PRACTICAL ORGANIC CHEMISTRY (II)

SECOND LEVEL

1444 - 1443

2022 - 2023

LECTURER: RAWNAQ THAMER

INDEX

SAFETY AND SECURITY RULES IN THE LABORATORY

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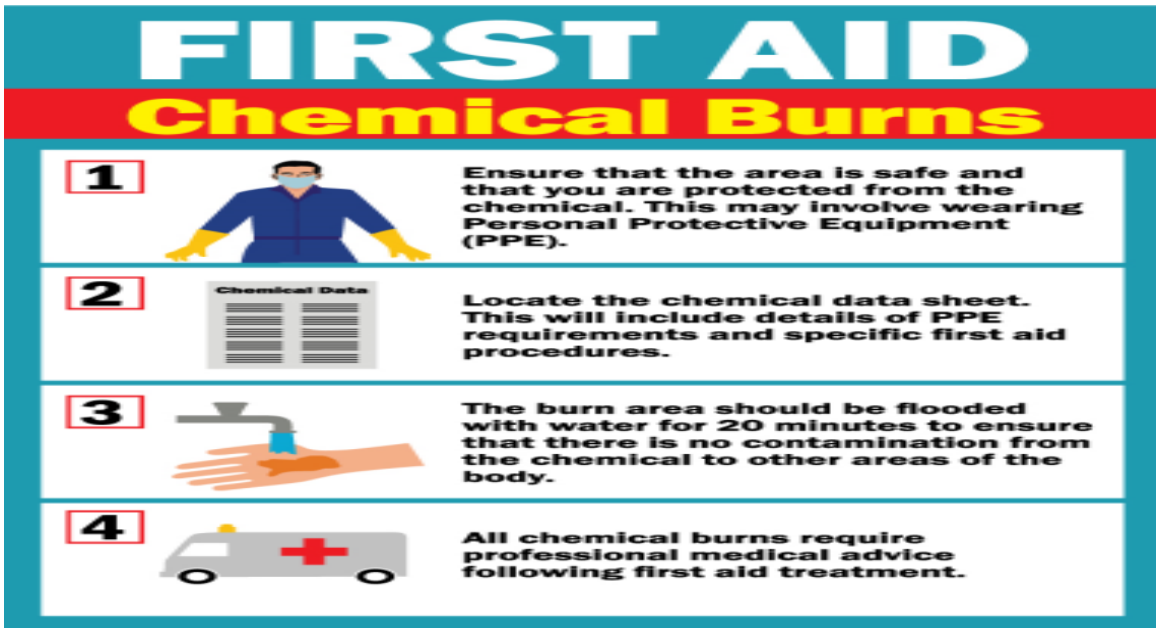
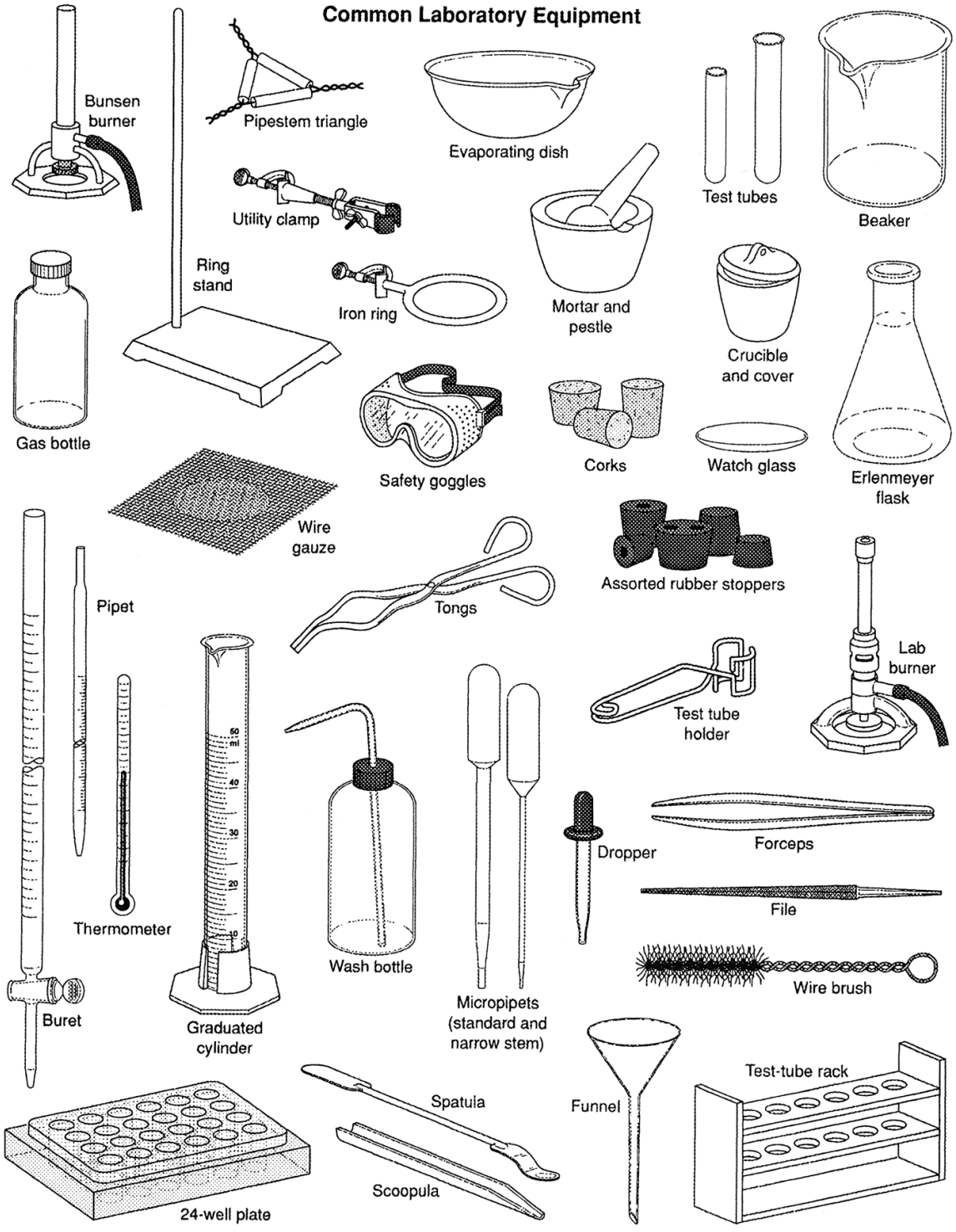



