

# **EXPERIMENT 1**

## **DETERMINATION OF SOLUBILITY CLASS**

Each functional group has a particular set of chemical properties that it to be identified.

Some of these properties can be demonstrated by observing solubility behavior, while others can be in chemical reactions that are accompanied by color changes, precipitation formation, or other visible affects.

According to the presence of their functional group, organic compounds can be classified in different families and class.

The types of compounds soluble in a particular solvent are summarized on the following pages:

1. Water Soluble Compounds
- 2 . 5% Sodium Hydroxide Soluble Compounds
- 3 5% Sodium Bicarbonate Soluble Compounds
4. 5% Hydrochloric Acid Soluble Compounds
5. 96% Sulfuric Acid Soluble Compounds
6. Solubility of Amphoteric Compounds

Much information can be obtained about an unknown substance by performing simple solubility and chemical tests.

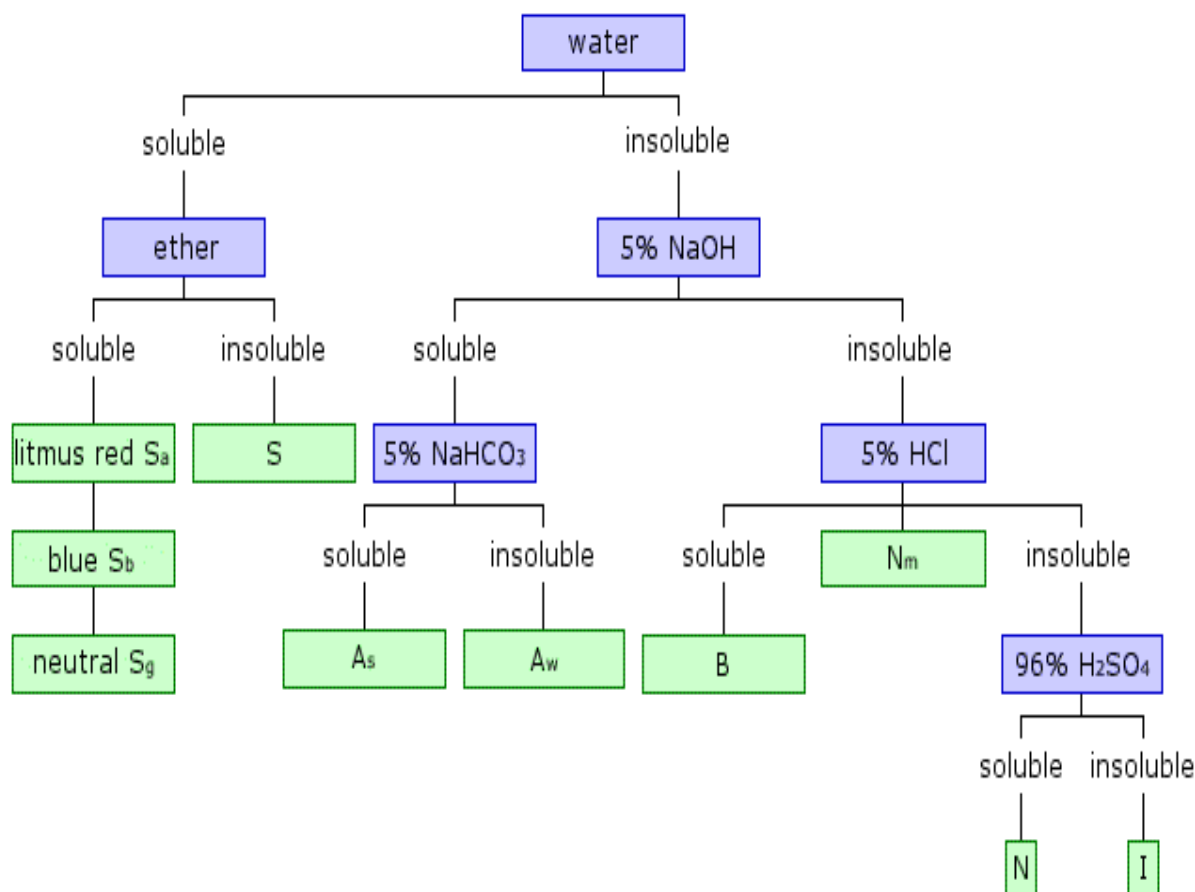
Determining the solubility behavior of an unknown in water, 5% sodium hydroxide solution, 5% sodium bicarbonate solution, 5% hydrochloric acid solution, and cold concentrated sulfuric acid will yield three kinds of information.

**First, the presence of a functional group is often indicated.** For instance, because hydrocarbons are insoluble in water, the mere fact that an unknown is partially soluble in water indicates that a polar functional group is present.

**Second, solubility in certain solvents often leads to more specific information about the functional group present.** For example, an unknown may be insoluble in water, but soluble in 5% sodium hydroxide solution. In this case, the solubility of a water insoluble unknown in basic solution is a strong indication that an acidic functional group (like a carboxylic acid) is present.

**Third, information about molecular size and shape can often be obtained.** For example, in homologous series of compounds, the members with fewer than five carbon atoms are water soluble, whereas the higher homologs are not. Additionally, in homologous series of compounds, chain branching and positioning of functional groups toward the center of the molecule increases water solubility.

## Flow Chart for Classes of Organic Compounds



### Class

### Functional Group Possibilities

S<sub>a</sub> monofunctional carboxylic acids (≤5C), arylsulfonic acids

S<sub>b</sub> monofunctional amines (≤6C)

S <sub>g</sub>	monofunctional alcohols, aldehydes, ketones, esters, nitriles, and amides (all ≤5C)
S	salts of organic acids, amine hydrochlorides, amino acids, polyfunctional compounds with hydrophilic functional groups
A <sub>s</sub>	strong organic acids: carboxylic acids (>6C), phenols with electron-withdrawing groups in the ortho and/or para position(s), β-diketones
A <sub>w</sub>	weak organic acids: phenols, enols, oximes, imides, sulfonamides, thiophenols (all >5C), β-diketones, nitro compounds with α-hydrogens
B	aliphatic amines (≥8C), anilines (only one phenyl group attached to N), some ethers
N <sub>m</sub>	miscellaneous neutral compounds containing N or S (>5C)
N	alcohols, aldehydes, ketones, monofunctional esters (>5C but <9C), ethers, epoxides, alkenes, alkynes, some aromatic compounds (with activating groups)
I	saturated hydrocarbons, haloalkanes, aryl halides, other deactivated aromatic compounds, diaryl ethers

## Procedure:

1. **Water Solubility.** Place 0.05 mL or 25 mg of compound in a small test tube, and add 0.75 mL of water in small portions. Shake test tube vigorously after the addition of each portion of solvent. If water soluble, go on to step 2; otherwise proceed to step 3.
2. **Ether Solubility.** Place 0.05 mL or 25 mg of compound in a small test tube, and add 0.75 mL of diethyl ether in small portions. Shake test tube vigorously after the addition of each portion of solvent. If the compound is both water and ether soluble, the acid-base properties of the compound should be determined with litmus.
  - litmus turns red - water soluble acidic compound (class S<sub>a</sub>)
  - litmus turns blue - water soluble basic compound (class S<sub>b</sub>)
  - litmus neutral - water soluble general compound (class S<sub>g</sub>)

If the compound is not ether soluble it is a salt, amino acid, or contains many hydrophilic functionalities (class S)

3. **5% NaOH Solubility.** Place 0.05 mL or 25 mg of compound in a small test tube, and add 0.75 mL of NaOH solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If NaOH soluble, go on to step 4; otherwise proceed to step 5.
  
4. **5% NaHCO<sub>3</sub> Solubility.** Place 0.05 mL or 25 mg of compound in a small test tube, and add 0.75 mL of NaHCO<sub>3</sub> solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If NaHCO<sub>3</sub> soluble, then it is a strong organic acid (class A<sub>s</sub>). If not NaHCO<sub>3</sub> soluble, then it is a weak organic acid (class A<sub>w</sub>).
  
5. **5% HCl Solubility.** Place 0.05 mL or 25 mg of compound in a small test tube, and add 0.75 mL of HCl solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If HCl soluble, then it is an organic base (class B). If not HCl soluble and (from elemental analysis) is found to contain nitrogen or sulfur, then it is a miscellaneous neutral compound (class N<sub>m</sub>). If not HCl soluble, then go on to step 6.
  
6. **96% H<sub>2</sub>SO<sub>4</sub> Solubility.** Place 0.6 mL of H<sub>2</sub>SO<sub>4</sub> in a small test tube, and add 0.05 mL or 25 mg of compound. Shake test tube vigorously. If H<sub>2</sub>SO<sub>4</sub> soluble, then it is a neutral compound (class N). If not H<sub>2</sub>SO<sub>4</sub> soluble, then it is an inert compound (class I).



## EXPERIMENT 2

### IDENTIFICATION OF ALCOHOLS

Alcohols are organic compounds containing an -OH functional group bonded to a carbon atom. There are three classes (types) of alcohols: primary, secondary, and tertiary as shown below.

**Primary alcohol:** RCH<sub>2</sub>OH the 'R', an alkyl group, and the -OH are attached to a primary carbon atom, a carbon bonded to one other carbon atom (highlighted).

