Experiment 3. Extraction

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Section: C02
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Procedure:

Refer back to pages 10 to 28 of the lab manual

Dr. Tony Durst et al. “Organic Chemistry Laboratory Manual 2018.”

Observations

Part A:

Extraction with 0.006M Methylene blue

- Separation was not easily visible to the eye
- Blue dye was observed to be in the aqueous layer in the test tube

Extraction with 0.006M Methyl red

- Separation was easily visible, clear solutions
- Red dye was seen in the organic layer

Both 0.006M methylene blue and 0.006M Methylene red

- Separation easily visible
- the red portion was at the top layer and the blue was at the bottom of the test tube

Crystal Violet and 1-Butanol(without salt)

- Solution was purple and clear
- Test tube felt cold

With 4.96 g of salt

- Separation was well seen as the to layer was very thin
- Top layer was a clear dark purple while the bottom layer was clear and colourless with the salt at the bottom

Part B:

0.73g Unknown 1, 10 mL dichloromethane

- The NaOH solution was seen at the top layer in the test tube
- Top solution was a little more clear than the bottom solution
- The organic solution was seen at the bottom of the test tube
- Gas escaping during venting made a sound
- If the stopper is left while extracting, the solution bubbles

HCl added to aqueous extracts

- White precipitate formed
- Litmus paper turns from blue to red

When in ice bath

- The solution was clumpy, white and had a jelly like texture

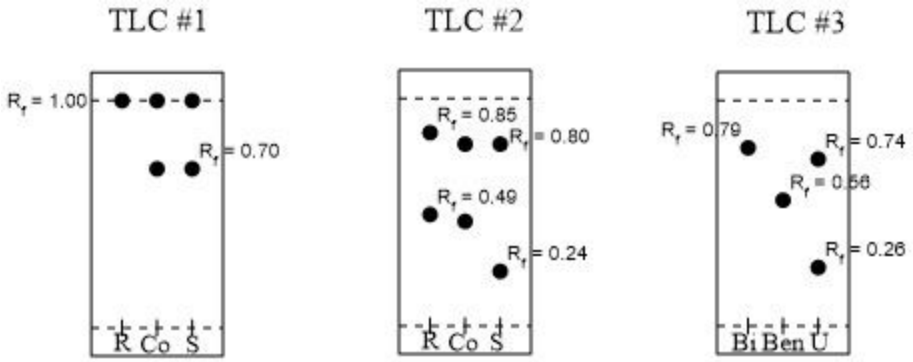
When filtering

- Water overflowed because the tube was connected to the wrong pipe
- Still a lot of product on filter paper seen

