Extraction

Experiment 3

TA: Daliane Regis

CHM1321 Z05

Written by

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Date: Friday, February 8, 2019

Procedure

As described in the lab manual. (Durst et al., *Organic Chemistry Laboratory Manual,* exp. 3, 2019)

Data

Table of Reagents

Reagent	Volume/mass	Molarity (M)
Ether	1 mL	-
Methylene blue	1 drop	0.006
Methyl red	1 drop	0.006
Aqueous crystal violet	1 drop	0.003
NaCl	1.20g	-
Unknown sample #56	0.53g	-
Dichloromethane	10 mL	-
NaOH	10 mL	2
HCl	-	10

Part A

#1

Image 1

Water,ether,methylene blue before mixed

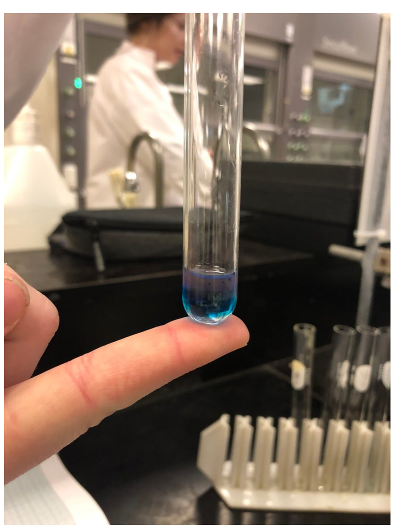


Image 2

Water,ether, methyl red before mixed

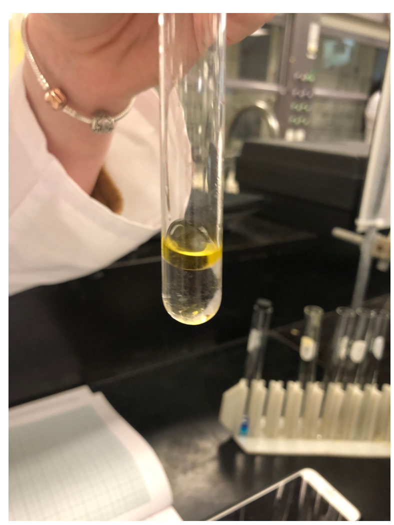
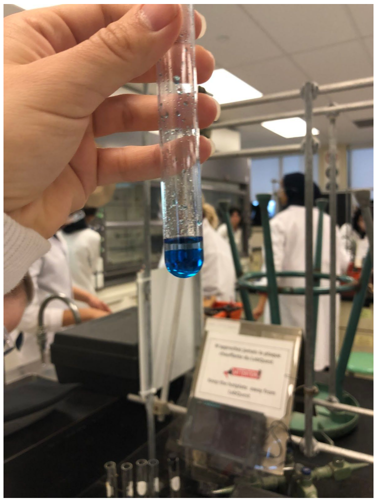


Image 3

Water, ether, methylene blue after being mixed

Image 5

Both test tubes added together



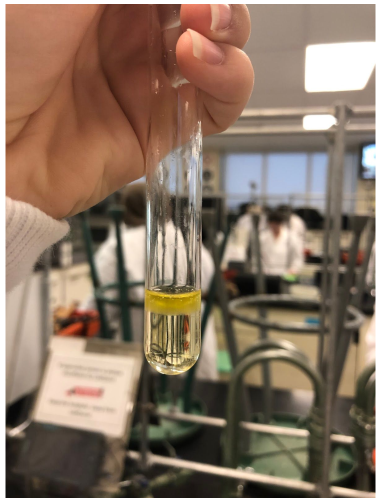
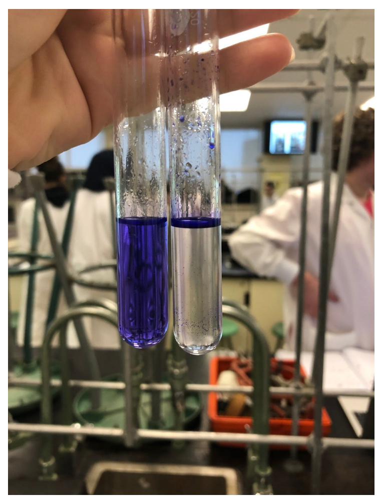