Chemical Symbols
Look Out For These Symbols
On The Chemical Containers
& Understand Their Meanings
Oxidizers
Corrosives
Toxicity (Severe)
Environmental Toxicity
Gases Under Pressure
Explosives Self Reactives
Carcinogen • Respiratosy Sensitizer
• Reproductive Toxicity • Target Organ
Toxicity • Mutagenicity • Aspiration
Toxicity
Flammables • Self Reactives
• Pyrophorics • Self-Heating
• Emits Flammable Gas
• Organic Peroxides
Irritant • Dermal Sensitizer
• Toxicity (Harmful) • Narcotic Effects
• Respiratory Tract


إشارات الخطر
Hazard signs
Corrosive
مواد أكالة
Toxic
سام
Irritant
مخرش
Hot surface
سطح ساخن
General warning
تحذير عام
Oxidising
مواد مؤكسدة
Explosion risk
مواد قابلة للانفجار
First aid
إسعاف أولي
High voltage
توتر عالي
Radiation
إشعاع
Flammable materials
مواد قابلة للاشتعال
Environmental hazard
خطر بيئي
Gas bottle
أنبوبة غاز
Biohazard
خطر حيوي
Laser radiation
أشعة ليزر

Common Laboratory Equipment



DISTILLATION

WHAT IS DISTILLATION?

