Organic Chemistry Lab 3: Extraction

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Procedure

The procedure was followed as it was described in the Introductory Organic Chemistry Lab Manual (CHM1321 Introductory Organic Chemistry Laboratory Manual 2018, Dr. Tony Durst, Dr. Tito Scaiano, Dr. William Ogilvie, and Dr. Alison Flynn, 2018, Exp. 3, p 28 to 33).

One minor modification to the procedure was made, and it is as follows:

- In Part B step 12
  - Pour the organic phase into the separatory funnel and extract twice more, unless there is no visible separation on the third attempt in which case allow the gasses to escape and collect the organic phase. Combine the basic and aqueous phases.

Observations

Part A:

- The methylene blue mixture mixed all the way through and created a sort of gradient royal blue being slightly darker near the top of the test tube - no clear delineation
- The methyl red mixture was actually more of an orange and separated into an orange layer on top and a clear transparent layer below which was much larger
- When they were mixed they separated into a small orange layer onto and a large royal blue later below, assumed to be the organic layer and aqueous layer respectively
- When salted:
  - 0.61g of NaCl was used
  - The solution with NaCl had a purple precipitate which rose to the top and was vibrantly purple
  - Some was suspended in the clear liquid below
  - In the solution without NaCl was very purple all the way through

Part B:

- unknown #1 with a mass of 0.81g
- White crystalline powder
- Mixed with 2:8 EtAOC and Hexanes which was clear and transparent with a strong odour
- NaOH then added, clear and colourless
- While mixing some got stuck to the side of the flask - created clear colourless solution where mixing was successful
- When aqueous HCl was added precipitate formed
- Ice bath allowed more precipitate formation
- Filtration resulted in a mass of precipitate of 0.20g but much remained stuck to side of original flask

Results

Table 1: The Rf values of the TLC plate spotted with the Organic Layer

TLC Plate	Reference Spot	Co-Spot	Sample Spot	Reference Spot - Rf	Co-Spot Rf	Sample Spot Rf
1	Unknown mixture	Unknown mixture & Organic Layer	Organic Layer	U1 - 0.29 U2 - 0.63	C1 - 0.24 C2 - 0.63 C3 - 0.90	0.63

Table 2: The Rf values of the TLC plate spotted with the Aqueous Layer

TLC Plate	Reference Spot	Co-Spot	Sample Spot	Reference Spot - Rf	Co-Spot Rf	Sample Spot Rf
2	Unknown mixture	Unknown mixture & Aqueous Layer	Aqueous Layer	U1 - 0.21 U2 - 0.67	C1 - 0.23 C2 - 0.63	S1 - 0.21 S2 - 0.65

Table 3: The Rf values of the TLC plate spotted with the Organic and Aqueous Layer

TLC Plate	First Lane	Second Lane	Second Lane	First Lane - Rf	Second Lane - Rf	Third Lane - Rf
3	Biphenyl	Benzophenone	Unknown mixture	1 - 0.65 2 - 0.79	1 - 0.08 2 - 0.58	U1 - 0.27 U2 - 0.63