Image 4
Water, ether, methyl red after being mixed

Image 6
#2
Mass NaCl added= 1.20 g

Test tube on the left: distilled water, 1-butanol, 1 drop of crystal violet
Test tube on the right: distilled water, 1-butanol, 1 drop of crystal violet and 1.2 g of NaCl



Part B

Image 7

Before the separation

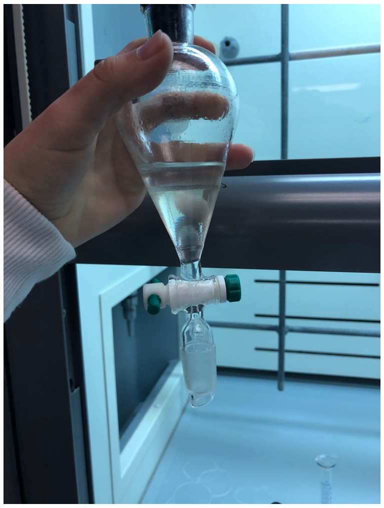


Image 8

Organic part of the separation

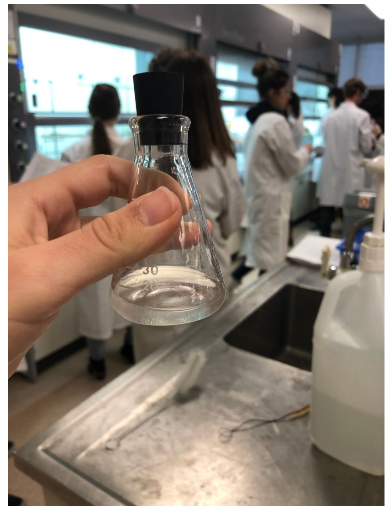


Image 9

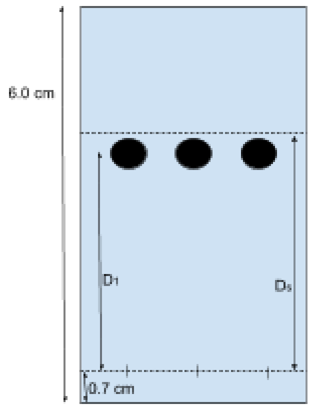
Aqueous part of separation

Image 10

After adding HCl to aqueous solution

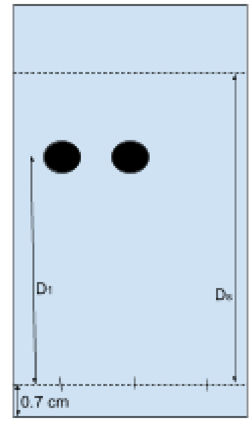
TLC #1:

D_s = 3.3 cm
D_1 = 3.0 cm



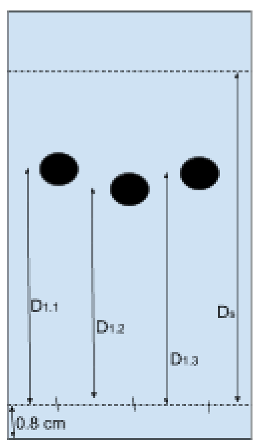
TLC #2:

D_s =4.8 cm
D_1 =2.7 cm



TLC #3:

D_s =4.6 cm
$D_{1.1}$ = 3.7 cm
$D_{1.2}$ =3.5 cm
$D_{1.3}$ =3.6 cm



Calculations

Calculations for TLC 1 and 2

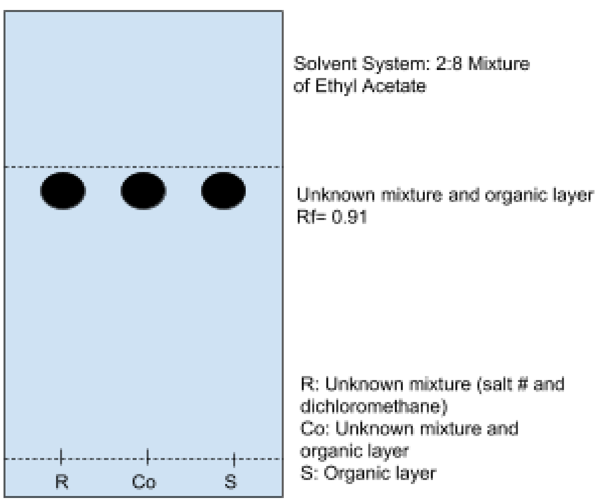
	TLC #1	TLC #2
D_1	3.0 cm	2.7 cm
D_s	3.3 cm	4.8 cm
Rf	0.91*	0.5625

