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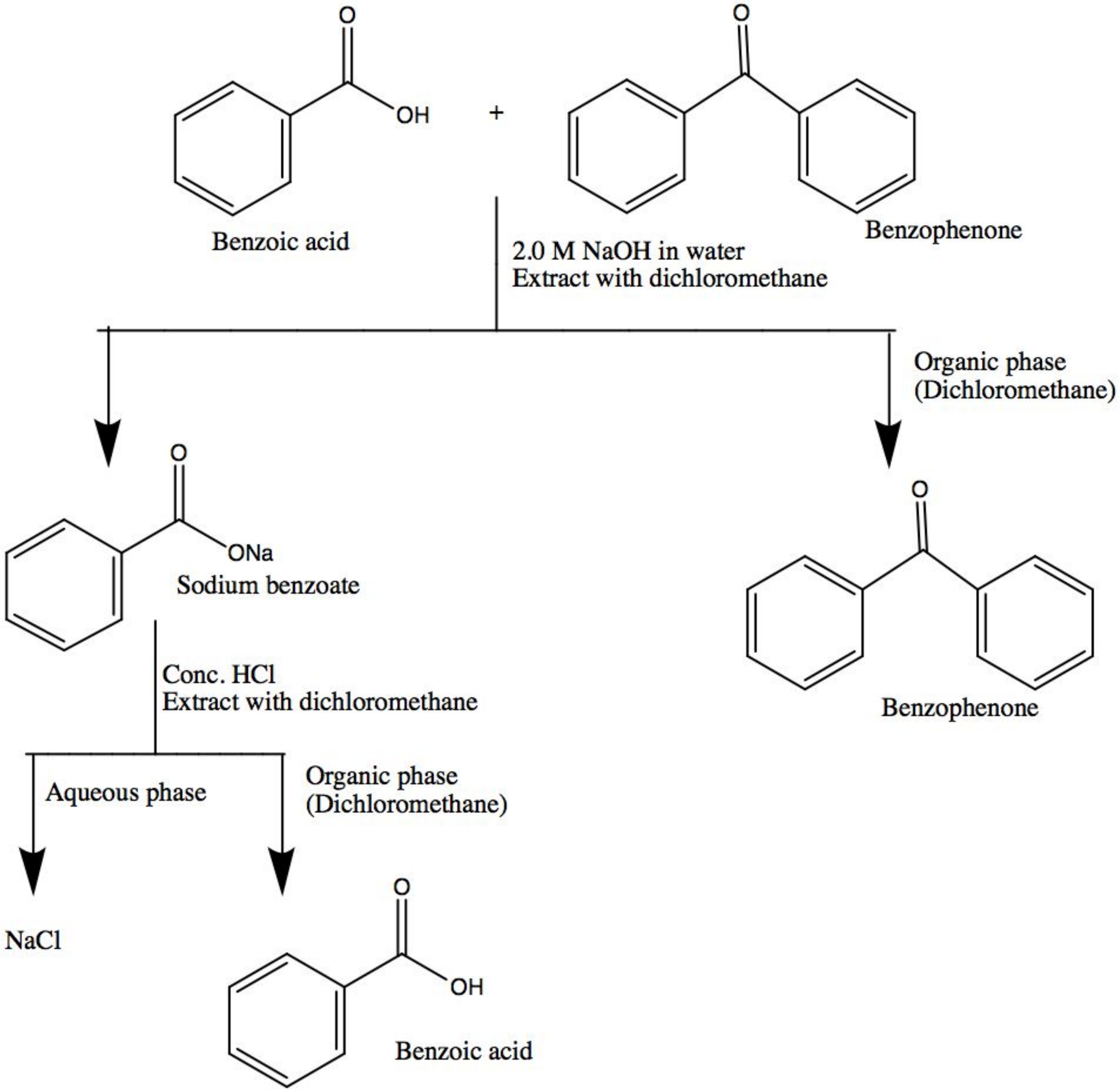
Lab Day (T/W/Th/F): W
Lab Week (even/odd): even
Lab time: 10:00

Experiment 3.
Extraction

Initials: H.P.

