جامعة الملك سعود
King Saud University
CHEMISTRY DEPARTMENT

ORGANIC PREPARATIONS

(CHEM-345)

Student Name	
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2024 G

Organic Preparations – CHEM 345	
Week and Date	Experiments
Week 1 18/08/2024	Instruction, equipment and safety
Week 2 25/08/2024	Explain the method of extractions, recrystallization and distillation Summary of functional group and practical detection
Week 3 01/09/2024	Preparation of Salicylic acid
Week 4 08/09/2024	QUIZ 1 2 - Preparation of Aspirin
Week 5 15/09/2024	3 - Preparation of Cyclohexanone-semi carbazone
Week 6 22/09/2024	National Saudi Day
Week 7 29/09/2024	4 - Preparation of Dimethyl Maleate
Week 8 06/10/2024	QUIZ 2 5 - Preparation of 2,3-Dibromo-3-phenyl propanoic acid
Week 9 13/10/2024	6 - Preparation of 9,10-Dihydroanthracene-endo α,β-Succinic anhydride (Diels-Alder Reaction)
Week 10 20/10/2024	7 - Preparation of tert-Butyl chloride from tert-Butyl alcohol
Week 11 27/10/2024	QUIZ 3 8 - Preparation of Acetanilide (Acetylation of Aniline)
Week 12 03/11/2024	9 - Preparation of Paracetamol
Week 13 10/11/2024	Fall Break
Week 14 17/11/2024	QUIZ 4 10 - Preparation of α-Glucose Pentaacetate
Week 15 24/11/2024	Final Practical and Theoretical Examinations

PLEASE READ THE FOLLOWING BEFORE YOUR FIRST DAY IN THE LAB

Course Description

Content:

Practical application of some common chemical reactions used in the preparation of organic compounds and methods of purification - Application of the foundations and rules of chemical calculations to calculate the theoretical outcome and percentage of chemical reactions - The use of physical and chemical methods and spectroscopy in the identification of prepared organic compounds - Preparation of some organic compounds such as Aspirin, Paracetamol, Salicylic acid ,……..

Course Objectives and Outcomes
Objectives:

1. Description of modern laboratory techniques
2. Preparation of organic compounds.
3. interpret the route of preparation of organic compounds
4. Characterization of an organic compound by interpreting spectra
5. Analyze the results and view the practical reports for each experiment.

Outputs:

At the end of this course, the student can:

- Describes the basic techniques of organic reactions.

- Learn about the steps of different organic reactions.

- Distinguish between specific reactants and perform basic calculations.

- It examines the solubility of any organic compound and suggests separation and - purification methods.

- Conducts the required experiments accurately.

- Writes reports clearly and accurately.

INTRODUCTION Date:

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.

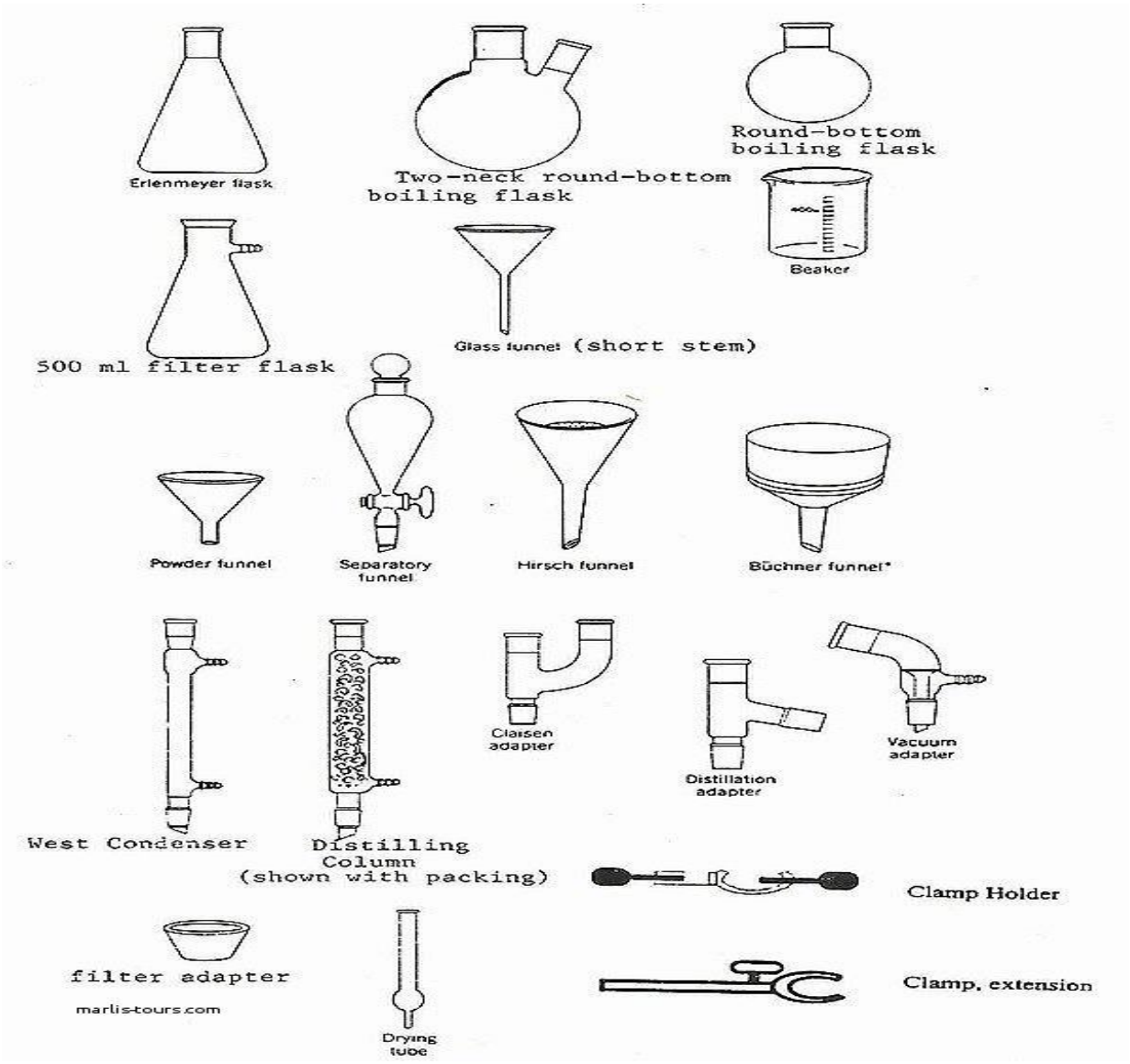
This grading will count towards your final grade and will help you to assess how well you are progressing in your practice of preparation organic chemistry. For this reason, your demonstrator will discuss the assessment with you when he grades the experiment at the end of the practical period.