* $Rf = \frac{D_1}{D_s} = \frac{3.0\ cm}{3.3\ cm}$ =0.90909091

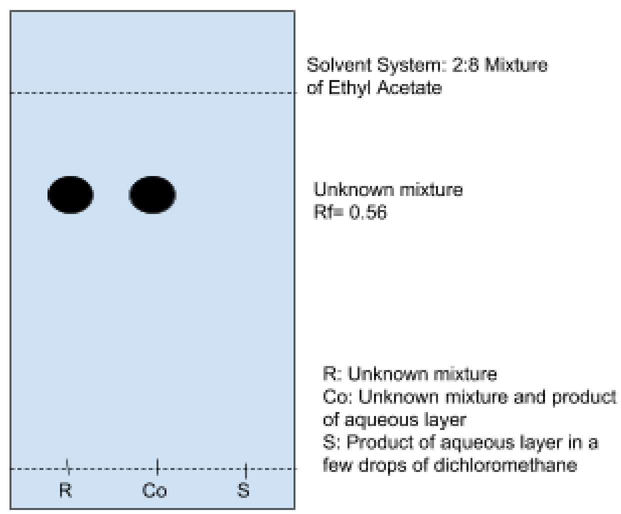
Calculations for TLC 3

	TLC #3
$D_{1.1}$	3.7 cm
$D_{1.2}$	3.5 cm
$D_{1.3}$	3.6 cm
D_s	4.6 cm
$Rf_{1.1}$	0.804348
$Rf_{1.2}$	0.76086957
$Rf_{1.3}$	0.7826087

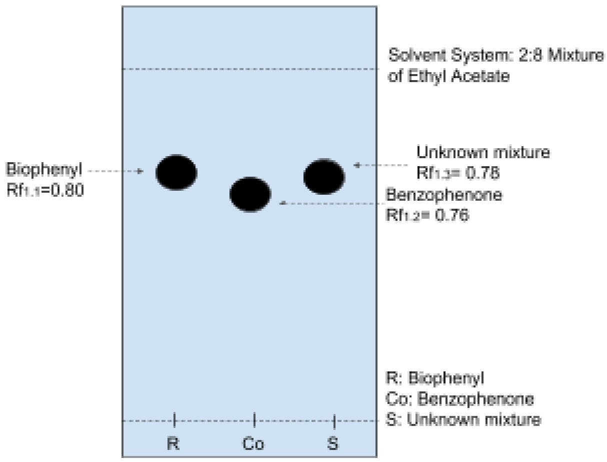
TLC #1:



TLC #2:



TLC#3:



Unknown #56
Mass of unknown= 0.53 g
Mass of aqueous and paper= 2.65 g
Mass of paper= 0.46 g
Mass of aqueous= 2.19 g

% composition of benzoic acid = $\frac{mass\ of\ unknown\ added}{mass\ of\ aqueous}$ *100%
= $\frac{2.19\ g}{0.53\ g}$ *100 %
=391%’

Observations

- ethene and water makes a clear solution
- ethene, water, and methylene blue makes a light blue transparent solution
- after shaking the ethene, water, and methylene blue, the organic layer was colourless and the aqueous layer was blue
- when ethene, water, and methyl red were mixed, the organic layer was yellow and translucent, and the aqueous layer was slightly yellow but transparent
- when the test tube containing the methyl red and the one with methylene blue were mixes, the aqueous layer turned transparent blue and the organic layer was colourless and clear
- after adding NaCl to a test tube containing distilled water, 1-butanol, and crystal violet, the solution went from being completely purple to only having purple in the top organic phase

Questions

1. Why would it be difficult to perform an extraction using acetone and water?
   It would be difficult to perform an extraction between acetone and water because acetone is miscible (can dissolve in water) therefore there would be no separation of the two, and there would only be one phase.

