# Chemistry of Natural products
# 436 chem – 3
# Level 7

# Part one

# Preparation of simple organic compounds

# Preparation of simple organic compounds

**In any experiment we must write**

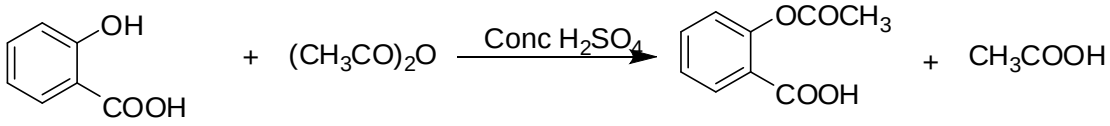
**1- Name of the reaction**
**2- Name of the prepared organic compound**
**3- Equation of reaction**
**4- Mechanism of reaction**
**5- Procedure of reaction**
**5- Calculation**
**6- Yield of the product**
**7- Melting point (m.p.) of the prepared compound**

# 1-Preparation of Aspirin

## Materials:

Salicylic acid
Acetic anhydride
Conc.Sulphuric acid

## Reaction:-




Salicylic acid Aspirin

## Procedure:-

1- In a conical Flask fitted well, place Salicylic acid (4 gm), acetic anhydride (10 ml) and conc.$H_2SO_4$ (1 ml**),** shake the mixture for 15 min.

2- Add water (20 ml) into the reaction mixture, then shake till ppt. formation.

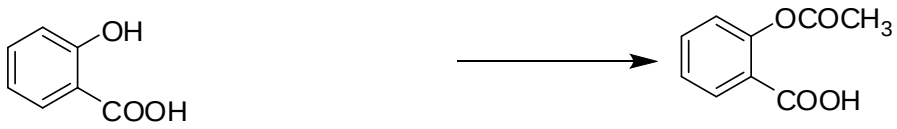
3- Filter the ppt., wash it with water, left it till dryness.

4- Weigh the ppt., and then calculate the percent

5- measure melting point.

## Calculation of Final Yieldof Aspirin:-

### Theoretical yield




**$C_7H_6O_3$** **$C_9H_8O_4$**

[7x12+6x1+3x16] [9x12+8x1+4x16]

138 180

4gm. X

X= Theoretical yield = [4x180]÷138= gm.

### Practical yield

Weight of filter paper= W1 gm.
Weight of filter paper + ppt= W2 gm.
Practical yield= weight of ppt= W2-W1=…… gm.

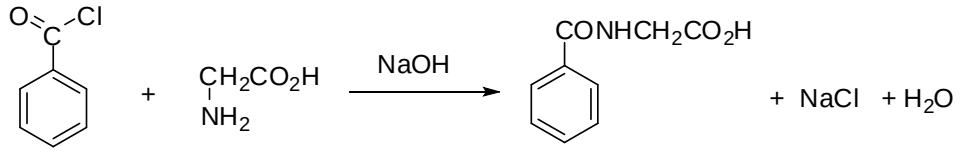
Final yield = practical yield ÷theoretical yield] x100= %

| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

## 2-Preparation of Hippuric acid (benzoylglycine)

**Materials:**
Benzoyl chloride
Glycine
Sodium hydroxide

**Reaction**




**Glycine** **Hippuric acid**

Procedure:

1- In a conical flask, dissolve glycine (7.5 g) in an aqueous sodium hydroxide soln. (10%, 75 ml) and add to it benzoyl chloride (14 ml) in a five or six installments.

2- After each addition, stopper the flask and shake vigorously until all chloride has reacted (the content may be warmed gently and thoroughly shaken until the smell of benzoyl chloride disappears).

3- Transfer the solution to a beaker containing crushed ice and add conc. hydrochloric acid slowly and with stirring until the solution becomes acidic.

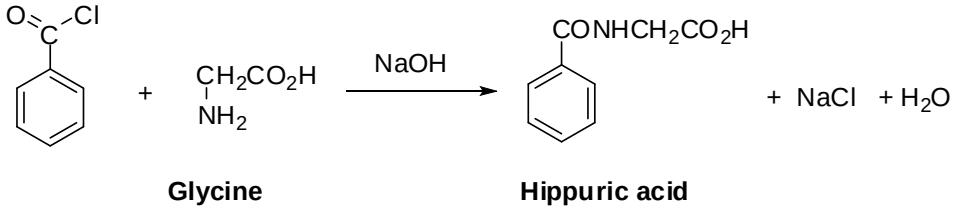
4-Filter the crude product (containing a little benzoic acid as impurity) by suction, wash well on the filter with cold water and boil the impure product gently with carbon tetrachloride (25 ml) for 10 minutes to remove the benzoic acid.