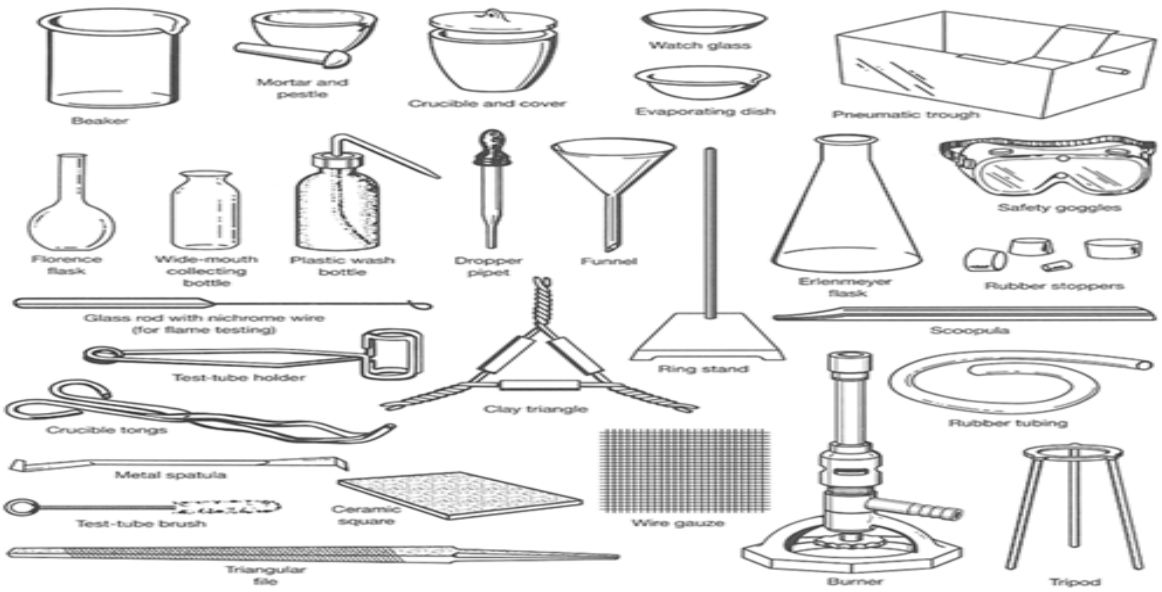
familiar with the organic chemistry involved before you carry out any reaction.

Distribution of Marks

Attendance	5 marks
Reports of preparation	٥٠mark
Drop quizzes Midterm	٥marks
Final Exam (Theoretical + Practical	40 mark
Total	100 mark

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.

safety means inside the laboratory

You should read it well

1. Wear a lab coat when you enter the lab and no student will allow me to work unless they wear a coat.
2. Glasses and gloves should be worn before starting work.
3. Clean your glass test tubes before and at the end of work.
4. It is strictly forbidden to eat and drink in the laboratory.
5. Read each test before you start working on it.
6. Write down your results in the report.
7. Before adding any particular detector, you must make sure of the name of the detector by reading the label on it.
8. Do not conduct any experiment that was not asked of you.
9. Flammable materials such as alcohol and acetone should not be heated on direct flames, but a water bath should be used.
10. A special dropper should be used for the same reagent bottle [wash with water if similar].
11. No reagent or solid salt should be returned to the original bottle from which it was taken, and if it is not used, it is preferable to dispose of it in the correct way.
12. Do not place the caps of reagent bottles on the surface of the bench so as not to be contaminated with other substances.
13. Experiments accompanied by the rise of toxic or smelly gases or vapors must be carried out in the gas cabinet.
14. If substances with acidic or alkaline properties fall or any substances come into contact with your hands, wash them several times with water.
15. When heating the tube on a direct flame, constantly move the tube with the pipe opening pointing to the opposite side away from your colleague's face.
16. It is forbidden to work during the preparation of derivatives except in the presence of the professor of the subject or the laboratory report.
17. In the event of any emergency Allah inform your classmates and the course instructor or call the university emergency in the event of a fire or explosion.
18. Shoes should be worn on the practical date due to the danger of chemicals to the body.
19. Wash hands thoroughly before leaving the laboratory.

University Emergency Number From any fixed line within the university: 950
From any other line: 0114670950
University Civil Defense Number: 950

Date:

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.

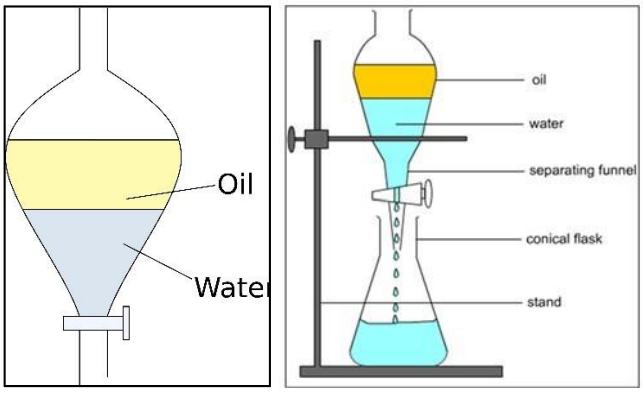


Figure 1: Diagram of separating funnel apparatus.

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.

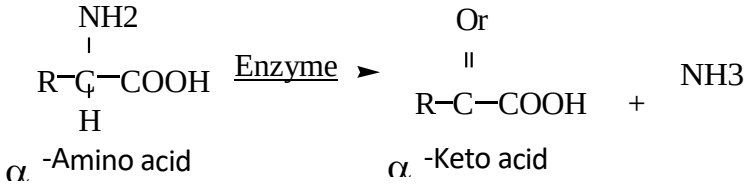
Solvent	**Formula**
Hexane (light petrol)	C6H14
Benzene	C6H6
Diethyl ether (ether)	$(C2H5)_{2O}$
Ethyl acetate	CH3COOC2H5
Chloroform	CHCl3
Carbon tetrachloride	CCl4

NOTE that methanol, ethanol, and acetone are miscible with water and cannot be used for extraction from water.