Figure 1: Image of TLC plates marked using UV light

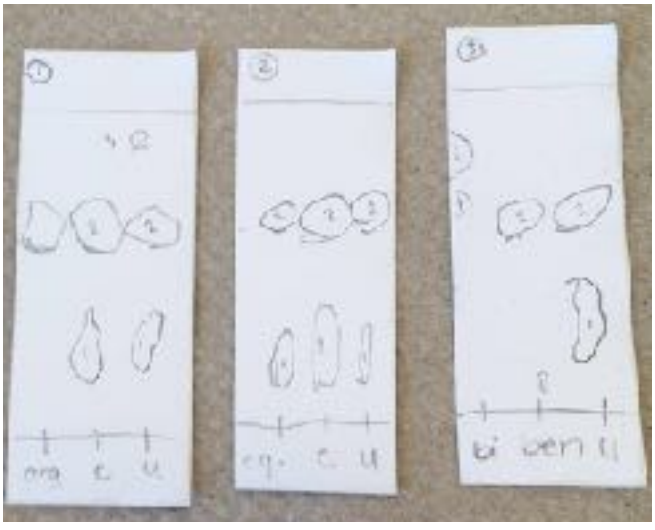


Figure 2: Computer drawing of TLC plate spotted with Organic Layer

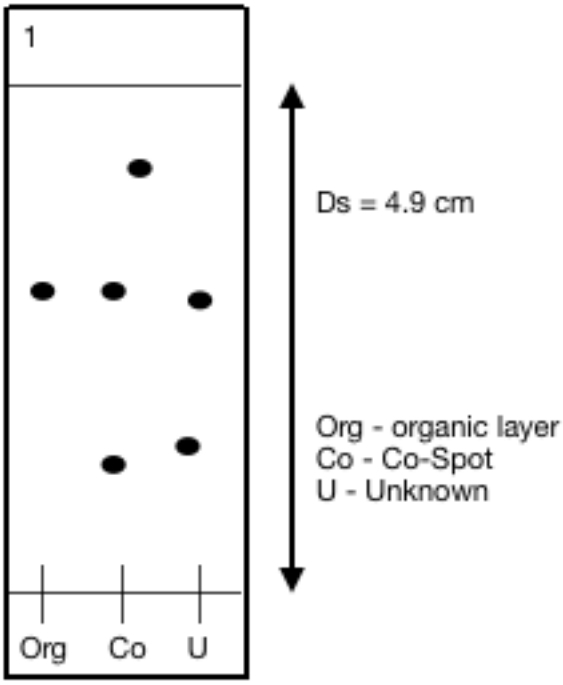


Figure 3: Computer drawing of TLC plate spotted with Aqueous Layer

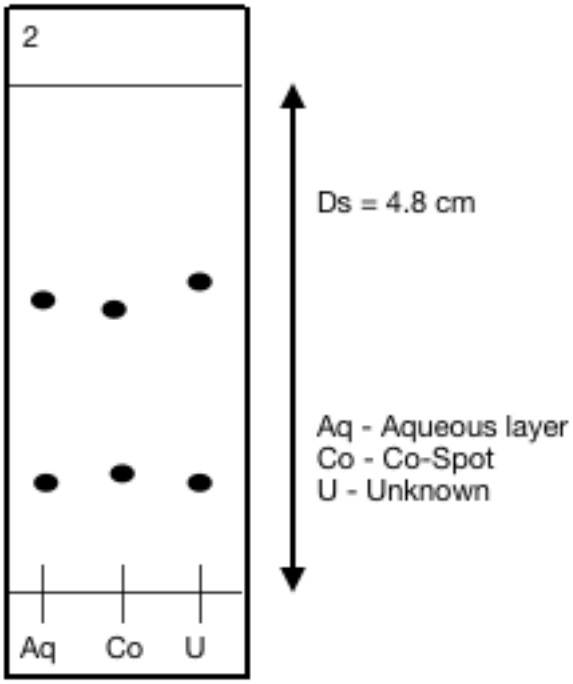
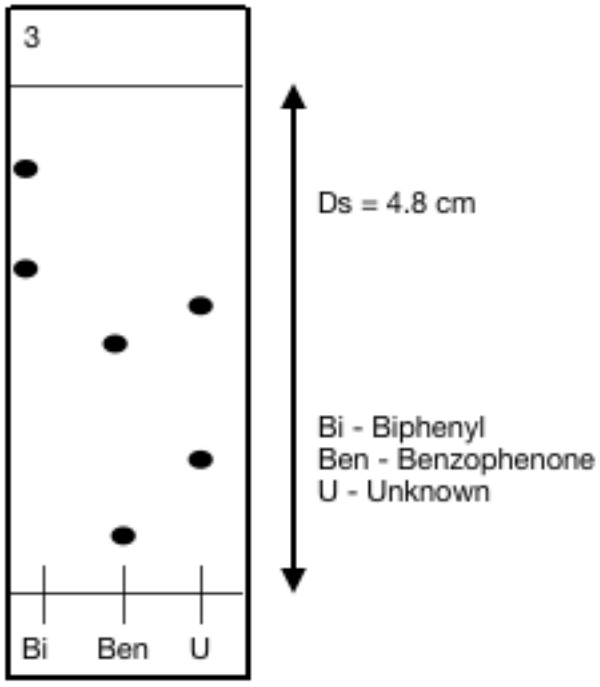


Figure 4: Computer drawing of TLC plate spotted with the Organic and Aqueous Layer



Results:

Unknown #	Initial Mass	Mass Obtained	Composition	% Yeild
#1	0.81g	0.20g	Benzoic Acid and Benzophenone	25%

Calculations

Percent Yield of Benzoic Acid:

Mass of unknown sample used: 0.81g
Mass of precipitate formed: 0.20g

Percent Yield of Benzoic Acid = (mass of product) / (mass of unknown sample) x100%
= (0.20g) / (0.81g) x 100%
= 24.69%

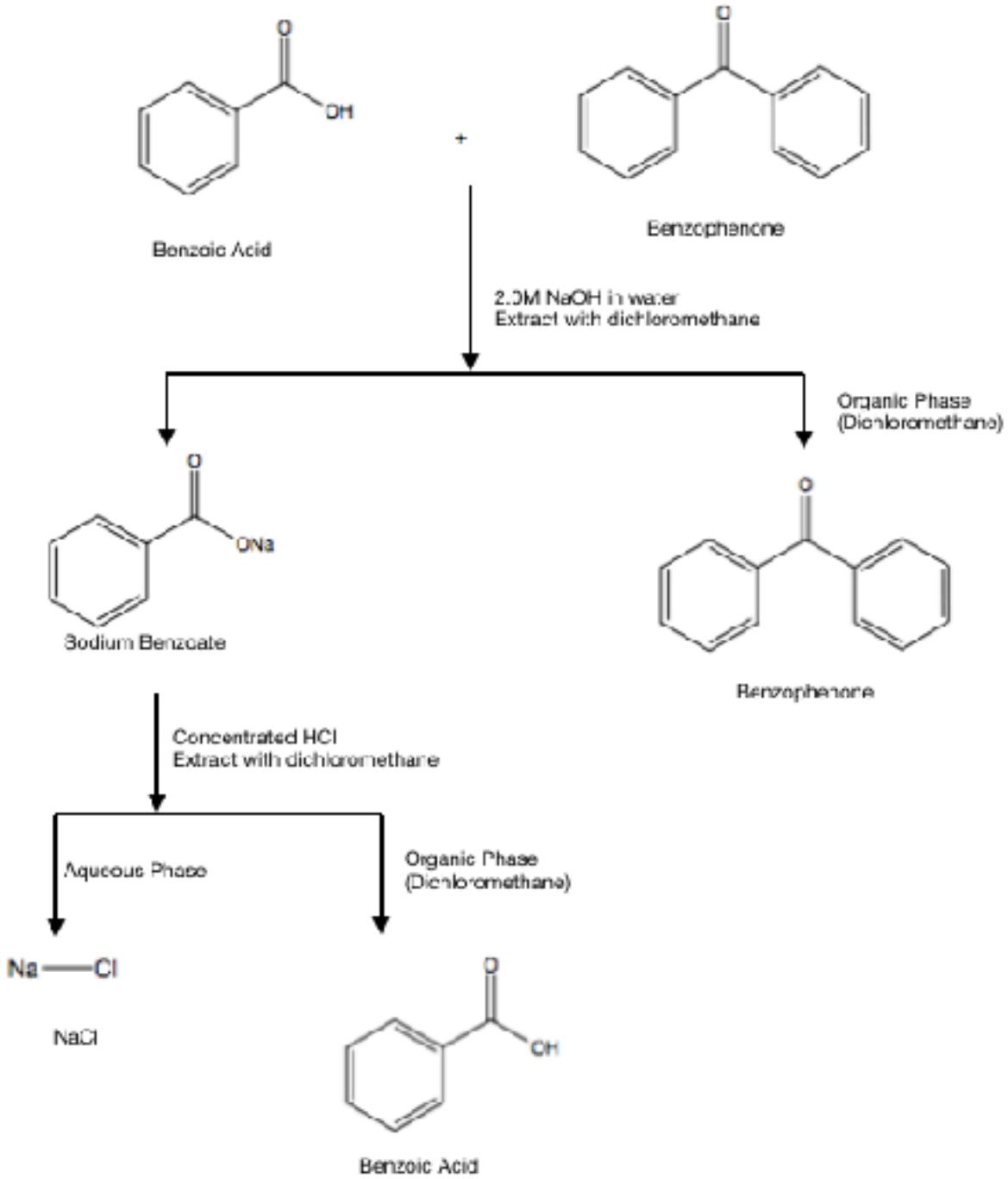
Therefore the percent yield of benzoic acid is approximately 25%.