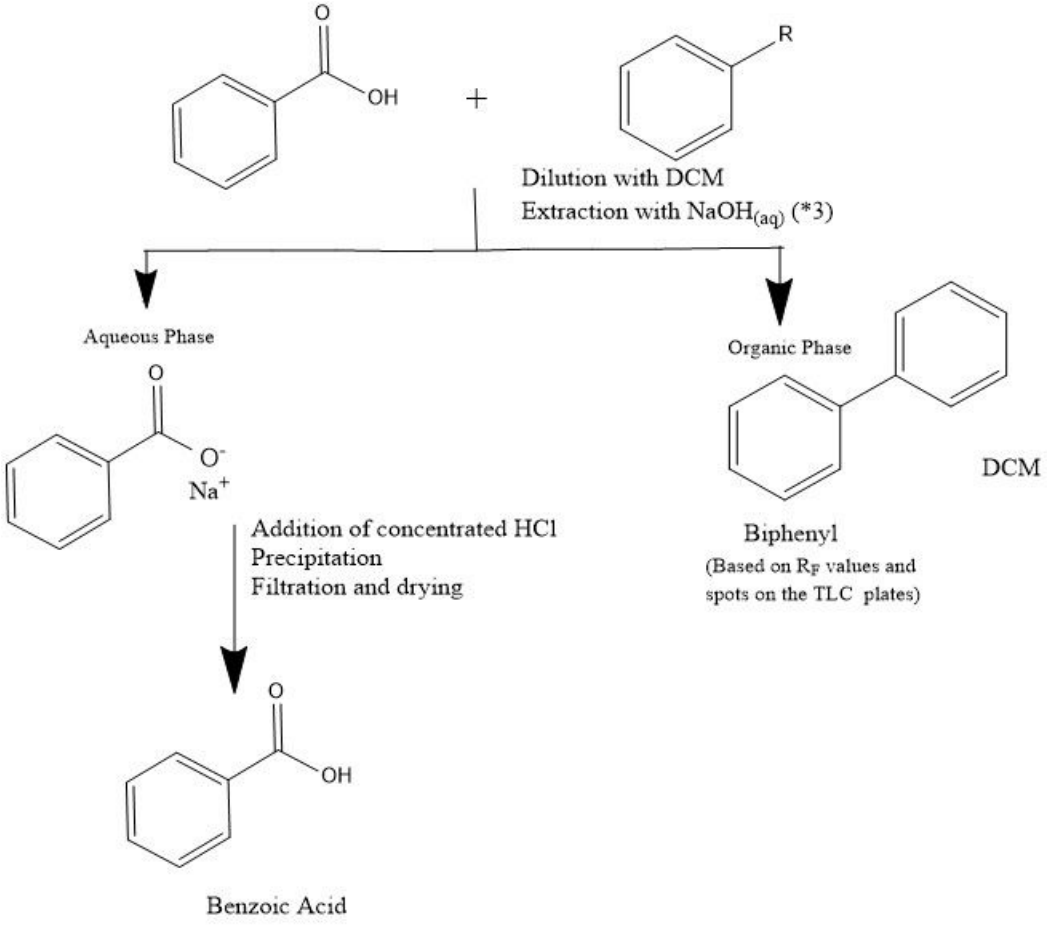
TLC Plates

- First TLC plate the organic solution was used as the reference point
- All the spots lined up and have approximately the same R_F value

- For the second plate the aqueous extract was used as the reference point
- Each spot made a streak going up the plate
- Also has a good separation between the spots
- For the third TLC plate, biphenyl and benzophenone were used as reference spots
- the separations were not as good
- The compound also left streaks while going up the TLC plate
- The unknown and the biphenyl spots lineup and have the same R_F



Flow Chart



Table

Unknown Number	Initial mass of watch glass (g)	Mass of Watch glass and product (g)	Total mass of product collected (g)	\Composition% (percent yield)
1	46.17	46.31	0.14	264.29%

Calculations

Part A:

R_F: 1st TLC plate

Total distance: 3 cm

Reference Point

Spot 1= 3cm

R_F= 3/3= 1 cm

Spot 2= 2.1 cm

R_F= 2.1/3= 0.7 cm

Part B:

Total product collected= (Mass of watch glass+product)-mass of watch glass

Total mass of product collected= 46.31g-46.17g= 0.14g

Percent Yield= (actual yield/ experimental yield)*100

Actual yield: 0.37

Experimental yield: 0.14g

Percent yield= (0.37g/0.14g)*100= 264.29

Discussion:

- In part A for the extraction with methylene blue, the separation was not easily visible to the eye because methylene blue has atoms that hydrogen bond with water which allows it to dissolve with water and settled in with the aqueous solution
- For the extraction with methyl red, the red dye was seen in the organic layer. This is because it has nonpolar bonds that counter the interaction between the molecules that hydrogen bond with water.
- When both solutions were mixed together, the separation was easily visible because the solutions were immiscible and therefore didn't mix
- When the crystal violet was mixed with the 1-butanol solution, the solution was clear and purple because crystal violet is soluble in both 1-butanol and water which won't cause a separation.

- The violet crystal was pushed towards organic phase when the salt NaCl was added because the organic compound that was slightly soluble in the aqueous phase would be pushed towards the organic phase due to the ionic strength caused by the NaCl (makes it more polar)
- This is a technique called salting out, which is used when an extraction is inefficient due to the organic compound being partly soluble in water.
- Salting out pushes the organic compound out of the aqueous layer since it is saturated by NaCl. This is why the separation was well seen.

