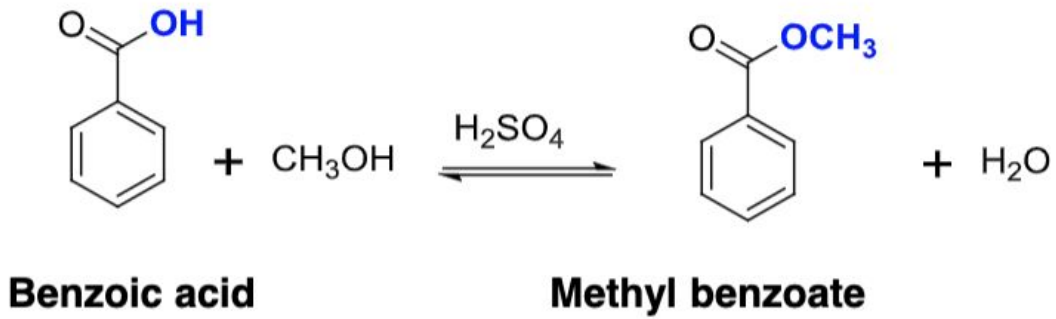
Exp 1: Preparation of methyl benzoate from benzoic acid

Magdalena Botrous, Yaseen Khdair, Emily Hyde, Max Shi, and Danielle Pierre

Purpose and Reaction

The purpose of this lab is to synthesize methyl benzoate from benzoic acid via fischer esterification. The reaction will be monitored using Thin Layer Chromatography.



Reagents

Name	**M.W.** (0.5 pts)	**Density** (0.5 pts)	**Amount (grams/mL)** (0.5 pts)	**Moles** (0.5 pts)	**Hazards/Precautions (MSDS data) and melting point or boiling point** (2 pts)	**Role of the reagent** (1 pts)*
Benzoic acid	122.12 g/mol	1.27 g/mL	2.5 g	0.0205 mol	Skin/eye irritation MP: 122.3C at 1atm	reagent
Methyl benzoate	136.15 g/mol	1.08 g/mL	2.787 g theoretical yield	0.0205 mol theoretical yield	Combustible liquid, harmful if swallowed MP: -15C at 1atm	Product

Reagents

Sulfuric acid	98.079 g/mol	1.83 g/mL	0.6 mL	0.0112 mol	Corrosive, eye damage, respiratory irritation, organ toxicity, reacts violently with water MP: 10C at 1atm	Catalyst
Methanol	32.04 g/mol	792 kg/m^3	8 mL	0.1978 mol	Flammable liquid and vapor, toxic by ingestion and inhalation, causes blindness or death if swallowed MP: -97.6C at 1atm	Reagent

Procedure

Exp 1_Methylbenzoate Preparation Procedure

1. Place 2.5 g of benzoic acid and 8 mL of methanol in a 25-mL round bottom flask.
2. Carefully pour 0.6mL of concentrated sulfuric acid down the wall of the flask.
3. Add boiling chips and reflux the mixture for 1 h using heating mantle (note: methanol boiling point is 64.7 0C at atmospheric pressure.
4. Cool the mixture to room temperature and pour into a separatory funnel containing 5 mL of water and 5 mL of dichloromethane.
5. Rinse the flask with 2-3 mL of dichloromethane and pour into separatory funnel.
6. Shake the mixture and separate the organic layer from the aqueous.
7. Wash the organic layer with 5 mL of water followed by 5 mL of 5% Sodium carbonate (base).
8. Note: Swirl the mixture gently at first without a stopper then insert the stopper and shake vigorously.
9. Separate and pour the organic layer into a Erlenmeyer flask. Note: Take TLC of the mixtures at 0, 30, 60 minutes and organic layer after Sodium carbonate wash.

TLC Procedure Procedure:

1. Take 4 vials.
2. Add ~ 1 mL of reaction mixture to vial or tube at the time points mentioned in the table below.
3. Take a TLC plate and draw line 1 cm from both top and bottom.
4. Mark 4 dots (spots) with a pencil on one of the lines.
5. Spot 1 reaction mixture at 0 minute (before you start heating)each of the three Calculate the Rf of each spot.
6. Let it dry in air for few minutes before you put in TLC chamber for development.