**Secondary alcohol:** R<sub>2</sub>CHOH, the 2 'R' alkyl groups, and the -OH are attached to a secondary carbon atom, a carbon attached to two other carbon atoms from the 2 'R' (CH<sub>3</sub>-),

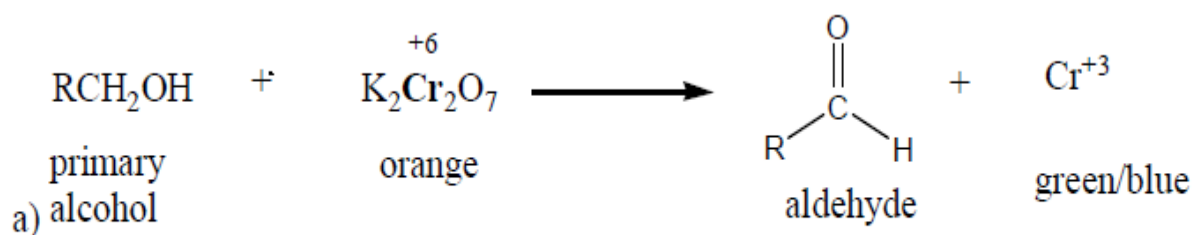
**Tertiary alcohol:** R<sub>3</sub>COH the 3 'R' alkyl groups, and the -OH are attached to a tertiary carbon atom (highlighted), a carbon attached to three other carbon atoms from the 3 'R' (CH<sub>3</sub>-) The International Union of Pure and Applied Chemistry (IUPAC) method of nomenclature for alcohols use the ending ol. Change the 'e' ending of an alkane to *ol* (i.e methane to methanol ethane to ethanol).

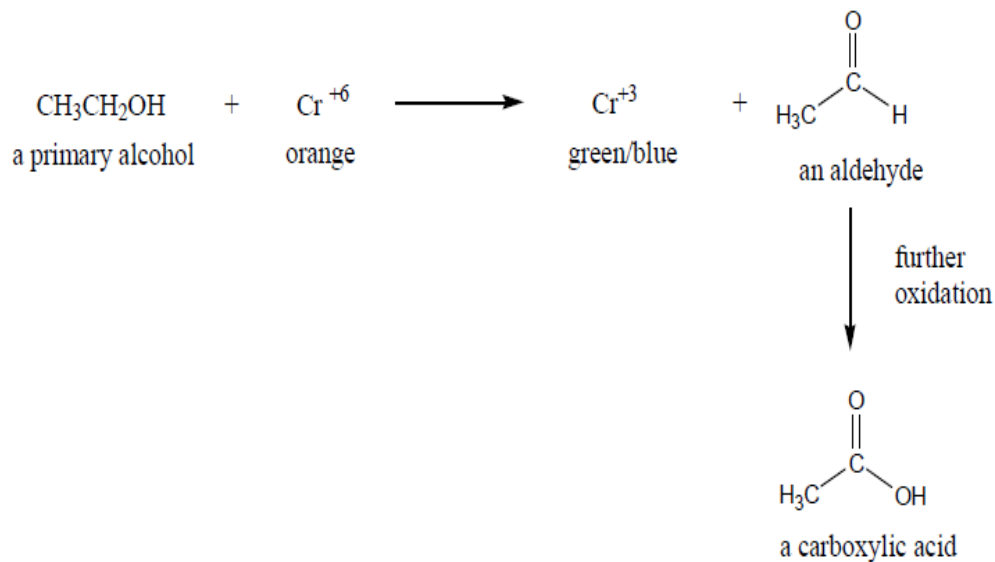
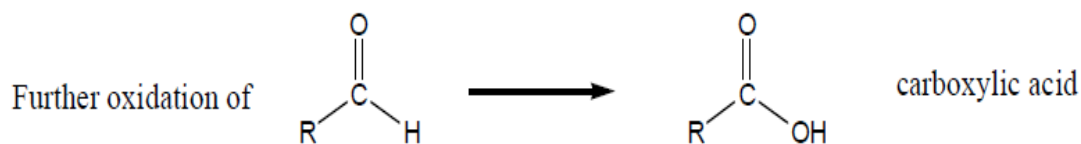
**R = represents any alkyl group**

### Oxidation

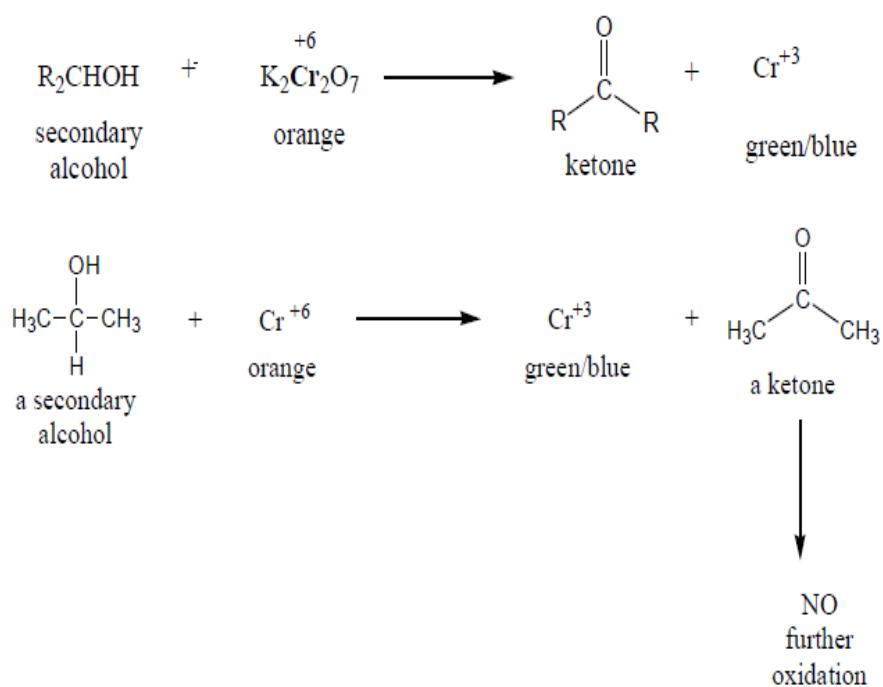
Oxidation of alcohols by strong oxidants such as K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in H<sub>2</sub>SO<sub>4</sub> is possible, but differs depending on the degree of alcohol.

Examples a, b, and c below show how a primary, secondary, and tertiary alcohol respectively respond to treatment of oxidants. If a reaction has occurred using K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in H<sub>2</sub>SO<sub>4</sub>, there is a color change from orange to green.

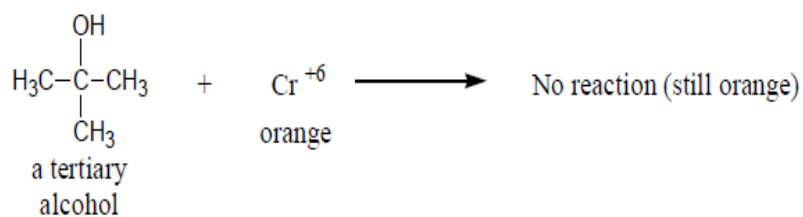




b)



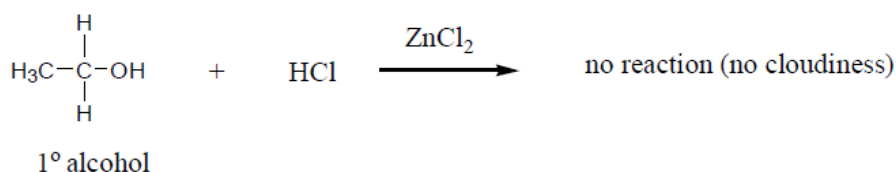
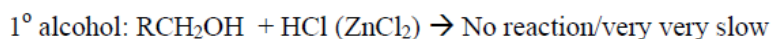
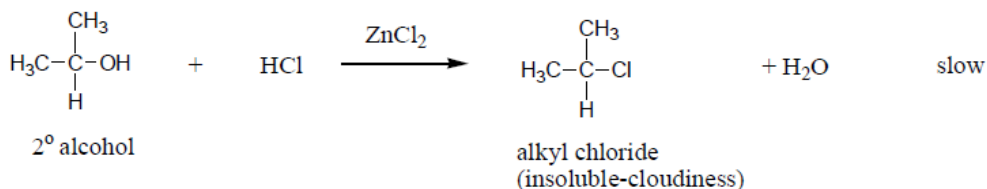
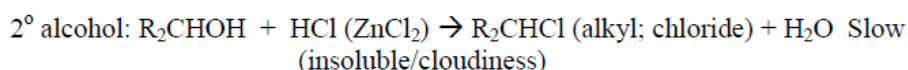
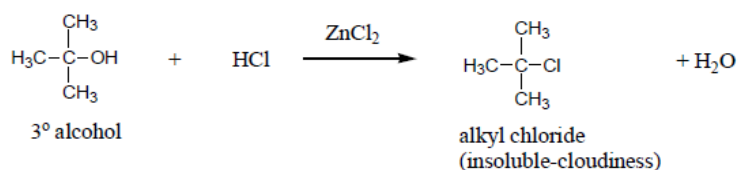
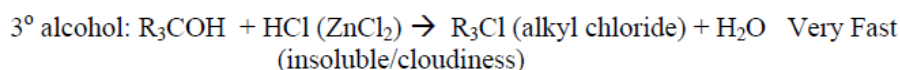
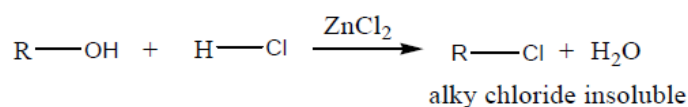
c) Tertiary alcohol:  $\text{R}_3\text{COH} + \text{K}_2\text{Cr}_2\text{O}_7 \rightarrow \text{NO OXIDATION}$



## Lucas Test:

This test is for low molecular weight alcohols and it distinguishes the rates of reaction of alcohols with the Lucas reagent (HCl and ZnCl<sub>2</sub>). Positive indicator of the reaction is the formation of a water insoluble alkyl chloride as cloudiness or a precipitate. The formation of an alkyl chloride with tertiary alcohol is very rapid, followed by the secondary alcohol that may take from 5 to 20 minutes to form visible cloudiness. Primary alcohols do not react with Lucas reagent or it may show very little result in a very long time.