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?
   Adding NaCl to the test tube would decrease the amount of dye in the aqueous layer as it would increase the ionic strength of water which pushes the dye out of the aqueous phase and into the organic layer.

3. Compound Y has the solubility of 2.0g/100 mL in water and 20.0 g/100 mL in ether. What mass of compound Y would be removed from a solution of 1.4 g of Y in 100 mL of water by a single extraction with 100 mL of ether?
   $K_D = \frac{Mass\ of\ ether/Volume\ of\ ether}{mass\ of\ water/Volume\ of\ water}$
   $= \frac{20/100}{2/100}$
   $= 10$
   $10 = \frac{y/100}{(1.4-y)/100mL}$
   $y = 1.27g$
   Therefore a mass of 1.27g of compound Y would be removed from solution.

4. What mass of compound Y would be removed from the original water solution in question 3 by two extractions using 50 mL of ether each time?
   $K_D = \frac{Mass\ of\ ether/Volume\ of\ ether}{mass\ of\ water/Volume\ of\ water}$
   $= \frac{20/100}{2/100}$
   $= 10$
   10= $\frac{y/50mL}{(1.4-y)/100mL}$
   $y = 1.17g$
   $1.4 - 1.17 = 0.23g\ remaining$
   $10 = \frac{y/50mL}{(0.23-y)/100mL}$
   $y = 0.19$
   Total removed= $1.17 + 0.19 = 1.36g$

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?

The student could add some water to determine which layer is the aqueous phase. When the water is added, if it forms a separate layer than the extracted phase, then that phase is the organic phase, as they would have two different densities so two different phases are formed. If the water does not make a layer separate to the phase it was added to than that phase is the aqueous phase.

6. Describe how you would separate a mixture of benzylamine (An organic base) and naphthalene. Both compounds are insoluble in water and soluble in ether.
   Dissolve both in dichloromethane and then extract three times with $HCl_{(aq)}$.
   The addition of the strong acid, HCl, will cause the organic base to react with it. The HCl will not react with the naphthalene as it is inert. When the benzylamine reacts with HCl it moves into the aqueous phase and the naphthalene will move into the organic phase, above the benzylamine as it is less dense. Ether is less dense than water, so it too will form the organic phase. Then, using a separatory funnel, the aqueous phase can be separated from the organic phase. Then, by adding a strong base such as NaOH, a precipitate will form from the deprotonated NH_3. Then this precipitate can be filtered out first via gravity and then via suction filtration.

Discussion

Part one of the lab was extracting water soluble dyes. In one test tube there was a mixture of ether, distilled water, and a drop of methylene blue. After the contents were thoroughly mixed together, two distinct phases were present. There was a clear organic phase sitting on top of the dyed aqueous phase. The aqueous phase is dyed because the methylene blue is polar and is therefore attracted to the aqueous phase to interact with the distilled water.In a second test tube, there was ether, distilled water, and a drop of methyl red thoroughly mixed together. After mixing, again there were two distinct phases. This time, the organic layer was yellow and translucent, and the aqueous phase was clear. This is because methyl red is a non-polar dye and therefore is repelled by the water into the organic phase with the ether. When the two test tubes were mixed together, the organic layer again turned it' yellow colour and the aqueous phase turned blue again. This proves that this is an effective way to separate the two phases as they are both seen in the final mixture of the two.

The next part of the experiment was the salting out effect. Two test tubes were prepared, each with distilled water, 1 drop of crystal violet and 1-butanol. After being thoroughly mixed, each solution was purple. When some NaCl was added to one of the test tubes and dissolved, all of the dye was pushed to the top of the water layer. This is because adding NaCl increases the ionic strength and therefore the polarity of the water by dissolving in it. The water molecules are more attracted to the Na^+ and Cl^- ions than the crystal violet, therefore pushing all of it to the top, decreasing the concentration of the dye in the aqueous layer.