How do we know we have prepared the methyl benzoate?

1. Take known benzoic acid and methyl benzoate solution (already prepared) in a beaker/tube or vial.
2. Take a TLC plate and draw line 1 cm from both top and bottom.
3. Mark 3 dots (spots) with a pencil on one of the lines.
4. Put Spots 1, 2 and 3 as mentioned in the table below.
5. Let it dry in air for few minutes before you put in TLC chamber for development.
6. Develop the TLC plate and compare spots on 3 with 1 and 2.

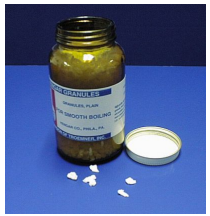
Stepwise Procedure

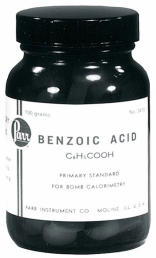
1. Place 2.5 g of benzoic acid and 8 mL of methanol in a 25-mL round bottom flask.

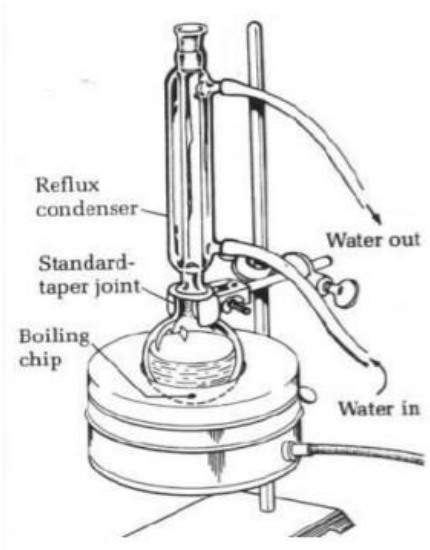
2. Carefully pour 0.6mL of concentrated sulfuric acid down the wall of the flask.

Stepwise Procedure

3. Add boiling chips and reflux the mixture for 1 h using heating mantle (note: methanol boiling point is 64.7 0C at atmospheric pressure.

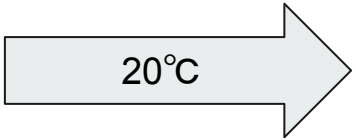


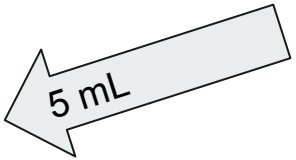


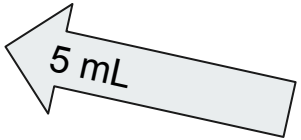


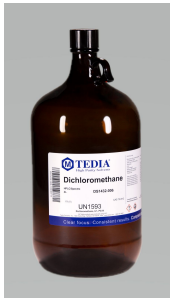
Stepwise Procedure (cont.)

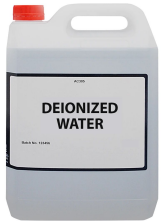
4. Cool the mixture to room temperature and pour into a separatory funnel containing 5 mL of water and 5 mL of dichloromethane.