Figure 1. Reactive extraction of benzoic acid and benzophenone



Part A: Extraction of water soluble dyes

Procedure:

- 1 mL of ether, 1.1 mL of distilled water and 1 drop of a 0.006 M methylene blue dye was placed into a test tube. A rubber stopper was inserted and the test tube was shaken vigorously
- The layers were allowed to settle
- The blue dye appeared in the aqueous layer but not very much in the organic layer. This likely means that the methylene blue dye is soluble in an aqueous phase but is insoluble in an organic phase.
- 1 mL of ether, 1.1 mL of distilled water and 1 drop of a 0.006 M methyl red dye was placed into a test tube. A rubber stopper was inserted and the test tube was
- The test tube remained as colourless and transparent. This may mean that methyl red is insoluble in both the ether or the distilled water. Since the liquids remained colourless after drops of methyl red were added, the methyl red are not a good indicator of which layer is organic and which is aqueous

Salting out effect

- Two test tubes containing 5 mL of distilled water, 0.5 mL of 1-butanol and 1 drop of 0.003 M aqueous crystal violet were prepared
- Rubber stoppers were inserted and both test tubes were shaken vigorously
- The entire content of the test tubes were violet in colour. This means that the crystal violet is soluble in both the 1-butanol and the distilled water
- A scoop of NaCl was added to one of the test tube and shaken vigorously
- The distilled layer layer was translucent and faintly violet while the butanol remained violet. This is due to the salting out effect, where a high concentration of the salt causes more molecules of water to interact with the ions and less interactions with the crystal violet. As a result, the solubility of the crystal violet in water decreases and migrates towards the butanol layer

Part B: Separating a mixture with reactive extraction

- 0.72 g of Unknown sample 1 was obtained
- 10 mL of dichloromethane was measured using a graduated cylinder and transferred to a 50 mL beaker
- The unknown sample was dissolved into the dichloromethane
- A small amount was kept for reference
- The separatory funnel was ensured for closure
- The solution was poured into the separatory funnel

- The beaker was rinsed with dichloromethane and transferred to the funnel
- 10 mL of 2.0 M NaOH was obtained using a graduated cylinder and added to the funnel
- A stopper was placed on the funnel and inverted
- The stoplock was opened to vent the contents and closed again
- The funnel was shaken for 30 seconds
- The stoplock was opened to vent the contents and closed again
- The funnel was placed on a wooden rack
- The stopper was removed and the layers were allowed to separate
- The lower organic layer was drained into a clean 50 mL beaker
- The upper aqueous layer was drained into a 125 mL Erlenmeyer flask
- 10.1 mL of 2.0 M NaOH was obtained using a graduated cylinder and added to the funnel
- The stopper was placed on the funnel and inverted
- The stoplock was opened to vent the contents and closed again
- The funnel was shaken for 30 seconds
- The stoplock was opened to vent the contents and closed again
- The funnel was placed on a wooden rack
- The stopper was removed and the layers were allowed to separate
- The lower organic layer was drained into the 50 mL beaker
- The upper aqueous layer was drained into the 125 mL Erlenmeyer flask
- 10.1 mL of 2.0 M NaOH was obtained using a graduated cylinder and added to the funnel
- The stopper was placed on the funnel and inverted
- The stoplock was opened to vent the contents and closed again
- The funnel was shaken for 30 seconds
- The stoplock was opened to vent the contents and closed again
- The funnel was placed on a wooden rack
- The stopper was removed and the layers were allowed to separate
- The lower organic layer was drained into the 50 mL beaker
- The upper aqueous layer was drained into the 125 mL Erlenmeyer flask
- Drops of concentrated HCl was added to the basic aqueous phase until the solution became translucent. There was precipitates of solid NaCl in the solution
- The pH was verified with litmus paper
- The mixture was cooled in an ice bath
- A test was attached to the nozzle of the water sink and to a suction flask
- A Buchner funnel was attached to the top of the suction flask with filter paper placed on it
- The aqueous mixture was carefully poured onto the funnel. Distilled water was added to ensure all the solids were on the funnel
- The solid NaCl was removed from the filter paper and onto weighing paper
- The solid NaCl was weighed. Approximately 0.20 g of NaCl were obtained