Sample Rf Value calculation:

Rf value = distance traveled by compound / distance traveled by eluting solvent
= Di / Ds
Rf value for TLC1 Org = 3.1cm / 4.9cm = 0.63

Therefore the Rf value for the organic phase on TLC1 in 2:8 EtAOC/Hexanes is 0.63.

Mechanism Describing the Reactive Separation of a Mixture Containing Benzoic Acid and Benzophenone



Discussion

- The extraction capitalizes on the tendons of the individual components of the mixture to dissolve in different solvents resulting in separation
- As in most extractions the two solvents involves were either organic or aqueous in phase, with water usually being the aqueous phase
- The organic phase is a carbon containing molecule that does not mix well with water

- Part A
  - Compound affinity to aqueous or organic layer was used to demonstrate separation
  - Methylene blue is charged
    - Has a sulphur anion and chloride cation and therefore reacts and dissolves in water
    - This means the dye should appear in the aqueous layer of the solution although it was observed to be present throughout being darker near the top
  - Methyl red is non polar
    - This means it should be immiscible in the aqueous phase and appear in the organic
    - It was observed to be solely residing in the top layer of the test tube
  - When mixed
    - There were clear blue and orange, lower and upper layers that clearly distinguished the layers into the aqueous and organic phases respectively
  - Crystal violet
    - In the mixture with NaCl, the NaCl reacted strongly with he aqueous layer forcing the purple dye out of the layer and into the organic layer
    - Without the NaCl there was uniform mixing of purple dye

- Part B
  - TLC plates 1 and 2, the organic and aqueous plates, indicates an ineffective separation between the two layers for the aqueous mixture
    - ie. there is some organic in the aqueous
  - The intended outcome should have been a single spot in each lane, not two
  - The spot with the Rf value 0.65 in the aqueous lane is clearly an unseparated portion of the unknown and is showing the organic layer because it has a very similar Rf value to the organic lane; 0.63
  - The goal would have been
    - the organic phase to have contained a single non polar compound
    - The aqueous phase to have contained a single highly polar compound
  - Our results were
    - Indeed a non polar compound (high Rf) for the organic phase
    - A mixture of a non polar compound and a polar compound (low Rf) due t the incomplete separation

- The streaking of the TLC plates on one component of the unknown (see figure 1) indicates it's high polarity
  - This affinity leads me to believe it is the benzoic acid
  - It would have high affinity to the silica gel coating the plate due to its polarity
- The other unknown spot (higher on the TLC plates), that did not streak, lines up quite well with the higher benzophenone spot (Rf values of 0.58 and 0.63 respectively) indicating it as the second component
  - I believe the benzophenone spot with Rf value 0.08 is an error due to possible contamination
- The reactive separation:
  - Successful due to the application fo acid / base properties and their tendencies to react
  - Allows for a clear distinction between which compound was the acid and which compound was organic
  - Because acids base reactions salt a separation between that uncharged organic substance and the acid can occur
  - The strong base NaOH reacted with benzoic acid when added to the solution
  - Sodium benzoate is formed and displays the properties of a salt
  - The conjugate base of benzoin acid rests in the aqueous phase and the organic compound rests in the organic which moves to the bottom of the separator funnel
  - To convert the salt back into its original acid form HCl is added
  - It allows for reprotonation of the salt which draws the acid out of the organic solution
  - Deprotonation of the acid and protonation of the base results in the exhibition of ionic properties and better capability to dissolve in non-organic solvents
- Yield
  - The extraction of NaCl yielded a rather low quantity
  - The experiment only has a yield of 25%
  - This could have been due to the inaccuracy of the collection, much of the precipitate was still stuck to the flask that the solution was made in before the filtration occurred, and then also stuck to the filter paper and filtration device

- Error
  - We did not have a 100% pure sample of the aqueous layer which caused an organic spot to appear on our aqueous lane on the TLC plate
  - It would appear as if there were also impurities or errors with biphenyl and benzophenone samples because small spots occurred in the lanes in non-logical places for their polarities (for example Rf value 0.08 for benzophenone on TLC pate 3, see figure 4)
    - These could be dud to unclean impurities, unclean lab equipment, or using the same capillary tube for different substances
  - The precision of the measuring instrument was to the nearest millimetre which could have been improved upon for such small scale measuring

Questions

1. **Ethanol and Water:**
   It would be difficultly or nearly impossible to preform an extraction with ethanol and water because the principal of extraction is that the compounds are separated by their polarity and therefore their miscibility in a solvent. If the solvents are able to mix with each other, as with Ethanol and water, the compounds may still separate but occupy the same general space and distinction would be incredibly difficult.

