarrow & \text{Ca}(\text{HCO}_3)_2 \\ & & \text{Soluble} \end{array}$$

(ii) The aqueous solution of mixture gives white ppt with NaCl solution and filtrate of this with H<sub>2</sub>S gives black ppt thus, mixture contains Pb<sup>2+</sup> ion.

$$\begin{array}{ccc} {\rm Pb(NO_3)_2 + 2NaC}; & \longrightarrow & {\rm PbCl_2 + 2NaNO_3} \\ & {\rm PbCl_2 + H_2S} & - & > & {\rm PbS + 2HCI} \end{array}$$

(iii) The aqueous solution of mixture on heating liberates reddish brown gas, so it should have NO<sub>3</sub> ion.

$$Pb(NO_3)_2 \longrightarrow PbO + \frac{2NO_2}{Red brown gas} + \frac{1}{2}O_2$$

(iv) The aqueous solution with NH<sub>4</sub>OH and excess of Na<sub>2</sub>HPO<sub>2</sub> gives a white crystalline opt, so mixture should have Mg<sup>2+</sup> tons.

$$\begin{array}{c} {\rm MgCO_3 - Na_2HPO_4 + NH_4OH} \longrightarrow \\ {\rm Mg(NH_4)PO_4} \downarrow + {\rm Na_2CO_2} + {\rm H_2O} \\ {\rm White} \end{array}$$