The chemical reaction involves replacing the –OH group of the alcohol with a chloride ion from hydrochloric acid (HCl), forming an alkyl chloride, as shown in the following equation.



## **PROCEDURE**

### **Part I – Reaction with sodium**

Under the hood place a small piece of sodium metal in to a 50 mL beaker of half filled with **water**. Observe the reaction and after completion of the reaction, add 1 or 2 drops of phenolphthalein indicator to the solution in the beaker. Write a balance equation for the reaction of sodium and water. Repeat the reaction of 2.0 mL alcohol with small piece of sodium metal in a large dry test tube. Note the reactivity of sodium metal with alcohol in compare (less or more reactive) to water. Test the resulting solution with phenolphthalein. Write the balance equations for reactions of sodium with **ethanol** and **1-butanol, phenol, and unknown**.

### **Part II – Solubility in water**

Place 2.0 mL of water in a small test tube, dropwise add **ethanol** and shake the mixture. Count the number of drops until the alcohol is no longer soluble in water, but do not use more than 10 drops. Repeat the same procedure using **2-propanol, 1-butanol, cyclohexanol, phenol, and unknown**. Record your results as very soluble (for 6-10 drops), soluble (for 2-5 drops), and insoluble (1 drop).

### **Part III. Lucas Test**

In a small test tube place 2.0 mL Lucas reagent, and add 4-5 drops of alcohol, shake the mixture well and observe the time required for the mixture to become cloudy or to form two different phases (layers). Try this test with **1-butanol, 2-butanol, 2-methyl-2-propanol (t-butyl alcohol), phenol, and unknown**. Based on your results of the Lucas test, determine the type of your unknown alcohols (1o, 2o, or 3o)

### **Part IV. Jones Reagent (Chromic acid test)**

In a small test tube, place about 1.0 mL acetone, 1.0 mL alcohol, and 2 –3 drops of the Jones reagent. Observe the color change, clear, orange (formation of Cr +6 as CrO<sub>3</sub>), or blue-green (formation of Cr 3+ ). Try this test with **1-butanol, 2-butanol, 2-methyl-2-propanol, phenol, and unknown**. Based on your results of the Chromic acid test determine the type of your unknown alcohol (1o, 2o, or 3o).



### **Part V. Esterification**

In a small test tube, mix ethanol (4-5 drops) and acetic acid (4-5 drops). Add one drop of concentrated sulfuric acid, warm the mixture in a hot water bath for about 5 minutes and then add 2.0 mL of cold water. Note the odor and write a complete equation for the reaction of ethanol and acetic acid.

In a small test tube, mix 1-butanol (4-5 drops) and acetic anhydride (4-5 drops). Stir well, and warm the solution in hot water bath. Add 2.0 mL of cold water and make mixture slightly basic by adding 4-5 drops dilute NaOH. Note the odor and write a complete equation for the reaction of 1-butanol and acetic anhydride.



## EXPERIMENT 3

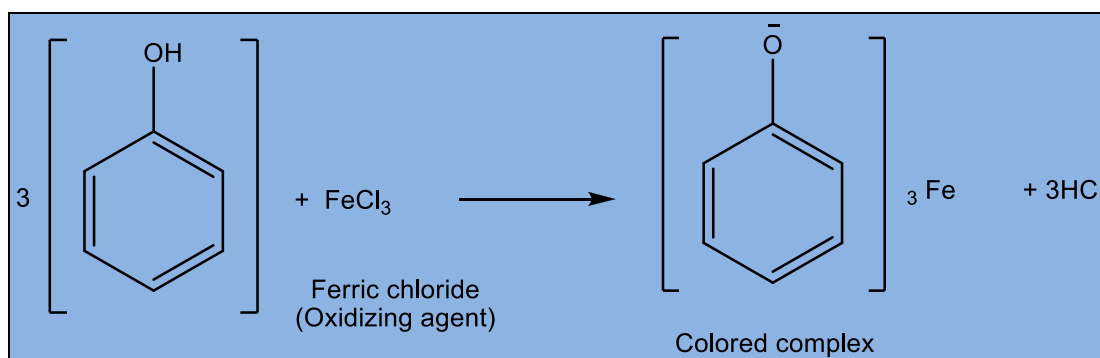
### IDENTIFICATION OF PHENOLS

Phenols are organic compounds with a hydroxyl group attached directly to an aromatic ring. They have the general formula Ar-OH. Examples of them include phenol, hydroquinone, resorcinol, *o*-cresol, *m*-cresol, *p*-cresol,  $\beta$ -naphthol, and catechol.

#### Chemical Reactions:

##### 1. Ferric chloride test.

Phenols react with ferric chloride to give colored compounds due to the presence of [-C=C-OH] (enol) group. Indeed this reaction is considered as a test for any compound with enol group.

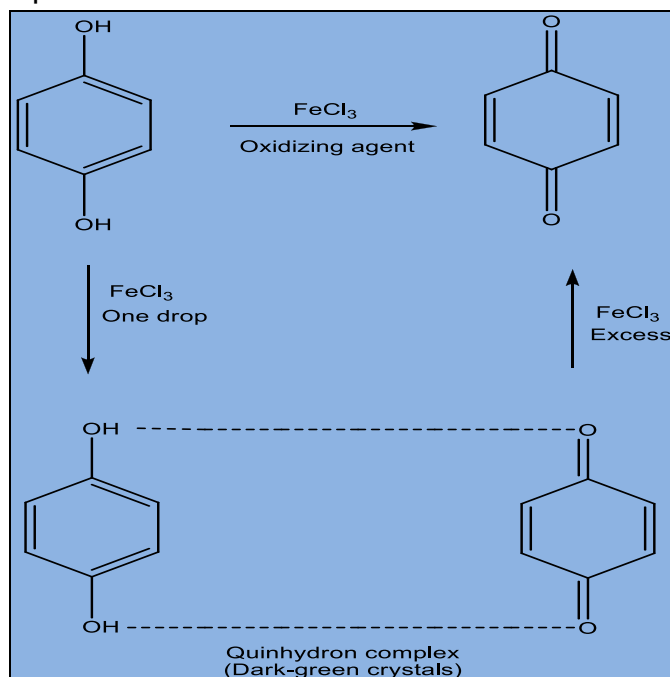


#### Procedure:

To a very dilute aqueous solution of phenol or to a few crystals of the solid phenol (0.1 gm) dissolved in water add 1 drop of ferric chloride solution and observe the resulting color:

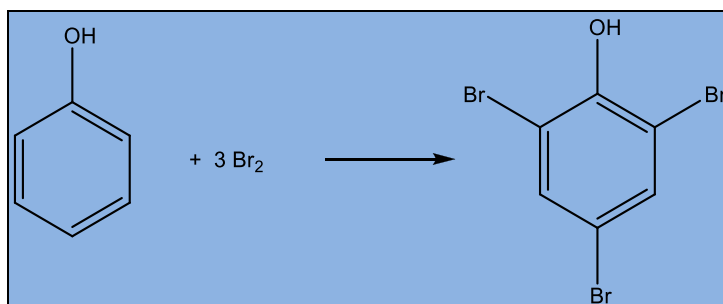
Phenols	Colors
phenol, <i>m</i> -cresol, resorcinol	violet or blue
<i>o</i> - and <i>p</i> -cresol	greenish blue
Hydroquinone	deep green
$\beta$ -naphthol	no special color

In the reaction of hydroquinone with ferric chloride as crystals may separate, and on further addition of ferric chloride solution a yellow solution of *p*-benzoquinone is produced:



## 2. Bromine water test.

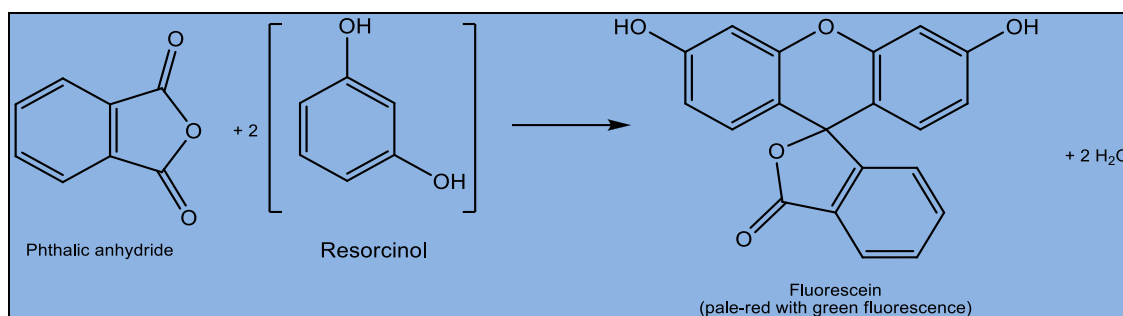
Phenols are generally highly reactive towards electrophilic reagents and are readily brominated by bromine water. e.g.



