INTRODUCTION TO ORGANIC CHEMISTRY (CHEM 109)

Student name:

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Student ID:

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Written by

Sulaiman Alkhamis

Ibrahim Aldossary

Prof. Abdullah Al-Anzi

Prof. Mohammed El-Newehy

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Table: 109 Chem Experiments (Common Year) –Semester 1445 H

Date	Experiment Title	Notes
	EXPERIMENT (0): Instruction and Equipment and Safety	
	EXPERIMENT (1): Solubility	
	EXPERIMENT (2): Extraction (Demo)	
	EXPERIMENT (3): Aliphatic hydrocarbons	
	EXPERIMENT (4): Aromatic hydrocarbons	Quiz (1): EXP (1,2,3,)
	EXPERIMENT (5): Hydroxy compounds (Alcohols & Phenols)	
	EXPERIMENT (6): Preparation of Aspirin (Demo)	
	EXPERIMENT (7): Aldehydes & Ketones	Quiz (2): EXP (4,5,6)
	EXPERIMENT (8): Carbohydrates	
	EXPERIMENT (9): Carboxylic acid & Their derivatives	Quiz (3): EXP (7,8,9)
	EXPERIMENT (10): Amino compounds	
	Final Exam Practical / Theoretical	

INTRODUCTION

Instruction, Equipment and Safety

PLEASE READ THE FOLLOWING BEFORE YOUR FIRST DAY IN THE LAB.

Aim and Purpose of the Course

Obviously the overall aim of the course is to give you experience in practical organic chemistry and help you generally to learn about organic chemistry as a part of medical/pharmacy course, but the purpose of the course will be clearer perhaps from the following five objectives:

1) Experience the process of discovery for yourself and relate it to the role played by experiment in scientific investigations.
2) Become familiar with isolating and purifying compounds by solvent/solvent extraction.
3) Handle as wide a variety of compounds bearing different functional groups as possible.
4) Exploit the opportunities for learning about organic chemistry during the practical time to the full by asking your demonstrator questions.

Continuous Assessment and the Practical Exam

You will receive a mark for your laboratory work after each experiment. Credit for an experiment will be based on attendance, and completion of the experiment to your demonstrators' satisfaction. He will be looking at a number of different things such your neatness and tidiness while working in the laboratory. Your demonstrator will grade your book like this:

A = Excellent result
B = Satisfactory
C = poor work

This grading will count towards your final grade and will help you to assess how well you are progressing in your practice of organic chemistry. For this reason, your demonstrator will discuss the assessment with you when he grades the experiment at the end of the practical period.

Experimental Method and Write-up

No note books are required. Spaces are provided at appropriate places in the manuals for you to record your observations, results etc. These must be written in during the laboratory period and graded by your demonstrator before you leave.

General Instructions

This course deals with a number of test tube reactions which illustrate many of the functional groups covered in the lecture course. These test tube experiments ae to be done during the first half period that you are in the lab.

The first experiments involve important practical techniques. These experiments are followed by experiments which follow the lecture course closely, so the lectures and practical are all part of the same organic chemistry course.

When you first come into the laboratory you will be allocated a certain cupboard containing most of the apparatus required for this terms work. Make sure all your apparatus is returned to this cupboard at the end of every practical.

Additional supplies of more special apparatus is also available for common use, but of limited supply. It is essential that this apparatus is returned clean at the end of each practical.

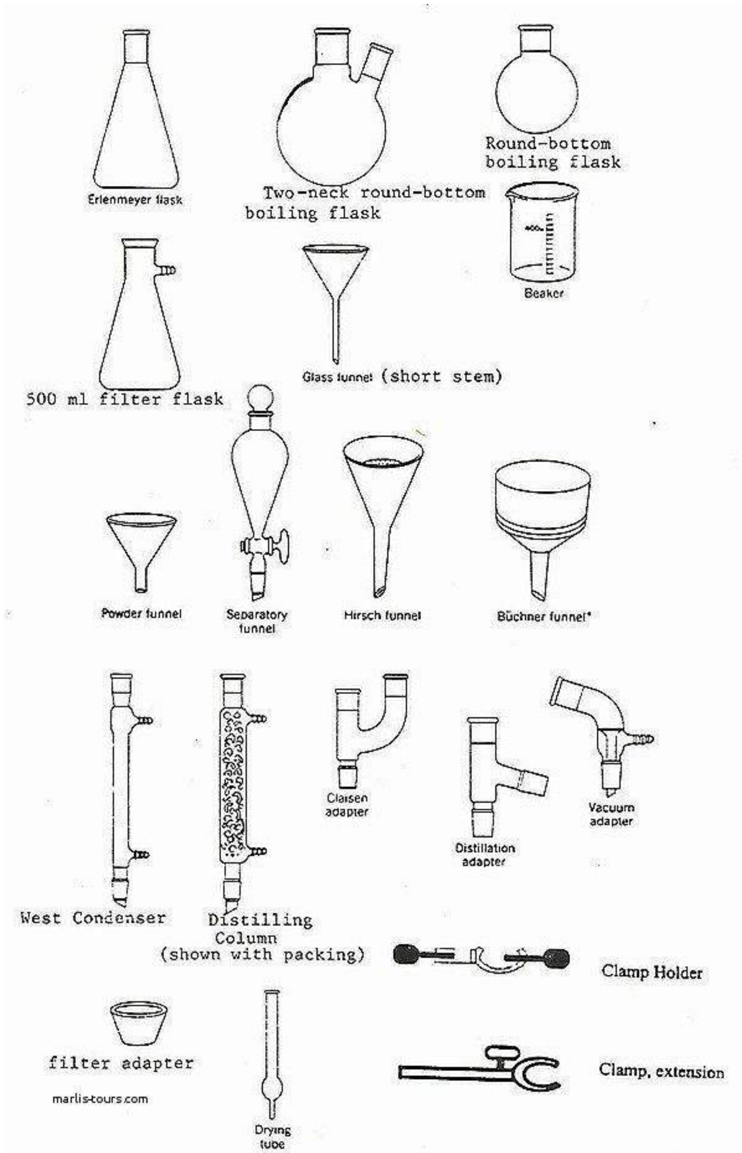
Please refer to the lecture material before you come to the laboratory so that you are familiar with the organic chemistry involved before you carry out any reaction.

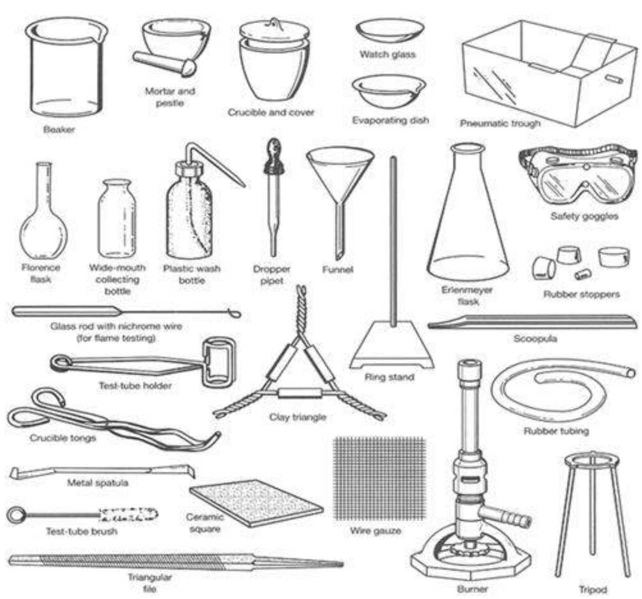
Grading

Thirty percent of your final grade will come from the practical part of the course. At the end of the course there will be a final practical exam. In addition, there will be five or three "drop quizzes", five of which will count towards your grade. These "drop quizzes" will be short 5-minute test which will come without warning. They are designed to encourage you to read the instructions concerning the experiment before you come tothe lab.

Attendance and Reports	10 degree
Drop quizzes Midterm	5 degree
Final Exam	15 degree

Laboratory Equipment





List of Glassware and Equipment

1) Condenser
2) Round-bottomed flask (50 mL and 100 mL)
3) Distilling head.
4) Adaptors.
5) Stoppers.
6) Separating funnels.
7) Beaker (50 mL, 100 mL, 250 mL and 400 mL).
8) Conical flask (100 mL and 250 mL).
9) Buchner funnel.
10) Buchner flask.
11) Funnel.
12) Glass rod.
13) Capillary tubes.
14) Test tubes.
15) Washing bottles.
16) Test tube rack.
17) Stand.
18) Clamps.
19) Test tube holder.
20) Heating Mantel 50 mL.
21) Watch glass.
22) Brush.

قائمة بالمختصرات الكيميائية

Sol.	Soluble	ذوبانية
Insol.	Insoluble	غير ذائبة
Eff.	Effervescence	فوران او تصاعد غاز
Ppt.	Precipitate	راسب
Dil.	Dilute solution	محلول مخفف
Conc.	Concentrate solution	محلول مركز
Obs.	Observation	المشاهدة
Res.	Result	النتائج
mL	Milliliter	ملليليتر
min	Minutes	دقائق
m.p	Melting point	درجة الانصهار
b.p	Boiling point	درجة الغليان
g	Gram	الجرام

Safety

A chemistry lab can be dangerous place. If you work with care, however you ought to survive and not eliminate your neighbors. Therefore: -

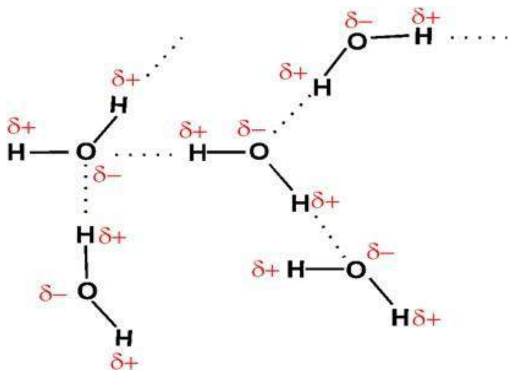
1) **ALWAYS** ware safety glasses in the lab. You may only be sitting at your bench writing in your manual, but your neighbor's distillation may explode.
2) **NEVER**, but never, allow ANY organic compound on your skin or breathe in the dust from a solid or spray from a liquid.
   Some compounds are labelled as particularly dangerous. Carcinogenic ones cause cancer, some apparently harmless ones, like benzene, are very toxic indeed. **ALL ARE LETHAL IN SUFFICIENT QUANTITY.**
   Rubber gloves are available to protect your hands.
3) **NEVER** boil organic compounds in the open lab or allow gases to escape, use the fume cupboard.
4) **BEWARE of FIRE**. Know where the fire extinguishers are. Look around you before lighting your burner. In particular, make sure no one is using ether or petrol nearly.
5) **HANDLE** glass-ware **CAREFULLY**. Use quick fit apparatus, and watch for chipped or cracked apparatus: return it to the preparation room.
6) **REPORT** all accidents to your demonstrator. You may think you are "all right" but it is not worth taking chances.

Keeping your bench clean is most important, always clean up at all times. You will have your grade reduced if you keep it in an untidy mess, the same applies to your locker.

EXPERIMENT 1

Solubility

General Rule: Like dissolve like



Water	1- polar 2- H-bond donor and acceptor 3-Solvates ions

Test-tube 1

- Place 1 mL of distilled water in the test tube.
- Add 0.5 mL of hexane ($CH_3CH_2CH_2CH_2CH_2CH_3$).
- Shake.

