Laboratory Task: Organic qualitative analysis, separation of compounds and preparation of derivative

At your work bench

Part I: Identification of functional groups in compounds A and B

▪ Droppers	4
▪ Cavity plate	1
▪ Chemicals	Compound A and B
	Sat. $NaHCO_3$,
	10% aq. NaOH,
	neutral $FeCl_3$ solution,
	Aq. $KMnO_4$,
	ammonical solution of $AgNO_3$.

Part II: Separation of the Given Mixture of Compounds A and B

▪ Beakers	2
▪ Dropper (plastic)	2
▪ Filter paper (circles)	1
▪ Funnel	1
▪ Glass rod	1
▪ Measuring cylinder (10mL)	2
(25 mL)	1
▪ Syringe (2mL)	1
▪ Wash Bottle	1
▪ Chemicals	Sat $NaHCO_3$, 30 mL
	1:1 HCl, 15 mL 
	pH paper
	Mixture of Compound A and B

Part III: Preparation of Derivative

- Conical Flask 2
- Cork 1
- Filter paper (circle) 1
- Funnel 1
- Glass rod 1
- Ice bath 1
- Measuring cylinder (50 mL) 1
  (10mL) 1
- Syringe (1mL) 1
- Wash Bottle 1
- Chemicals Acetone, 5 mL 
  Ethanol, 30 mL
  NaOH, 35 mL

Part IV: TLC

- Beaker 1
- Fusion Tubes 2
- Capillary 2
- TLC plate 1
- Watch glass 1
- Chemicals Pet Ether and Choloroform mixture (90:10)
  (solvent system for TLC)
  acetone (solvent for dissolution of sample)

Laboratory Task 3 (40 Marks)

Organic qualitative analysis, separation of compounds and preparation of derivative

Chalcones are 1,3-diphenyl-2-propene-1-one, in which two aromatic rings are linked by an aliphatic three carbon chain. There are important for a variety of. These are found in edible plants and are considered to be important in various biological compounds. It is also of great importance in pharmaceutical industry as a variety of novel heterocylcles can be designed.

Chalcones are generally coloured compounds because of the presence of the chromophore -CO-CH=CH-, which depends in the presence of other auxochromes. Chalcones and their derivatives are known for their anti-diabetic, anti-inflammatory, anti-parasitic, anti-histaminic, anti-malarial, anti-oxidant, anti-fungal properties.

The current experiment tests the student on various aspects of organic chemistry, such as functional group analysis, separation of two groups and their regeneration, synthesis and thin layer chromatography. Some of the skills are known to the students and some skills are learnt in the process of carrying out the experiment. The students also are asked to check the purity of the product synthesized using TLC technique.

Part I

You are expected to complete Part I of this task in first 30 minutes and return the paper to the lab expert. At the end of this part, you will be given the paper for part II.

Part I: Identification of functional groups in compounds A and B

You are given two compounds Compound A (molecular mass 122) and Compound B (molecular mass 106). Following reagents are given to you: Sat. $NaHCO_3$, 10% aq. NaOH, neutral $FeCl_3$ solution, aq. $KMnO_4$, ammonical solution of $AgNO_3$.

Conduct appropriate tests with the reagents provided to you and identify the functional groups present in compound A and B. Write your observations in the following table.

Compound A

Test	Observation	Functional group present

(3 marks)

Compound B

Test	Observation	Functional group present

(3 marks)

From your conclusions regarding the functional groups present in compound A and B, suggest a scheme for their separation from their mixture. (hint: one of the reagents supplied can be used for the separation).

(2 marks)

Part II: Separation of the Given Mixture of Compounds A and B

A mixture of compounds A and B is supplied to you in a 100 mL beaker. Add 20 mL of saturated $NaHCO_3$ solution to this mixture and stir the solution for 5 minutes (till effervescence ceases). Carefully, transfer the contents of the beaker to 25 mL measuring cylinder supplied to you. Use another 5 mL of saturated $NaHCO_3$ to transfer the content completely. Allow the two layers to separate clearly. Remove the organic layer, with the help of a syringe and transfer it to a clean 10 mL measuring cylinder. Note the volume of the organic layer (compound B). You will be using this layer for the preparation of new compound (see Part III).

