



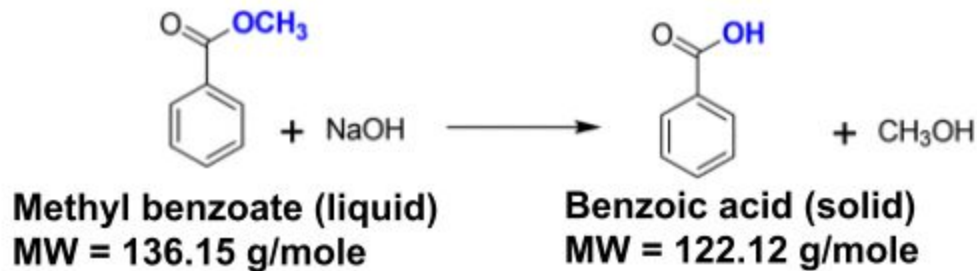
CH 246

Exp 8: Hydrolysis of Ester

I pledge my honor that I have abided by the Stevens Honor System.
Magdalena Botrous, Max Shi, Ahmed Ghani, and Logan Buddenbaum

Purpose and Reaction

The objective of this experiment is to prepare benzoic acid from methyl benzoate and sodium hydroxide via the hydrolysis of an ester.



Reagents

Name	M.W.	Density	Amount (grams/mL)	Moles	Hazards/Precautions (MSDS data) and melting point or boiling point	Role of the reagent
Methyl benzoate	136.15 g/mol	1.08 g/mL	2.5 g	0.0184 mol	BP: 199 C at 1atm may irritate skin, eyes, and digestive tract; slightly toxic by ingestion	Reagent
2M aq. NaOH	39.997 g/mol	2.13 g/mL	18.4 mL (double of what is needed to drive reaction)	0.0368 mol (double of what is needed to drive reaction)	very corrosive, irritation to eyes, skin, and mucous membrane, eye and skin burns BP: 1388C at 1atm	Reagent
Benzoic acid	122.12 g/mol	1.27 g/mL	2.247 g formed	0.0184 formed	Eye damage, irritation of the skin, rash, redness, burning feeling, irritation to nose, throat, and lungs if inhaled BP: 249.3C at 1atm MP: 122.3C at 1atm	Product
Methanol	32.04 g/mol	0.792 g/mL	10mL (used as solvent)	0.247 mol	Highly flammable, toxic, direct ingestion of more than 10mL can cause permanent blindness MP: -114.1C at 1atm BP: 78.37C at 1atm	Solvent and Product
HCl	36.458 g/mol	1.2 g/mL	-	-	Highly corrosive; will cause skin burns; vapors are dangerous and deadly to inhale BP: -85.05C MP: -114.2C	Reagent



Procedure

1. Dissolve 2.5 g methyl benzoate in 10 mL methanol in round bottom flask.
2. Add 18.4 mL NaOH to round bottom flask, and set up reflux apparatus. Add stir bar. Take small sample of solution for TLC.
3. Heat reaction to reflux for 30 minutes. Take sample of solution for TLC.
4. Cool reaction mixture and add to separatory funnel.
5. Add 10 mL dichloromethane and 10 mL water to separatory funnel. Extract aqueous layer (top layer).
6. Add hydrochloric acid to aqueous layer to precipitate benzoic acid. Add until pH on pH paper shows a reading of 3.
7. Perform vacuum filtration and wash with water.
8. Dry product and weigh.
9. Take TLC to confirm formation of benzoic acid product.

Stepwise Procedure

1. Dissolve 2.5 g methyl benzoate in 10 mL methanol in round bottom flask.
2. Add 18.4 mL NaOH to round bottom flask, and set up reflux apparatus. Add stir bar. Take small sample of solution for TLC.