5-Cool the mixture slightly and filter by gentle suction. Wash the residue with carbon tetrachloride (3-4 ml) and recrystallize from hot water. The yield is 13 g and the m.p

is 187°. Check purity by TLC also. Record IR spectra of glycine and the product .

## Calculation of Final Yieldof Hippuric acid:-

### Theoretical yield




**$C_7H_5OCl$** **$C_9H_9O_3N$**

[7x12+5x1+1x16+35] [9x12+9x1+3x16+15]

140 180

W=dv X

X= Theoretical yield = [Wx 180]÷140 = gm.

### Practical yield

Weight of filter paper = W1 gm.

Weight of filter paper + pp t= W2 gm.

Practical yield= weight of ppt = W2-W1=…… gm.

Final yield = practical yield ÷theoretical yield] x100= %

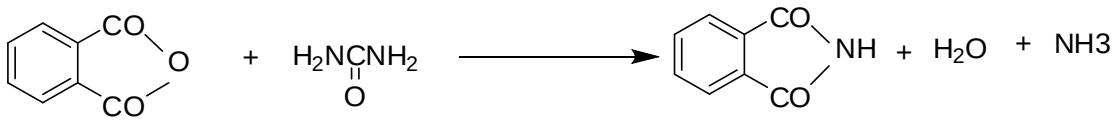
| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

## 3-Preparation of Phthalimide

**Materials:**

phthalic anhydride
urea

**Reaction**




phthalic anhydride urea Phthalimide

**Procedure:-**

1-In a conical Flask fitted well, place a mixture of phthalic anhydride (15gm.) and urea (3.5gm.) and heat the flask.

2-The reaction begin with the melting of contents, effervescence which gradually increase in vigor.

3-Allow it to cool, add water to disintegrate the ppt. in the flask.

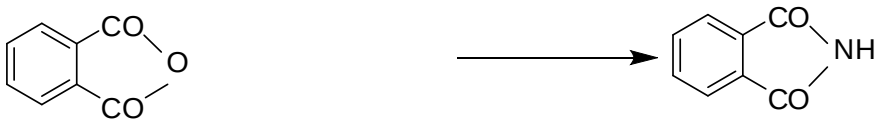
4-Filter the ppt., wash it with water, left theppttill dryness.

5-Weigh the ppt., then calculate the percent.

6-Measure the melting point

## Calculation of Final Yieldof Phthalimide:-

### Theoretical yield




$C_8H_4O_3$ $C_8H_5O_2N$

[8x12+4x1+3x16] [8x12+5x1+2x16 +15]

148 148

15 X

X= Theoretical yield = [15x 148]÷148= gm.

### Practical yield

Weight of filter paper= W1 gm.

Weight of filter paper + ppt= W2 gm.

Practical yield= weight of ppt= W2-W1=…… gm.

Final yield = practical yield ÷theoretical yield] x100= %

| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

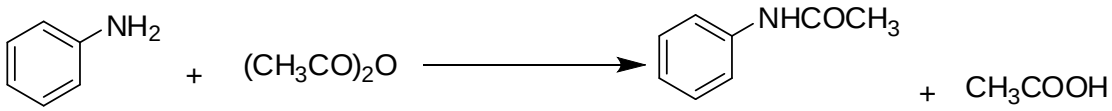
## 4-Preparation of Acetanilide

**Materials:**

Aniline

Acetic anhydride

**Reaction**




Aniline Acetanilide

**Procedure:-**

1-In a conical Flask fitted well, place a mixture of aniline (10 ml) and acetic anhydride (10 ml.)

2-Boil the mixture for 5-10 min., and then Cool the hot liquid in ice with stirring

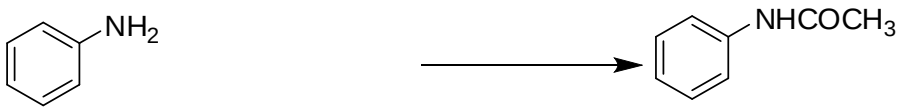
3- Filter the ppt., wash it with water, left the ppt till dryness.