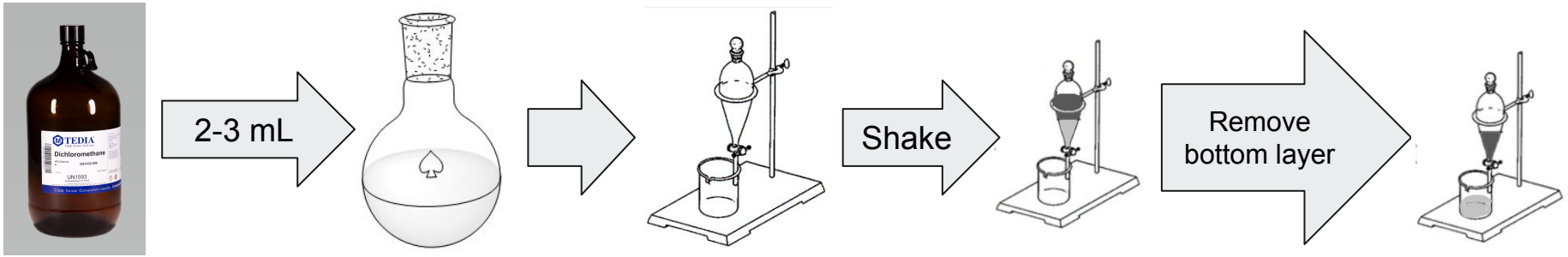






Stepwise Procedure (cont.)

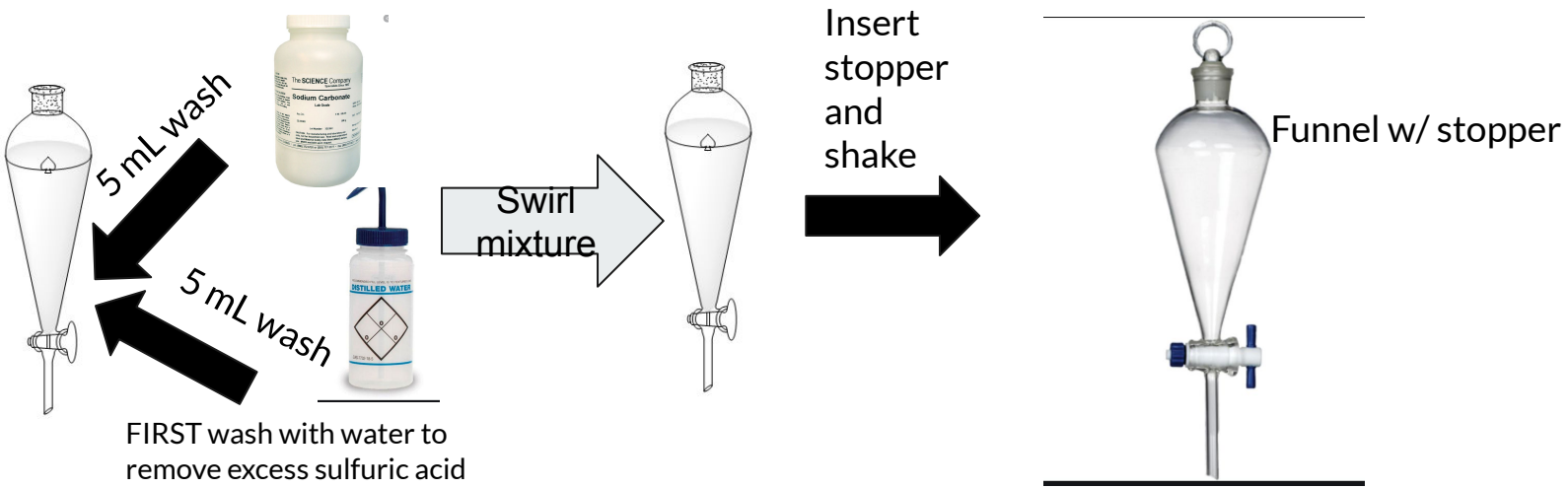
5. Rinse the flask with 2-3 mL of dichloromethane and pour into separatory funnel.
6. Shake the mixture and separate the organic layer from the aqueous.



Stepwise Procedure (cont.)

7. Wash the organic layer with 5 mL of water followed by 5 mL of 5% Sodium carbonate (base).
8. Note: Swirl the mixture gently at first without a stopper then insert the stopper and shake vigorously.