Hexane is:

1) non-polar.

2) non- H- bonding.

3) does not solvate ions.

Test-tube 2

- Place 1 mL of distilled water in the test tube.
- Add 0.5 mL of ethanol (CH_3CH_2OH).
- Shake.

Test-tube 3

- Place 1 mL of distilled water in the test tube.
- Add 5 drops of 1-hexanol ($CH_3CH_2CH_2CH_2CH_2CH_2OH$)
- Shake.

In hexanol, the ratio of water insoluble “part” to water soluble “part” is high.

Test-tube 4

- Place 1 mL of distilled water in the test tube.
- Add 1 mL of diethylether ($CH_3CH_2OCH_2CH_3$).
- Shake.
- In the same tube add 1 drop of ($KMnO_4$).
- Add 1 mL of ethanol.
- Shake well.

Test-tube 5

- Place 1 mL of distilled water in the test tube.
- Add 1 mL of chloroform ($CHCl_3$).
- Shake.

Ether and chloroform are good solvents for extraction from aqueous solutions.

Test-tube 6

- Place 1 mL of chloroform in the test tube.
- Add 1 mL of water.
- Add 1 mL of methyl red solution (in water).
- Shake.

Therefore, the methyl red is preferentially soluble in …………………………..

- Add 3 drops of 5% NaOH solution to the methyl red/$CHCl_3$/H_2O mixture and shake well

HA = methyl red

$$HA + NaOH \rightleftharpoons Na^+A^- + H_2O$$

HA (light yellow) soluble in water / $CHCl_3$

Na^+A^- (deep yellow) soluble in water / $CHCl_3$

Report number 1

Test	Observation	Result
H_2O + Hexane		
H_2O + Ethanol		
H_2O + Hexanol		
H_2O + Ether		
H_2O + Ether + Ethanol		
H_2O + Chloroform		
H_2O + Chloroform + methyl red		
3 drops of 5% NaOH solution to the mixture		

Questions:

1) Which of these organic solvents will form one layer with water:

a) Benzene.

b) Propanol.

c) Methanol.

d) Dipropylether.

e) Octanol.

EXPERIMENT 2

Extraction

It is evident from the test-tube reactions that a compound dissolved in one solvent can be extracted into another solvent if:

a) It is more soluble in the second solvent; and
b) The two solvents are immiscible.

On a preparative scale these extractions are carried out in a "separating funnel", which is constructed to allow easy separation of two immiscible solutions.

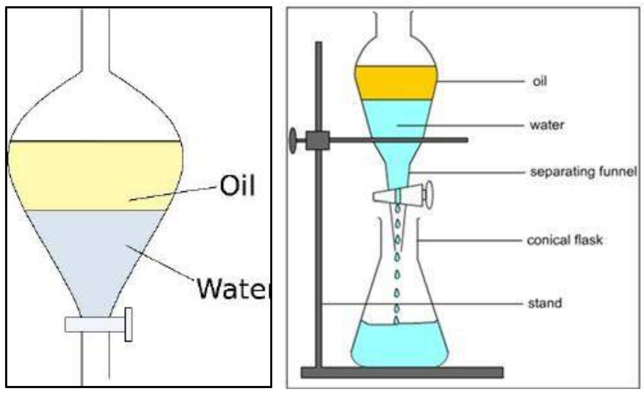


Figure 1: Diagram of separating funnel apparatus.

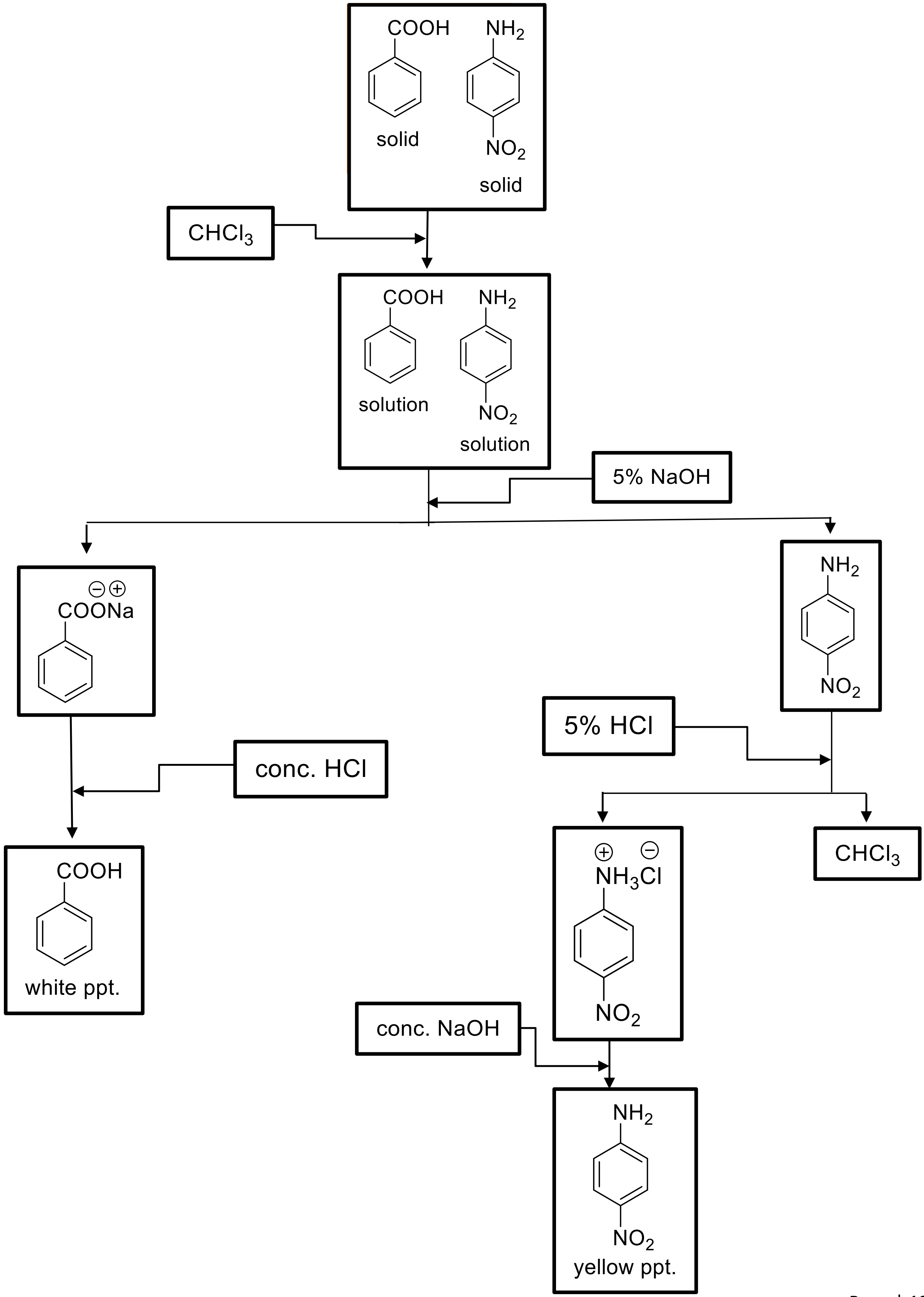
The objective of this experiment is to make you familiar with this technique.

Table 1: List of common solvents that can be used for extraction from water.

Solvent	**Formula**
Hexane (light petrol)	C_6H_{14}
Benzene	C_6H_6
Diethyl ether (ether)	$(C_2H_5)_2O$
Ethyl acetate	$CH_3COOC_2H_5$
Chloroform	$CHCl_3$
Carbon tetrachloride	CCl_4

<u>**NOTE**</u> that methanol, ethanol, and acetone are miscible with water and cannot be used for extraction from water.

Separation of Mixture of Basic and Acidic Compounds



<u>Caution</u> (Before you start)

- Since most organic chemicals are hazardous in some way (e.g. inflammable, poisonous vapor or poisoning through skin) take care not to get any on your hand.
- The *m*-nitroaniline is highly colored and toxic so you will be able to observe any contamination.
- If you get any, on your skin wash your skin with soap and water. Do not wash with an organic solvent, this would assist passage of the contaminant through your skin.

Procedure

- In a 50 mL conical flask weigh approximately 2 g of a mixture of benzoic acid (C_6H_5COOH), HA and *m*-nitroaniline.
- Add 25 mL of chloroform.
- Transfer the solution to a 50 mL separating funnel, using a funnel (Figure 1).

Solvent Extraction General Procedure

- Clamp the separatory funnel securely with the tap lubricated with Vaseline and closed.
- Pour in the chloroform solution through a funnel and use in a little extra chloroform to wash in the last traces of a powder sticking to the walls of the flask and to the funnel.
- Add 20 mL of 10% sodium hydroxide solution to the separatory funnel through the funnel.
- Insert the stopper (lubricated with Vaseline) and shake the separating funnel to mix the layers thoroughly, releasing the pressure via the top at frequently intervals.
- Allow the layers to separate.
- Remove the stopper. Run off the lower layer into a 50 mL conical flask; not to fast or you will create a vortex. Close the tap when the bore of the tap contains the last of the lower layer.

  The chloroform layer contains?

  ……………………………………………………………………………………..…….

  The aqueous layer contains?

  ………………………..……………………………………..……………………..

- Run the upper (aqueous) layer into a 100 mL conical flask.
- Add to upper layer conc. HCl until no whiter precipitate forms. It may be necessary to cool the solution.

  The precipitate is

  ……………………………………………………………………………………………

Allow the solution to cool and then suction filter off the solid, wash it with a little cold water and dry in an oven.

Suction Filtration

Apparatus: Buchner funnel, flasks and filter paper (Figure 2).

1) Choose the correct size of filter paper, wet it all over with water and place it centrally in the funnel.
2) Briefly apply suction to secure the paper firmly in place.
3) Pour in the slurry of crystals. Do not fill to more than 2/3.
4) Use filtrate (mother-liquor) to wash in any crystals which remains stuck to the walls of the flask.
5) To wash the crystals with cold water, first release the vacuum slurry the crystals taking care not to damage the filter paper then reapply the suction.

To dry, place the crystals on a clean filter paper, and cover with another piece of filter paper.

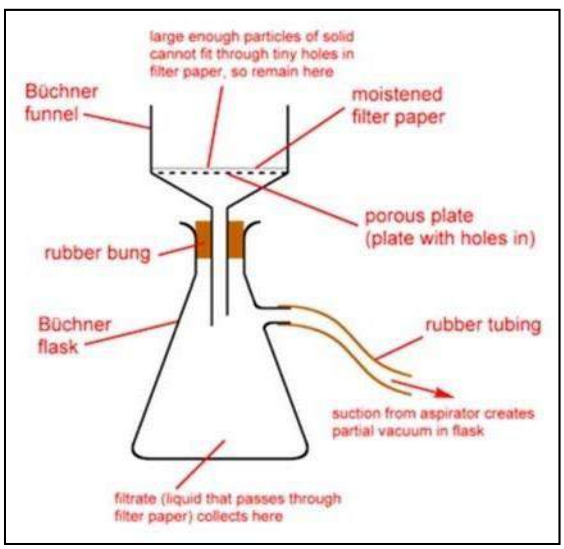


Figure 2: Buchner funnel.