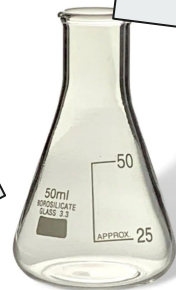


Sodium Hydroxide

18.4 mL



Cool to room temperature

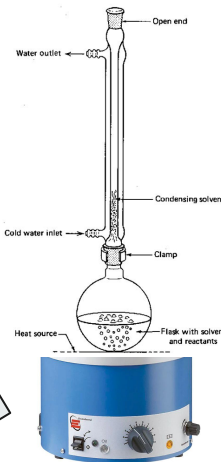


With swirling

Add contents

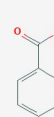


10 mL



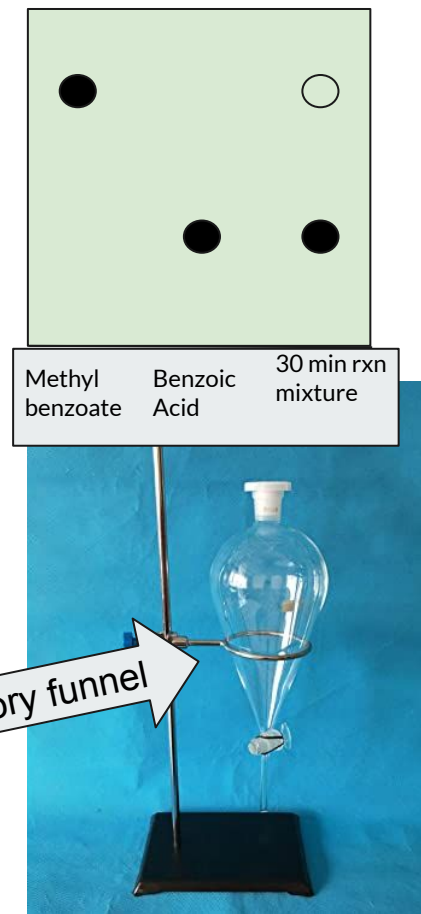
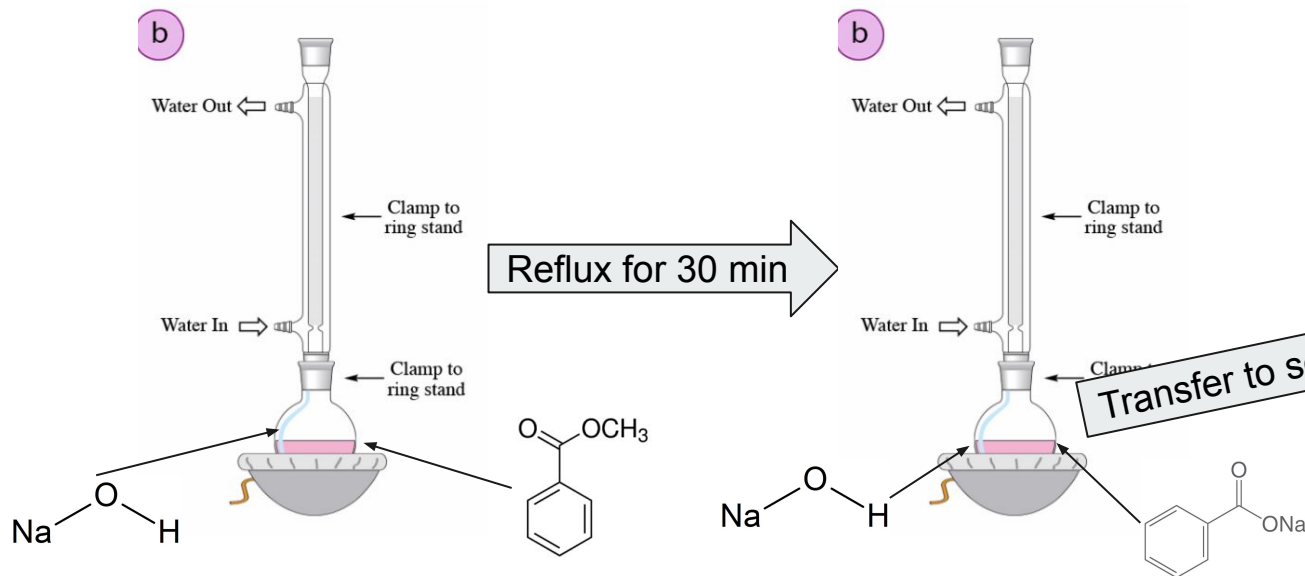
2.5 g

Methyl benzoate



Stepwise Procedure

3. Heat reaction to reflux for 30 minutes. Take sample of solution for TLC.
4. Cool reaction mixture and add to separatory funnel.



Stepwise Procedure

5. Add 10 mL dichloromethane and 10 mL water to separatory funnel. Extract aqueous layer (top layer).
6. Add hydrochloric acid to aqueous layer to precipitate benzoic acid. Add until pH on pH paper shows a reading of 3.

The diagram illustrates the stepwise procedure for precipitating benzoic acid. It begins with a separatory funnel containing a mixture of dichloromethane (CH₂Cl₂) and water. Arrows indicate the addition of 10 mL of dichloromethane and 10 mL of water. The aqueous layer (top layer) is extracted. The extracted aqueous layer is then added to a beaker. Hydrochloric acid (HCl) is added to the aqueous layer until the pH is 3, as indicated by the text "Add until pH=3". The pH is monitored using pH paper, as indicated by the text "Use pH paper". The resulting precipitate is benzoic acid, as indicated by the text "Precipitate benzoic acid". The final product is shown in a beaker.