Part B:

- Point of this was to do a separation of biphenyl and benzoic acid
- When 0.73g of the unknown was mixed with 10 mL of dichloromethane, the NaOH solution(aqueous) was well separated from the organic solution. This is because aqueous and organic phases are immiscible solvents
- Sound was heard when venting because that was the gas being vented which is completely normal.
- When the funnel was being shaken, NaOH reacted with benzoic acid in the organic phase and converted to sodium benzoate
- When the stopper was removed in the separatory funnel, there was a cloudy layer in the middle which is called an emulsion which is a mixture of the top and bottom solutions and disappeared because the blobs separated.
- The organic layer was seen at the bottom layer because chlorinated solvents like dichloromethane are denser than water and would be seen at the bottom.
- If a solvent is less dense than water then they will form the layer at the top
- When HCl was added to the aqueous extracts, it converted the solution back to benzoic acid
- White precipitate formed when the HCl was added because of this conversion of acidifying the aqueous phase. This is also the reason why the litmus paper turns from blue to red. Showing that it has been acidified.
- After the solution was placed in a iced bath, it was filtered. While filtering water overflowed in the flask due to connecting the tube to the wrong pipe and some product was lost. This is why the percent yield is so high.
- After the extraction process, there was still some water in the organic solution. The water was removed by a drying agent which is a salt that forms hydrated crystals to the organic phase.
- These hydrated crystals are what was filtered off.
- All three of the TLC plates helped prove that biphenyl was in the unknown.
- The Rf of the biphenyl and the unknown were approximately the same value on the third TLC plate. This shows that the unknown contains biphenyl.

- The first TLC plate showed that the unknown solution contained the organic portion since it lined up with the spot for the sample column.
- The first plate also showed that the unknown mixture contains another solution(the second spot at the bottom of the plate).
- The second plate proved that the unknown mixture also contained the compounds in the aqueous layer.
- This was proved because the sample spot was the aqueous solution(benzoic acid) and that spot had approximately the same Rf as the second spot on the unknown.
- This is the same spot that was also seen on the first TLC plate
- In conclusion, the organic portion that was in the unknown mixture was proven to be biphenyl as seen in the third TLC plate as well as the Rf values.

Questions

1. **Why would it be difficult to perform an extraction using ethanol and water?**

- The two solvents must be immiscible because the compound to be extracted must have a higher solubility in one solvent relative to the other
- Water should be used as the polar phase but ethanol is also polar which means “like dissolves like” and they would be miscible
- Once the two miscible solvents are used, separation can not occur and no layers would be formed
- Possess similar chemical properties so extraction and other techniques such as distillation would be difficult

2. Would adding NaCl to a test tube containing water, ether and methylene blue increase or decrease the amount of dye in the aqueous layer?

- It would decrease the amount of methylene blue in the aqueous layer because water would dissolve the salt
- Also due to the salting out effect it would repel and push out the methylene blue from the aqueous layer.
- In similarity with to the crystal violet, where the salting out technique increases the ionic strength of the water and pushing the organic compound out of the aqueous layer

3. Compound Y has a solubility of 2.0g/100ml in water and 20.0 g/100ml in ether. What mass of compound Y would be removed from a solution of 1.8g of Y in 100 ml of water by a single extraction with 100 ml of ether?

KD= [Y] Organic/ [Y]Aqueous

KD=10

[Y]Aqueous= 0.02g/mL

[Y] Organic=0.2g/mL

$$10 = \frac{W_1/V_1}{W_2/V_2}$$

Let W_1 and W_2 equal the masses of the compounds dissolve in volumes V_1 and V_2 of solvents 1 and 2.
W_2=1.8g
10= (W_1 /100ml)/ (1.8g/100ml)
W_1 =18.0/11.0= 1.64
1.64 g of compound Y will be removed by a single extraction with 100 mL of ether.

4. What mass of Y would be removed from the original water solution in question 3 by two extractions using 50 mL of ether each time? KD= 10
1st extraction: W_1 is the mass of compound Y removed after extraction 1
KD= [Y] Organic/ [Y]Aqueous
KD=10
10=(W_1 /50)/(W_2/100)
5=W_1 /1.8-W_1
9=6W_1
W_1 =1.5
2nd Extraction: W_1 is the mass of compound Y removed after extraction 2
10= (W_1 /50mL)/(W_2 /100mL)
5=W_1 /(0.3-W_1)
1.5=6W_1
W_1 =0.25g
The total mass removed of compound Y is 1.75g. Since 1.5g was removed during the first extraction and 0.25g was removed during the second one.
5.During an extraction a student loses track of which layer is the organic layer. How could she determine which layer is the aqueous phase?