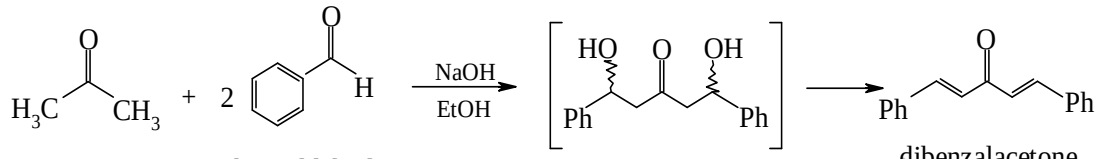
Volume of the organic layer (compound B)

Regeneration of compound A from the aqueous extract

Transfer the aqueous layer from the measuring cylinder to a 100 mL beaker. Add 50% HCl solution in small lots to the beaker with stirring till the solution is distinctly acidic (use pH paper). Note the volume of HCl added. Filter the precipitate (compound A) through filter paper. Use minimum amount of water for transferring and washing of the precipitate. The expert will collect your compound for drying.

Volume of acid needed for regeneration of compound A

The organic layer obtained in part II above (in the measuring cylinder) contains compound B, that is, benzaldehyde. You will be preparing dibenzalacetone from benzaldehyde. The reaction is presented below:



acetone benzaldehyde dibenzalacetone

The density of benzaldehyde = 1.04 g cm^{-3} and that of acetone = 0.787 g cm^{-3}.

Using the reaction given above and the density data, calculate the volume of acetone needed to be added for the preparation.

Volume of acetone needed for derivative preparation:

Part III: Preparation of Dibenzalacetone from benzaldehyde

To the organic layer in measuring cylinder, add the volume of acetone calculated by you with the help of a syringe supplied to you (Keep the tip of the needle of the syringe immersed in the organic layer while adding the acetone). Add 3 mL of ethyl alcohol to the same flask. In another conical flask, take 30 mL of 10 % NaOH and add 24 mL of ethanol to it. Stir the contents and keep the flask in ice bath for 5 minutes. With help of a syringe (used for transferring organic layer in part II), add half the quantity of benzaldehyde-acetone-ethanol mixture. Keep shaking the flask intermittently for about 15 minutes, without removing it from the ice bath. After this, transfer the remaining portion of benzaldehyde-acetone-ethanol mixture. Keep shaking the flask intermittently for another 15 minutes. Filter the product and wash it with 50 mL of water.

The laboratory expert will collect your products in Part II and III for drying.

Laboratory Task Answersheet

Part II

1 Mass of the empty butter paper ____ g

Mass of butter paper + compound A ____ g

Mass of compound A ____ g

(4 marks)

Part III

2 Mass of the empty butter paper ____ g

Mass of butter paper + dibenzalacetone ____ g

Mass of dibenzalacetone ____ g

(4 marks)

3 (a) Colour of dibenzalacetone ____

(b) Appearance

Crystalline ☐ Amorphous ☐

(Mark X in the correct box) **(1 mark)**

4 (a) Theoretical yield on the basis of the mass of benzaldehyde ____

(1 mark)

(b) The yield obtained as a percentage of theoretical yield.

____ %

5 Why is the reaction in Part III carried out in an alkaline medium?

(1 mark)

Part IV: TLC

Procedure for TLC

Dissolve a drop of benzaldehyde in a small quantity of acetone in a sodium fusion tube. Similarly prepare a solution of your product. Obtain a TLC plate from a laboratory expert. Draw a faint line, at a distance of about 1cm from the edge of the plate. Using a thin capillary tube, place a drop of the benzaldehyde solution on the line drawn on the plate. Allow it to dry. Then in a similar manner, spot the product solution on the same plate. Take care that the two spots do not merge into one another. Allow this spot also to dry. Then place the plate in the beaker, containing the eluant (supplied to you). Cover the beaker with a watch glass, and allow the solvent to rise appreciably (approximately 1 cm away from the top). Remove the plate from the beaker and mark the solvent front immediately. Mark the spots after exposing the plate to UV light (laboratory expert will help you for UV chamber). Calculate the R_f values using the formula given below and record the results.

$$R_f = \frac{\text{distance travelled by the compound}}{\text{distance travelled by the solvent front}}$$

a) R_f of benzaldehyde:

(3 marks)

b) R_f of dibenzalacetone:

(3 marks)

Submit your TLC plates to the expert before leaving the laboratory.