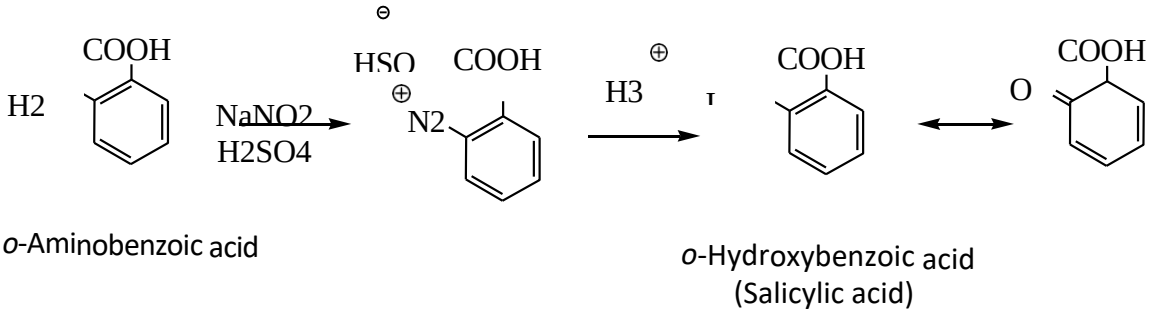
PREPERATION 1

Preparation of salicylic acid from anthranilic acid

When an animal is starved for glucose, fats are used as energy source. If starvation is prolonged the body's stocks of fat begin to run down and finally they are exhausted. The only remaining substances present in the body which form oxidizable substances are protein. Such energy as the protein can provide comes through the oxidation of its constituent amino acids. The amino acid is deaminated (that is the amino-nitrogen is lost) and the resulting molecule is converted into substances on the direct pathway of either glucose or fatty acid oxidation.

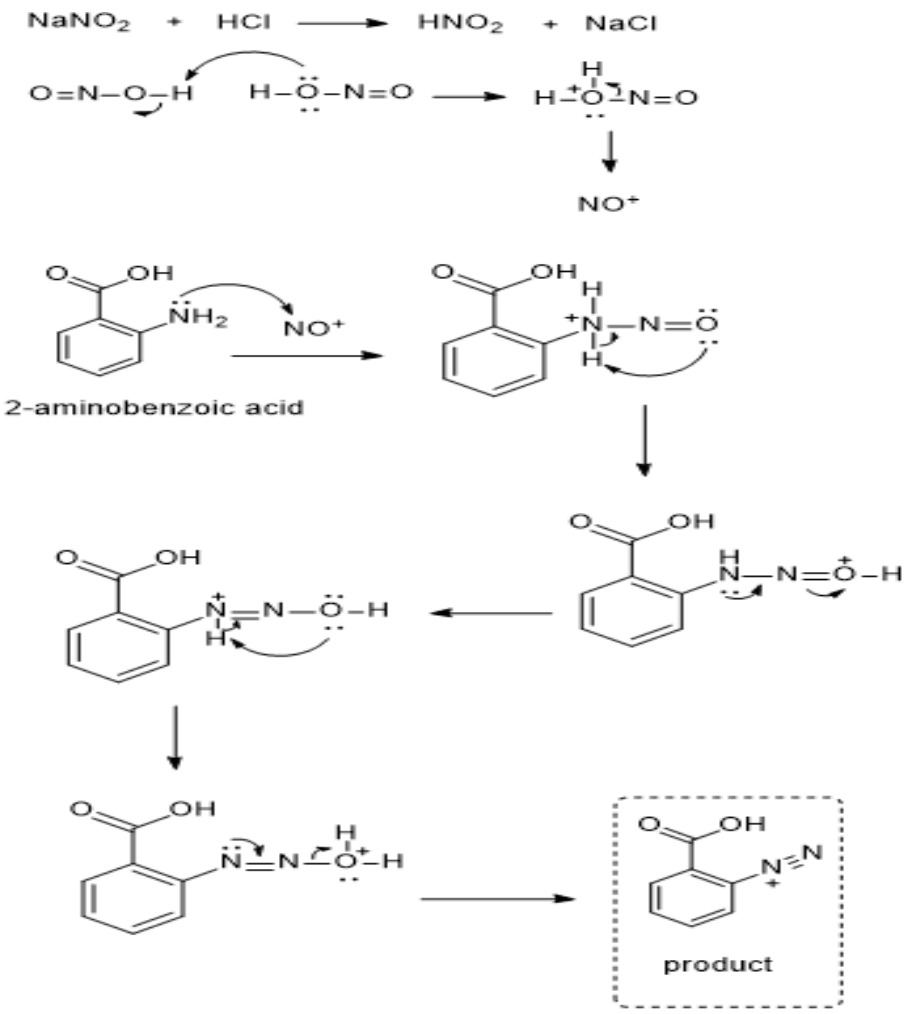


In today's experiment we will deaminating an amino acid (o-aminobenzoic acid) using nitrous acid.



Diazotization (reaction with nitrous acid) of amino group will give a diazonium salt. The diazonium salt will react with water to give a phenol. So in this experiment we are deaminating β–amino acid to give the equivalent β–keto acid in its more stable enol form.

reaction mechanism



Procedure

- In a 100 mL conical flask, warm a mixture of 2 g of *o*-aminobenzoic acid (anthranilic acid) and 20 mL of 10% sulfuric acid until the solid (the hydrogen sulfate salt of the basic amino group) just dissolves.
- Cool the reaction mixture in ice bath for a minute.
- Add 6 mL of 20% sodium nitrite solution to this mixture, with vigorous swirling. There is a vigorous, instant reaction.
- Without delay, pour this solution (now containing the diazonium salt) into a beaker containing 40 mL of 10% H2SO4 in a 500 mL beaker.
- Stir the foamy product and heat on a hotplate until boils for 15 minutes, stir continually.
- Then add 50 mL of water and stir until a uniform color appears.
- Suction filter the product and recrystallize it from hot water.
  Weight of *or*-hydroxybenzoic acid (salicylic acid) =..... g.
  m.p. of salicylic acid = ……. C^0

Questions

What is happen if you add sodium nitrate to aniline solution dissolved in sulfuric acid and why?