Then put chloroform layer back into separating funnel.

Extract with 20 mL of 10% HCl solution.

The chloroform layer contains? ………………………………………….

The aqueous layer contains? …………………..……………………..

Separate the layers.

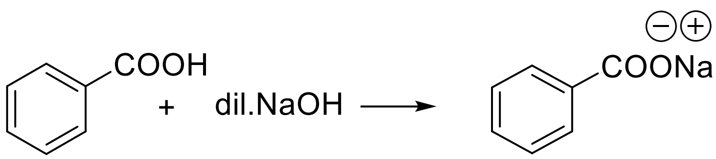
Keep the upper aqueous layer and put the lower chloroform layer into the residue bottle.

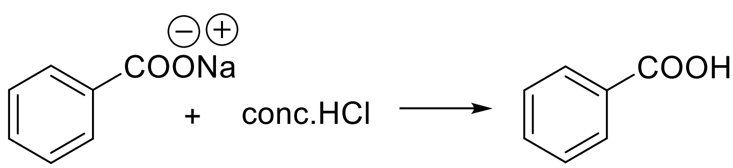
To the aqueous layer, add 10% NaOH until no further precipitation takes place. The precipitate is …………………………….…………………………

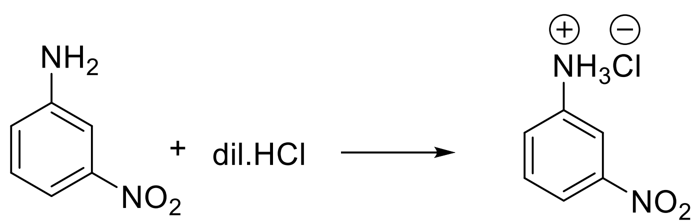
Suction filter the solid, wash with a little water and dry in the air at room temperature.

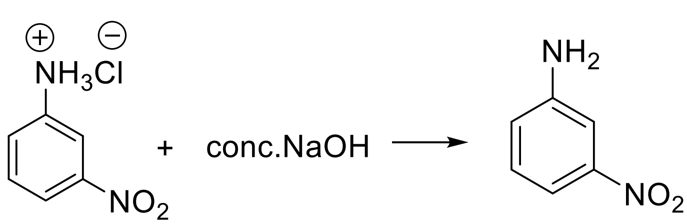
Submit your sample to your demonstrator before you leave.

Chemical equation:

1) 

2) 

3) 

4) 

Questions

Predict the outcome of the following extractions?

1) Benzoic acid dissolved in ether:
   a) Extract with aqueous HCl.
   b) Extract with H_2O.
   c) Extract with aqueous NaOH.
2) Aniline dissolved in ether:
   a) Extract with aqueous HCl.
   b) Extract with H_2O.
   c) Extract with aqueous NaOH.

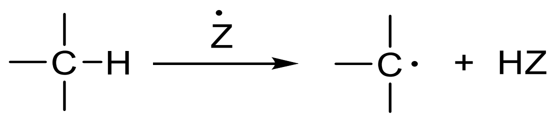
EXPERIMENT 3 Date:

Aliphatic Hydrocarbon (Bonding)

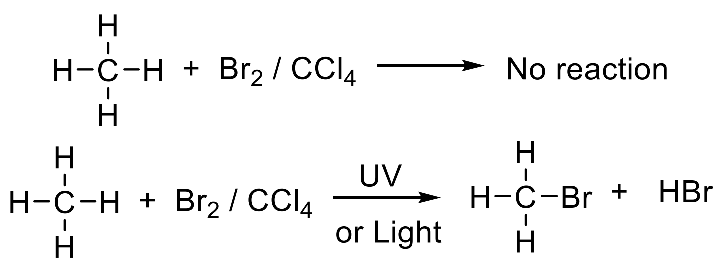
(Alkanes and Alkenes)

- ## Alkanes

In general C – H and C – C bonds in alkanes are unreactive towards polar reagents. Reaction generally occurs by the formation of radicals in the presence of asuitable initiator (Z·). This type of reaction is called **Free Radical Substitution**.



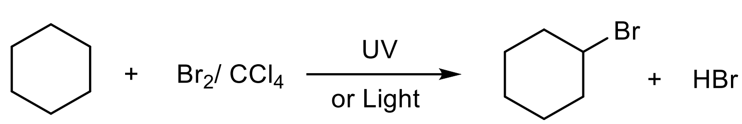
- ## Bromine to an Alkane:



Test-tube 1

- Take 1 mL of cyclohexane.
- Add 3 drops of Br_2/CCl_4 solution.
- In the presence of light from the sun or a UV lamp, the bromine undergoes hemolytic fission and readily reacts with the cyclohexane.

Chemical equation:



- Dip a clean glass rod into conc. NH_4OH and hold the glass rod over the mouth of the test tube. **What happened?**

  ……………………………..……………………………………………………
  ……………………………………………………………………………………..

<u>The observing is the formation of NH_4Br which is a test for the presence of HBr.</u>

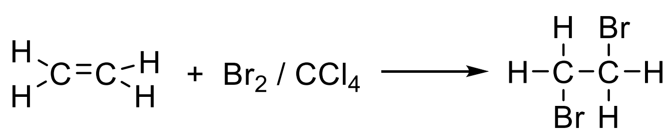
Chemical equation:

$$HBr + NH_4OH \longrightarrow \overset{+}{N}H_4\overset{-}{B}r + H_2O$$

- ## Alkenes

Alkenes undergo **polar addition reactions** initiated by electron rich system of the double bond.

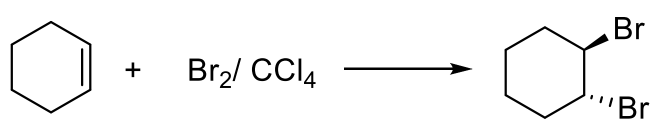
- ## Addition of Bromine to an Alkene



<u>Test-tube 2</u>

- Take 3 drops of cyclohexene.
- Add 3 drops of Br_2/CCl_4 solution.

Chemical equation:



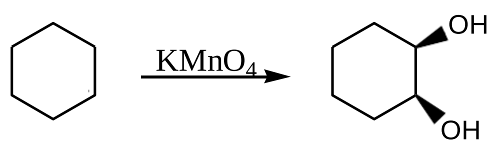
- **Alkene with Potassium Permanganate ($KMnO_4$)**

Reaction with dil. $KMnO_4$ solution is also a test for the presence of carbon-carbon double bonds. The double bond of an alkene is easily oxidized to a di-OL.

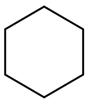
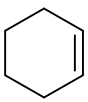
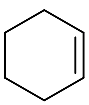
Test-tube 3

- Take 3 mL of dil. $KMnO_4$ solution.
- Add 3 drops of cyclohexene.
- Shake.

Chemical equation:

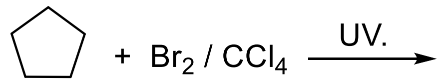


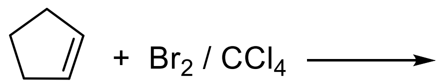
Report number 2

Test	Observation	Result	Chemical equation
 + Br_2 / CCl_4 (light or UV.)			
 + Br_2 / CCl_4 (Direct)			
 + $KMnO_4$			

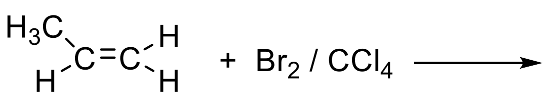
Questions:

Complete the following reactions:

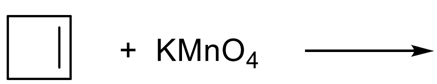
1) 

2) 

3) $CH_3CH_3 + Br_2 / CCl_4 \xrightarrow{\text{Light}}$

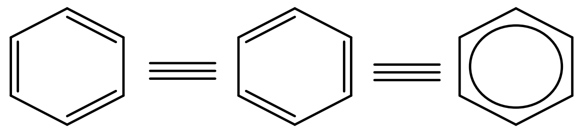
4) 

5) 

6) 

EXPERIMENT 4 Date:

Aromatic Hydrocarbons



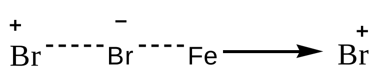
Benzene

Aromatic compounds have multiple double bonds; these compounds do not undergo addition reactions. Their lack of reactivity toward addition reactions is due to the great stability of the ring systems **that result from resonance.**

- **Bromine with Benzene**

Test-tube 1

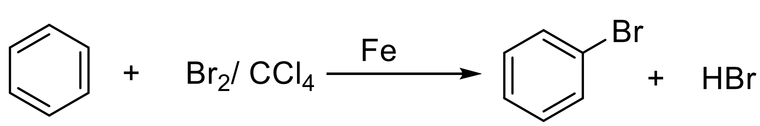
- Take 1 mL of 90% acetic acid (solvent).
- Add 5 drops of benzene.
- Add 5 drops of Br_2/CCl_4.
- Pour the contents of the tube into the vial containing the iron powder, shake. Polarization of the bromine occurs



The positive bromine species is a better electrophile than molecular bromine.

The type of reaction in benzene is **electrophilic aromatic substitution.**

Chemical equation:

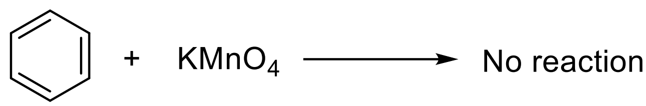


- **Benzene with Potassium Permanganate ($KMnO_4$)**

Test-tube 2

- Take 1 mL of dilute aqueous $KMnO_4$ solution.
- Add 2 drops of benzene.
- Shake.

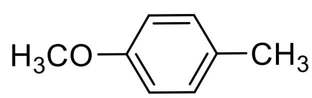
Chemical equation:



- **Oxidation of Alkyl Side-Chains**

alkylbenzenes are oxidized to benzoic acids (-COOH), as long as the alkyl group contains at least one hydrogen at the benzylic position.

p-Methoxytoulene with $KMnO_4$

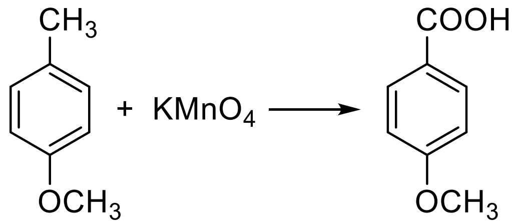


p-Methoxytoulene

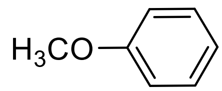
Test-tube 3

- Take 1 mL of dilute aqueous $KMnO_4$ solution.
- Add one drop of *p*-methoxytoulene.
- Shake.

Chemical equation



Anisole (Methoxybenzene) with $KMnO_4$



Methoxybenzene

Test-tube 4

- Take 1 mL of dilute aqueous $KMnO_4$ solution.
- Add 2 drops of anisole (methoxybenzene).
- Shake.