The final part of the experiment was to determine what the unknown compound #56 was comprised of. This was done via thin layer chromatography (TLC). On the first TLC plate was the original mixture (unknown compound) as the reference and the organic phase as sample, the second TLC plate was the original mixture as the reference and the product from the aqueous layer as the sample, and the third TLC was biphenyl, benzophenone, and the unknown, respectively. On the first TLC, there was a slight error although it is unclear why. The TLC plate was left in the solvent for over 20 minutes and the solvent front still did not continue to elute up the plate. Due to time constraints, the TLC had to be removed. The result was a TLC plate with the reference, co spot, and sample all at the top of the solvent front, about 3.3 cm, each with a resulting R_f value of 0.91. The TLC should have looked about the same only with the solvent front moving further up the plate, with the co spot and reference leaving a streak behind it. The second TLC also had an error. There were 2 spots, the reference and the co spot, at the same height on the plate and each with a bit of a streak following it, and nothing in the sample lane. The likely explanation for this occurrence is either that when the sample spot was applied it was too small, or that the sample was too diluted. There should have been a streak in the sample lane that looked the same as the ones in the reference and co spot lanes. The third TLC was a success. Three spots total, the biphenyl and the unknown were at the same height and the benzophenone was slightly lower than the two. By looking at the third TLC, it is easy to determine that the unknown compound contained biphenyl, as the biphenyl and the unknown had the same R_f value.

Possible sources of error for this experiment are:

- Mixing up aqueous and organic phase flasks during the experiment. If this happens the way to determine what each flask contains is to add a few drops of water. If the drops diffuse into the phase than that is the aqueous phase, and if the drops do diffuse and make a layer above the phase than that is the organic phase.
- When spotting the TLC plates, it is possible that too much or too little is spotted in some of the lanes. If there is too much, the dot under UV will be too big after the solvent front elutes through the plate, and will be too difficult to read. If the spot is too small, there may be no spots that appear at all after the solvent front moves to the top of the TLC plate. Both of these occurrences could deter results. To ensure that this portion of the experiment goes smoothly, after spotting the TLC plates but before put into the solvent, put them under the UV light to see how big or small the spots are. If a spot is too small simply re-spot it. However, if it is too big the TLC plate should be thrown out and completely redone.
- Not completely drying out the benzoic acid will create an artificially high percent yield. To ensure that the benzoic acid is completely dried out, make sure that it is left in both the gravity filter and the suction filtration long enough. When taking the filter paper out of the Buchner funnel, if it is still stuck to the paper and still wet, it should be put back in the funnel and allowed more time to dry.
- During the first part of the experiment, it is possible to not have enough of one of the dyes in either of the test tubes. This could deter the conclusion from that part of the experiment. For example, after the test tubes were mixed, the organic layer was clear, and the aqueous layer was blue. The organic layer should have retained its colour as well. From these results, the conclusion would be that the experiment is not a good way to separate the two solutions. However, if another drop or two of the methyl red was added to the final mixture, the organic layer would have had its colour, thus changing the results and the conclusion.

Conclusion

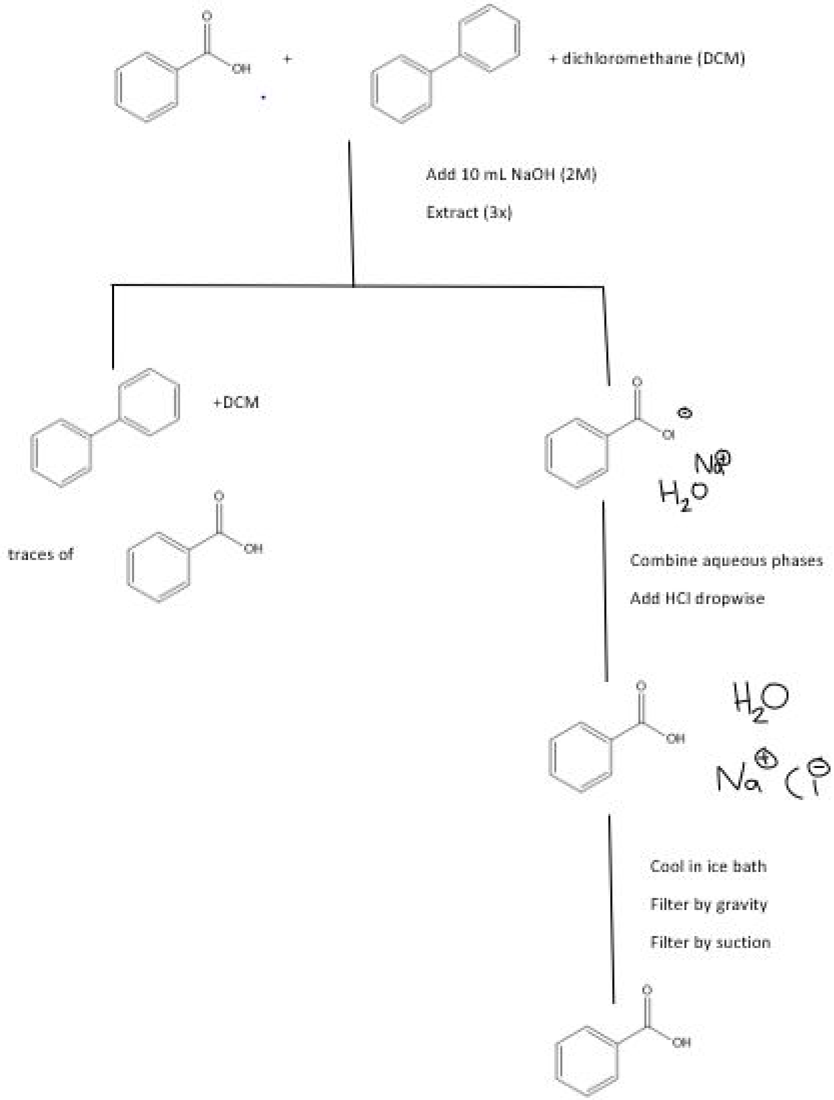
In conclusion, the first two labs went smoothly, as the desired results were obtained.