2. **NaCl:**
   NaCl dissolves well in water and it's free floating ions would interact with it readily, more so than methylene blue which is only slightly charged. The NaCl ions would saturate the available spaces in the water and force the dye to go to the organic phase. This process is called "salting out" and causes the amount of dye in the aqueous later to decrease.

3. **Compound Y:** The ratio of the solubility of the compound in each of the phases is called the distribution coefficient or KD
   KD = Solubility in water / solubility in ether
   KD = (2.0 g/100mL / 20.0g/100mL)
   KD = 0.1

   Let A represent the mass of compound Y in the aqueous phase
   Let E represent the mass of compound Y in the organic phase

   A + E = 1.8g E = 1.8g - A
   0.1 = (A / 100mL) / (1.8g - A / 100mL)
   A = 0.08g
   E = 1.8g - 0.08g
   = 1.72 g

   Therefore there will be 1.72g of compound Y removed from the solution by ht single extraction with 100mL ether.

4. **Extraction 1:**
   Let A represent the mass of compound Y in the aqueous phase
   Let E represent the mass of compound Y in the organic phase

   0.1 = (A / 100mL) / (1.8g - A / 50mL)
   A = 0.30g
   E = 1.8g - 0.30g
   = 1.50 g

   Therefore after one extraction 1.50g of compound Y will be removed from the solution of water by extraction will 50mL ether.

Extraction 2:

0.1 = (A / 100mL) / (0.30g - A / 50mL)
A = 0.05g
E = 0.30g - 0.05g
= 0.25 g

Therefore after two extractions an additional 0.25 g of compound Y will be Y will be removed from the solution of water by extraction will 50mL ether.

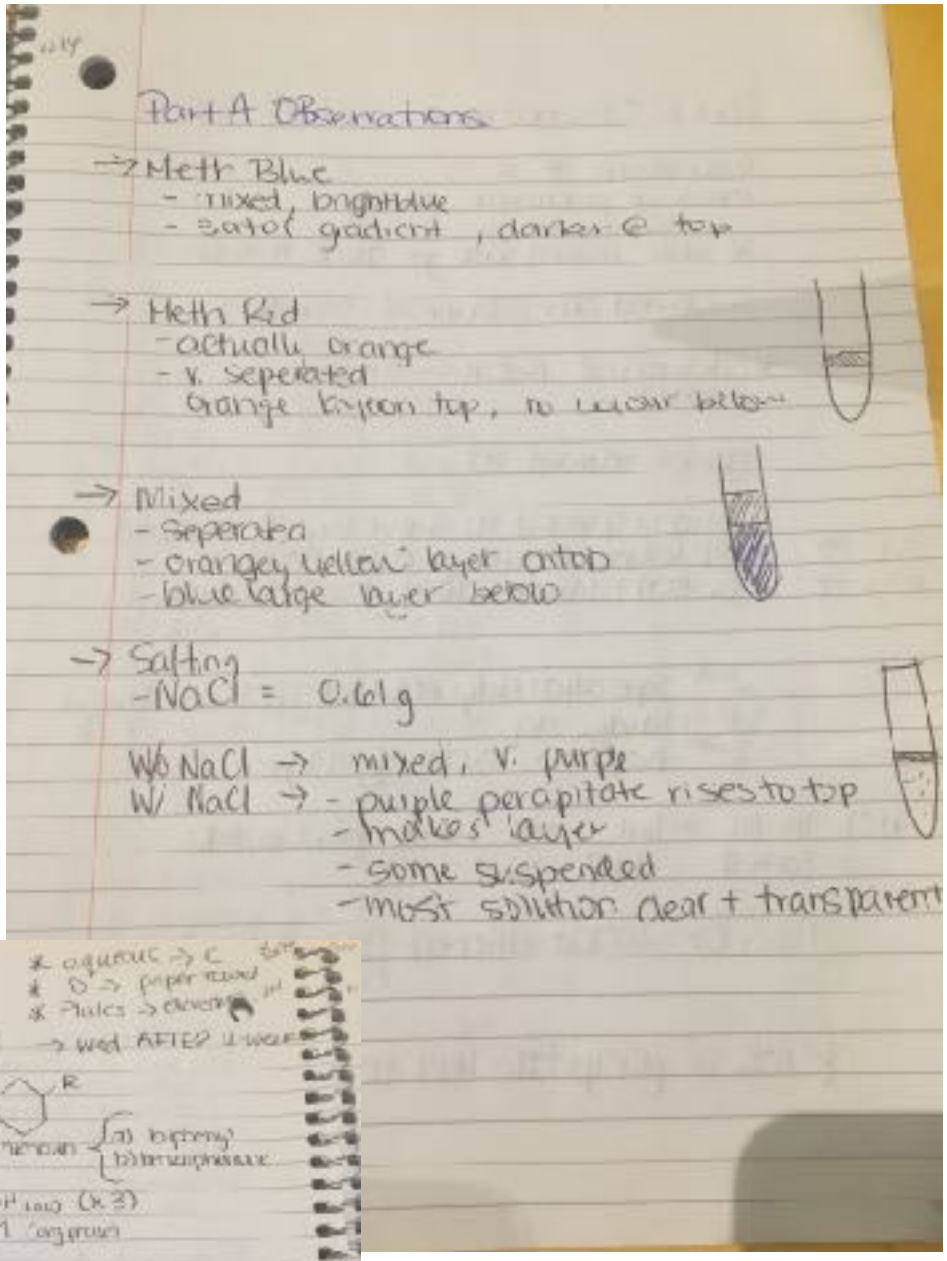
Total :
Total Mass of Y removed = 1.50g + 0.25g
= 1.75g