Distillation refers to the selective boiling and subsequent condensation of a component in a liquid mixture. It is a **separation technique** that can be used to either increase the concentration of a particular component in the mixture or to obtain (almost) pure components from the mixture. The process of distillation exploits the difference in the **boiling points** of the components in the liquid mixture by forcing one of them into a gaseous state. It is important to note that distillation is not a chemical reaction but it can be considered as a physical separation process.

- The liquid to be distilled is called "Distilland".
- The liquid obtained after condensation of the vapors is called "Distillate" or " Condensate".
- The apparatus used for distillation is called " Distillator" or "Still".
- The arrangement used to cool the vapors is called "Condenser"
- The temperature at which the vapor pressure of a liquid is equal to the atmospheric pressure is called "boiling point". It is different from the evaporation point

OBJECTIVE OF DISTILLATION

1- Separation of fluids from mixtures.
2- Extraction of active chemical constituents.
3- Product purification.
4- Recovery of expensive solvent.
5- Recycling of the reactant .
6- Extraction of volatile oils.

TYPES OF DISTILLATION

Some important types of distillation include:

A- Simple distillation

B- Fractional distillation

C- Steam distillation

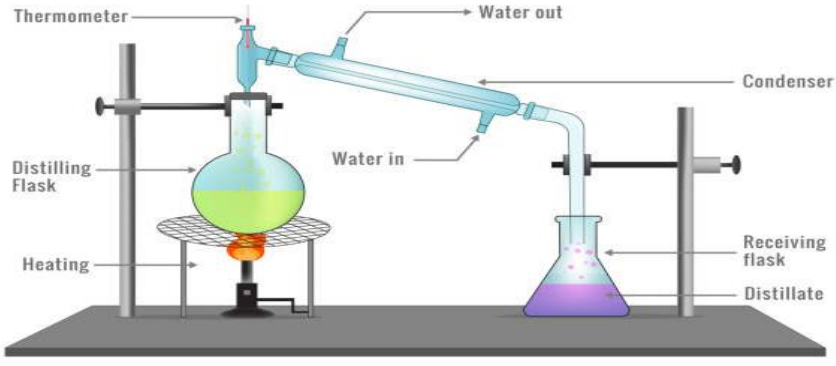
D- Vacuum distillation

E- Reflux

A- SIMPLE DISTILLATION

- Simple distillation involves heating the liquid mixture to the boiling point and immediately condensing the resulting vapors.
- This method is only effective for mixtures where in the boiling points of the liquids are considerably different (a minimum difference of 25^{o}C).
- Examples : Benzene (B.p. = 80^{o}c) and Aniline (B.p.= 184^{o}c)

  Chloroform (B.p.= 61^{o}c) and Aniline (B.p.= 184^{o}c)



Figure(1): Simple distillation device

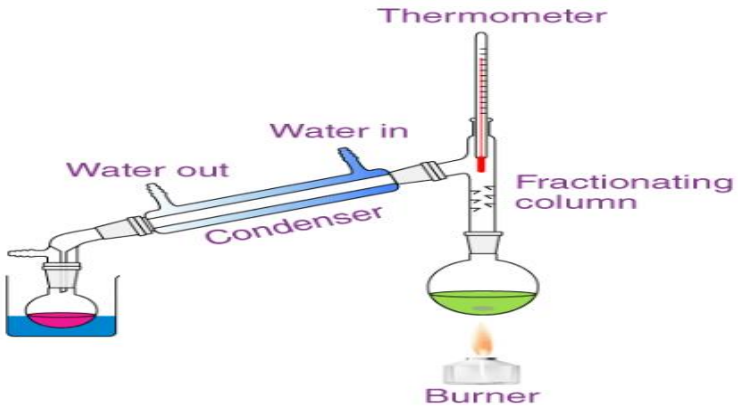
B- FRACTIONAL DISTILLATION

Fractional distillation is often used to separate mixtures of liquids that have similar boiling points. It involves several vaporization-condensation steps (which takes place in a fractioning column). This process is also known as rectification. The fractional distillation apparatus is composed of:

- Round-bottom flask or distilling flask
- A source of heat, which can be a fire or a hot bath.
- Receiving flask to collect the condensed vapors
- Fractioning column
- Thermometer to measure the temperature in the distilling flask
- Condenser

When heated, the liquid mixture is converted into vapors that rise into the fractioning column. The vapors now cool and condense on the walls of the condenser. The hot vapors emanating from the distilling flask now heat the condensed vapor, creating new vapors.

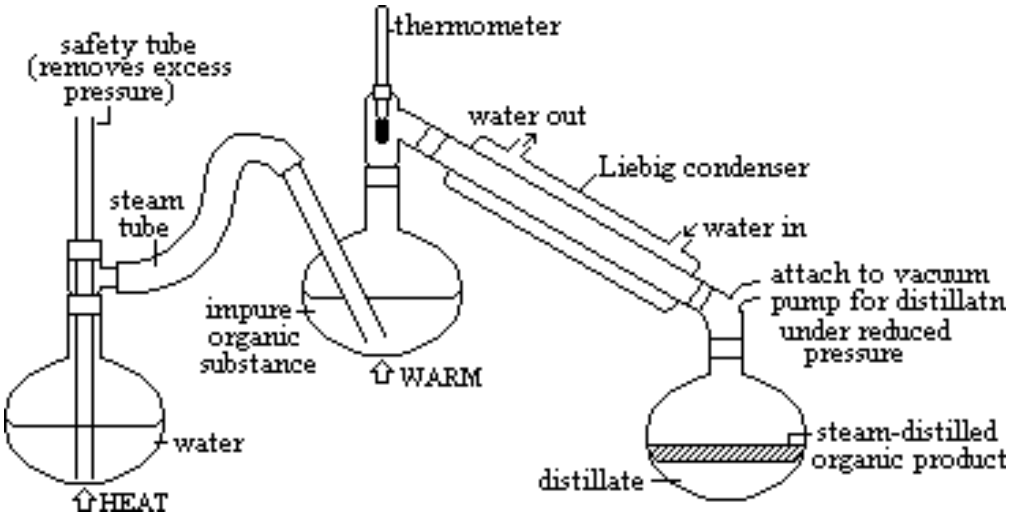
Many such vaporization-condensation cycles take place and the purity of the distillate improves with every cycle. An illustration depicting a fractional distillation setup is provided below.



Figure(2): Fractional distillation device

C- STEAM DISTILLATION

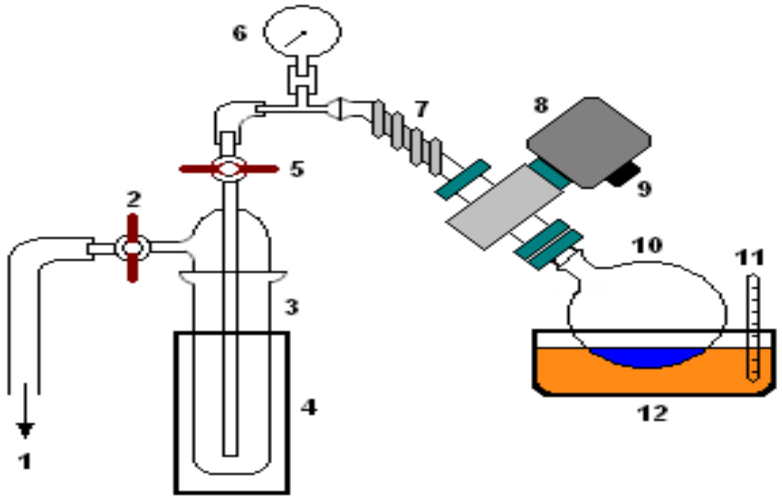
- Steam distillation is often used to separate heat-sensitive components in a mixture.
- This is done by passing steam through the mixture (which is slightly heated) to vaporize some of it. The process establishes a high heat-transfer rate without the need for high temperatures.
- The resulting vapor is condensed to afford the required distillate.
- The process of steam distillation is used to obtain essential oils and herbal distillates from several aromatic flowers/herbs.