Dissolve or suspend about 0.05 g of the compound in 2 mL of dilute hydrochloric acid and add bromine water drop wise until the bromine color remains. A white precipitate of the bromophenol may form. Solid bromophenol derivatives can be used for the confirmation of the structure of a phenol.

## 3. Phthalein test.

Many phenols yield Phthalein which give special colors in alkaline solutions when reacting with Phthalic anhydride and a little amount of concentrated sulfuric acid. An example is the case with resorcinol:



The fluorescence is because of the oxygen linkage between the two phenolic nuclei (in basic medium).

**Procedure:**

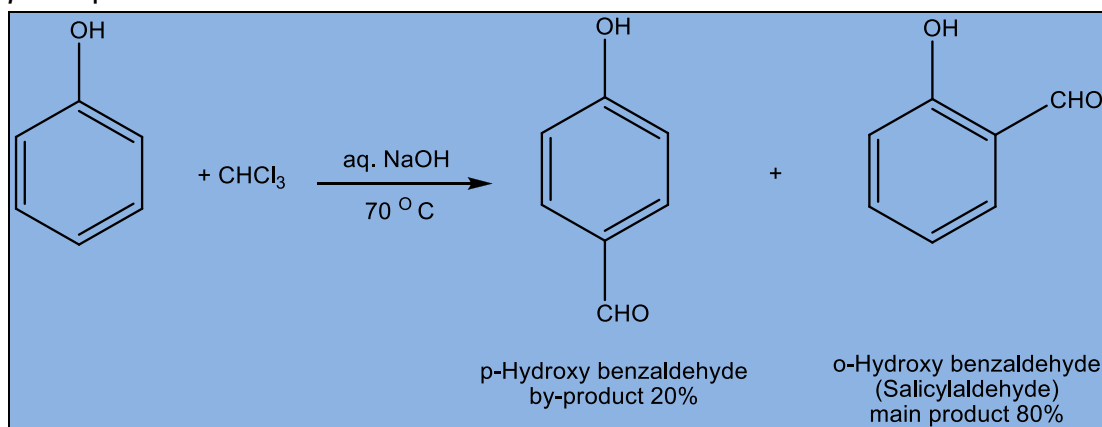
In a dry test tube put about 0.1 gm of the compound and an equal amount of Phthalic anhydride or Phthalic acid, mix well, and add 1-2 drops of conc. H<sub>2</sub>SO<sub>4</sub>. Heat on a direct flame for 1 minute until the crystals of the mixture melts. Then cool the test tube and add excess of 10% sodium hydroxide solution. Results should be as follows:

Phenols	Colors
<i>β</i> -naphthol	very pale green with slight fluorescence
Phenol	red to pink
<i>o</i> -cresol	red – violet
<i>m</i> -cresol	blue to pink
Resorcinol	pale red color with green fluorescence
Hydroquinone	iodine color

If the resultant color is not so clear you can dilute with water.

#### 4. Riemer - Tiemann reaction.

Treatment of phenol with chloroform and aqueous sodium hydroxide solution introduces an aldehyde group (-CHO) into the aromatic ring at the *ortho*- or *para*- positions:



#### Procedure:

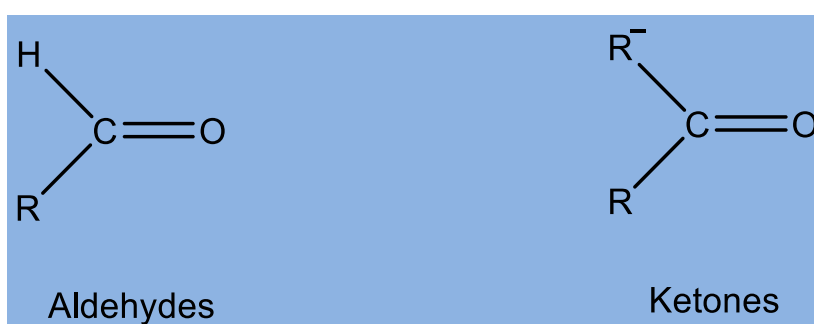
To about 0.2 gm of the phenol add 1 ml of 30% NaOH solution and 1 ml of chloroform, heat on water bath, and observe the color of the aqueous layer:

Phenols	Colors
phenol	yellow or no color
<i>o</i> -cresol	deep orange
resorcinol	red color with a little fluorescence
hydroquinone	deep brown
$\beta$ -naphthol	deep blue that turns to green
<i>m</i> -cresol	pale orange
<i>p</i> -cresol	yellow

## EXPERIMENT 4

# IDENTIFICATION OF ALDEHYDES AND KETONES

Aldehydes are compounds of the general formula RCHO; ketones are compounds of the general formula RCOR'. The groups R and R' may be aliphatic or aromatic, and in one aldehyde, formaldehyde, R is a hydrogen atom.



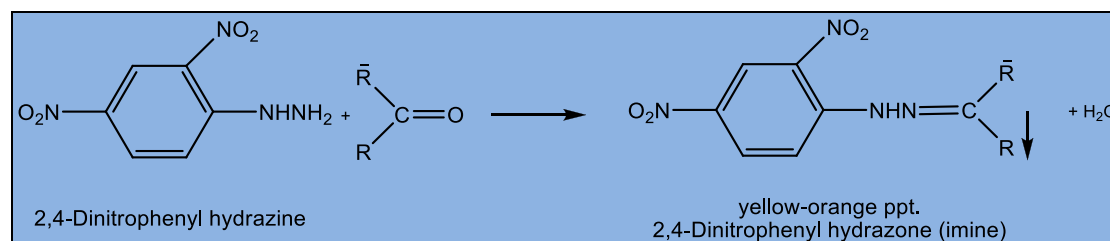
Both contain the carbonyl group (C=O), which lends to their chief chemical and physical properties.

Examples of them include formaldehyde, acetaldehyde, propionaldehyde, benzaldehyde, salicylaldehyde, acetone, acetophenone, benzyl methyl ketone, and benzophenone.

### Chemical Reactions:

#### 1. General test (2, 4-Dinitrophenylhydrazine).

Both aldehydes and ketones give yellow or orange precipitate with 2,4-Dinitrophenylhydrazine reagent.



### Procedure:

Add to 2 drops of the compound 3 drops of the reagent, a yellow or orange precipitate will be formed. If the compound is insoluble in water, dissolve it in 1 ml of methanol and then add the reagent

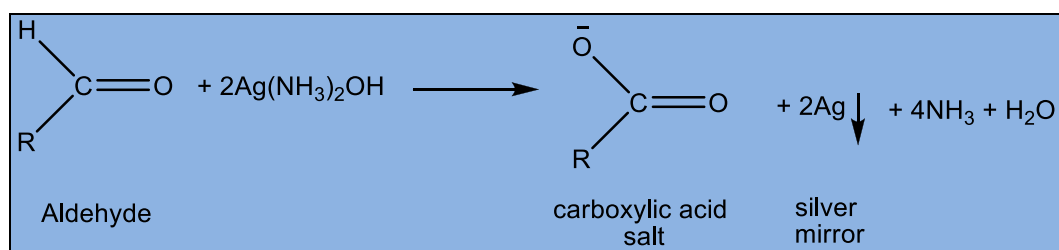
## 2. Tests for differentiation between aldehydes and ketones.

Differentiation between aldehydes and ketones is achieved by taking the advantage of the fact that aldehydes can easily oxidized while ketones cannot (they need stronger oxidizing agents).

Two reagents can be used for this purpose, Tollen's reagent or Fehling's reagent. Only aldehydes give positive results with these two reagents.

### A. Tollen's test (Reduction of ammonical silver nitrate).

Tollen's reagent is the combination of silver nitrate solution with ammonium hydroxide in the presence of sodium hydroxide solution. This reagent gives a silver mirror in the presence of aldehydes because the reaction between them involves the oxidation of the aldehyde to the corresponding carboxylic acid with an accompanying reduction of silver ion from this reagent to silver element in the form of a silver mirror.



The oxidation process needs alkaline medium; therefore sodium hydroxide solution is used, and in order to overcome the formation of the brown silver oxide precipitate ( $\text{Ag}_2\text{O}$ ), ammonium hydroxide is used to serve as a complexing agent for this precipitate making it a water soluble complex.

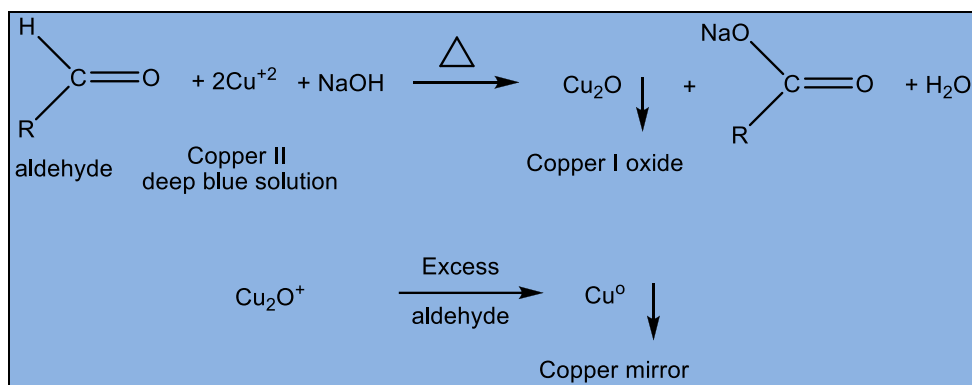
Note that since the medium is alkaline, salts of the produced carboxylic acid are formed rather than the acid itself.