……

……

……

……

Preparation 2

Preparation of Aspirin

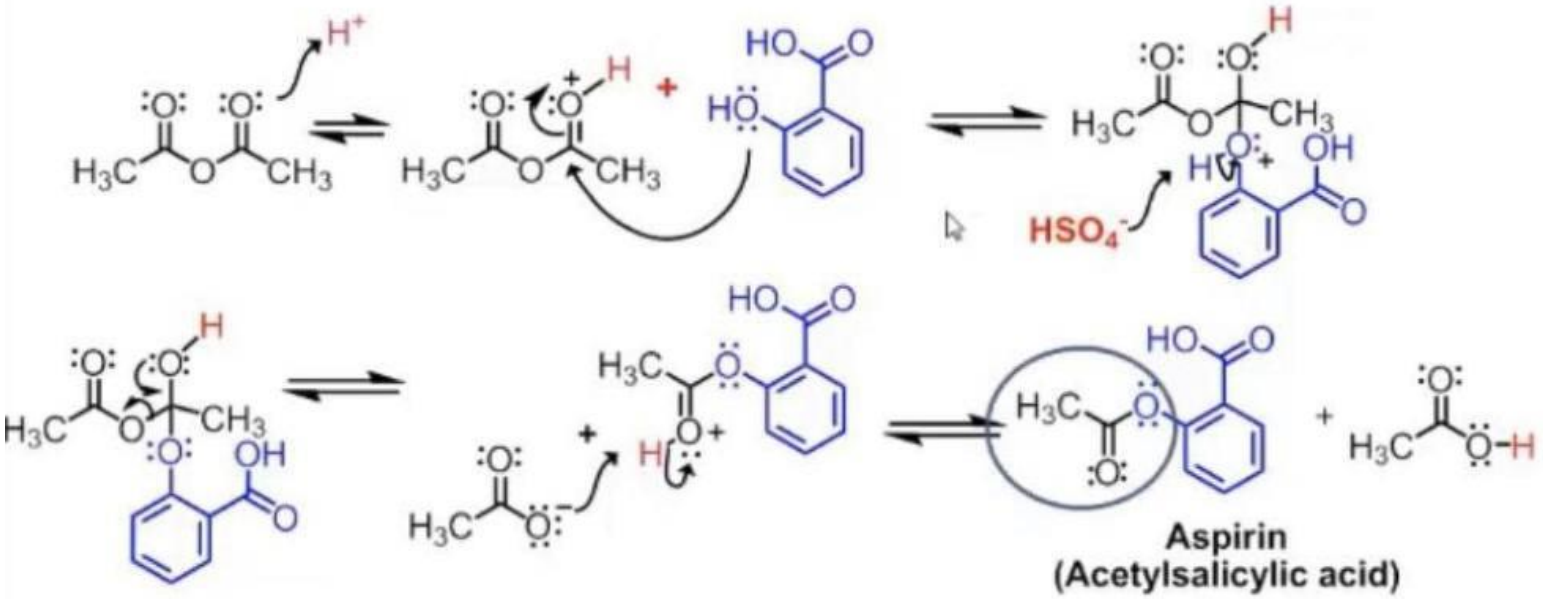
Salicylic acid and many of its derivatives are antipyretics and analgesics but the acetyl derivative is not having any bad side-effects. Willow bark tea, which contains salicylic acid, was known to Hippocrates as a valuable analgesic ("Salicylic acid" is derived from the Latin Salix, willow).

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 reactions, in particular the secondary defense mechanism (its substances including histamine, kinins and prostaglan

Aspirin Synthesis and Mechanism



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 Test for Phenol

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

……

……

Preparation No 3

Cyclohexanone semi carbazide.

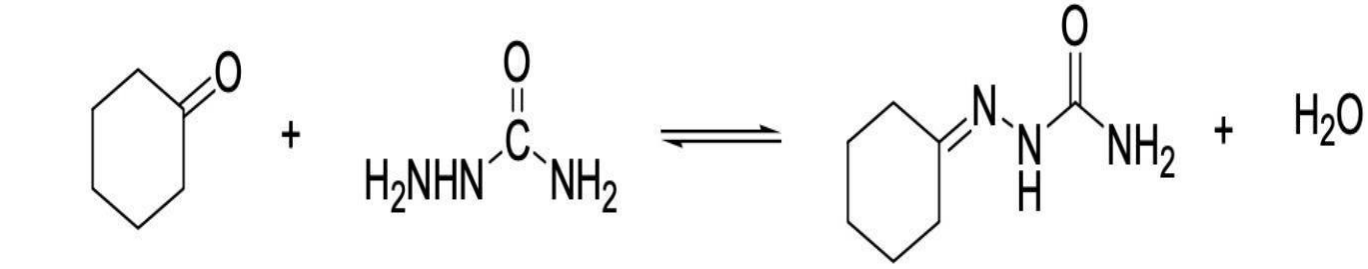
Aim:

Preparation of Cyclohexanone semicarbazone with high yield.

Materials:

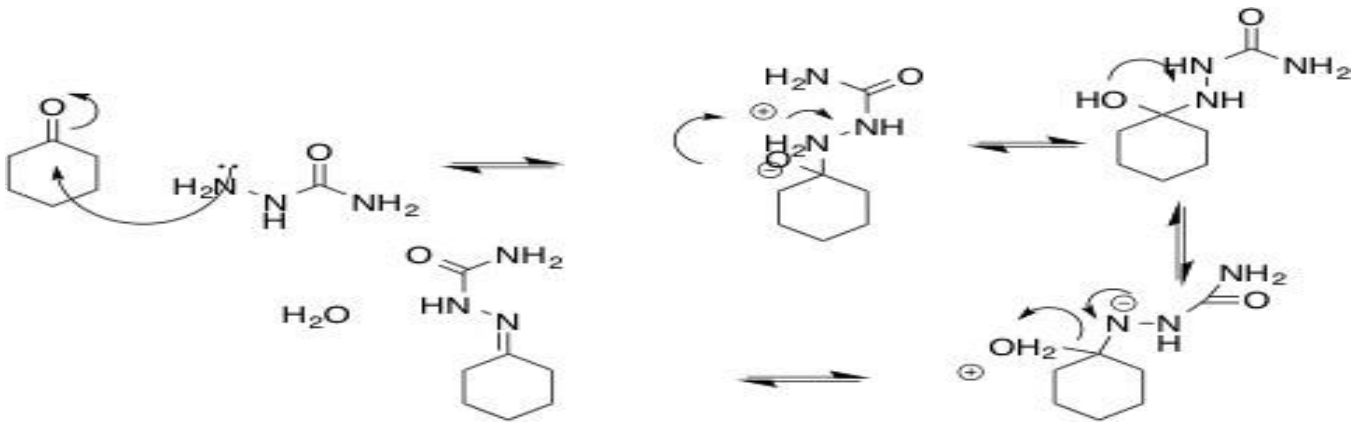
- Semi carbazide hydrochloride, Cyclohexanone, Sodium bicarbonate
- Distilled water

Reaction equation:



Cyclohexanone (2) + Semicarbazide (1) ⇌ Cyclohexanone semicarbazone + H_2O

Reaction Mechanism:



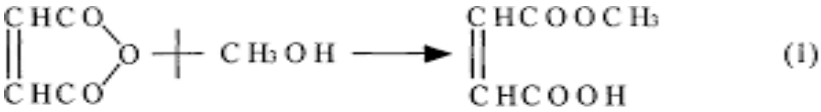
Procedures:

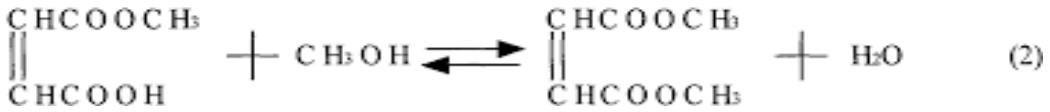
1. Weight 1g of Semi carbazide hydrochloride in conical flask(100mL).
2. Add 2g of NaHCO3 and 25mL of distilled water.
3. When the bubbles are over, add 1ml of Cyclohexanone quickly.
4. After that, shake the mixture for 15-20 minutes.
5. Then we are filtering by Büchner funnel (we washed the precipitate with cold distilled water) and take the weight of the Semicarbazon and melting point.