Chemical structures shown:

- Dichloromethane: ClCCl
- Water: O
- Sodium hydroxide: [Na]OH
- Sodium benzoate: c1ccccc1C(=O)[O-][Na+]
- Hydrochloric acid: Cl

Labels in the diagram:

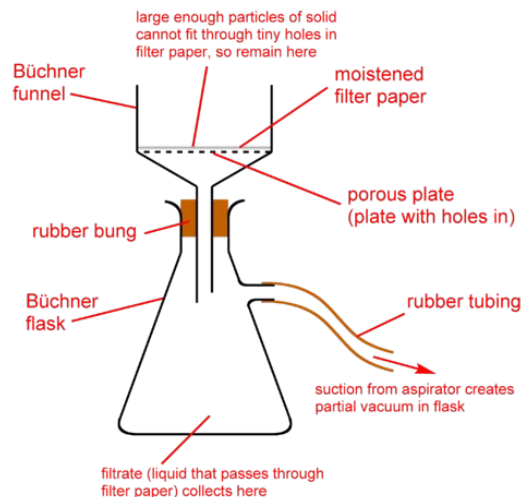
- 10 mL
- 10 mL
- Extract top layer
- Add until pH=3
- Use pH paper
- Precipitate benzoic acid

Stepwise Procedure

7. Perform vacuum filtration and wash with water.
8. Dry product and weigh.



Vacuum filtrate
+ wash with
water



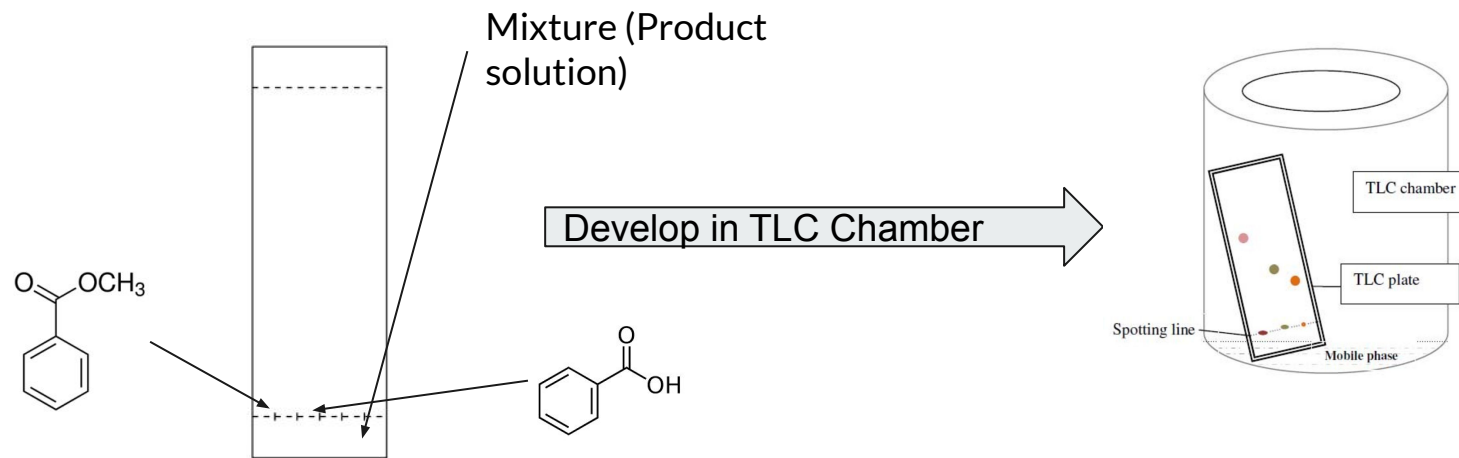
Let product dry at room
temperature and then weigh