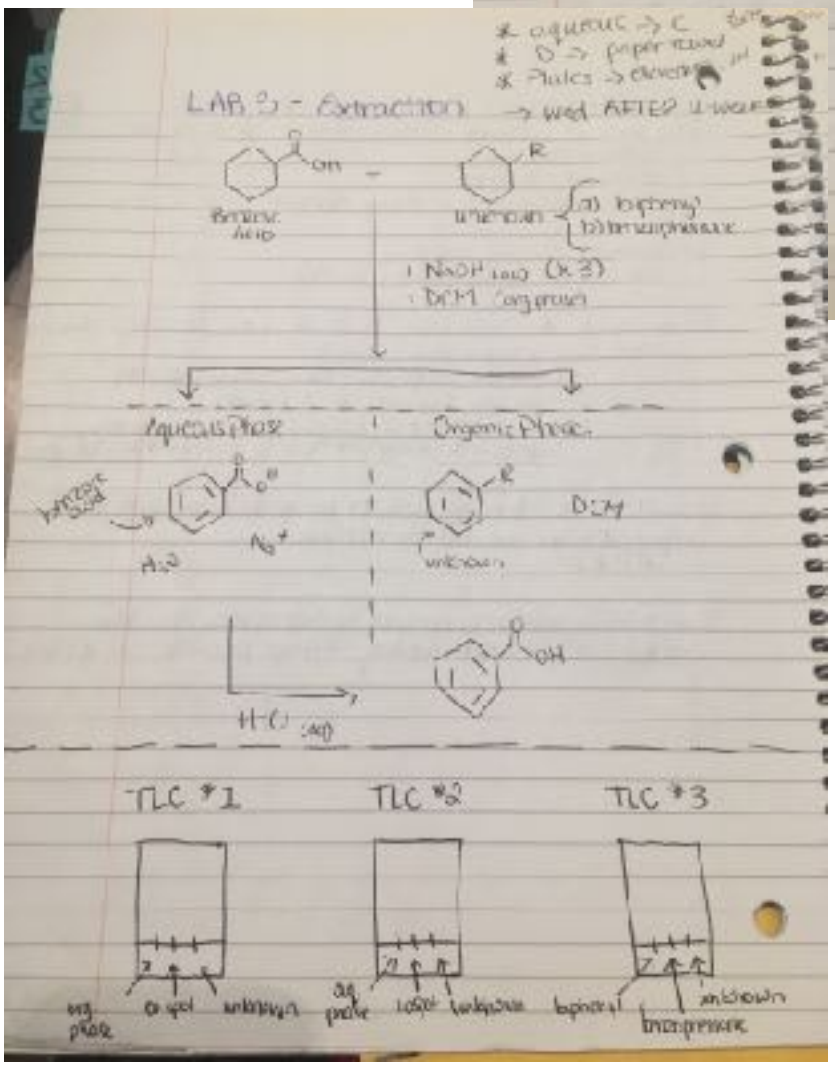
Therefore the two extractions will remove 1.75g from the solution.