• Nitration of Benzene

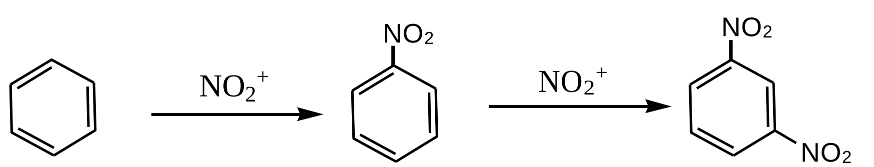
Nitration occurs when one (or more) of the hydrogen atoms on the benzene ring is replaced by a nitro group, NO_2. Nitrating mixture is concentrated nitric acid (HNO_3) with sulfuric acid (H_2SO_4).

$$HNO_3 + H_2SO_4 \rightleftharpoons NO_2^{\oplus} + HSO_4^{\ominus} + H_2O$$

Test-tube 5

- Take 1 mL of nitrating mixture (conc. H_2SO_4 + conc. HNO_3).
- Add 5 drops of benzene.
- Shake well.
- Take about 20 mL of distilled water in a small beaker.
- Pour reaction mixture into this water.

Observation



Benzene a liquid less dense than water

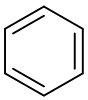
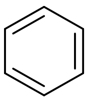
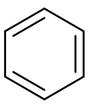
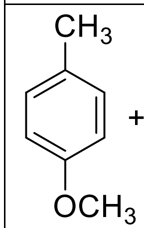
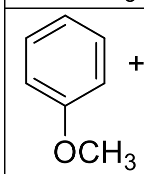
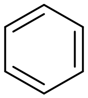
Nitrobenzene a liquid more dense than water

m-Dinitrobenzene solid

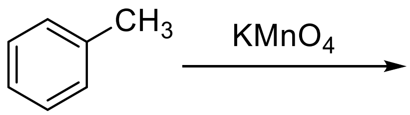
The main product of my reaction is ……………………………………………..

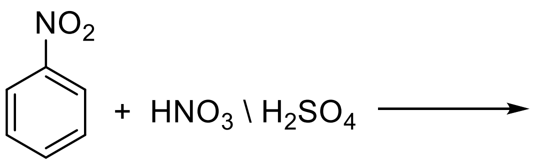
The first nitro group deactivates the ring to further attack and directs the second nitrogroup exclusively to the meta position.

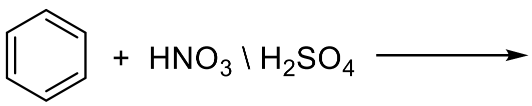
Report number 3

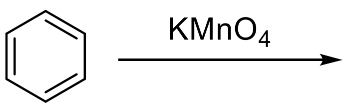
Test	Observation	Result	Chemical equation
 + Br_2 / CCl_4 (Direct)			
 + Br_2 / CCl_4 (Fe)			
 + $KMnO_4$			
 + $KMnO_4$			
 + $KMnO_4$			
 + HNO_3 \ H_2SO_4			

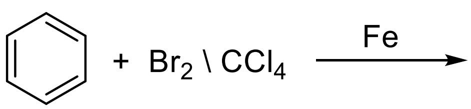
Questions

1) 

2) 

3) 

4) 

5) 

EXPERIMENT 5 Date:

Hydroxy Compounds (Alcohols and Phenols)

- ## Preparation of Alkyl Halide from Alcohol

In practice:

Reaction 1

$$ROH + SOCl_2 \longrightarrow RCl + SO_2 + HCl$$

Thionyl choride

Reaction 2

$$ROH + PCl_5 \longrightarrow RCl + POCl_2 + HCl$$

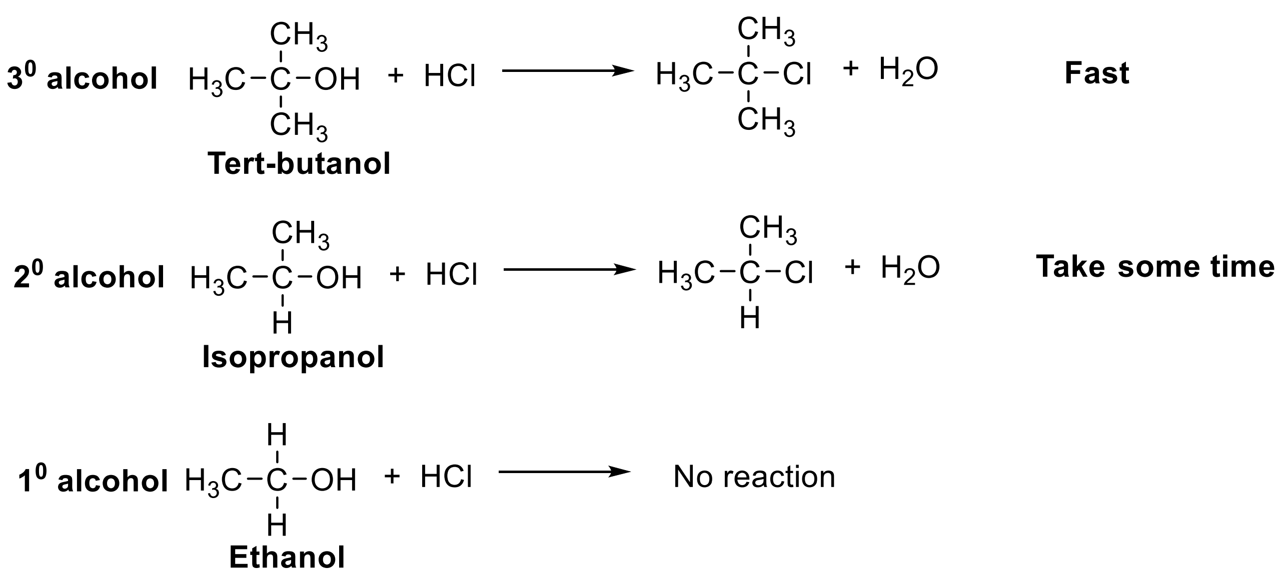
pentachlorphosphors

- ## Lucas' reagent

Test-tube 1

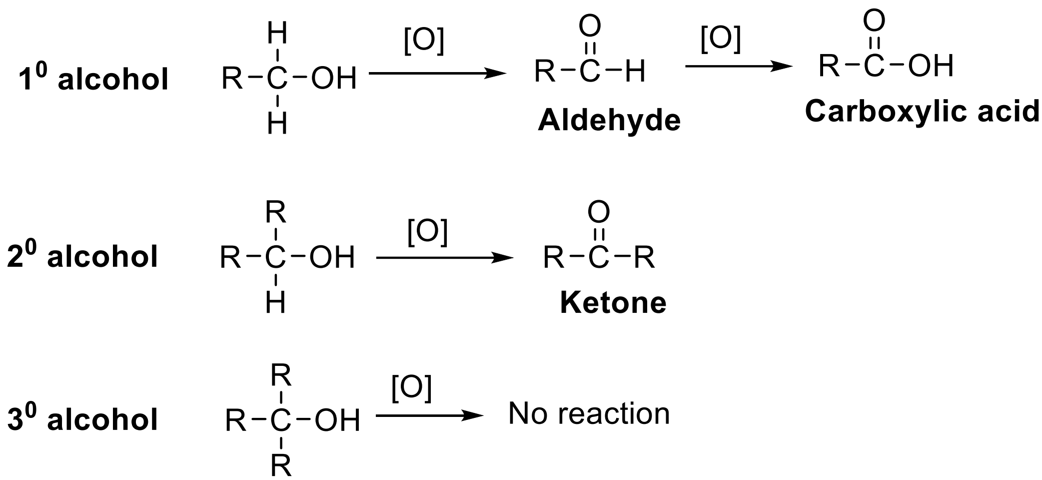
- Take 1 mL of conc. HCl in three test tubes.
- Add 10 drops of *t*-butanol in test tube 1.
- Add 10 drops of isopropanol in test tube 2.
- Add 10 drops of ethanol in test tube 3.
- Shake.

Chemical equation:



- ## Oxidation of Alcohols

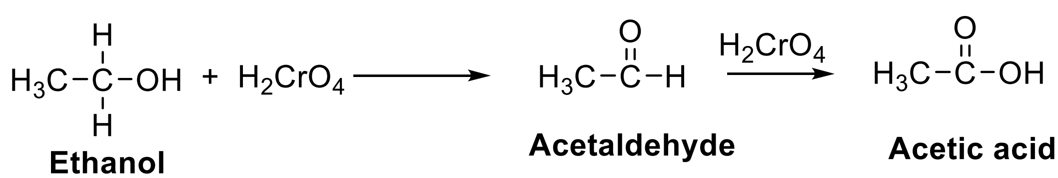
Reaction with an oxidizing agent e.g. $KMnO_4$, H_2CrO_4, is an important synthesis of aldehydes, ketones and carboxylic acids.



Test-tube 2

- Take 1 mL of chromic acid (H_2CrO_4).
- Add 5 drops of ethanol (primary alcohol).
- Place the tube in hot water bath.

Chemical equation:

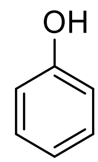


Test-tube 3

- Take 1 mL of chromic acid (H_2CrO_4).
- Add 5 drops of *t*-butanol (tertiary alcohol).
- Place the tube in hot water bath.

- **Phenols**

Phenols are compounds that possess a hydroxyl group directly attached to an aromatic ring.



Phenol

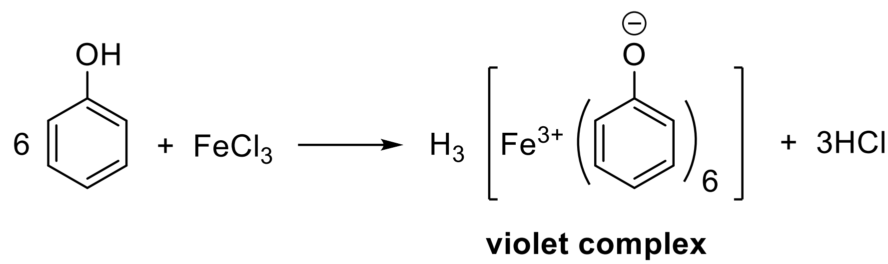
- **Ferric chloride ($FeCl_3$)**

phenol group will form a blue, violet, purple, green, or red-brown color upon addition of aqueous ferric chloride ($FeCl_3$). This reaction **can be used as a test for phenol group.**

Test-tube 4

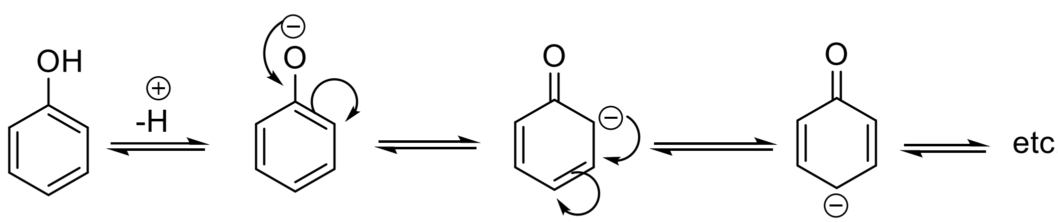
- Take 3 drops of the aqueous phenol solution.
- Add 2 drops of $FeCl_3$ solution.

Chemical equation:



- **Acidity of Phenols**

Resonance stabilization of the charge in the anion is responsible for phenols being weak acids.