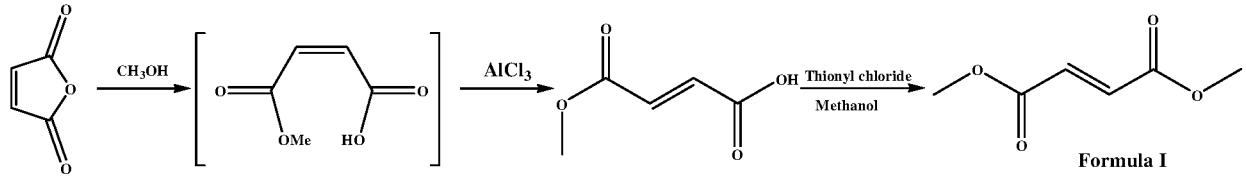
Preparation No 4

Preparation of Dimethyl Maleate

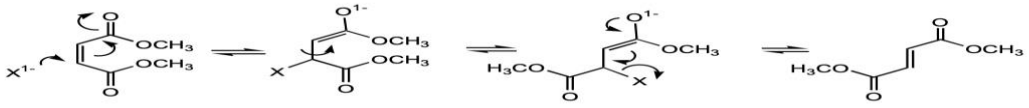
Dimethyl maleate is an organic compound with the formula C6H8O4. It is the dimethyl ester of maleic acid.
This experiment includes preparing dimethyl maleate from anhydrous maleic anhydride and methanol, then reacting it with piperidine to obtain an unknown compound (X). The student should identify the nature of this compound.

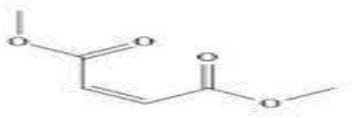


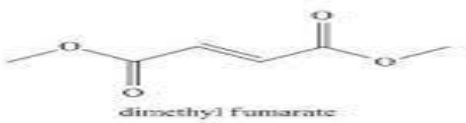




Mechanism G (nucleophilic attack at the π bond)




dimethyl maleate


dimethyl fumarate

Synthesis

Dimethyl maleate can be synthesized from maleic anhydride and methanol, with sulfuric acid as catalyst, via a nucleophilic acyl substitution for the monomethyl ester, followed by a Fischer esterification reaction for the dimethyl ester

Applications

Dimethyl maleate is used in many organic syntheses as a dienophile for diene synthesis. It is used as an additive and intermediate for plastics, pigments, pharmaceuticals, and agricultural products. It is also an intermediate for the production of paints, adhesives, and copolymers

Dimethyl maleate has also found use in applications where improvements in the hardness and toughness of polymer films are desired. This includes, in particular, the improvement of anti-blocking properties of copolymers of vinyl acetate with DMM. It is also used as an internal modifier to increase the glass transition temperature of styrene or vinyl chloride polymers

Chemistry

Hydrolysis of dimethyl maleate gives maleic acid, or possibly the maleic acid monomethyl ester. Hydration of the same compound gives malic acid

Procedure:

1- Dimethyl maleate First: Prepare Weigh 10 g of maleic anhydride,

2- then place it in raound bottome flask (flask) with a capacity of 100 ml .

3- Add 40 ml of methanol and 2 ml of concentrated sulfuric acid. Stir the mixture well,

4- then install a condenser on the flask vertically (so that the condensation process can take place Refluxs). . Heat the mixture in the flask gently in a water bath for 15 minutes.

(Remember to pass water inside the condenser during the heating process).

5- Cool the solution .

6- Pour this solution into a 250 ml conical flask containing 20 saturated solution of sodium chloride (Brine). Why?

7- The product was extracted with methylene chloride (CH2Cl2) three times each Once in an amount of 10 CH2Cl2 (3*10ml= 30 ml) why?

8- Wash the organic layer after collecting it in a beaker with 15ml of a dilute solution of sodium bicarbonate (Na HCO3 5%). Why? Use a separating funnel in the washing process.

9- Separate the organic layer and dry it with anhydrous sodium sulfate anhydrous sodium sulphate Na2SO4

10. Filter the solution into a clean, dry, pre-weighed cup.

11. Evaporation on a water bath or electric heater to eliminate methylene chloride{ in the gas cabinet} B.P.= 41°C

12- After evaporation of me thylene chloride (CH_{2Cl2}), dimethyl maleate remains

13- Find the weight of the product.

:

Second:

Converting the compound Dimethyl maleate to the unknown compound (X)

1-In the beaker (50 ml) Dissolve the dimethyl maleate product In 10 ml of Methanol

2- . Add 2 ml piperidines to the gas cabinet.

.3- Heat the mixture to boiling in a water bath (in a gas cabinet.)

4- Cool the solution with ice.

5- . Collect the precipitated product (X) by filtration on a Buechner funnel.

6. Compound (X) can be recrystallized from methanol.

Determine the resulting compound and find its weight and melting point.

Results:

Weight of the resulting compound (X) = g

Melting point of the resulting compound (X) M.P =

Questions:

1. What is the formula of the compound (X).

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PREPARATION NO 5

ADDITION OF BROMINE TO *TRANS*-CINNAMIC ACID

Adding bromine to cinnamic acid

.

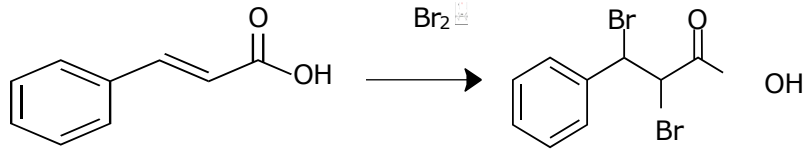
Bromine is added to alkenes by means of qualitative analysis and detection of the double bond in them. Bromine has a red color - it is grown from, while the resulting alkenes and dibromide compounds are colorless. Therefore, a dilute solution of bromine dissolved in carbon tetrachloride (CCl) or from Chloroform to alkene, its color disappears quickly.