5. I**f a student looses track of which sample** is the organic layer and which sample is the aqueous layer the student can test the mixture's reaction to a solvent. A good choice for this would be water because it is polar, so it will mix with an aqueous layer and not with an organic layer. If the layers are too close in appearance to water to tell, the student could determine the densities of the aqueous and organic layer and determine which would reside on top and bottom when mixed. The less dense would sit on top of the more dense layer.

6. **To separate a mixture** of the organic base, Benzyl Amine and Naphthalene one must add an acid such as Hydrochloric acid to the mixture containing consisting of the mixture with ether. The interaction of the acid causes the donation of a proton to the amine group of benzyl amine causing it to become an ammonium ion and thus water soluble. This will cause it to migrate from the organic phase into the aqueous phase. The naphthalene will remain unaffected in the organic phase and the separation can be perform in the the separators funnel. The resulting aqueous phase has NaOH added to it which accepts a proton and the resulting salt is removed through suction filtration. The benzyl amine can be recovered by using the separator funnel again containing dichloride thane and water in which the benzyl amine will reside in the organic dichloromethane layer.

Raw Data


Part A Observations
→ Meth Blue
- mixed, bright blue
- sort of gradient, darker @ top
→ Meth Red
- actually orange
- v. seperated
orange layer on top, no color below
→ Mixed
- seperated
- orangey yellow layer on top
- blue beige layer below
→ Salting
- NaCl = 0.61 g
W/o NaCl → mixed, v. purple
W/ NaCl → - purple precipitate rises to top
- makes 3 layer
- some suspended
- most solution clear + transparent


LAB 3 - Extraction
Aqueous Phase
Organic Phase
DCM
TLC #1
TLC #2
TLC #3

Part 1: Observations

Unknown: # 1
mass of unknown: 0.5g

* while mixing some got stuck to side
- created pink colorless solution

* addition of NaOH → student + observes

- gases released x3

- when replaced in stand layers seperated
aq → - top layer cloudy + colourless
org → - bottom layer clear + colourless.

- 2nd seperation only resulted in thin layer of cloudy aq
- 3rd had no cloudy layer

HCl 7# 11 - 1ml → white percipitate formed dropi

Mass of suction filtered percipitate: 0.20g

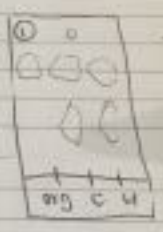
* lots of precipitate left in 25ml flask

TLC Plates

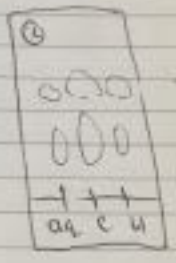
went in 12:05 - 12:11 : 6 min

Rf values * Rf = D1/D2

① Ds - 1.9
Rf org = 3.1/4.9 = 0.63
Rf C1 = 1.2/4.9 = 0.24
Rf C2 = 3.1/4.9 = 0.63
Rf U1 = 1.4/4.9 = 0.29
Rf U2 = 3.1/4.9 = 0.63
Rf C3 = 4.4/4.9 = 0.90



② Ds = 4.8
Rf Aq1 = 1.0/4.8 = 0.21
Rf Aq2 = 3.1/4.8 = 0.65
Rf C1 = 1.1/4.8 = 0.23
Rf C2 = 3.0/4.8 = 0.63
Rf U1 = 1.0/4.8 = 0.21
Rf U2 = 3.2/4.8 = 0.67



③ Ds - 4.8
Rf biphenyl 1 = 3/4.8 = 0.625
Rf biphenyl 2 = 3.8/4.8 = 0.79
Rf benzo 1 = 0.4/4.8 = 0.08
Rf benzo 2 = 2.8/4.8 = 0.58
Rf U1 = 1.3/4.8 = 0.27
Rf U2 = 3.0/4.8 = 0.63