The first lab resulted in the separation of the aqueous and organic substances. In the first test tube, the methylene blue dye went into the aqueous layer, and in the second the methyl red dye went into the organic layer. It is easy to distinguish the two layers by adding a few more drops of distilled water into the test tubes and seeing which layer will expand. That layer had more water added to it, and is the aqueous layer. When these two test tubes were added together it created a clear organic layer on top, and a blue/green colour in the aqueous solution. More red dye should've been added to the second test tube so when both test tubes were added together, the aqueous layer on the bottom was blue and the organic layer on top was yellow.

In the second lab, two test tubes were prepared with distilled water, 1-butanol, and one drop crystal violet. When NaCl salt was added to the second test tube, the desired result occurred. All the dye got pushed up into the organic layer on top resulting in it being a deep violet and the aqueous layer on the bottom became clear.

In the last part of the lab, the unknown was determined to be biphenyl by comparing the Rf of the unknown with the Rf of the known substances. When doing this part of the lab, many source of errors came up. This created a range of errors from a high percent yield, to the TLC plate possibly getting messed up from spotting. In the end it was still able to be determined the unknown given was biphenyl.

Flow Chart


+ dichloromethane (DCM)
Add 10 mL NaOH (2M)
Extract (3x)
+DCM
traces of
Combine aqueous phases
Add HCl dropwise
Cool in ice bath
Filter by gravity
Filter by suction

3v

Raw Data

Hope’s Raw Data

Exp. 3 Feb. 6

Part A

1. ethene + water → clear
   1ml + 1ml + 1 drop 0.006M methylene blue
   ↳ light blue
   ↳ dye is in aqueous layer

2. ethene + water → Clear
   1ml + 2ml + 1 drop 0.006M methyl red
   ↳ yellow layer on top
   ↳ dye is in organic layer

3. when mixed the organic layer became ~~clear~~ ~~yellow~~ clear
   + the aq. layer remained blue
   ∴ not a good way to separate methyl red + methylene blue

4. Salting out
   5ml distilled water, 1 drop of 0.003M crystal violet
   1.20g NaCl in one test tube

   Test tube w/o NaCl → purple throughout
   With NaCl → purple layer on top, translucent bottom
   ↳ buton

   explain: the NaCl made the water more polar.
   Salt dissolves in water + separates the phases, dye moves to organic layer

RCS

Part B.