Test-tube 5

- Take 1 mL of aqueous phenol solution.
- Add 1 drop of bromothymol blue.
  (**pH color** change of bromothymol blue; (yellow at pH 6.2) to (blue at pH 7.6).

Observation

The color of the solution …………………..…………………………………………….

The solution has a pH of …………………..……………………………….………….

Test-tube 6

- Take 1 mL of aqueous phenol solution.
- Add 1 drop of bromophenol blue.
  (**pH color** change of bromophenol blue; (yellow; pH 3.0 to blue; pH 4.6).

Observation

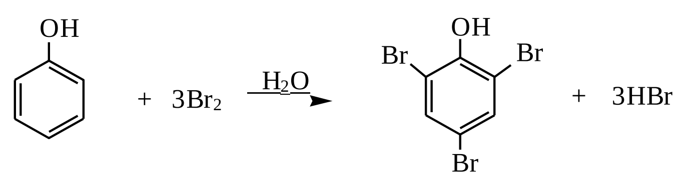
The color of the solution ………………….……….………………………….

The solution has a pH of …………………………..…………………………...

Conclusion

The phenol solution therefore has a pH somewhere between ………. and ………

- **Electrophilic Substitution in Phenol**

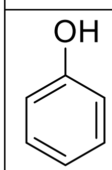
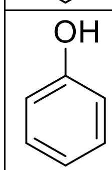


2,4,6-Tribromophenol
(white ppt)

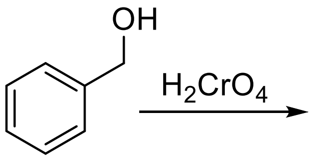
Test-tube 7

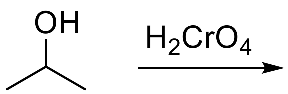
- Take 3 drops of the aqueous phenol solution.
- Add 2 mL of bromine water.

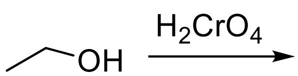
Report number 4

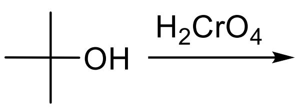
Test	Observation	Result	Chemical equation
1^{o} alcohol + Conc.HCl			
2^{o} alcohol + Conc.HCl			
3^{o} alcohol + Conc.HCl			
1^{o} alcohol + H_2CrO_4			
3^{o} alcohol + H_2CrO_4			
 + $FeCl_3$			
 + Br_2 / H_2O			

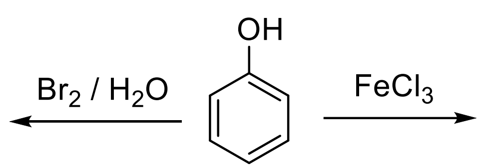
Questions

1) 

2) 

3) 

4) 



EXPERIMENT 6 Date:

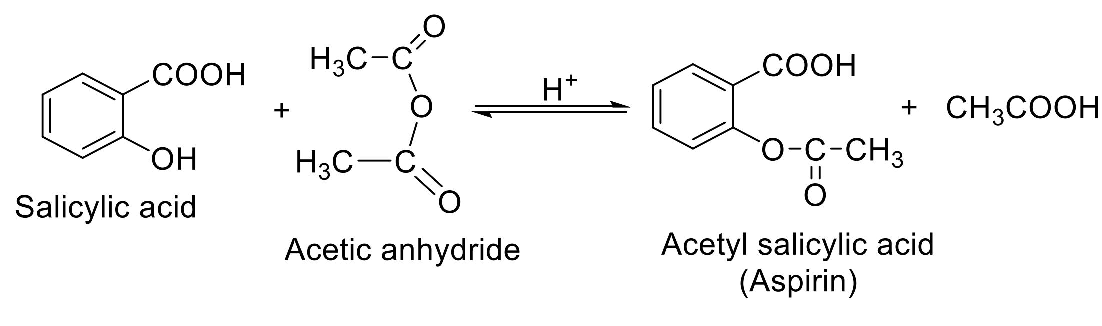
Preparation of Aspirin

Salicylic acid and many of its derivatives are antipyretics and analgesics but the acetyl derivative is not having any bad side-effects. Willow bark tea, which contains salicylic acid, was known to Hippocrates as a valuable analgesic ("Salicylic acid" is derived from the latin salix, willow).

Acetylsalicylic acid was first prepared in 1854 by a German called Gerhardt, but it was not until forty years later that its medicinal value was recognized when Hoffmann, a German chemist, tried it on his father and found that it eased the old man's rheumatic pains.

Aspirin is therefore one of the earliest examples of the alternation in chemotherapeutic properties that can result from a seemingly minor alteration in molecular structure. Of course, aspirin itself can cause undesirable side effects in some people (gastric irritation and skin rashes) but these effects are not nearly (headache, dizziness, mental confusion, nausea, etc.).

According to one textbook "over 27 million pounds of aspirin are consumed yearly in the United States (sufficient to treat over 17 billion headaches)". Thus though aspirin is widely used its mechanisms of action are still unknown. Both the antipyretic and the analgesic effects for many years thought to be central; that is, its drug somehow affects the temperature control center (the hypothalamus) and the pain center (subcortical regions, probably the thalamus) of the brain. More recently a new hypothesis of action has been advanced. The drug may act locally to reduce fever, pain and inflammation by inhibiting cellular release of a chemical which mediates these defense reaction, in particular the secondary defense mechanism (its substances including histamine, kinins and prostoglandines).



Procedure

- Weigh out 5 g of salicylic acid.
- Place the salicylic acid in a 125 mL conical flask.
- Add 10 mL of acetic anhydride in the hood.
- Add 1-2 mL of conc. H_2SO_4.
- Stopper flask with plug of cotton wool.
- Swirl the reaction mixture for 10 minutes.
- After 10 minutes, add 25 mL of ice water and swirl for 10 minutes.
- Collect the precipitate in a small Buchner funnel.
- Wash it with 10 mL of ice water.
- Dry the product in the air.
- Weigh and determine the melting point.

- **Ferric Chloride ($FeCl_3$)**

Ferric chloride forms complexes with enols and phenol. These complexes are usually dark in color, dark purple and dark green being common. These colored complexes form the basis of a test for phenols.

Procedure

- Place 2 mL of ferric chloride solution in the test-tube (Ferric chloride solution has a pale yellow color).
- Add to it a few crystals of salicylic acid.

Repeat the test but instead of using salicylic acid, add a few crystals of your product.

Observation

……………………………………………………………………………………

Weight of Aspirin obtained = ……….. g.

Meting point of Aspirin =..............°C.

Questions

Q1) What is the structure of Aspirin?

Q2) Write the chemical equation for preparation of Aspirin?

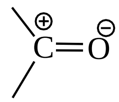
Q3) Why does $FeCl_3$ react with Salicylic acid but does not react with aspirin?

EXPERIMENT 7 Date:

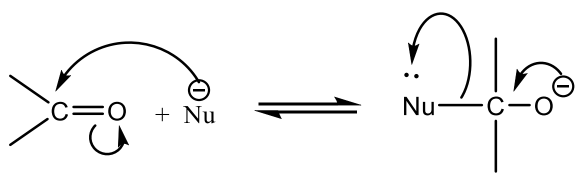
Aldehydes and Ketones

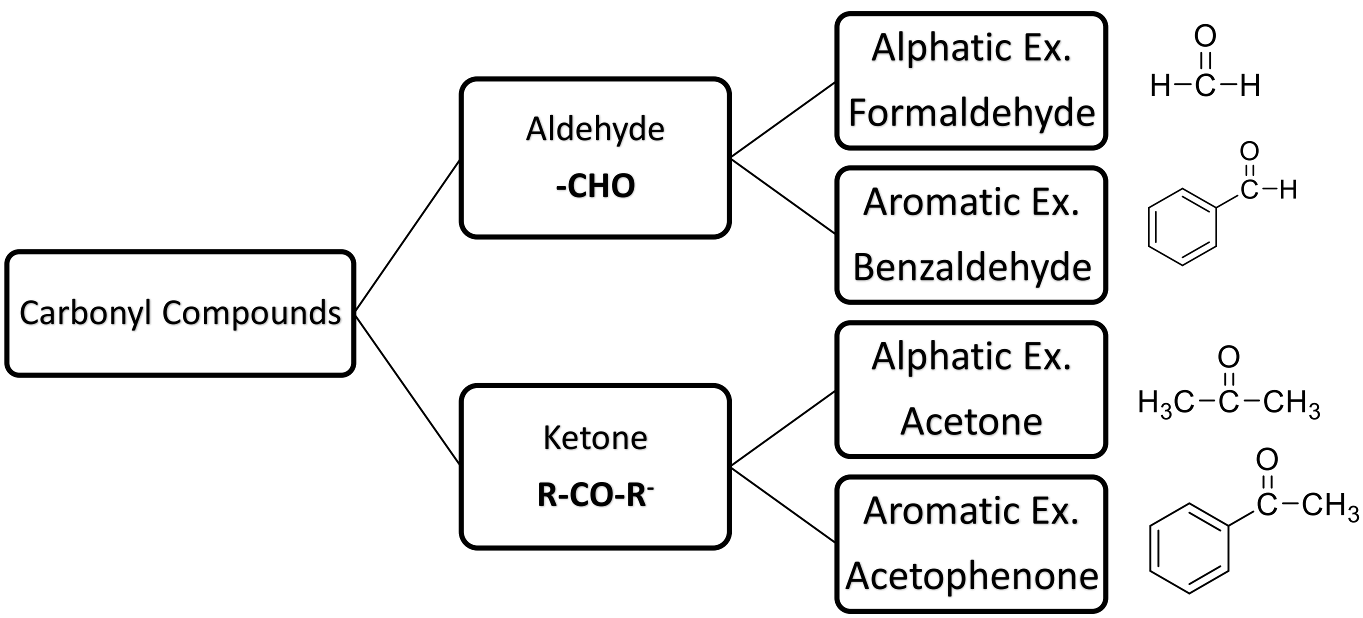
Aldehyde Ketone

Aldehydes and ketones are characterized by carbonyl group-polarized double bond.



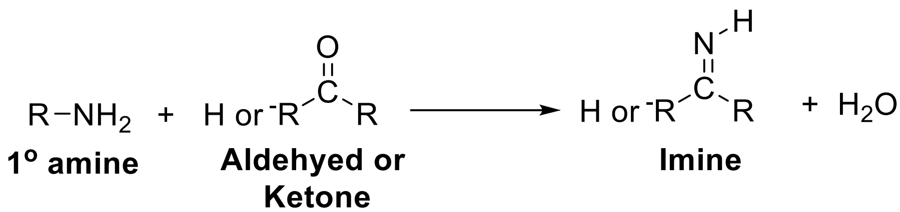
Attack by nucleophiles therefore occurs at the C^+ end.