The reaction involves the formation of the bromonium ion as an intermediate, which quickly reacts with nicklonyl 8 to form, trans Almighty brocid.

In this experiment you will react *trans*-cinnamic acid with bromine to form 2,3-dibromo-3 phenyl propanoic acid

. You will use your textbook and your knowledge of organic chemistry to predict

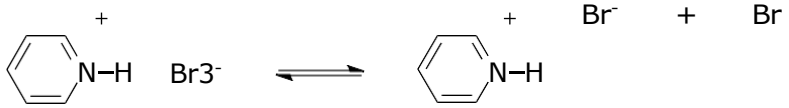
the stereochemical outcome of the product. You will then perform the reaction and use the melting point of the product to test your hypothesis.



trans-cinnamic acid **2,3,-Libromo-3-phenyl propoionic acid**

mixture. Pyridinium tribromide exists in equilibrium with pyridinium hydrobromide and bromine, and

thus, we will use it to generate the bromine needed for our reaction.

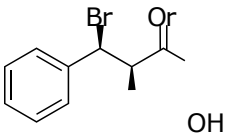


pyridinium tribromide

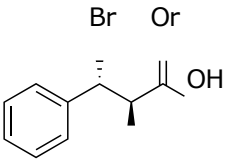
pyridinium hydrobromide

completing the reaction and purifying the crude product by recrystallization, you

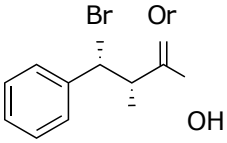
will use melting point



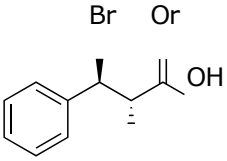
(2R,3R)-2,3-dibromo-3-phenylpropanoic acid



(2R,3S)-2,3-dibromo-3-phenylpropanoic acid



(2S,3S)-2,3-dibromo-3-phenylpropanoic acid



(2S,3R)-2,3-dibromo-3-phenylpropanoic a

Bromine is toxic and burning to the skin. Avoid contact with or inhalation of the skin warning

Method of work:

1- Mix 3.7 grams of cinnamicacid (Qurna) and put it in a 100 ml conical flaske.

2- Add 20 ml of Chloroform $CHCl_3$ to it.

3- Heat the flask in a hot water bath until the cinnamic acid dissolves in the Chloroform .

4- The solution was then cooled by the Ice, and the solid began to precipitate.

5-- Add a solution of 0.2 g of bromine dissolved in 5 ml of chloroform

,(which you prepared in advance during the attack)

7- quickly add bromine solution with strong shaking and stirring

8- Leave the solution in the beaker containing ice for 30 minutes until the product crystallizes.

NOTE - It is preferable to cover the Beakar with vouel during the previous waiting period.

9- Collect the product by filtration over a funnel.

10-Dry Prouduct and find the M.P

Preparation No 6:

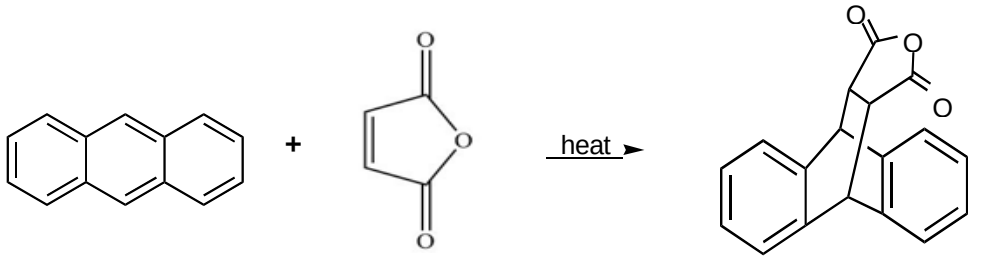
Preparation of Anthracene-9.10-endo-α,β-Succinic Anhydride by Diels-Alder Reaction

- The purpose of this experiment is to form 9, 10-dihydroanthracene-9,10-α, β- succinic anhydride by way of a Diels Alder reaction.
- Purifying the product to perform recrystallization.
- Characterize the product using melting points and the infrared spectrum.

Introduction:

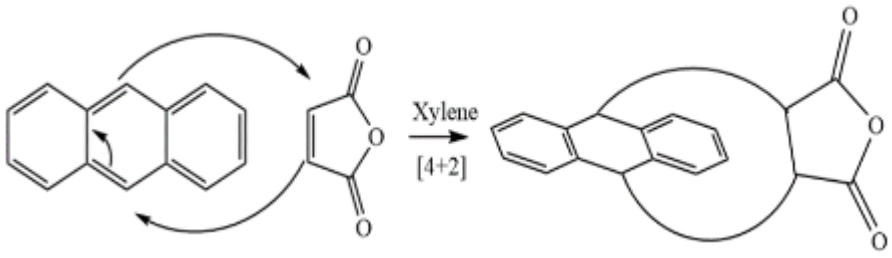
The Diels Alder reaction between anthracene and maleic anhydride to form 9, 10- dihydroanthracene-9,10-α, β-succinic anhydride occurred via the Diels Alder mechanism. Anthracene served as the diene and maleic anhydride was the dienophile. Following the reaction

The crude product will be recrystallized. Melting points of both the crude product and recrystallized product will be taken and compared with the literature value. Also, an infrared spectrum of the recrystallized product will be taken. Both melting points and the infrared spectrum will be used to characterize the product.



Anthracen + **Maleic Anhydride** → **Diels Alder Adduct**

Diels Alder Adduct



Procedure:

Part 1- preparation:

- Weigh 2.0 gm of anthracene and 1.1 gm maleic anhydride and place them in the reaction flask. Add 25.0 mL of xylene.
- Fit the vial with a condenser,
- Turn on the water to the condenser Use a 200°C sand bath to heat the mixture at reflux for 30 minutes.
- Cool the mixture to room temperature and prepare an ice bath using a 150-mL beaker. Then cool the mixture in the ice bath for approximately 5 minutes

Collect the recrystallized solid by vacuum filtration using a Hirsch funnel and rinse the crystals with 1 mL of ice-cold xylene.

- Weigh the crude product and set aside a small sample to dry.
- This sample will later be used to measure the melting point of the crude product.

Part 2- purification:

Purifying the Product To perform recrystallization.