SECOND wash with sodium bicarbonate to remove remaining excess sulfuric acid and benzoic acid

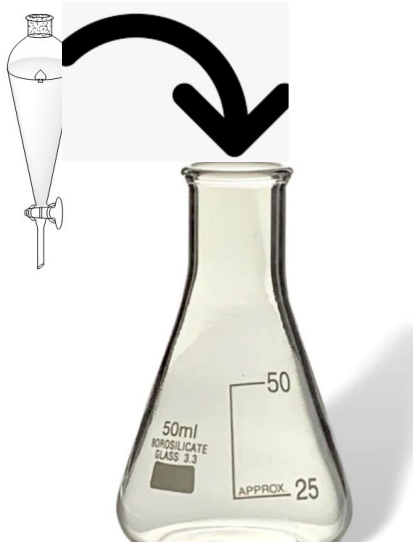


FIRST wash with water to remove excess sulfuric acid

Stepwise Procedure (cont.)

9. Separate and pour the organic layer into a Erlenmeyer flask. Note: Take TLC of the mixtures at 0, 30, 60 minutes and organic layer after Sodium carbonate wash.

Flask containing organic layer of previous mixture

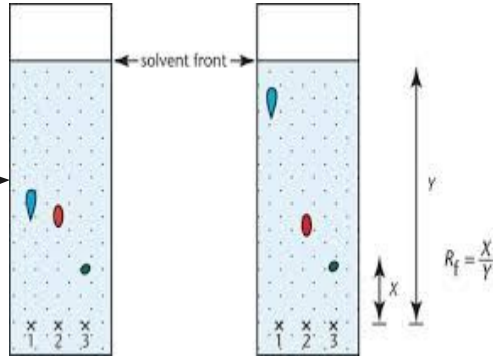


Take TLC at 0,30,60 minutes

1. Take known benzoic acid and methyl benzoate solution (already prepared) in a beaker/tube or vial.
2. Take a TLC plate and draw line 1 cm from both top and bottom.
3. Mark 3 dots (spots) with a pencil on one of the lines.
4. Put Spots 1, 2 and 3 as mentioned in the table below.

Beaker containing benzoic acid and methyl benzoate solution

Draw line 1 cm from top and bottom of TLC paper and make 3 spots



Allow the solvent to reach the top pencil line by capillary action

5. Let it dry in air for few minutes before you put in TLC chamber for development.
6. Develop the TLC plate and compare spots on 3 with 1 and 2.
7. Repeat TLC steps with 4 spots of reaction mixture at 0, 30, 60 min of heating and after sodium bicarbonate wash

Let air-dry before putting into TLC chamber for development

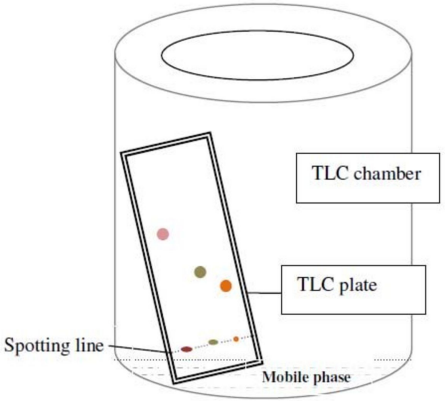
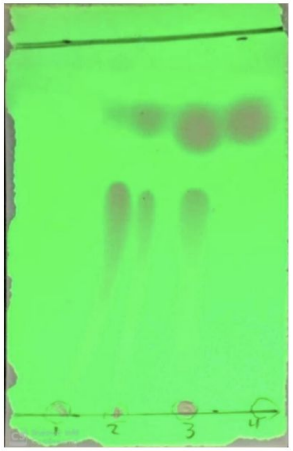
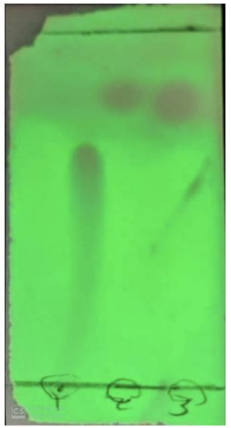