- ## Carbonyl Compounds with Amines

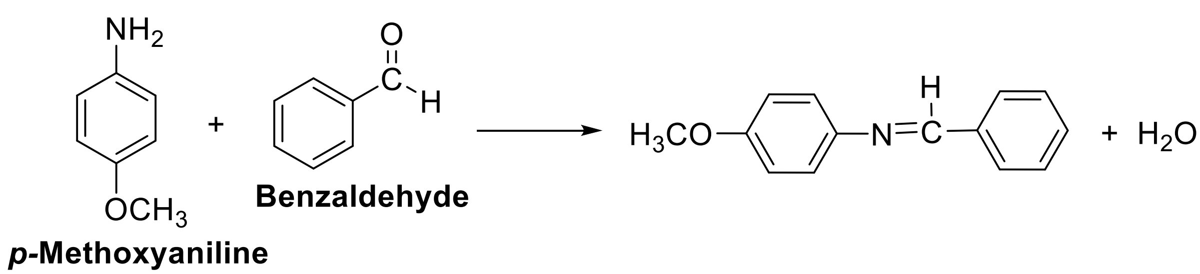
Aldehydes and ketones could react with ammonia or 1º-amines to form imine derivatives, also known as Schiff bases (compounds having a C=N function).



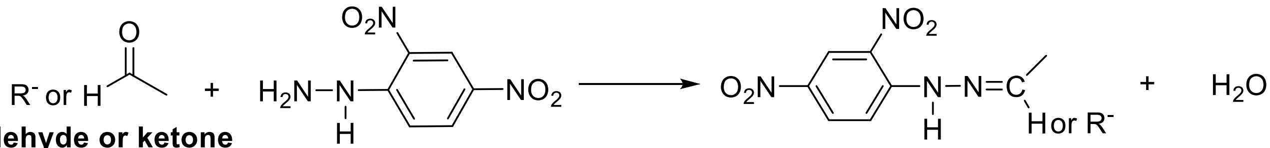
Test-tube 1

- Take 0.5 mL of ethanol.
- Add 5 drops of benzaldehyde.
- Add 1 mL of *p*-methoxyaniline (*p*-Anisidine) in ethanol-water.
- Shake.
- Place in a hot water bath.
- Cool the tube and add 1 drop of water.

Chemical equation:



- **2,4-Dinitrophenylhydrazone Formation**



Aldehyde or ketone

2,4-Dinitrophenylhydrazine (2,4D.N.P.)

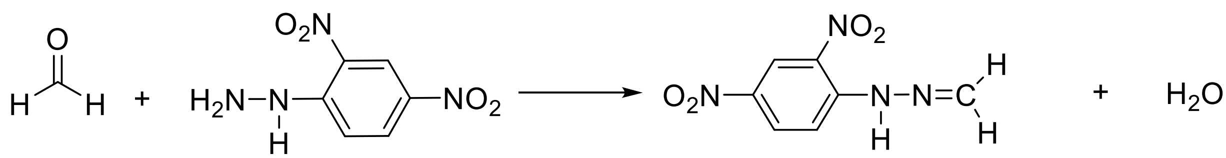
2,4-Dinitrophenylhydrazone of aldehyde or ketone

The reaction is used to identify aldehydes and ketones.

<u>Test-tube 2</u>

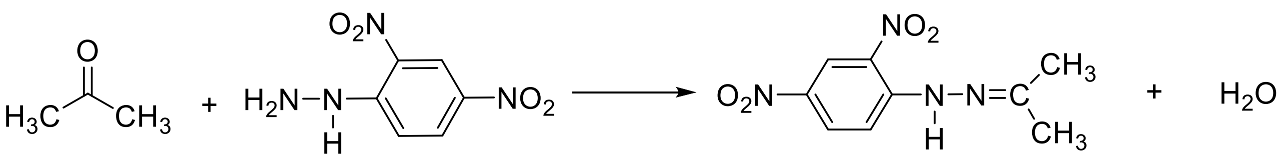
- Take 1 mL of 2,4-dinitrophenylhydrazone (2,4D.N.P.) reagent.
- Add 1 drop of acetaldehyde or acetone.

Chemical equation:



Acetaldehyde

2,4-Dinitrophenylhydrazine (2,4D.N.P.)

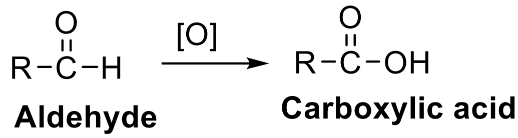


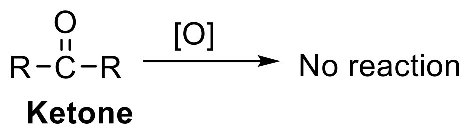
Acetone

2,4-Dinitrophenylhydrazine (2,4D.N.P.)

- ## Oxidation of Carbonyl group

Aldehydes could oxidize to form carboxylic acid while ketone does not oxidize.

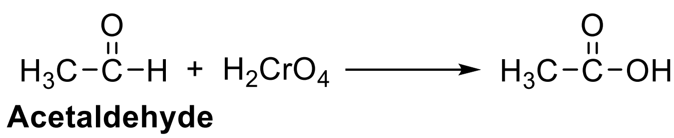


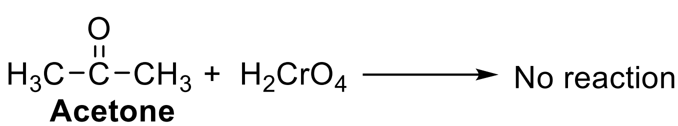


<u>Test-tube 4</u>

- **Take two test tubes** add 5 drops of acetaldehyde and the other add 5 drops acetone.
- Add 10 drops of chromic acid for the two test tubes (take care).
- Place the tubes in the hot water bath.

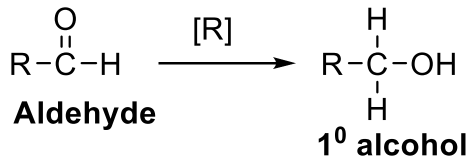
Chemical equation:



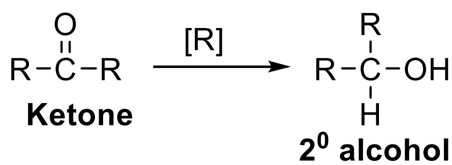


- **Reduction of Carbonyl group**

Addition of a hydride anion ($H:^-$) to an aldehyde or ketone gives an alkoxide anion, which on protonation yields the corresponding alcohol. Aldehydes produce 1º-alcohols and ketones produce 2º-alcohols.



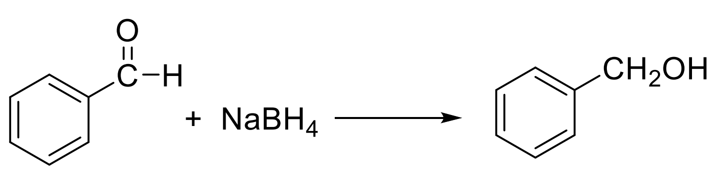
Reducing reagent: $NaBH_4$



Test-tube 3

- Take 1 mL of ethanol.
- Add 5 drops of benzaldehyde.
- Add 2 mL of $NaBH_4$ solution.
- Allow to stand.
- Add a few pieces of ice to a small beaker.
- Pour into the reaction mixture.

Chemical equation:



- **Distinguishing Test between Aldehyde and Ketone**

Tollen's reagent (a weak oxidizing agent) – ammonical silver nitrate with NH_4^+ as the oxidant.

Test-tube 5

- Take 2 mL of $AgNO_3$ solution.
- Add 8 drops of 5% NaOH solution.
- Add ammonium hydroxide dropwise and with mixing until the black precipitate just dissolves. (This is Tollen's reagent)
- Divide the solution into 2-test-tubes.

Reaction E (i)

- Add 1 drop acetaldehyde of to the sample of Tollen's reagent in the test-tube.
- Place the tube in the hot water bath.

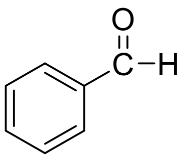
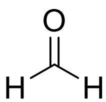
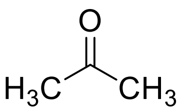
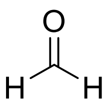
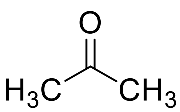
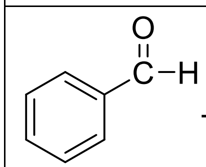
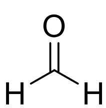
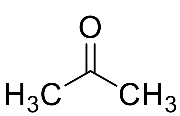
Chemical equation:

$$H_3C-\overset{\overset{\displaystyle O}{\|}}{C}-H + 2\left[Ag\,(NH_3)_2\right]^{\oplus}\overset{\ominus}{O}H + H_2O \longrightarrow H_3C-\overset{\overset{\displaystyle O}{\|}}{C}-OH + \downarrow 2Ag + 4NH_3 + 2H_2O$$

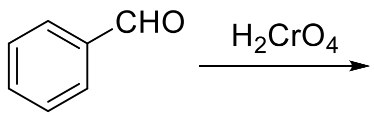
Reaction E (ii)

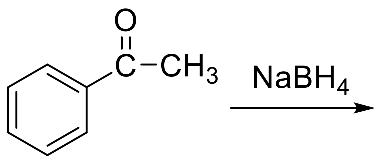
- Add 1 drop of acetone to the sample of Tollen's reagent in the test-tube.
- Place the tube in the hot water bath.

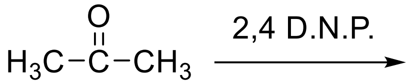
Report number 5

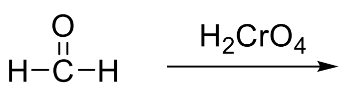
Test	Observation	Result	Chemical equation
 + p-methoxyaniline (p-Anisidine)			
 or  + 2,4 D.N.P.			
 + H_2CrO_4			
 + H_2CrO_4			
 + $NaBH_4$			
 + Tollen's reagent			
 + Tollen's reagent			

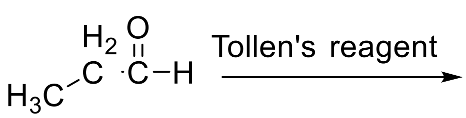
Questions

1) 

2) 

3) 

4) 

5) 

EXPERIMENT 8 Date:

Carbohydrates

Carbohydrates are compounds containing C, H, O and the H and O are present in the same proportion as in water. The word carbohydrate can be expressed as hydrates of carbon because molecular formulas of these compounds.

Example; Glucose has the molecular formula $C_6H_{12}O_6$, which might be written as $C_6(H_2O)_6$.

The chemistry of carbohydrates is mainly the combined chemistry of two functional groups: the hydroxyl group and the carbonyl group.

Carbohydrates are usually classified according to their structure as;

1) Monosaccharides: (glucose, galactose and fructose).
2) Disaccharides: (sucrose, maltose and lactose).
3) Polysaccharides: (starch and inulin).

The three classes of carbohydrates are related to each other through hydrolysis.

$$\text{polysaccharide} \xrightarrow[H^+]{H_2O} \text{oligosaccharides} \xrightarrow[H^+]{H_2O} \text{monosaccharides}$$

$$\underset{\substack{\text{starch} \\ \text{(a polysaccharide)}}}{[C_{12}H_{20}O_{10}]_n} \xrightarrow[H^+]{n\,H_2O} \underset{\substack{\text{maltose} \\ \text{(a disaccharide)}}}{n\,C_{12}H_{22}O_{11}} \xrightarrow[H^+]{n\,H_2O} \underset{\substack{\text{glucose} \\ \text{(a monosaccharide)}}}{2n\,C_6H_{12}O_6}$$

Monosaccharides (or simple sugars, as they are sometimes called) are carbohydrates that cannot be hydrolyzed to simpler compounds.