Figure(3): Steam distillation device

D- VACUUM DISTILLATION

- Vacuum distillation is ideal for separating mixtures of liquids with very high boiling points.
- In order to boil these compounds, heating to high temperatures is an inefficient method. Therefore, the pressure of the surroundings is lowered instead.
- The lowering of the pressure enables the component to boil at lower temperatures. Once the vapor pressure of the component is equal to the surrounding pressure, it is converted into a vapor.
- These vapors are then condensed and collected as the distillate. The vacuum distillation method is also used to obtain high-purity samples of compounds that decompose at high temperatures.

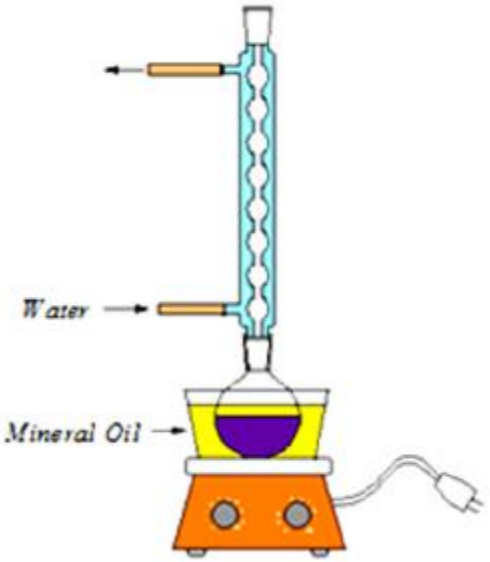


Figure(4): Vacuum distillation device

E- REFLEX:

Reflex is a type of distillation technique involves the condensation of vapors & the return of this condensate to the system from which it originated.

This method is used to keep or prevent the reactants from loss by evaporation during a reaction.



Figure(5): Reflex distillation device

NOTE

1- **In the Google search engine, you can type "handbook the purification in laboratory" or "Handbook of the Distillation in Laboratory" or similar words; You will find more information.**
2- **You can use this book "Purification analytical chemistry, second edition"**
3- **You can use this book "Purification of laboratory chemicals. Sixth edition".**

QUESTION:

Define distillation and write its application in pharmacy.