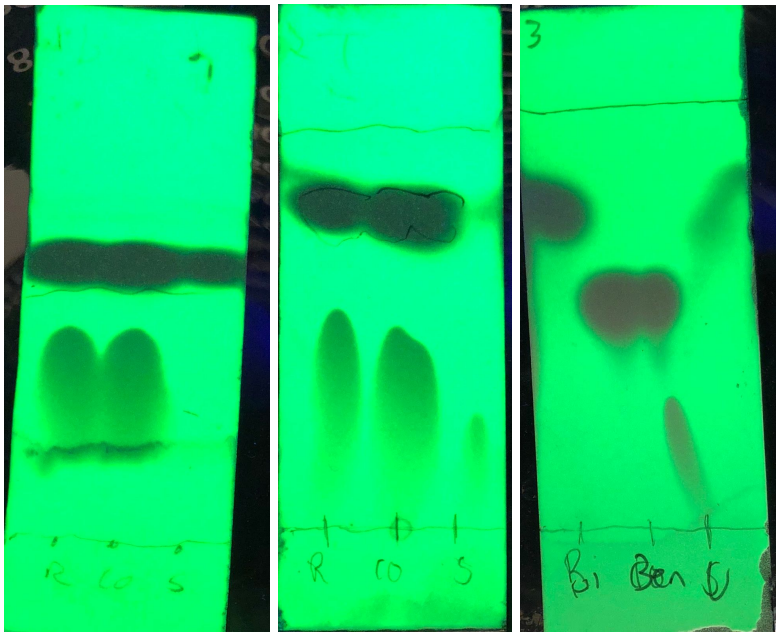
- Perform a water test by adding a few drops of water and if it is miscible it is the aqueous layer and if it is immiscible it is the organic layer

6. Describe how you would separate a mixture of benzyl amine (an organic base) and naphthalene. Both compounds are insoluble in water and soluble in ether.

- Benzyl amine and its salt has different solubility. The salt of benzyl amine is soluble in water. Therefore adding an acid would turn benzyl amine into its salt.
- Once this is done, the separations of the layers can be done using ether and water
- Then to recover the benzyl amine, we add a base to the benzyl amine salt
- This will cause it to precipitate out, then proceeding with filtering out the water

Raw Data

TLC Plates:



References

Dr. Tony Durst et al. “Organic Chemistry Laboratory Manual 2018.”

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Experiment 3.

	observations
0.006 M methylene blue	dye is in the both aqueous layers
0.006 M methyl red	dyes in ether so the organic layer.
when both mixed	good way to seperate. red at top blue at bottom.

4.

Crystal violet + 1-butanol (without salt)	Purple, clear., cold
With salt (4.96 g),	seperated top layer thin Purple and, bottom layer is clear

Part B.

0.13g Unkown 1: 10 ml dichloromethane
bottom is organic { NaOH on top, top solution is a little less clear than bottom solution
gas escaping during venting made sand.

2nd time reusing.
- same observations
- bubbles if stopper left in

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160(0.05)

Adding HCl (8ml) to aqueous extracts
observations
- pH, white
- litmus paper turned red from blue

When in Ice Bath.

Clumpy, white, jelly texture

When filtering water overflowed bc connected wrong tube

watch glass: 46.17 g.

→ had enough product on filter paper

watch glass + product = 46.31

|Δ| = 0.14 g

↑ total product collected

TLC PLATES

1st sample - bottom layer
2nd sample - top layer

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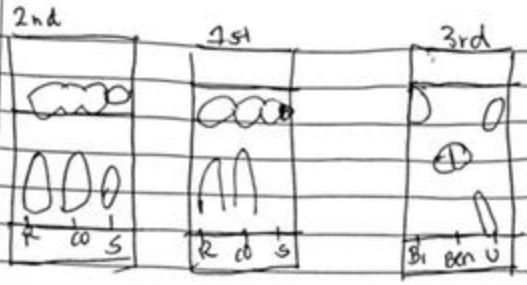

RTs

2nd
Td= 4.1 cm
R=
① 3.5/4.1
② 2.0/4.1cm
CO:
① 3.3/4.1
② 1.9/4.1
S:
① 3.3/4.1
② 2.0/4.1

1st
Tk = 3 cm
R
① 3.0/3 = 1
② 2.1 cm/3 cm
CO
① 3/3 = 1
② 2.1/3
S:
① 3/3 = 1
② 2.1/3

3rd
Td. 4.3
R-Bi
① 3.4/4.3
CO-Bcn
① 2.4/4.3
S-UNC.
1.1/4.3
3.2/4.3