Polysaccharides contain many monosaccharide units—sometimes hundreds or even thousands. Usually, but not always, the units are identical. Example; starch and cellulose, contain linked units of the same monosaccharide, glucose.

Oligosaccharides (from the Greek oligos, few) contain at least two and generally no more than a few linked monosaccharide units. They may be called disaccharides, trisaccharides, and so on, depending on the number of units, which may be the same or different. Example; Maltose is a disaccharide made of two glucose units. Sucrose is made of two different monosaccharide units: glucose and fructose.

- **Physical properties**

Colorless solids, which decomposes on heating and therefore have no definite melting points. All are soluble in cold water except starch.

- **General Reactions:**

❖ Molisch's Test

Test-tube 1

- Take concentrated solution of the carbohydrate in water.
- Add equal amount of α-naphthol solution.
- Carefully add excess amount of concentrated H_2SO_4 inside the wall of the tube to form a heavy layer at the bottom.

Observation

A deep violet ring is produced at the interface.

Shake the solution.

Observation

The violet color spreads through the whole solution.

❖ Reduction of Fehling's Solution (A+B)

Test-tube 2

- Take 1 mL of sugar solution.
- Add 1 mL of Fehling's solution (A+B).
- Boil the soln. for one minute.

Observation

- Glucose, Fructose, Maltose (Malt sugar), and Lactose (Milk sugar); Reduction takes place and a red ppt. of cuprous oxide is formed.
- Starch and Sucrose give –ve result.

❖ Barfoed′s Test: (Copper Acetate in Acetic Acid Solution)

Test-tube 3

- Take 1 mL of sugar solution.
- Add 1 mL of Barfoed's reagent.
- Boil the soln. for one minute.

Observation

- Glucose and Fructose give red ppt. of cuprous oxide appears.
- Maltose gives red ppt. only after prolonged heating (more than 15min) as it is hydrolyzed to glucose.
- Lactose (Milk sugar) gives red ppt. only after prolonged heating (more than 15 min) as it is hydrolyzed to glucose and galactose.
- Starch and Sucrose give –ve result.

❖ Rapid Furfural Test

Test-tube 4

- Take 1 mL of dil. sugar solution.
- Add 1 mL of α-naphthol solution.
- Add 3 mL of conc. HCl.
- Boil in a water bath.

Observation

- Glucose, Maltose, and Lactose (Milk sugar); a violet color appears after some time.
- Fructose and Sucrose (Can sugar); an immediate violet coloration on boiling.

❖ Iodine Test(I_2 Solution)

Test-tube 5

- In two test tubes take 1 mL of dil. Sugar (Sucrose and Starch) solution.
- Add 1 mL of I_2 solution.

Observation

- Starch gives blue-black color
- Sucrose –ve

❖ Tollen's reagent (Ammoniacal Silver Nitrate)

Test-tube 6

- Take 1 mL of sugar solution.
- Add 1 mL of dil. ammoniacal silver nitrate (Tollen's reagent).
- Boil the soln. for one minute.
- Place the test tube on a water bath.

Observation

- Glucose, Fructose, Maltose (Malt sugar) and Lactose (Milk sugar); a silver mirror is produced in 1-2 minutes.

Starch and Sucrose (Can sugar) give –ve result.

❖ Osazone formation

Test-tube 7

- Take solid sugar, phenyhydrazine hydrochloride, and sodium acetate in the ratio of 1:2:3.
- Add 5 drops of water.
- Place in a hot water bath for 10-15 minutes.

Observation

- Glucose and Fructose; yellow ppt. of the osazone appears after 10-15 minutes. Examine under the microscope, yellow needles aggregated in the form of sheaves.
- Maltose (Malt sugar); Forms yellow ppt. of osazone soluble in hot water, the yellow crystals of the osazone will appears only after cooling the soln. Examine the crystals under the microscope.
- Lactose (Milk sugar); Forms yellow ppt. of osazone soluble in hot water, the yellow crystals of the osazone will appears only after cooling the soln. Examine the crystals under the microscope.
- Starch and Sucrose (Can sugar); –ve result.

Glucosazone or Fructosazon

Maltosazone

Lactosazone

❖ Water Solubility Test

<u>Test-tube 12</u>

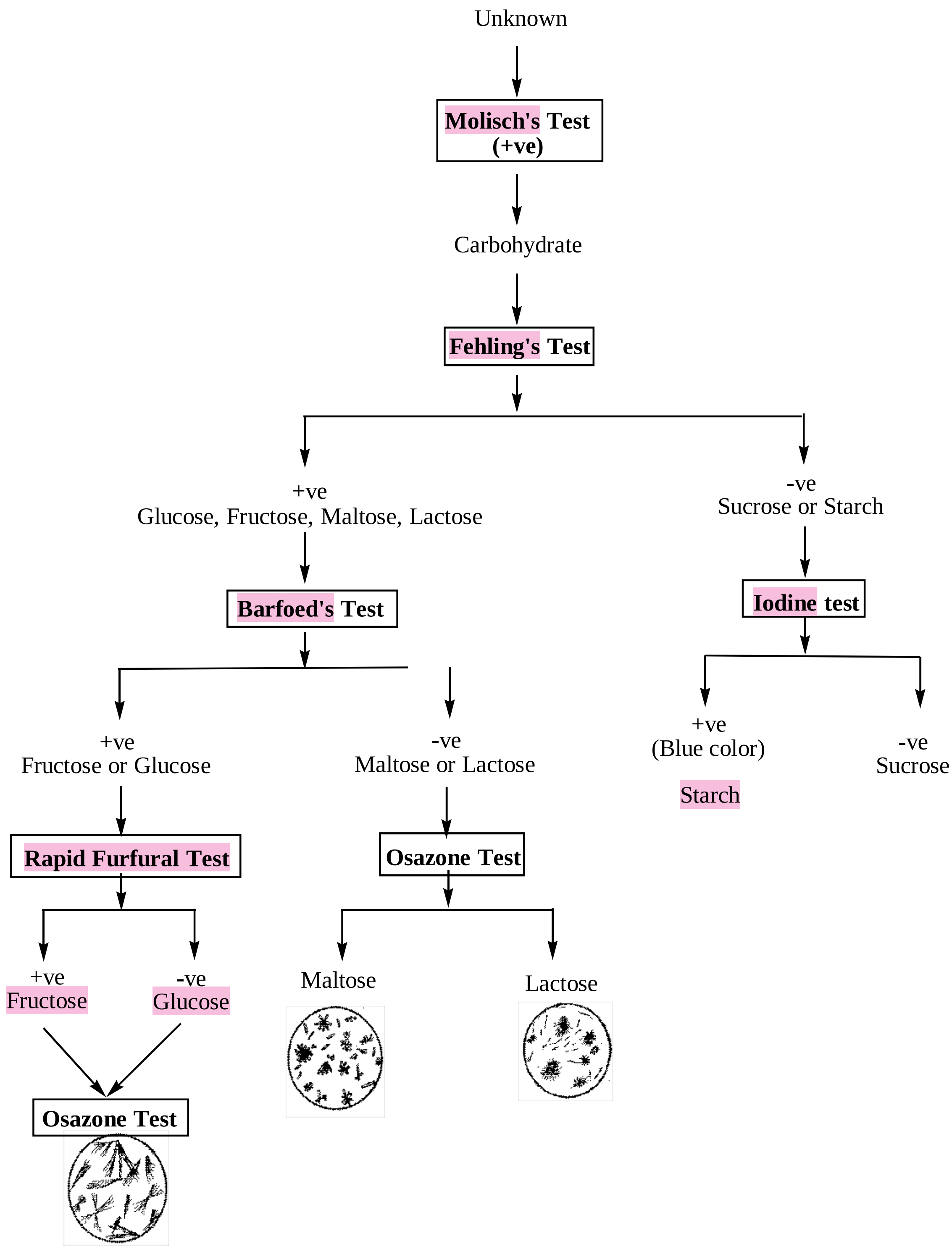
- Take solid sugar.
- Add water.
- Shake.

<u>Observation</u>

- Mono-and disaccharides are soluble in water.
- Starch insoluble in water.

General Scheme for Identification of Carbohydrates

Starch, Sucrose, Glucose, Fructose, Maltose, Lactose



Report number 6 **Sugar name:**

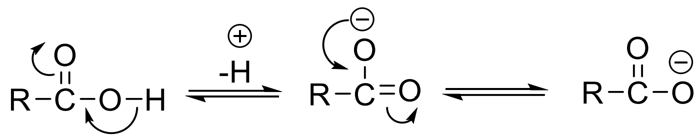
Test	Observation	Result	Chemical equation
Molisch's Test			
Fehling's Solution (A+B)			
Barfoed's Test			
Rapid Furfural Test			
Iodine test			
Tollen's reagent			
H_2O			

EXPERIMENT 9 Date:

Carboxylic acids and Their Derivatives

- ## Reaction of Carboxylic acid with Sodium bicarbonate

Carboxylic acids are stronger acids than phenols.



Test-tube 1

- Take 1 mL of 4% sodium bicarbonate solution ($NaHCO_3$).
- Add one drop of 90% acetic acid solution.

Note the CO_2 evolved (effervescence).

Chemical equation:

$$H_3C-\overset{O}{\overset{||}{C}}-OH + NaHCO_3 \longrightarrow H_3C-\overset{O}{\overset{||}{C}}-\overset{\ominus\oplus}{ONa} + \uparrow CO_2 + H_2O$$

- ## Relative Acidities of Some Substituted Benzoic Acid

a) Electron withdrawing substituents in Ar will reduce the charge on the carboxylateanions.
b) The anion will therefore be stabilized.
c) The acid will therefore show a greater tendency to dissociate.
d) The acid will therefore be stronger if Ar is electron withdrawing than if Ar iselectron donating.

Test-tube 2, 3, 4

- Place 1 mL of *p*-nitrobenzoic acid in test-tube 2.
- Place 1 mL of benzoic acid in test-tube 3.
- Place 1 mL of *p*-methoxybenzoic acid in test-tube 4.
- Add 1 drop of bromophenol blue solution to each tube.
  (**pH color change** of bromophenol blue; yellow (pH 3.0), blue (pH 4.6).
- Add 1 drop of 0.1M NaOH to each tube.

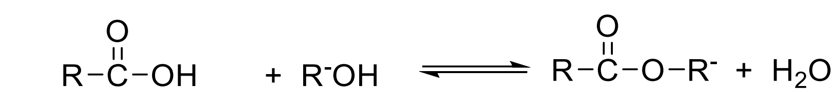
Observation

1) Color of p-nitrobenzoic acid …………………………………………
2) Color of benzoic acid ………………………………………………….
3) Color of p-methoxybenzoic acid ……………………………………

Order of acidity:

……………………………………………………………………………

- **Esterification**



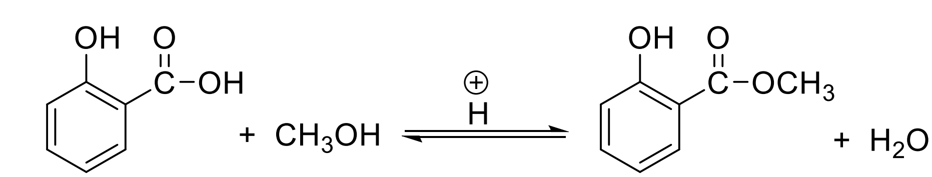
Carboxylic acid **Alcohol** **Ester**

Test-tube 5

- Take 2 mL of the solution of sulfuric acid in methanol.
- Add 5 drops of the solution of salicylic acid in methanol.
- Place the tube in hot water bath.Take about 15 mL of cold water in a small beaker.
- Add the contents of the tube to the beaker.