Unknown: 56 0.53g

dichloromethane - 9.5ml

upper cloudy organic layer

HCl by drop ~150 drops

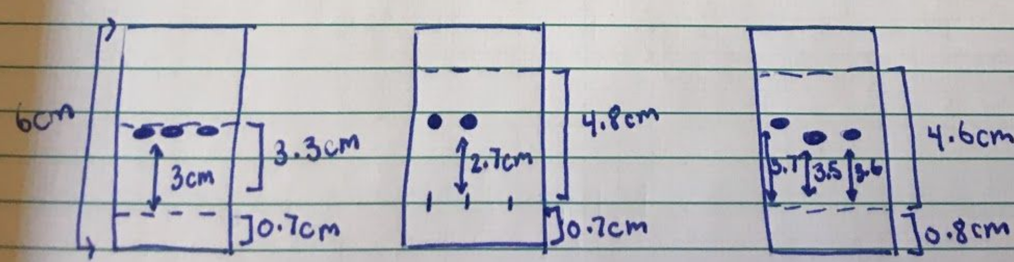
white precipitate

Paper + aqueous = 2.65g

paper = 0.46g

aqueous =

TLC 1 TLC 2 TLC 3



Hannah's Raw Data

Experiment 3 Feb 6/19

Part A

- Ether has a strong smell (bad)

1. Ether + water → clear + drop → blue
   ↳ dye is in aq. layer (water) *added water to check where it went to
   ↳ ether is on top

2. Ether + water → clear + drop → red
   ↳ ether is on top
   ↳ dye is in organic layer ether is less dense then water

3. When the 2 solutions are mixed we get:
   - organic layer on top (clear)
   - water (aq.) on bottom (greenish - blue)

~~*org layer should be yellow~~ → good way to ~~seperate~~

did NOT dissolve well. not a good way to seperate

DRCS

Feb 6/19

Part B.2

m_0 = 1.20 g NaCl

added NaCl makes 2 layers → bottom = translucent
→ top = purple

- Salt makes water more polar
- Salt dissolves in water + seperates the phases, dye moves to organic layer

Feb 6/19

Part B

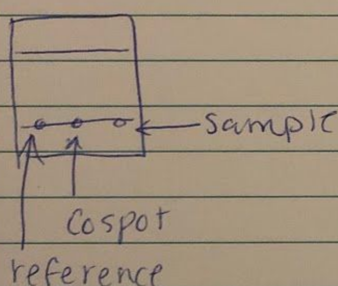
Unknown 5b m = 0.53 g

paper + aqueous : m = 2.65 g
paper : m = 0.46 g
aqu. m = 2.19 g

Volume dichloro = 9.5 mL

Organic layer = bottom

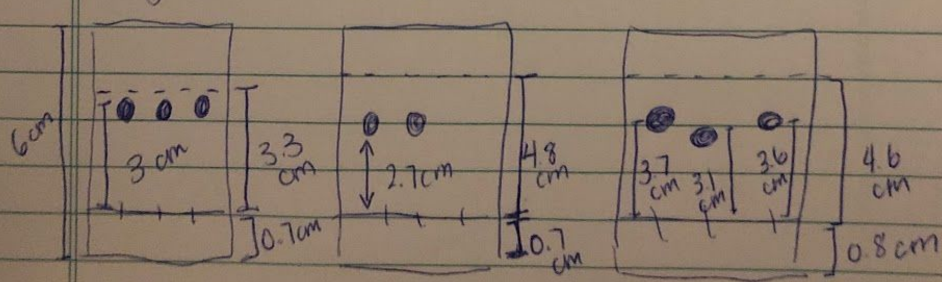
HCl drops ~150



TLC #1 — unknown + organic

TLC #2 — unknown + aqueous

TLC #3 — biophenyl, unknown, benzophenone



Peer Evaluation

COURSE: CHM 1321 TA Name: Daliare Regis
YOUR NAME (PRINT): Hope Boyle SIGNATURE: Hope Boyle

CONFIDENTIAL PEER EVALUATION FORM FOR EXPERIMENT 3

Each team member must submit one assessment form evaluating each other member of the team.
Teams will consist of 2 (max 3) members for reports.
You may edit this form.
Do not share or discuss the contents or possible contents of this assessment with others.

In assessing the work of your fellow team members, consider the following aspects:

- Quality of work
- Contribution to the work as a whole
- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade (/5)
Hannah Prazak	Great partner	5

A – Excellent (5) B: Great (4) C: Good (3) D: Fair(2) F: Poor (1)

Note: Do not evaluate yourself on this form