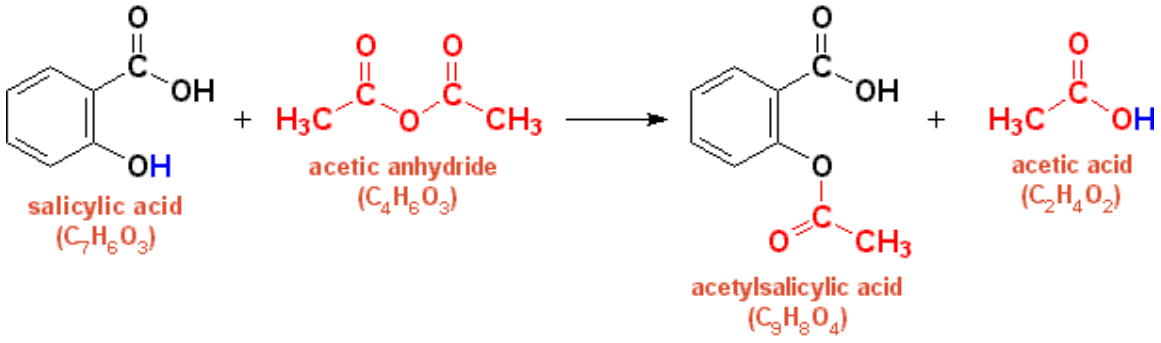
PREPARATION OF ASPIRIN

ASPIRIN

- The common name for the compound acetylsalicylic acid is aspirin.
- It was widely used as a fever reducer and a pain killer.
- Salicylic acid, whose name comes from Salix, the willow family of plants, was derived from willow bark extracts.
- In folk medicine, willow bark teas were used as headache treatments.
- Nowadays, salicylic acid is administered in the form of aspirin which is less irritating to the stomach than salicylic acid.

PREPARATION

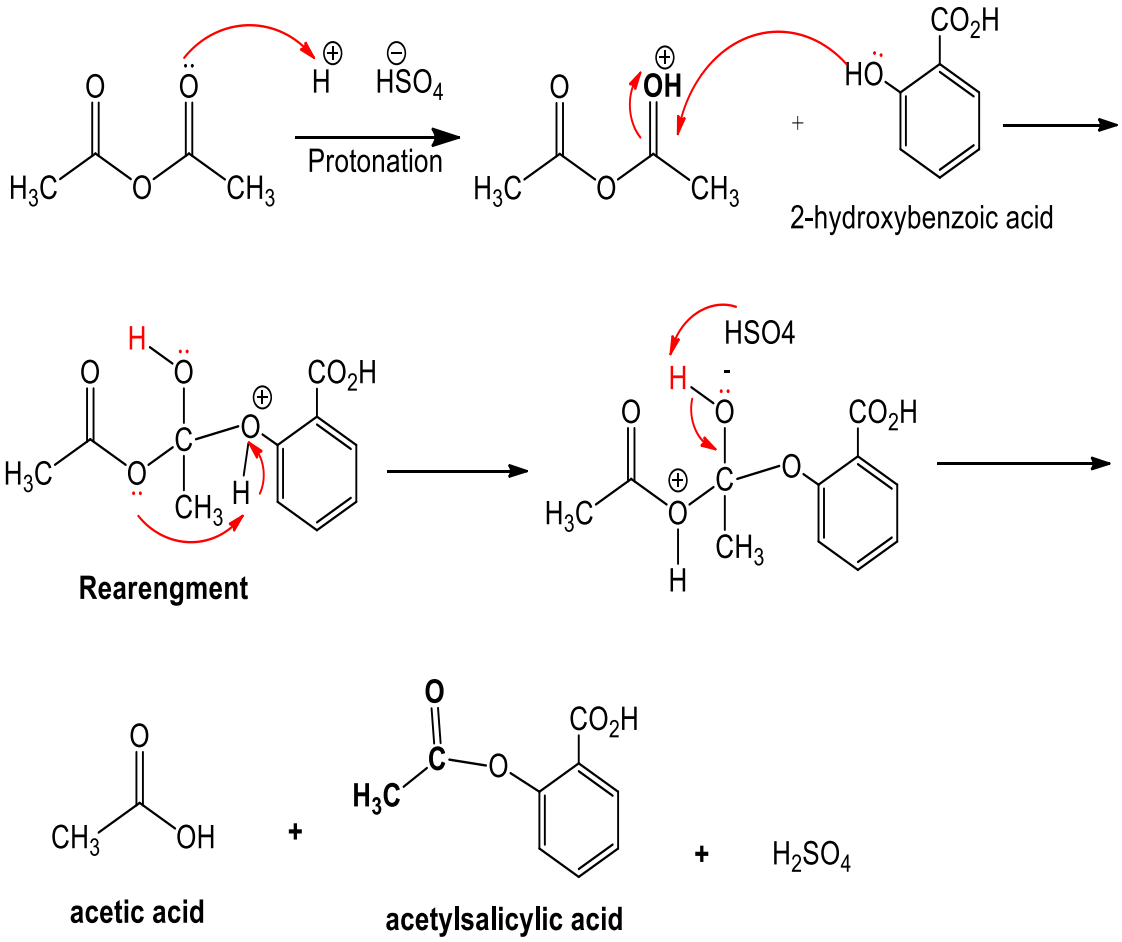
- To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride.
- A small amount of a strong acid such as sulfuric acid used as a catalyst which speeds up the reaction.
- The excess acetic acid will be reduced with the addition of water.
- The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added.
- The synthesis reaction of aspirin is shown below:



Equ.(1): Preparation of aspirin

- A “purified product” can be obtained through recrystallization of the crude product in hot ethanol.
- In this experiment, the crude product will be the desired product.
- The melting point range of pure aspirin is 138-140^0 C and the melting point range of the salicylic acid starting material is 158 161^{0}C.
- If impurities are present in your crude sample, the melting point range for your product will be lower than the range of pure aspirin.
- Also, your melting point range may be greater than 2 degrees.

Mechanism:



SAFETY

- Salicylic acid is able to penetrate and break down fats, causing moderate chemical burns to the skin.
- Acetic anhydride flammable, harmful by inhalation and if swallowed, causes burns.
- Sulfuric acid causes severe burns.
- So you must wear protective gloves, protective clothing, and eye protection.

PROCEDURE

1.Place about 2 g of Salicylic acid in the flask . In the fume hood, add 5.0 mL of acetic anhydride from into the flask. Add 5 drops of conc. H_2SO_4 (catalyst) to the flask.

2. Fit the flask with a reflux and heat the mixture to (50-60)℃. Stir if needed to dissolve the salicylic acid. Heat the water to boiling, and shut off the flame. Keep the flask in the hot water bath for 15 minutes

3 . While the flask is still in the water bath, slowly add 2ml of distilled water to the flask to decompose any excess acetic anhydride.

4. Cool the mixture to room temperature or under the tap water.

5. Add (35 ml) of water with stirring.

6. Collect the product by filtration and wash it with distill water.

7.Transfer the filter paper and aspirin to a pre weighed watch glass and allow to dry in your locker until the next lab.

PURIFICATION

RECRYSTALLIZATION OF ASPIRIN

- The crude product was dissolved in 5ml of EtOH (Ethanol) in a 100ml conical flask. The solution will be warmed if necessary. The solution was added with 30ml of hot distilled water.
- The mixture is warmed until the solid dissolves completely in the solution.
- The solution is cooled.
- Weigh the dry product to obtain the yield of the reaction. Calculate the theoretical yield and percent yield of the reaction.

MELTING POINT

1. Pack a few crystals of your aspirin product in a melting point capillary tube.
2. The melting point of aspirin is determined. The melting point range is the temperature when you first notice the aspirin crystals melting up until the temperature when no crystals remain.

RESULTS AND CALCULATION

- Mass (molecular wight) of salicylic acid (a) =138.121 g/mol
- Mass of filter paper & watch glass (b) = 34.29 g(Check it out for yourself)
- Mass of dried, recrystallized aspirin, filter paper & watch glass (c) = 35.86g
- Mass of dried, recrystallized aspirin
- (practical)= (d) = (c) -(b)
- (d) = 35.86g - 34.29g = 1.57g
- Percent Yield= Practical weight/theoretical weight x100%.

	M.wt.	weight
salicylic acid	138	2g
aspirin	180	x

x= 180 x 2 / 138 = 2.6g(theoretical)

%yield = Practical / Theoritical

%yield = 1.57g /2.60g*100%

= 57.6%

QUESTION:

- What are Pharmaceutical uses of aspirin?
- What are side effect of aspirin?

NOTE

1- **In the Google search engine, you can type "handbook the Preparation of aspirin" or similar words; You will find more information.**
2- **You can use this book "Aspirin and the Salicylates K D Rainsford"**