### Procedure:

- Tollen's reagent.  
Add 2 – 3 drops of the compound to 2 – 3 ml of Tollen's reagent, a silver mirror will be formed. If no reaction occurs, warm the test tube in water bath for few minutes.

### B. Reduction of Fehling's reagent.

This test, like Tollen's test, is used to distinguish aldehydes from ketones. Only aldehydes can reduce Fehling's reagent (a deep blue solution) to give a red cuprous oxide precipitate ( $\text{Cu}_2\text{O}$ ).

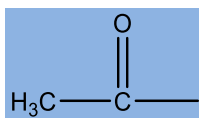


### Procedure:

- Add 5 drops of the compound to 1 ml of Fehling's solution, and then heat in water bath for 5 minutes (with shaking for water insoluble compounds).

Aldehydes change the color of Fehling's solution from blue to green, orange precipitate, and then red precipitate or copper mirror. Ketones don't change the color of this reagent. On the other hand, this test does not give sharp results with aromatic aldehydes.

### **3. Special tests for aldehydes and ketones containing a terminal methyl group.**



These include acetaldehyde, acetone, acetophenone, and benzyl methyl ketone.

#### **A. Iodoform (Haloform) test.**

For details about the procedure of this test see Iodoform test for alcohols that contain a terminal methyl group.

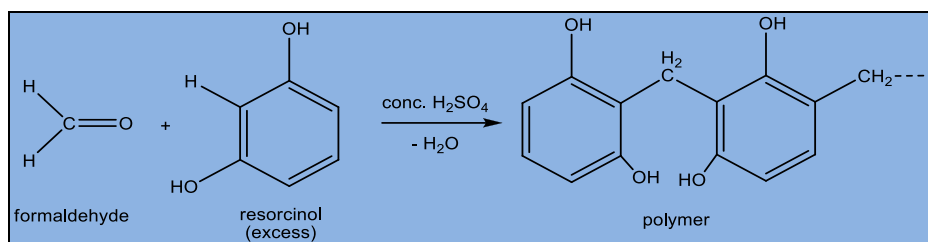
#### **B. Sodium nitroprusside test (Na<sub>2</sub> [Fe (CN)<sub>6</sub> NO].2H<sub>2</sub>O).**

To few drops of the compound add 1 ml of sodium nitroprusside solution and excess of 30% NaOH solution, a red color complex is a positive test.

### **4. Polymerization reaction.**

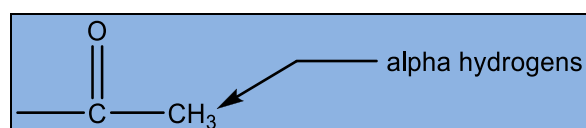
To 0.5 ml of formaldehyde or salicylaldehyde add 0.2 gm of resorcinol and drop by drop concentrated sulfuric acid to get a red or reddish violet color, or a white ring that changes to a reddish violet ring.



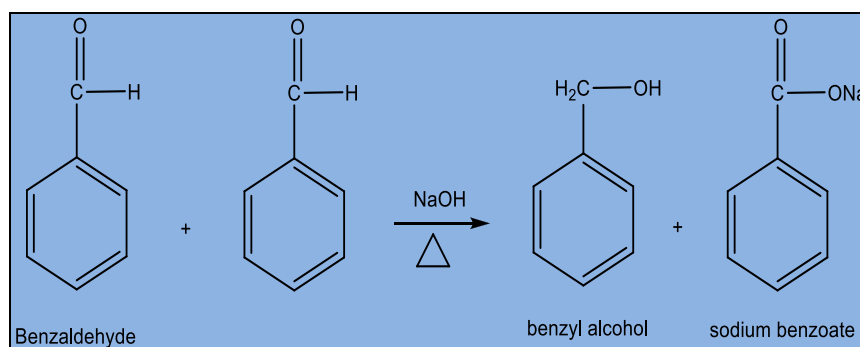


### 5. Cannizzaro reaction.

Benzaldehyde, salicylaldehyde, and formaldehyde can undergo this reaction because they do not contain an alpha hydrogen atom.



In this type of reactions the aldehyde undergoes a self oxidation-reduction in the presence of a strong basic medium to yield a mixture of the corresponding alcohol and the salt of the corresponding carboxylic acid (or the acid itself). Therefore, one molecule of the aldehyde serves as the oxidizing agent while the other serves as the reducing agent.



### Procedure:

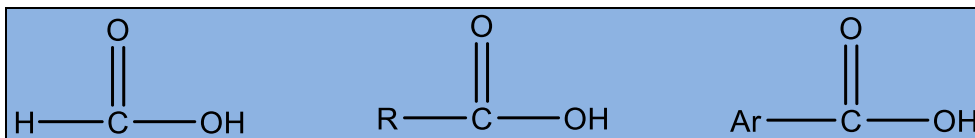
To a few drops of benzaldehyde (or the other aldehydes) add 0.5 ml of 30% sodium hydroxide solution and heat slowly with shaking for five minutes. A precipitate of sodium benzoate is produced. Dissolve this precipitate by adding few drops of distilled water, and then add drops of concentrated hydrochloric acid to liberate benzoic acid as a white precipitate.

As mentioned earlier formaldehyde can undergo this reaction; however, this reaction can not be considered for testing formaldehyde since the acid produced formic acid, is liquid and can not be seen as compared to the solid benzoic acid resulted from benzaldehyde.

## EXPERIMENT 5

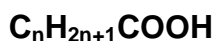
### IDENTIFICATION OF CARBOXYLIC ACIDS

Carboxylic acids are organic compounds that have a carboxyl group (-COOH) attached to hydrogen (HCOOH), to an alkyl group (RCOOH), or to an aryl group (ArCOOH).



They may be mono carboxylated such as formic acid and acetic acid, multi carboxylated such as oxalic acid and succinic acid, hydroxylated such as lactic acid and citric acid, or they may be aromatic such as benzoic acid and salicylic acid

Carboxylic acids that have long, straight-chain hydrocarbon group (with 12 or more C atoms) are commonly classified as **fatty acids**, and several are biological importance. Fatty acids occur in animal or vegetable tissue and nearly always have an even number of C atoms. Those that have only single bonds between C atoms are **saturated fatty acids**, while those with one or more C=C double bond are **unsaturated fatty acids**. The general formula of saturated fatty acid is:



Stearic acid is saturated fatty acids with 17 C atoms in the hydrocarbon group, R, so general formula is:



#### **General test (Ferric chloride test).**

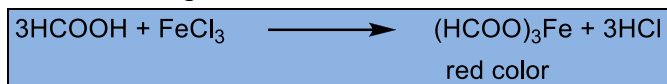
The acid solution should be made neutral before performing the test with ferric chloride solution. This is achieved by adding dilute ammonia solution drop by drop with shaking to a solution of about 0.5 gm of the acid in water until the medium becomes basic as indicated by changing the color of litmus paper to blue or changing the color of phenolphthalein indicator from colorless to pink, in which case the characteristic odor of ammonia is predominant.

As mentioned above the solution should be neutral and the excess ammonia should be removed by heating the test tube until the odor of ammonia disappears.

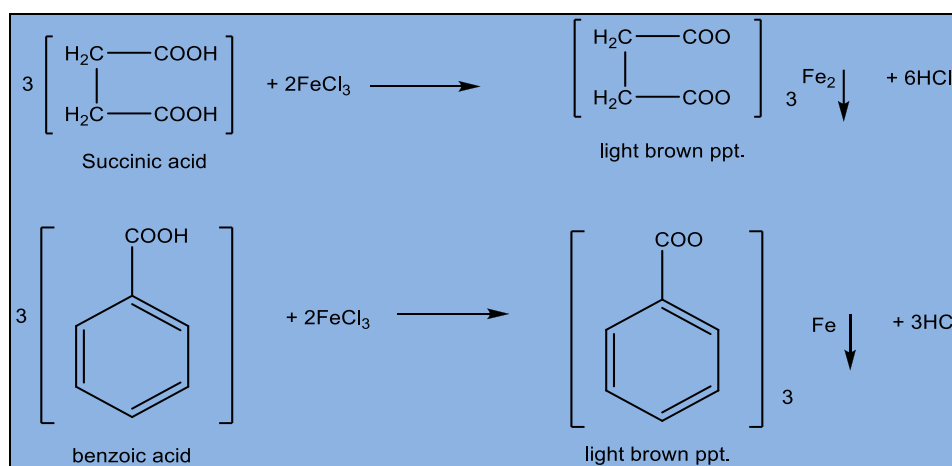
(Note: In case of oxalic acid keep part of its neutral solution to be used in its special test as will be seen later).