The ester, methyl salicylate (oil of wintergreen) has a distinctive smell.

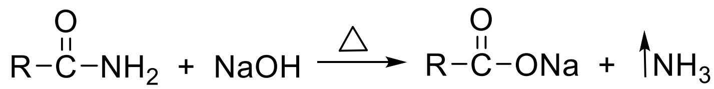
Chemical equation:



Salicylic acid **Methanol** **Methyl salicylate**

- **Amide Hydrolysis**

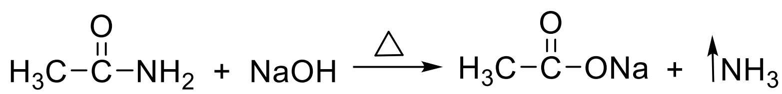
  Susceptible to basic (and acidic) hydrolysis but are less reactive than esters.



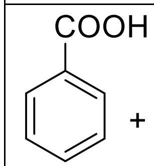
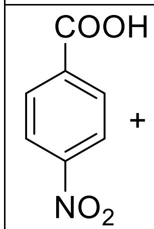
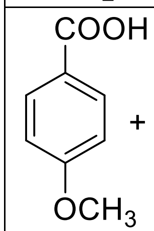
Test-tube 6

- Take 1 mL of the ethanol solution of acetamide.
- Add 1 mL of 10% NaOH solution.
- Then take red litmus and keep it on the top of the tube.
- Place the tube in the hot water bath.

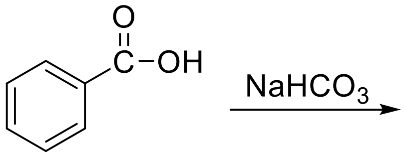
Chemical equation:

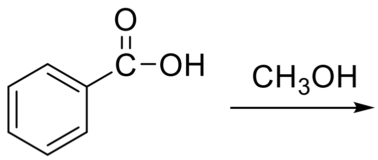


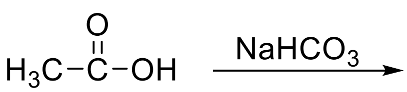
Report number 7

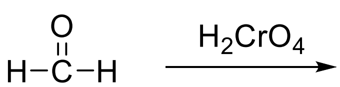
Test	Observation	Result	Chemical equation
Acetic acid + $NaHCO_3$			
 + Bromophenol blue			
 + Bromophenol blue			
 + Bromophenol blue			
Esterification			
$H_3C-C(=O)-NH_2$ + 10% NaOH with red litmus paper			

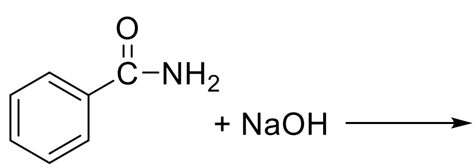
Questions

1) 

2) 

3) 

4) 

5) 

EXPERIMENT 10 Date:

Amino Compounds

Basicity of Amines

Test-tube 1

- Take 1 mL of aqueous aniline.
- Add 2 drops of phenolphthalein.
  (pH color change of phenolphthalein; colorless pH 8.0 to pink pH 10.0).

Observation

Aniline: Color …………………. ∴ pH is ……………….…

- Add 2 drops of *p*-nitrophenol solution to the aniline tube.
  (**pH color change** of *p*-nitrophenol; colorless pH 5.0 to yellow pH 7.0.)

Observation

Aniline: Color …………………. ∴ pH is …………………

Test-tube 2

- Take 1 mL of aqueous methylamine.
- Add 2 drops of phenolphthalein.
  (pH color change of phenolphthalein; colorless pH 8.0 to pink pH 10.0).

Observation

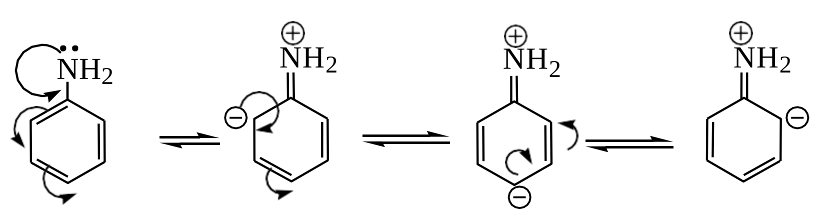
Methylamine: Color ………………. ∴ pH is ……………

The pH of the aniline solution is between …………… and ………………..…

Aniline is **(less - more)** basic than methylamine.

The lone pair of electrons on the nitrogen of aniline is delocalized over the ring.

etc.



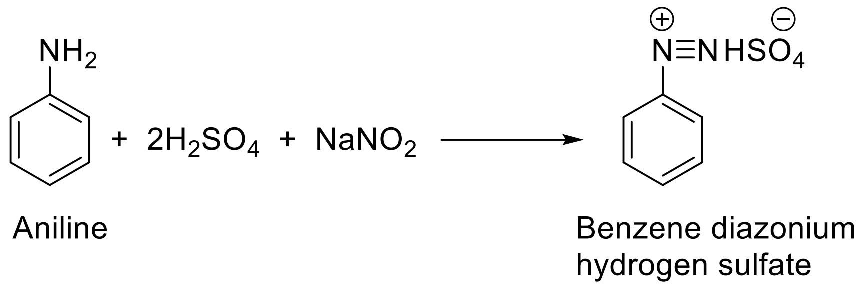
Protonation of this nitrogen is therefore less favorable than in the case of methylamine (no resonance possible).

- **Diazotization (Reaction with Nitrous Acid)**

Test-tube 3

- Take 1 mL of the solution of aniline in 10% H_2SO_4.
- Slowly add 5 drops of $NaNO_2$ solution.
- Shake the tube after adding each drop.
- Set the tube aside.

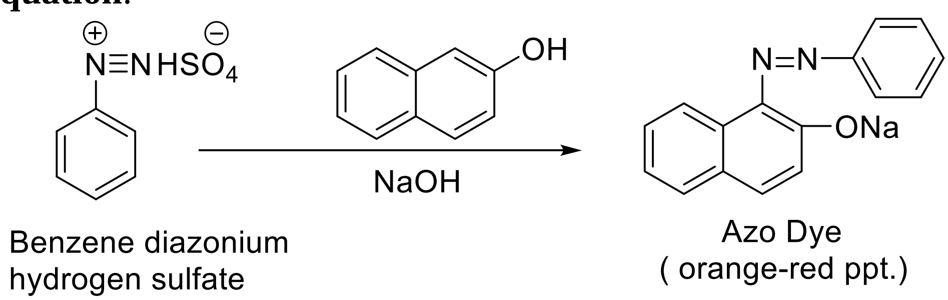
Chemical equation:



Test-tube 4

- Take 1 mL of 2-naphthol solution in sodium hydroxide.
- Pour a little (less than half) of the diazonium salt solution in Test-tube 4 into the 2-naphthol solution.

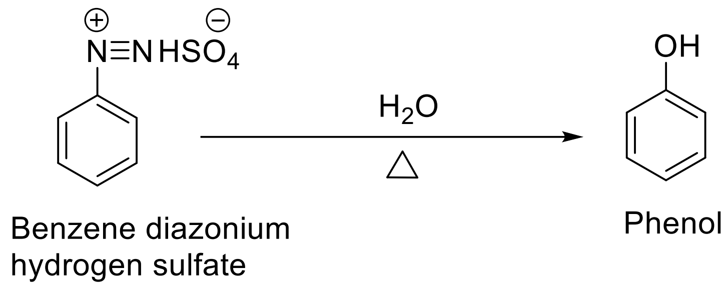
Chemical equation:



Test-tube 5

- Take 10 mL of hot water.
- Add rest of the diazonium salt solution from Test-tube 4.
- Heat and swirl.

Chemical equation:



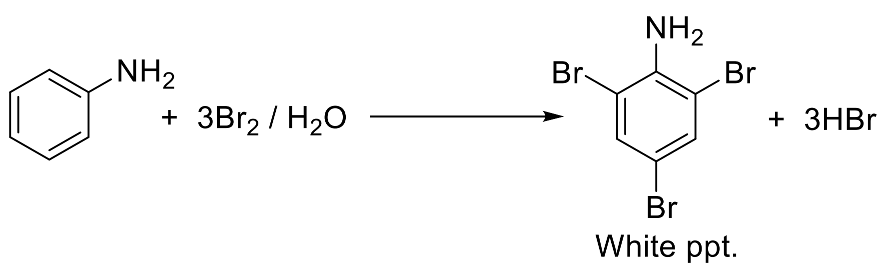
- **Bromination of Aniline**

Aniline is highly reactive to electrophilic substitution reactions (-NH_2 is a strong electron-donor).

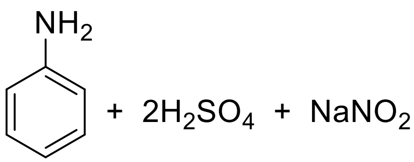
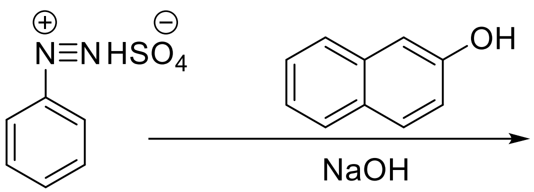
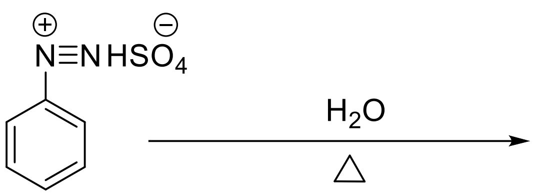
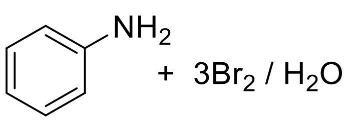
Test-tube 6

- Take 3 drops of aqueous aniline solution.
- Add 1 mL of bromine water.

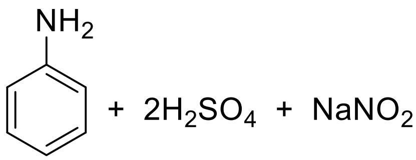
Chemical equation:

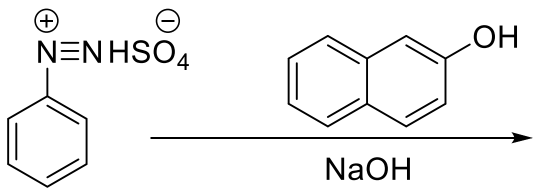


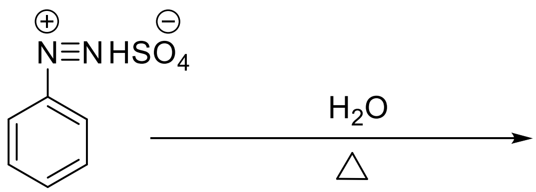
Report number 8

Test	Observation	Result	Chemical equation
			
			
			
			

Questions

1) 

2) 

3) 

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