- place the product in a 10-mL Erlenmeyer flask.
- Add ethyl acetate and heat the mixture gently in a water bath until the ethyl acetate boils.
- Gradually add more ethyl acetate until all the product dissolves or until no more appears to be dissolving.
- Transfer the solution (filtration) to another 10-mL flask containing a small amount of boiling ethyl acetate.

- Allow the solution to cool to room temperature and then cool the solution in an ice bath for 5 minutes. If it's necessary, scratch the bottom of the flask with a glass rod to induce crystallization.
- Collect the crystallized solid by vacuum filtration, using a Hirsch funnel.
- Spread the product crystals thinly over a clean watch glass and allow them to dry for approximately fifteen minutes.

Preparation .07

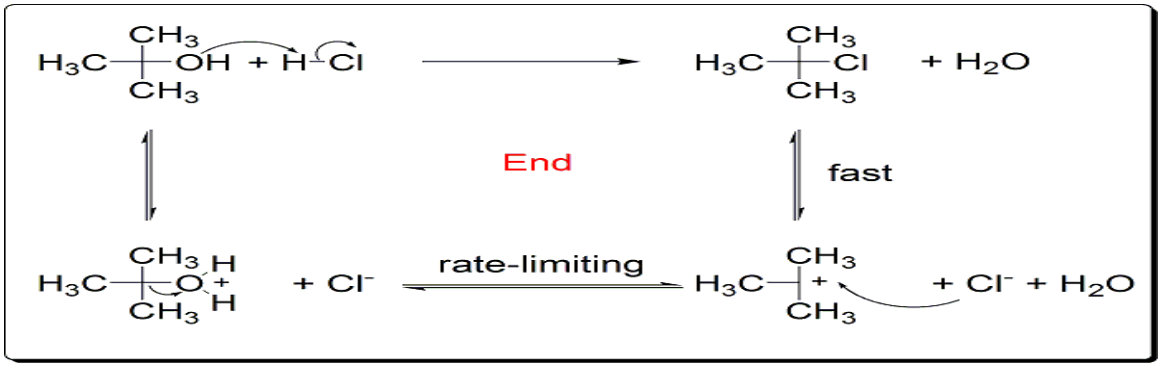
Preparation of t-butyl chloride

Objectives:

-Prepare *tert*-butyl chloride (2-chloro-2-methylpropane) from *tert*- butyl alcohol (*tert*-butanol) using an acid catalyzed dehydration reaction. (Note: the correct IUPAC name for this compound is 2-methyl-2-propanol).

We will also learn how to use a separatory funnel, the use and purpose of a drying agent and the technique of distillation.

The first step of the overall reaction is an acid-base reaction between the *t*-butanol and the hydrochloric acid. The *t*-butanol is a weak base and the hydrochloric acid is a strong acid. The alcoholic oxygen becomes fully protonated and so the equilibrium lies far to the right. In the second step we have the slow loss of water to form a carbocation intermediate. This species is very reactive and is immediately attacked by the chloride ion liberated in the first step to form the final product. This is an example of an SN1 reaction (Substitution Nucleophilic Unimolecular).



Experimental Procedure

- 1- In your separatory funnel (250 mL), place 125 mL of concentrated hydrochloric acid that has first been cooled to 5°C in an ice bath.
- 2- Add 28 mL (22 g) of t-butanol and swirl gently with the stopper off the separatory funnel, release the pressure after every swirl for 20 minutes.
- Once the layers have been thoroughly mixed,
- 3-allow the separatory funnel to stand with the stopper off. You will gradually see two layers forming. (What are they?)

  4- Drain off the bottom layer. This is the aqueous layer. It is waste, but be sure to save it until you have finished with the experiment.
- 5- Add 100 mL of cool Na_2CO_3 5% to the separatory funnel and shake again several times to mix the two layers. Again, separate the aqueous layer and set it aside. (You can save this with the first aqueous layer.)
- 6- Add 10 Ml anhydrous sodium bicarbonate to the separatory funnel. Be careful here. The sodium bicarbonate is a weak base. The purpose for adding this is to neutralize any hydrochloric acid that may be remaining.
- {- When it reacts with the hydrochloric acid, carbon dioxide gas is given off. Pressure can build up very quickly in a closed separatory funnel and your solution will shoot out of the funnel }
- 7- Filter through a small filter paper into a 100 ml distilling flask , add 2—3 chips of papers porcelain and distill .
- 8- collect the fraction boiling point at 48 ---53 C^0
- 9 - Determine the yield .

Preparation 08:

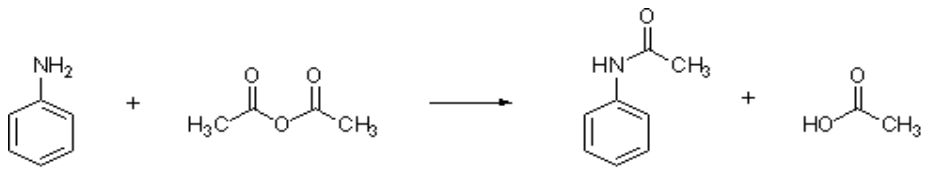
Preparation of Acetanilide

Objectives:

- To synthesis acetanilide by reaction of aniline and acetic anhydride (amine to amide).
- To purify acetanilide by crystallization method using water.
- Purity check by melting range and TLC.
- Acetanilide characterization using IR spectrum.

Discussion:

This experiment involves four functional groups common in organic chemistry. The substrate (reactants) are both liquids and one of the products is solid. The reaction of aniline with acetic anhydride is a transformation in which products, acetanilide and acetic acid, are obtained.



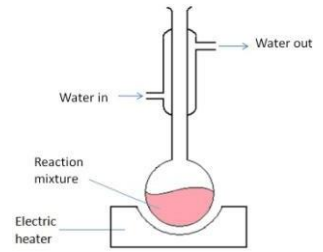
The substrate (reactants):

Compound	MP (BP)	Density	Hazards
Aniline	(184 ºC)	1.022 g/mL	Irritating (eyes/skin). Harmful if inhaled/ingested. Possible carcinogen.
Acetic Anhydride	(138 ºC)	1.082 g/mL	Irritating (eyes/skin). Toxic by inhalation, Flammable (fop 49 ºC).