5-Weigh the ppt., then calculate the percent.

6-Measure the melting point

## Calculation of Final Yieldof Acetanilide

### Theoretical yield




**$C_6H_7N$** **$C_7H_9ON$**

[6x12+7x1+15] [7x12+9x1+16+15]

94 124

W=dv X

X= Theoretical yield = [Wx 124] ÷ 94= gm.

### Practical yield

Weight of filter paper= W1 gm.

Weight of filter paper + ppt= W2 gm.

Practical yield= weight of ppt= W2-W1=…… gm.

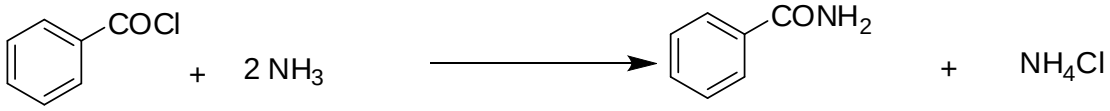
Final yield = practical yield ÷theoretical yield] x100= %

| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

## 5-Preparation ofBenzamide

**Materials:**
Benzoyl chloride
Ammonia

**Reaction**




Benzoyl chloride Benzamide

**Procedure:-**

1-In a conical Flask fitted well, place ammonia (5 ml) and set the flask in ice bath.

2-AddBenzoyl chloride(2.5ml) into it drop wise with frequent shaking for 10 min.

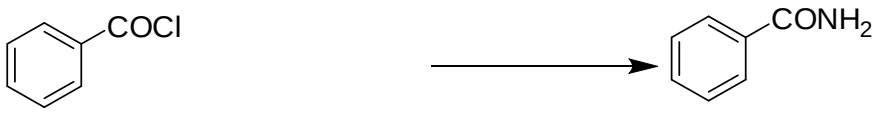
3- Filter the ppt., wash it with water, left the ppt till dryness.

5-Weigh the ppt., then calculate the percent.

6-Measure the melting point

## Calculation of Final Yield of Benzamide

## Theoretical yield




**$C_7H_5OCl$** **$C_7H_7ON$**

[7x12+5x1+16+35] [7x12+7x1+16+15]

140 122

W=dv X

X= Theoretical yield = [W x 122] ÷ 140= gm.

## Practical yield

Weight of filter paper= W1 gm.

Weight of filter paper + ppt= W2 gm.

Practical yield= weight of ppt= W2-W1=…… gm.

Final yield = practical yield ÷theoretical yield] x100= %

| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

## 6- Preparation of Benzanilide

**Materials:**

Benzoyl chloride
Aniline

**Reaction**


Aniline

Benzoyl chloride Benzanilide

**Procedure:-**

1-In a conical Flask fitted well, place aniline (2 ml) and sodium hydroxide solution 10% (30ml).

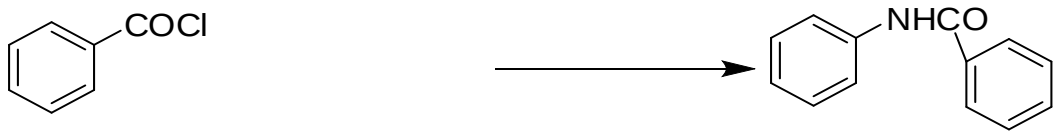
2-Add Benzoyl chloride (3ml) into it drop wise with frequent shaking for 10 min.

3- Filter the ppt., wash it with water, left the ppt till dryness.

5-Weigh the ppt., then calculate the percent.

6-Measure the melting point

**Calculation of Final Yield:-**




| $C_7H_5OCl$ | $C_{13}H_{11}ON$ |
|---|---|
| [7x12+5x1+16+35] | [13x12+11x1+16+15] |
| 140 | 198 |
| W=dv | X |

X= Theoretical yield = [W x 198] ÷ 140= gm.

**Practical yield**

Weight of filter paper= W1 gm.
Weight of filter paper + ppt= W2 gm.
Practical yield= weight of ppt= W2-W1=…… gm.