Cool the solution, and then add few drops of ferric chloride solution to get different colors of solutions or precipitates as follows:

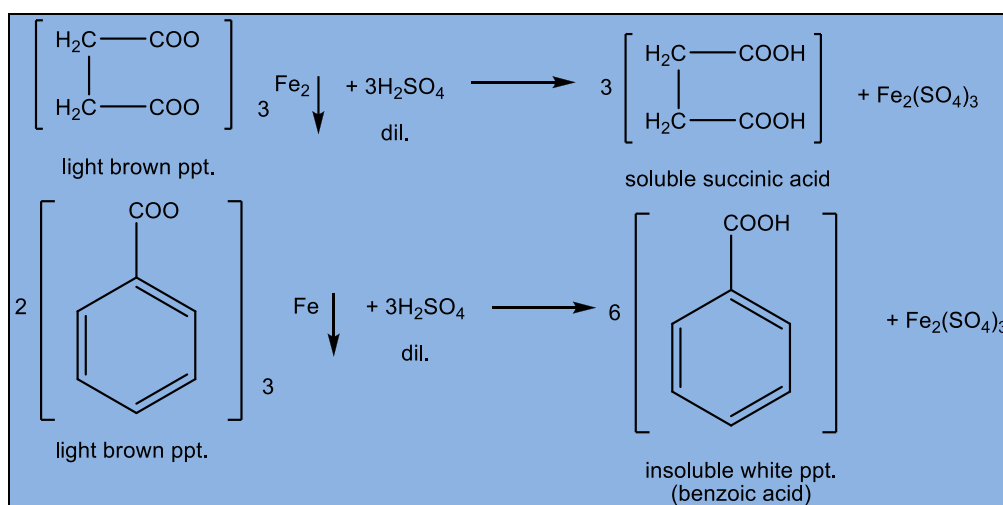
Formic acid and acetic acid give a red colored solution.



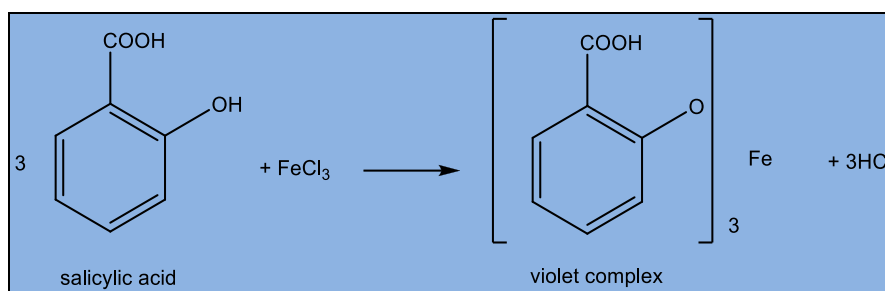
Succinic acid and benzoic acid give a light brown precipitate.



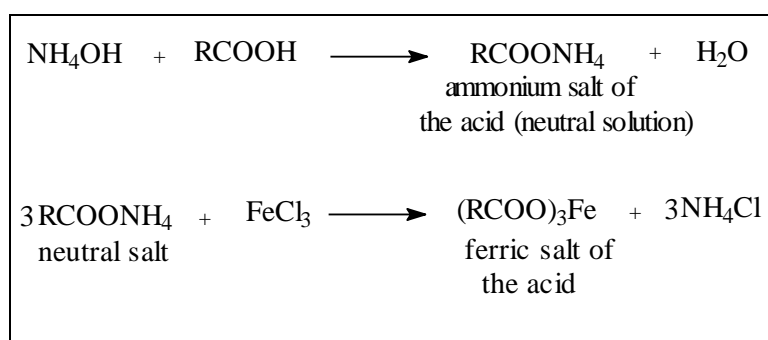
To distinguish between these two acids add to the precipitate few drops of dilute sulfuric acid to liberate the free carboxylic acid again. In one case the liberated acid is water soluble and it is succinic acid which is aliphatic. On the other hand benzoic acid is liberated as a white precipitate because it is insoluble in water since it is aromatic.



Salicylic acid gives a violet color.



Oxalic, tartaric, citric, and lactic acids don't give a special change. Again the details of the general reaction are:

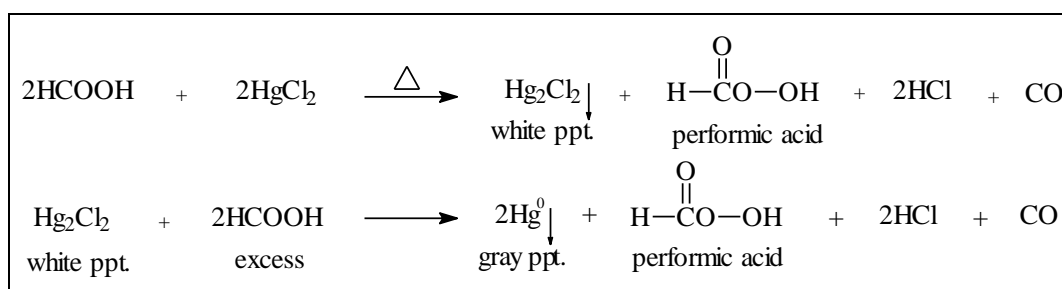


### Special tests for formic acid.

Formic acid has the group ( $\text{HC}=\text{O}$ ) so it can reduce certain compounds while being oxidized:

#### A. $\text{HgCl}_2$ test.

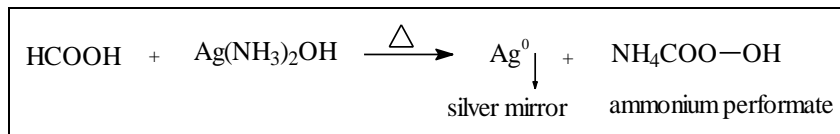
Formic acid reduces mercuric chloride to mercurous chloride (white precipitate) and, in the presence of excess acid, to mercury element (gray precipitate).



To few drops of the acid add few drops of mercuric chloride solution, and Then heat to get a white precipitate. Add excess of the acid with heating to get the gray precipitate of elemental mercury.

## B. Tollen's test.

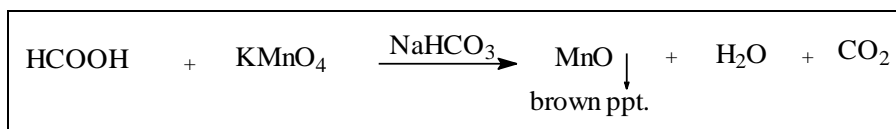
For procedure and preparation of Tollen's reagent refer to this test (page 18).



## C. Alkaline $\text{KMnO}_4$ test.

Formic acid reacts with potassium permanganate solution, a strong oxidizing agent, in alkaline medium causing decolourization of the reagent.

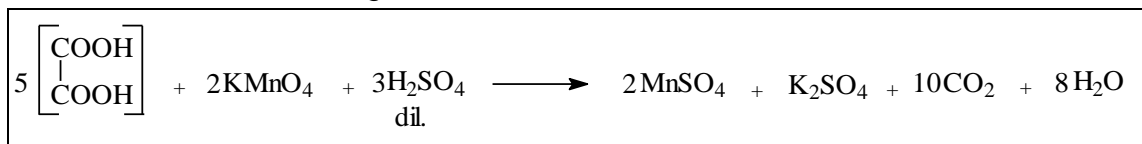
Mix 2 - 3 drops of the acid with 5 ml of sodium bicarbonate solution, and then add 1% potassium permanganate solution drop by drop and observe the disappearance of a brown precipitate of manganese oxide.



## Special tests for oxalic acid.

### A. Acidic $\text{KMnO}_4$ test.

Oxalic acid doesn't react with alkaline potassium permanganate solution. With acidic potassium permanganate solution it reacts causing decolourization of the reagent:

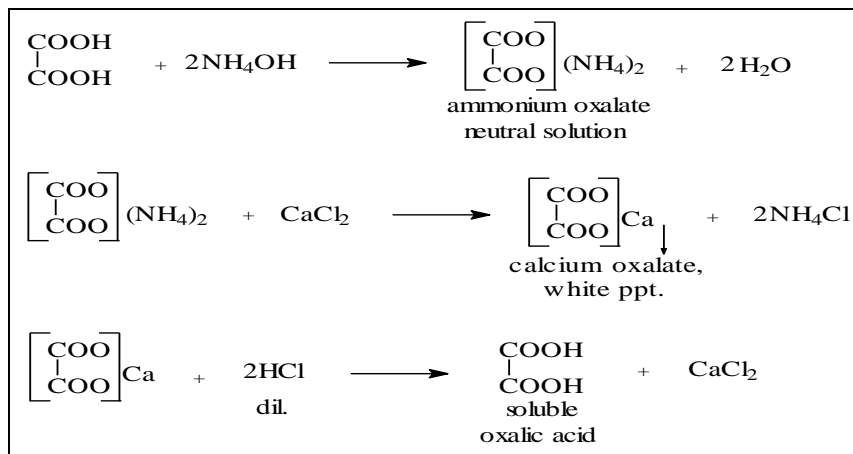


Dissolve 0.5 gm of the acid in 2 - 3 ml of distilled water and add 2 - 3 ml of dilute sulfuric acid. Heat gently (water bath), and then add potassium permanganate solution drop by drop and observe the disappearance of the violet color of the reagent.