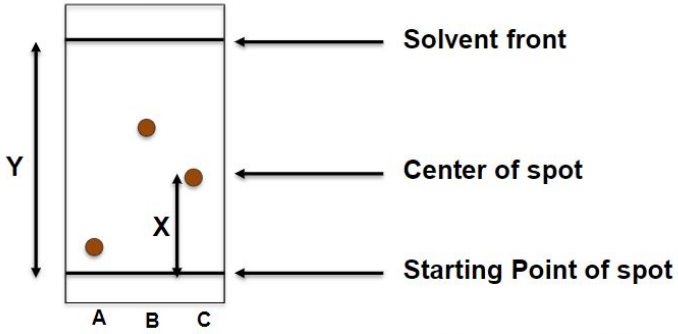
Plate 1



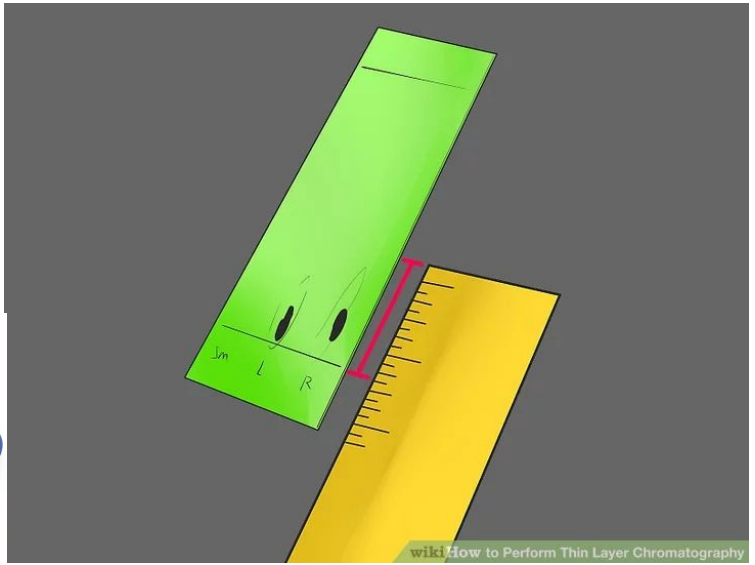
Develop TLC and compare Rf values of each spot and repeat with 4 spots of reaction mixture after 0, 30, and 60 minutes of heating, and after sodium bicarbonate wash

Plate 2





Rf (spot C) = X/Y



Measure solvent front distance and distance travelled by the compounds and calculate Rf

Results: Plate 1

TLC Results:

Time	TLC plate 1 (Spot number)	Rf Values	Benzoic acid	Methyl benzoate
0 min	1	Rf_1	0.64	0.82
30 min	2	Rf_2	0.62	0.82
60 min	3	Rf_3	0.60	0.84
Post-Wash	4	Rf_4	0	0.84