Final yield = practical yield ÷theoretical yield] x100= %

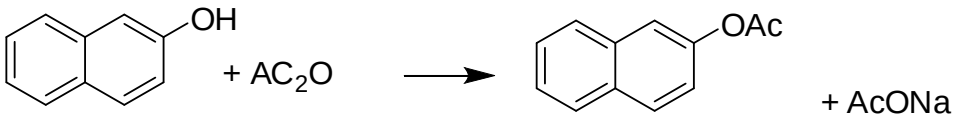
| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

## 7-Preparation of B-naphthyl acetate

**Materials:**

β -Naphthol
Acetic anhydride
Aq. Sodium hydroxide 10 percent

**Reaction**




**Procedure:-**

1-In a conical Flask place β -Naphthol (10g), add aqueous Sodium hydroxide 10 percent (50ml) into it and shake the mixture until all β -Naphthol dissolve.

2- Cool the solution in an ice bath and add into it crushed ice and then acetic anhydride (11.5ml).

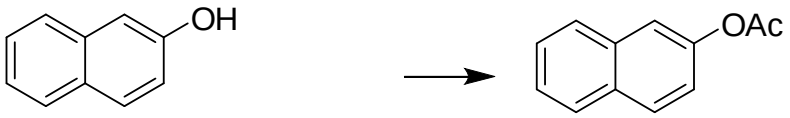
3- Shake the reaction mixture vigorously for about 30 min.

4- Filter the ppt., wash it with water, left the ppt till dryness

5-Weigh the ppt., then calculate the percent.

6-Measure the melting point

**Calculation of Final Yield:-**




| $C_{10}H_8O$ | $C_{12}H_{10}O_2$ |
|---|---|
| [10x12+8x1+16] | [12x12+10x1+16x2] |
| 144 | 186 |
| 10 | X |

X= Theoretical yield = [10 x 186] ÷ 144= gm.

**Practical yield**

Weight of filter paper= W1 gm.
Weight of filter paper + ppt= W2 gm.
Practical yield= weight of ppt= W2-W1=…… gm.

Final yield = practical yield ÷theoretical yield] x100= %

| | |
|---|---|
| **Theoretical Yield** | **gm** |
| **Practical Yield** | **gm** |
| **Final Yield** | **%** |

# Part two

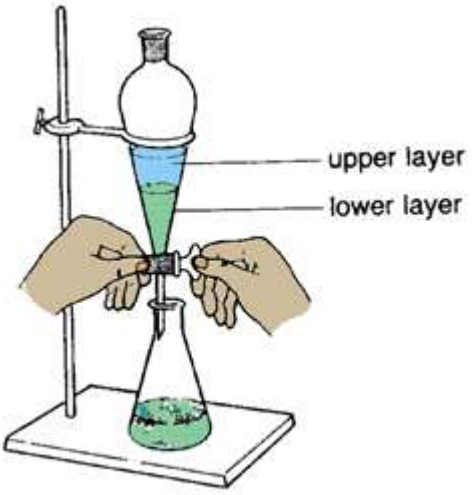
# Extraction of natural products

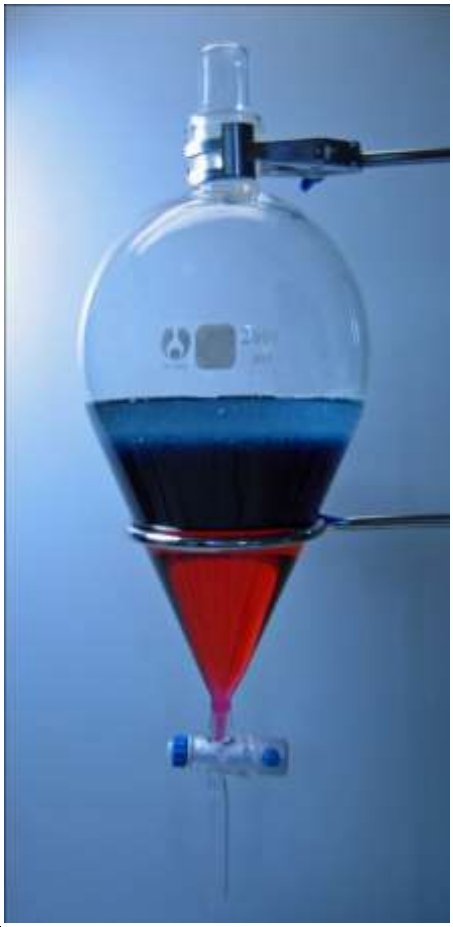
**Experimental Glossary: Extraction**

**1-separatory funnel**. It used to separate organic molecules out from those that are not organic .Example organic solvents (such as chloroform or ether) and aqueous solvents (such as H2O) are used. . In a separatory funnel, the organic solvent and its dissolved substances will form a distinguishable layer from the aqueous layer and its dissolved substances.