Benzoic acid, (excess) HCl, and
sodium chloride

Stepwise Procedure

9. Take TLC to confirm formation of benzoic acid product.



20% ethyl
acetate/hexane

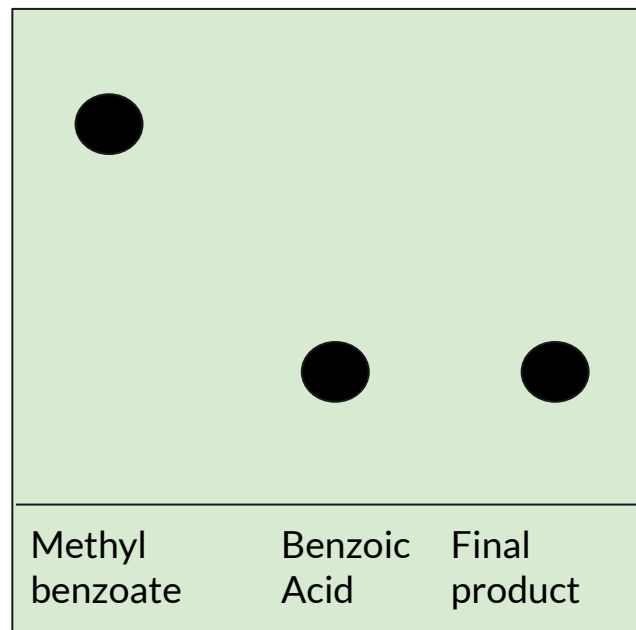
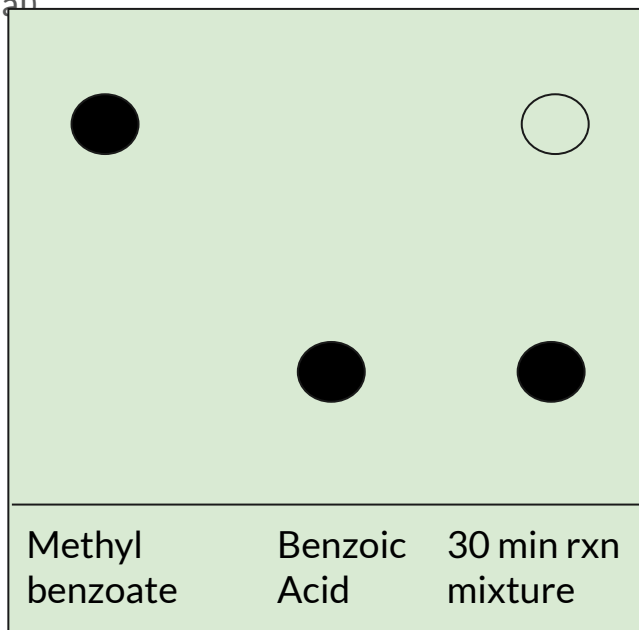
Results

	Benzoic Acid	Methyl Benzoate	Final Product
Rf value	0.37	0.84	0.37

- 1) % Yield of Benzoic Acid: 1.124 g = 50% yield
- 2) TLC results below/left (after 30 minutes) and to the right (final)

Final TLC results:
product is benzoic acid

TLC results after 30 minutes:
product is split between benzoic acid and methyl benzoate, but mostly benzoic acid





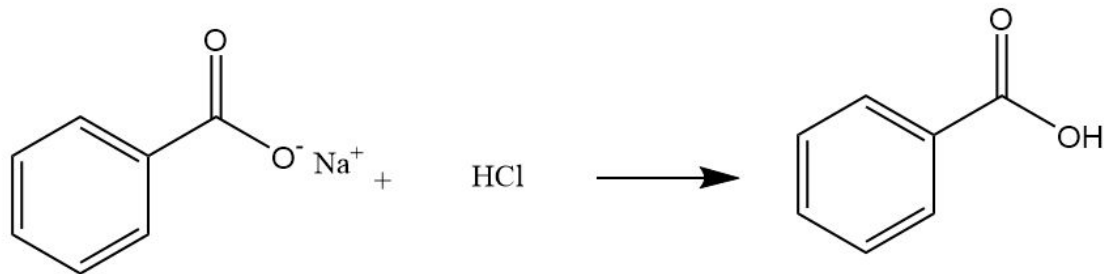
Conclusion

This lab demonstrated the preparation of benzoic acid and methanol from sodium hydroxide and methyl benzoate via a hydrolysis of an ester reaction. Spectroscopy was conducted to view the spectra of the methyl benzoate and benzoic acid. The results of the experiment were seen using Thin Layer Chromatography, or TLC. As expected, it was observed that the benzoic acid product had the lower R_f value, and any remaining unreacted methyl benzoate would have the higher R_f value. This result makes sense because of the relative polarities of the two compounds. There were no issues during the experiment. There are no further recommendations, since the lab was conducted remotely. A practical application for this type of reaction is saponification, which is a reaction used to prepare soaps from fats and oils.

Post-Lab Questions

(1) What was the role of concentrated HCl in the procedure? Draw the reaction happened after addition of HCl. (2 point)

The role of HCl in this procedure was to protonate the sodium benzoate and allow for the formation of benzoic acid.



Post Lab Question



(2) Based on Exp 1 and Exp 8 you performed, describe the reactions to convert methyl benzoate to ethyl benzoate. Need two steps.(2point)

Step 1:

Take methyl benzoate and perform a hydrolysis reaction by adding NaOH, along with separating the products and protonation of the potassium benzoate to form benzoic acid.

Step 2:

Take the benzoic acid and perform a fischer esterification reaction using the benzoic acid formed in Step 1, ethanol, and sulfuric acid to get ethyl benzoate.