Plate 1

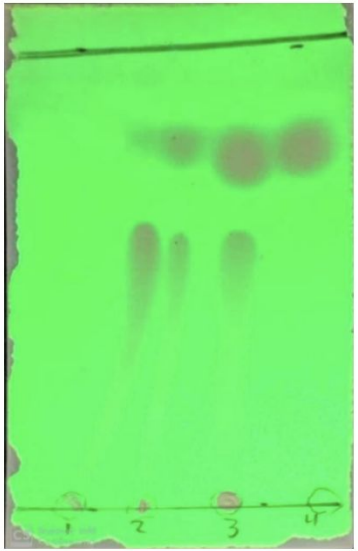


Plate 2

Results: Plate 2

Distance traveled by solvent: 5cm

Spot 1: distance traveled by benzoic acid = 3.25cm

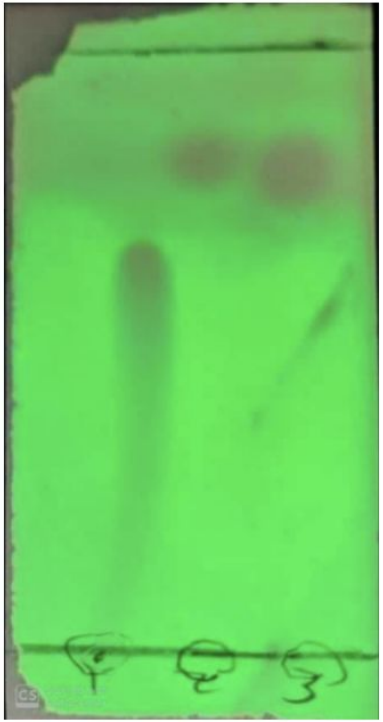
Rf = 3.25/5 = 0.65

Spot 2: distance traveled by methyl benzoate = 4.25cm

Rf= 4.25/5 = 0.85

Spot 3: distance traveled by experimental = 4cm

Rf = 4/5 = 0.80



Conclusion

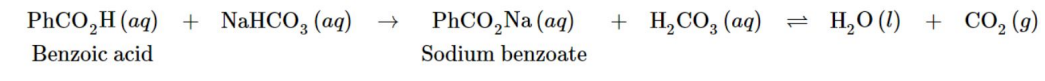
This lab demonstrated the preparation of methyl benzoate from benzoic acid and methanol using sulfuric acid as a catalyst. This reaction is an example of a fischer esterification reaction and the results are shown using thin layer chromatography. The TLC results show that the benzoic acid has a lower Rf value due to its higher polarity than the methyl benzoate product. There were no issues during the experiment. There are no further recommendations, since the lab was conducted remotely. A practical application for the fischer esterification reaction is to convert carboxylic acids in the presence of alcohol and a strong catalyst to form an ester. This is a typical practice for creating volatile esters used in perfumes and cosmetics, as well as solvents for paints, lacquers, and varnishes.

Post-Lab Questions

1. Sodium bicarbonate solution can react with benzoic acid and bring it to the aqueous layer from the organic layer. Provide an explanation and the reaction between benzoic acid and sodium bicarbonate. (1 point)

An acid-base extraction can be used to extract carboxylic acids from the organic layer into the aqueous layer. Sodium hydroxide can be used to convert a carboxylic acid into its more water-soluble ionic carboxylate form. However, if the mixture contains a desired compound that can react with sodium hydroxide, a milder base such as sodium bicarbonate should be used. By using sodium bicarbonate, the byproduct (carbonic acid) can decompose to water and carbon dioxide gas.

The equation is as follows:

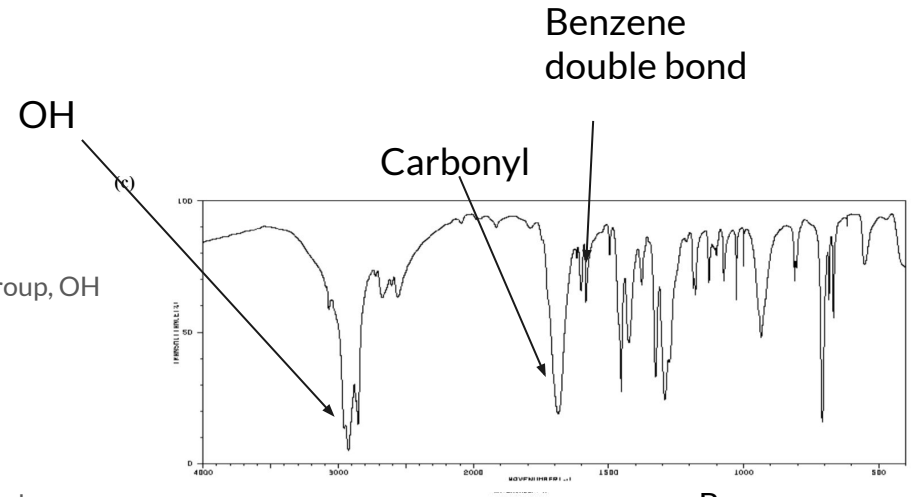


Post Lab Questions

2.

Which of the following is an IR of the benzoic acid. Label Carbonyl group, OH and benzene double bond in the IR of the benzoic acid. (1.5 points)

Option C



Which of the following is an IR of the methyl benzoate. Label Carbonyl group, OCH3 group and benzene double bond in the IR of the methyl benzoate (1.5 points) Note: IR are given in the next page.

Option B