Whether or not the organic layer is above or below the aqueous layer in the separatory funnel depends on its density relative to the density of the aqueous layer. If chloroform and H2O are used, the organic layer will be below the aqueous layer because chloroform is more dense, therefore heavier, than H2O .Whereas ether is used and H2O are used, the organic layer will be above the aqueous layer because ether is less dense.

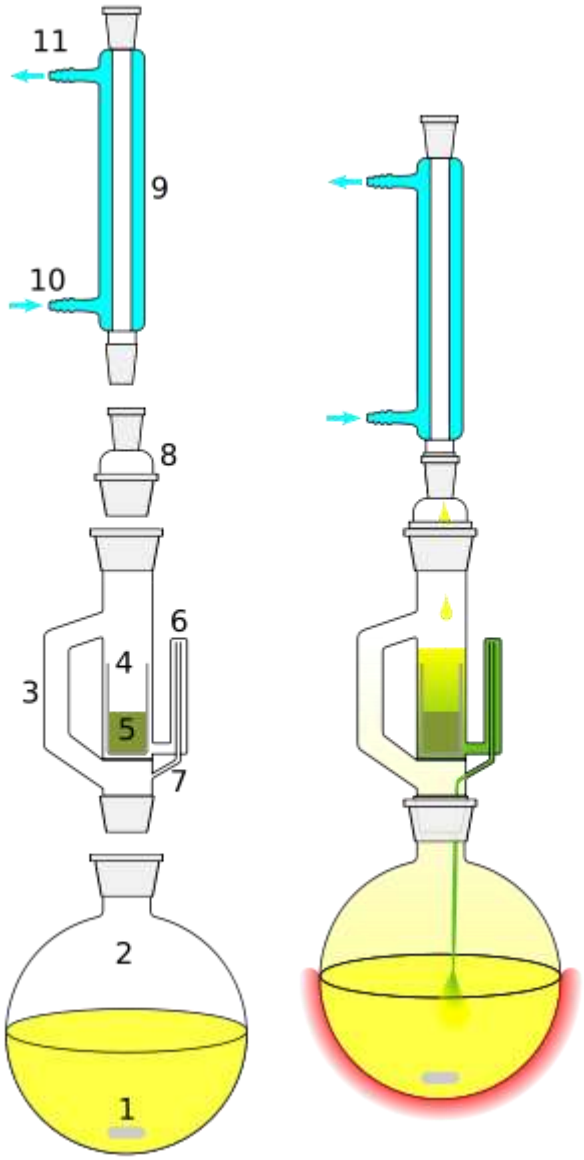
This apparatus can be seen in the diagram below.
Once the two layers are discernable, they can be released from the separatory funnel and collected into their respective containers. To ensure the purity of the extracted organic layer, extractions can be performed multiple times.



**2- Extraction apparatus, soxhlet**

2-A **Soxhlet extractor** is laboratory apparatus. Typically, a Soxhlet extraction is only required where the wanted compound has a *limited* solubility in a solvent, and the impurity is insoluble in that solvent.

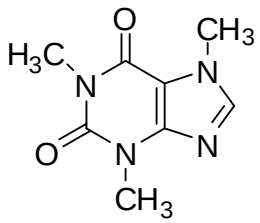



A schematic representation of a Soxhlet extractor
**1:** Stirrer bar **2:** flask **3:** Distillation path **5:** Solid **6:** Siphon top **7:** Siphon exit **8:** Expansion adapter **9:** Condenser **10:** Cooling water in **11:** Cooling water out

## 8- Extraction of caffeine from green tea

**Materials:**

Green tea
Calcium carbonate
Hexane
Calcium chloride




**Caffeine:** it is alkaloidal compound naturally occuring

**Procedure:-**

1-In a conical Flask place green tea (25gm) and water (150 ml) and calcium carbonate (10gm). Boil on hot plate for 20 min.

2-filter tea extract and let it till cool, place the extract in a separating funnel, then add hexane.

3- Shake the separating funnel and let it for few min. you will observe presence of two layers organic and aqueous layers.

4-Isolate organic layer from aqueous and repeat step 3 several times till complete extraction of caffeine from tea extract.

1- Filtere organic layer over anhydrous calcium chloride
2- Evaporate hexane till dryness , and weigh the residue ( caffeine).
3- Calculate the percent of caffeine.