- Three TLC plates were prepared
- An indeterminate volume of acetone was obtained in a 50 mL beaker
- The original sample was spotted onto the reference and the co-spot lane of the first two TLC plates using a capillary
- The capillary was cleaned by dipping the capillary into the acetone
- The organic phase was spotted onto the sample and the co-spot lane of the first TLC plates
- The capillary was cleaned by dipping the capillary into the acetone
- The aqueous phase was spotted onto the sample and the co-spot lane of the second TLC plates
- The capillary was cleaned by dipping the capillary into the acetone
- The third TLC plate was spotted with biphenyl, benzophenone and the unknown mixture
- The plates were developed in a 2:8 mixture of EtOAC and hexane as eluants
- The solvents were allowed to proceed so that the solvent front is approximately 1 cm from the top of the plate
- The plates were removed from the developing jar and dried
- A pencil line was immediately marked where the solvent front ends
- The plates were visualized under a UV light
- Photos of the plates were taken
- A circle was gently drawn around each spot and the displacement was measured

Data:

Table 1: Experimental data of TLC of original sample compared with organic phase

Measurement	**Displacement (cm)**
Solvent front (R_s)	5.2
Displacement of Original Sample	3.6
Displacement of Co-spot	3.4
Displacement of Organic Phase	3.4

Figure 2: TLC plate of unknown mixture compared with organic phase

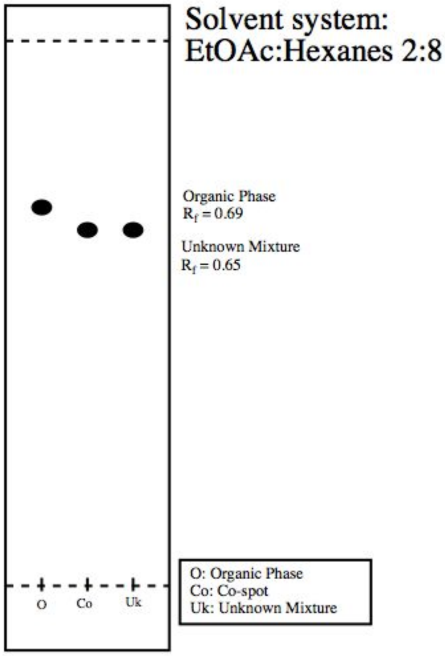


Table 2: Experimental data of TLC of original sample compared with aqueous phase

Measurement	**Displacement (cm)**
Solvent front (R_s)	5.8
Displacement of Original Sample	4.7
Displacement of Co-spot	3.4
Displacement of Aqueous Phase	3.5

Figure 3: TLC plate of unknown mixture compared with aqueous phase

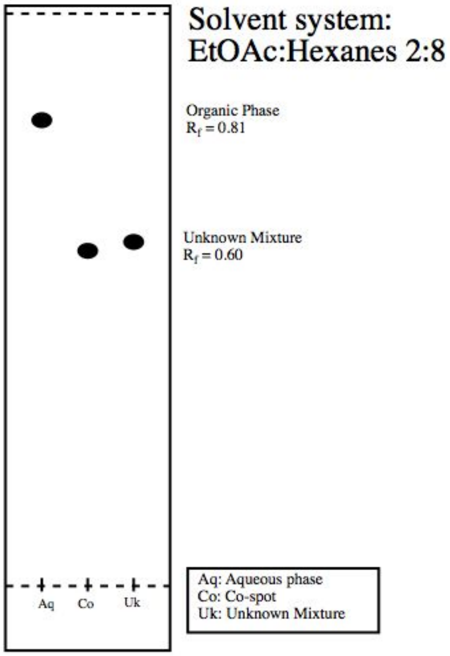
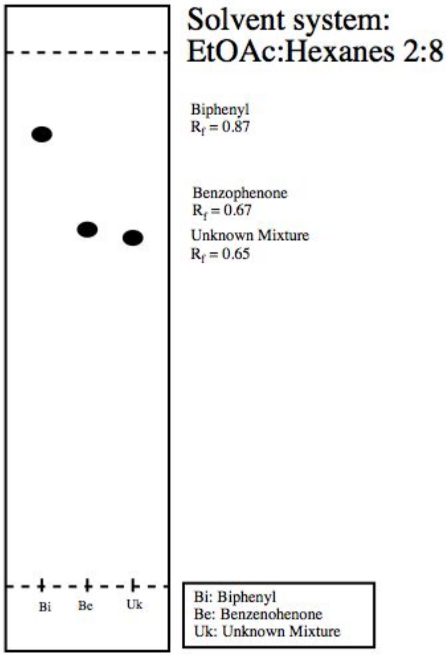