## B. CaCl<sub>2</sub> test.

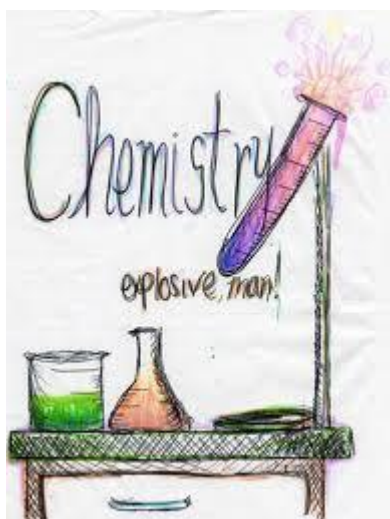
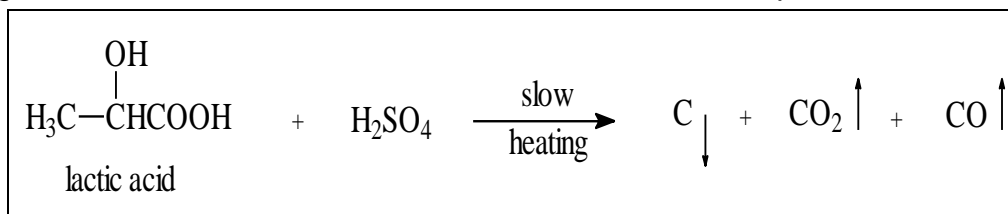
To the neutral solution of the acid (see the general test) add few drops of calcium chloride solution; a white precipitate of calcium oxalate is formed. This precipitate dissolves in dilute hydrochloric acid and not in dilute acetic acid.



## Special test for lactic acid.

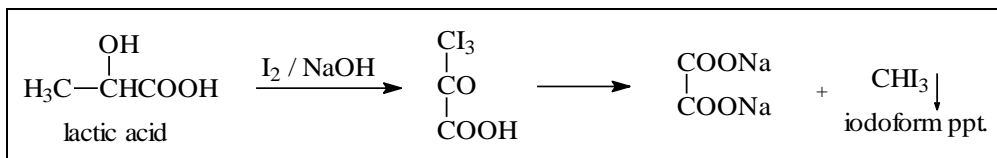
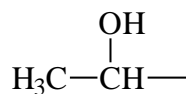
### A. H<sub>2</sub>SO<sub>4</sub> test.

Add 1 ml of concentrated sulfuric acid to 0.5 ml of the acid and heat slowly to get bubbles of carbon dioxide and then a black color is produced.



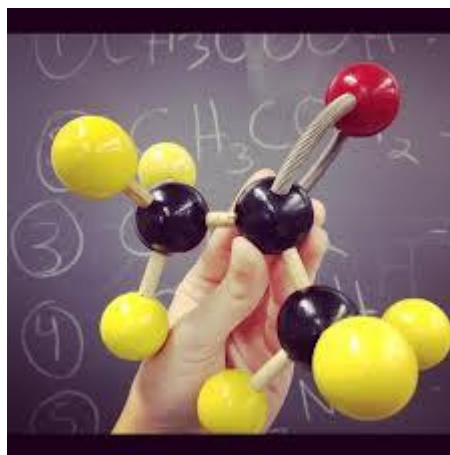
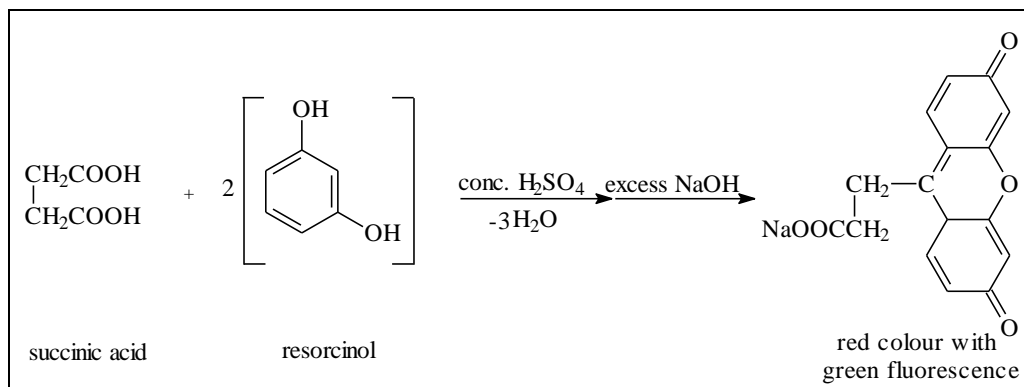
### B. Iodoform test.

Lactic acid can undergo Iodoform formation reaction since it contains a terminal methyl group;



### Fluorescence test for succinic acid.

In a test tube mix equal quantities of succinic acid and resorcinol with 2 drops of concentrated sulfuric acid. Heat the mixture on direct flame for 1 minute until the mixture melts. Cool and add excess of 10% sodium hydroxide solution to get a red color with green fluorescence. If the color is not so clear dilute with water.



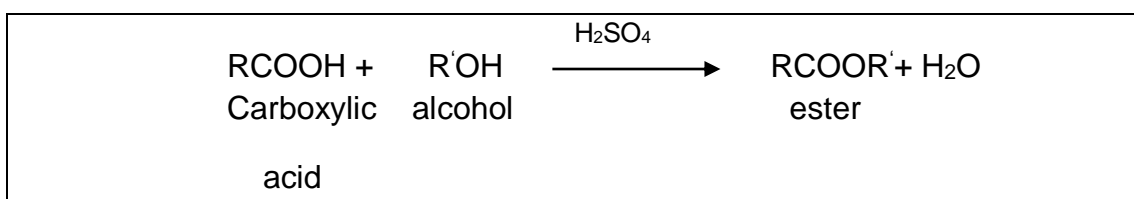
## EXPERIMENT 6

# THE PROPERTIES AND PREPARATION OF ESTERS AND SOAP

In this experiment we are going to prepare several esters and not their characteristic aromas. we will also prepare soap an carboxylic acid salt

### Esters:

The general formula for an ester is  $\text{RCOOR}'$ . An ester can be prepared by reacting an alcohol with carboxylic acid. The R group in the general formula was part of the acid. The other group, symbolized  $\text{R}'$  was part of the alcohol. Sulfuric acid catalyzes the reaction.

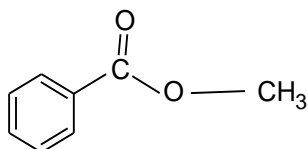


The names of esters are similar in form to those of the salt of carboxylic acids.

$\text{HCOOCH}_3$   
Methyl formate

$\text{CH}_3\text{COOCH}_3$   
methyl acetate

$\text{CH}_3\text{COOCH}_2\text{CH}_3$   
ethyl acetate



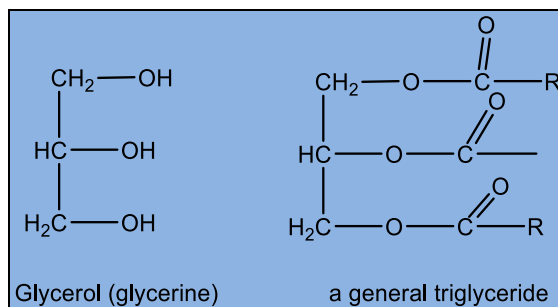
Methyl benzoate

Many esters are colorless liquids with pleasant, fruity aromas. The aroma and flavor of many foods are due to the presence of esters.

### Triglycerides:

Triglycerides are triesters, containing three ester functional groups per molecule. They are large molecules that can be considered the products of the reaction of glycerol (an alcohol containing three  $-\text{OH}$  groups that is also called glycerine) with three fatty acids. Fats and oils are triglycerides.



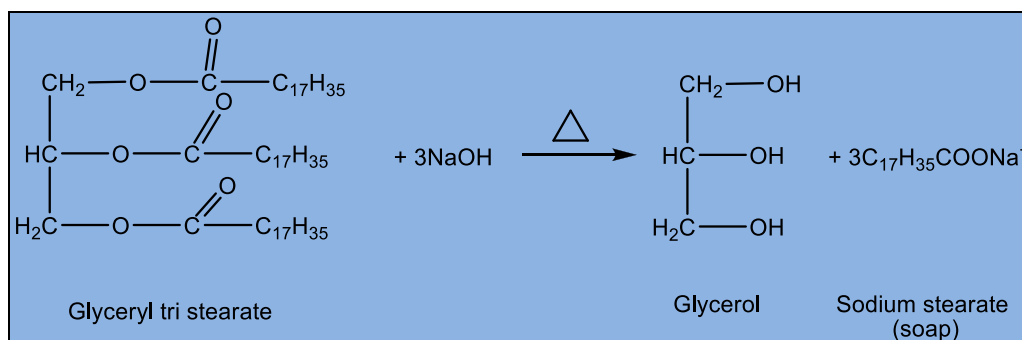


### The preparation of soap - Saponification

Soap is prepared by the hydrolysis of triglycerides in the presence of strong base like NaOH or KOH. Instead of obtaining a fatty acids as products of the hydrolysis. The Na or K salt of the fatty acids is obtained. If a fatty acid did form in the hydrolysis, it would quickly be neutralized by the NaOH or KOH to form the soap.

The soluble salt of a fatty acid is soap. The reaction that produces soap is called

**Saponification** and it is of great industrial importance. Sodium stearate is produce when glyceryl tri stearate, from animal fat, is heated in the presence of aqueous NaOH. Sodium stearate is the primary component of Ivory soap.