A solid product is often desirable since it may be recrystallized and a melting point determined. Recrystallization is a widely used technique to purify a solid mixture. The desired product is isolated from its impurities by differences in solubility. Insoluble impurities and colored impurities can be removed from hot solvent through the use of activated carbon and filtration. Soluble impurities remain in the cold solvent after recrystallization. The desired product should be as soluble as possible in hot solvent and as insoluble as possible in cold solvent. The selection of solvent is, therefore, critical to the

Experimental Procedures

First Method:

Step	Procedures	
1	Place 0.1 molo of aniline (d=1.022 g/mL) in a spherical flask(100mL).	
	Add 20 mL of glacial acetic acid and 20 mL of acetic anhydride.	
	Heat the mixture under reflux for 10-15 min.	
2	Cool the reaction mixture and transfer it into beaker contain 50 mL of NaOH 5% with stirring in ice bath.	
3	Collect the product by vacuum filtration using Buchner funnel.	
4	Purify acetanilide by crystallization method using water.	
5	Allow the sample to dry completely. Weigh the dry product, calculate the percentage yield and determine its melting point. Collect to product in a paper and write your name and submit it with the report.	
	% Yield acetanilide = mass acetanilide recovered x100 Theoretical mass of acetanilide	

Second Method:

Preparation 9

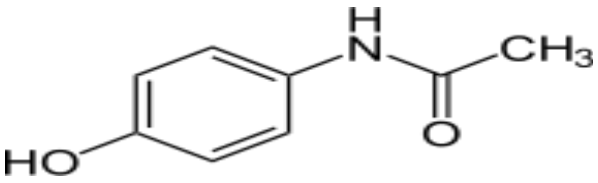
PREPARATION OF PARACETAMOL

INTRODUCTION:

Paracetamol is a pain killer and an antipyretic, that is used widely, because of its high effectiveness, and low side effects.

Paracetamol is a benzene ring, that contains hydroxide and an amide (acetamide) group on the para position (1,4).

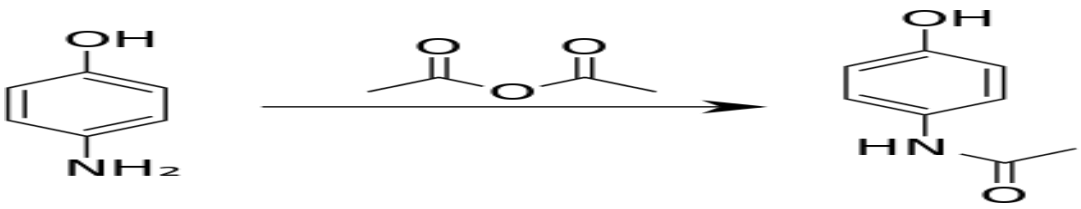
Overdosing on paracetamol may cause damage to the liver.



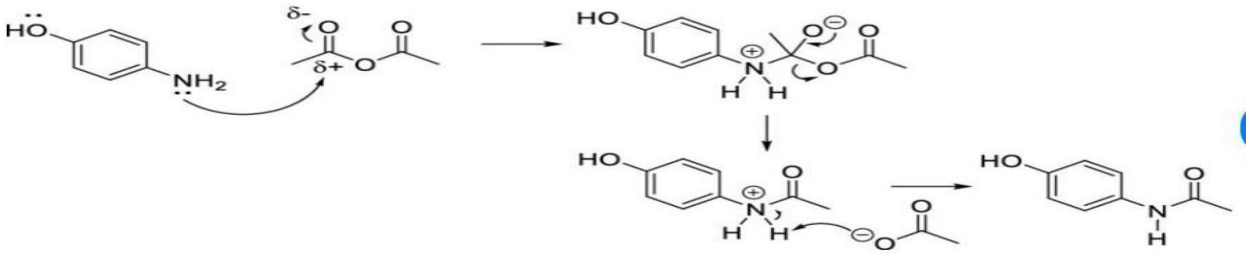
Structure of paracetamol

Materials used: 1- p-amino phenol 2- acetic anhydride 3- distilled water 4- a condenser 5- round bottom flask 6- Buechner funnel.

Equation



Reaction mechanism:



Procedure:

1- In the round bottom flask, weight about 1.375g of p-aminophenol
2- Add 4 ml of distilled water.
3- Add 1.5 ml of acetic anhydride, add drop by drop.
4- Reflux for 20 minutes, note that the water goes from down the condenser and out from the top (revertive of the gravity).
5- After 20 minutes, let the mixture cool off, and sperate the residue by Buechner funnel.

Calculations and results:

- Weighting the residue was found to be ……. g.
- When measuring the melting point, theoretically it will be 169C-170C.
- To calculate the percentage error, we need to know how many grams of paracetamol should be produced (mass theoretically),

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D-glucose pentaacetate

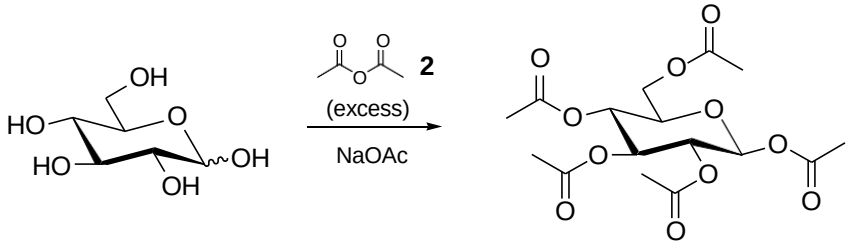
Aims

Carbohydrates such as D-glucose are essential biomolecules that play important roles in metabolism. They are also organic molecules that feature alcohol functional groups and, as such, undergo many of the reactions of simple alcohols such as ester formation. In this experiment you will form five esters at one go by reacting D-glucose with acetic acid in the presence of a base catalyst. The product D-glucose pentaacetate is an important intermediate in synthetic carbohydrate chemistry.

Reaction

D-Glucose (**1**) has three different types of alcohol present; the primary alcohol at C-6, the three secondary alcohols at C-2, C-3, and C-4, as well as the hemiacetal OH group at C-1. All of these will react with acetic anhydride (**2**) to form the pentaacetate (**3**).

Reaction Equation ;



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Procedure

- Weigh out α-glucose (5 g) and sodium acetate (4 g) and place them into a 100 mL round bottomed flask.
- Add a boiling chip and then ***carefully*** add acetic anhydride (25 mL).
- Heat the mixture to ~90 ^{oC} for 90 minutes with occasional swirling to make sure the chemicals mix.
- Cool the flask and then ***very carefully*** pour the reaction mixture (with stirring) into a 500 mL beaker that contains 250 mL of ice water.
- Filter the product and dry for 10 minutes under vacuum. Weigh the crude product.
- Recrystallize the material from a mixture of methanol and water (~1:2) and then filter the solid once it has completely reprecipitated. Dry for 10 minutes on the vacuum filter. Weigh the pure product and obtain the melting point.