Procedure:

#### A. Preparation of some esters

1. Place the following reagents in the labeled test tubes:

- Test tube A: 20 drops of ethanol + 20 drops of glacial acetic acid + 10 drops of concentrated H<sub>2</sub>SO<sub>4</sub>.
- Test tube B: 20 drops of pentanol + 20 drops of glacial acetic acid + 10 drops of concentrated H<sub>2</sub>SO<sub>4</sub>.
- Test tube C: 20 drops of benzoic acid + 20 drops of ethanol + 10 drops of concentrated H<sub>2</sub>SO<sub>4</sub>.

2. Stir the contents of each test tube. When the water bath has reached 85° C, turn off the burner and place stoppered test tube in the hot water.

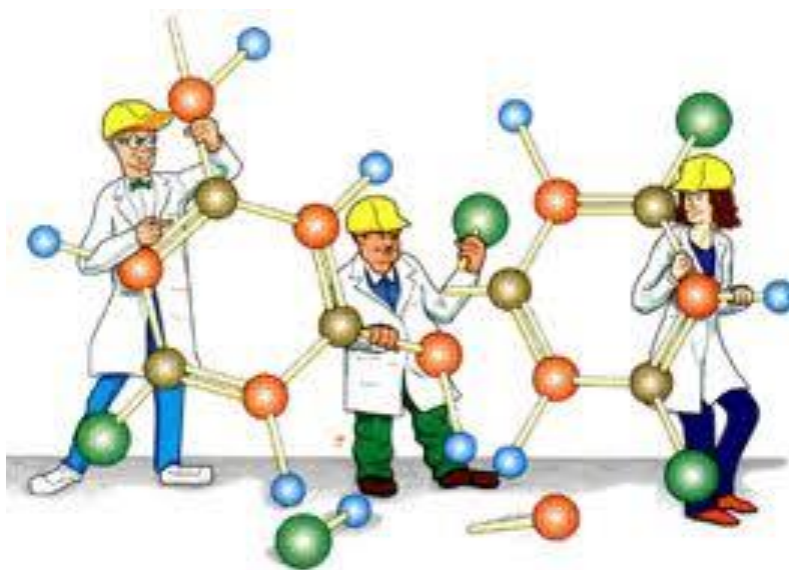
3. After 10 min. removes the stopper from each tube and notes the odor should product. You be able to detect the aroma of banana, butter rum, fingernail and minty scent of wintergreen. Record the aroma of each mixture on the report sheet, then complete the equation and name the ester.

### **B. Preparation of soap**

1. In an evaporating dish, mix 2 ml of vegetable oil and 3 ml of ethanol, this is a good solvent for both triglyceride (the vegetable oil) and NaOH. Add 20 drops of 50 % NaOH.

2. Heat the mixture, while stirring, with a moderate, almost luminous flame until it becomes a thick paste. Allow the evaporating dish to cool. Note the appearance of the

product on the report sheet, then complete the equation and name the products of the saponification reaction.



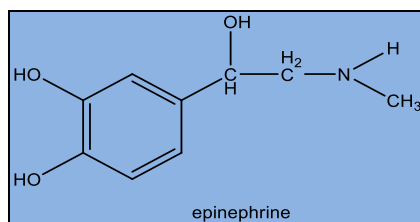
## EXPERIMENT 7

### IDENTIFICATION OF AMINES

Amines are organic compounds and functional groups that contain a basic nitrogen atom with lone pair. Amines are derivatives of ammonia, wherein one or more hydrogen atoms have been replaced a subsistent such as an alkyl or aryl group. Important amines including amino acids, biogenic amines, trimethyl amine and aniline. For aliphatic amines name groups attached to N; use suffix -amine.

#### Biological Activity:

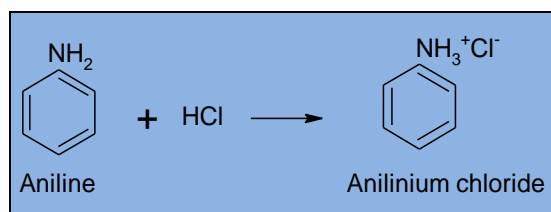
Amines have strong, characteristic odors, and are toxic. The smells of ammonia, old fish, urine, rotting flesh, and semen are mainly composed of amines. Many kinds of biological activity produce amines by breakdown of amino acids. Many hormones like epinephrine, nor epinephrine, and dopamine, are amines.



#### Chemical Reactions:

##### 1. General test (The hydrochloric acid test).

Amines are characterized chiefly through their basicity. A water insoluble compound that dissolves in cold dilute hydrochloric acid or a water-soluble compound whose aqueous turn litmus blue.



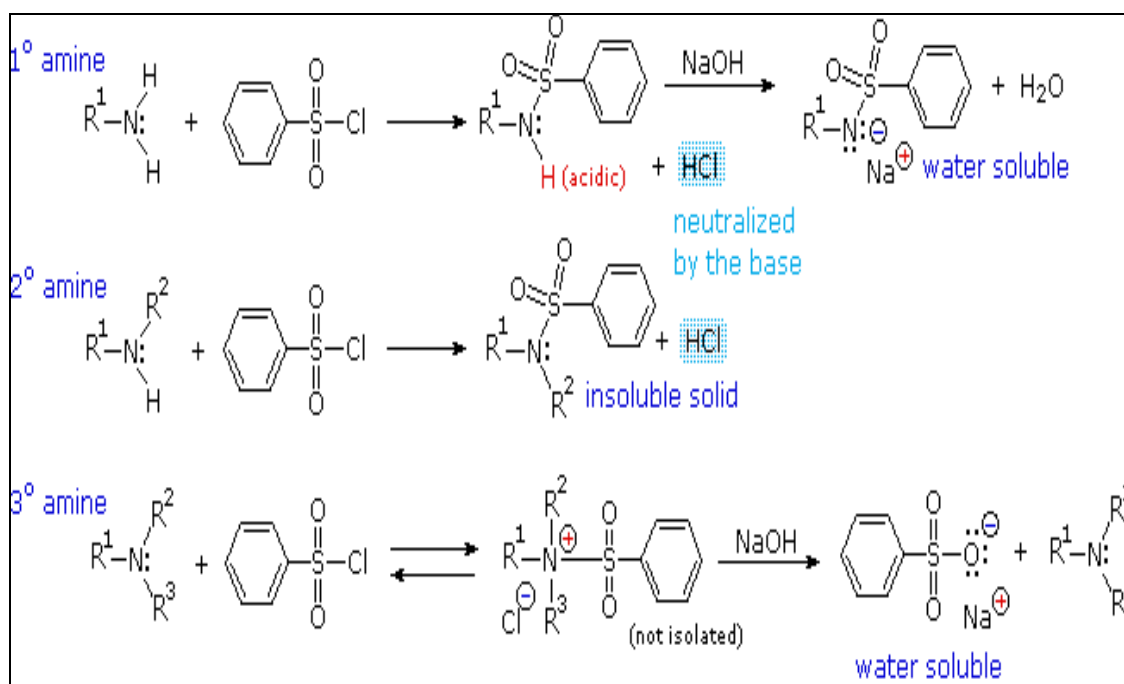
#### Procedure:

Place 3 ml of water in a clean test tube. Add one drop or a spatula tip of your unknown. Swirl the mixture. If your unknown is soluble, test the pH of the solution. An alkaline pH is indicative of an amine. If your unknown is not soluble in water, add 3 ml of 5% HCl solution. If your unknown dissolves in the acid solution, an amine is indicated.

## 2. Tests for differentiation between. Primary and secondary amines.

### The Hinsberg Test .

An electrophilic reagent, benzenesulfonyl chloride, reacts with amines in a fashion that provides a useful test for distinguishing primary, secondary and tertiary amines (the Hinsberg test). As shown in the following equations, 1° and 2°-amines react to give sulfonamide derivatives with loss of HCl, whereas 3°-amines do not give any isolable products other than the starting amine. In the latter case a quaternary "onium" salt may be formed as an intermediate, but this rapidly breaks down in water to liberate the original 3°-amine (lower right equation).



The **Hinsberg test** is conducted in aqueous base (NaOH or KOH), and the benzenesulfonyl chloride reagent is present as an insoluble oil. The amine dissolves in the reagent phase, and immediately reacts (if it is 1° or 2°), with the resulting HCl being neutralized by the base. The sulfonamide derivative from 2°-amines is usually an insoluble solid. However, the sulfonamide derivative from 1°-amines are acidic and dissolve in the aqueous base. Acidification of this solution then precipitates the sulfonamide of the 1°-amine. Benzenesulphonyl chloride reacts with primary and secondary but not with tertiary amines to yield substituted sulphonamides. The substituted sulphonamide formed from a primary amine dissolves in the alkali medium whilst that produced from a secondary amine is insoluble in alkali.

**Procedure:**

Place 0.5 mL (or 0.5 g) of the compound, 15 - 10 mL of 5% NaOH and 1 mL of benzenesulphonyl chloride in a test tube, stopper the tube and shake until the odor of the sulphonyl chloride has disappeared. The solution must be kept alkaline (if no reaction has occurred, the substance is probably a tertiary amine).

If a precipitate appears in the alkaline solution, dilute with about 10 mL of water and shake; if the precipitate does not dissolve, a secondary amine is indicated.

If there is no precipitate, acidify it cautiously to congo red with concentrated hydrochloric acid (added drop wise): a precipitate is indicative of a primary amine.