Table 3: Experimental data of TLC of unknown mixture compared with biphenyl and benzophenone

Measurement	Displacement (cm)
Solvent front (R_s)	5.4
Displacement of Reference Solution	4.6
Displacement of Co-spot	3.6
Displacement of Unknown Sample	3.5

Table 4: TLC plate of unknown mixture compared with biphenyl and benzophenone



Calculation:

<u>Retention Factor:</u>

<u>TLC 1</u>

Retention factor of Organic phase

$$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = \frac{3.6\ cm}{5.2\ cm} = 0.69$$

Retention factor of Co-spot

$$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.65$$

Retention factor of Unknown Sample

$$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.65$$

<u>TLC 2</u>

Retention factor of Aqueous phase

$$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = \frac{4.7\ cm}{5.8\ cm} = 0.81$$

Retention factor of Co-spot

$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.59$

Retention factor of Unknown Sample

$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.60$

TLC 3

Retention factor of Benzophenone

$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = \frac{3.6\ cm}{5.4\ cm} = 0.85$

Retention factor of Biphenyl

$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.67$

Retention factor of Unknown mixture

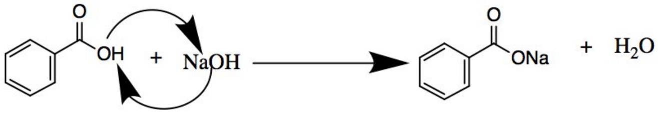
$R_f = \frac{Displacement\ of\ Substance}{Displacement\ of\ solvent\ front} = 0.65$

Percent Yield:

Experimental mass: 0.20 g

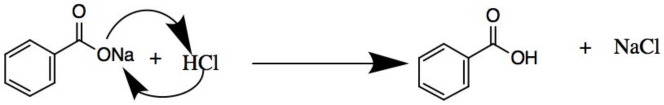
Moles of NaOH = (0.0301 L) × (2.0 M of NaOH) = 0.0602 moles of NaOH

Considering the chemical equation:



Every one mole of NaOH produces one mole of sodium benzoate

Considering the following chemical equation:



Every one mole of sodium benzoate produces one mole of NaCl

Overall, 0.0602 moles of NaCl were produced.

Theoretical yield (mass) = moles of NaCl × molar mass of NaCl

= 0.0602 moles of NaCl × 58.44 g/mol = 3.53 g of NaCl

Percent Yield = $\frac{Experimental\ Yield}{Theoretical\ Yield} \times 100\%$
$= \frac{0.2\ g\ of\ NaCl}{3.52\ of\ NaCl} \times 100\% = 5.66\%$

The percent yield of sodium chloride is 5.66%.

Discussion:

During the extraction of methyl blue dye, the aqueous layer of the mixture turns to a blue colour while the organic layer remained transparent after the test tube was shaken. This means that the methyl blue is soluble with water. Meanwhile, the methyl red had no effect on the mixture, as the ether and the aqueous layers remained colourless. This may be resolved if additional momentum had been applied to the test tube contains the mixture For the extraction of crystal violet dye, both the organic and aqueous layers became a violet colour, indicating solubility in both organic and aqueous solvents. When the salt was added to the mixture, being an electrolytic substance, the ions of the salt dissociate in the aqueous layer but not the organic layer. With the increasing amount of solutes in the aqueous layer and the water molecules having a better affinity to the ions, the crystal violet became less soluble in the water but not the ether. It results in the aqueous layer being a faint violet colour while the ether remains a bold violet.

The reactive separation proceeds as follows: the mixture of benzoic and an another aromatic molecule is poured into the separatory funnel in dichloromethane solvent along with an aqueous solution of sodium hydroxide. The hydroxide ions of the sodium hydroxide then attacks the alcohol component of the carboxylic acid group of the benzoic acid but not the other aromatic molecule. This results in the formation of benzoate ions, which is soluble in water but not in the dichloromethane and hence, migrates to the aqueous layer while the aromatic molecule remains in the dichloromethane. The dichloromethane layer is then extracted (being a denser solvent) in one container while the aqueous layer containing benzoate ions is extracted into another container. This aqueous phase is treated with an acid (hydrochloric acid) which donates a proton to the carboxyl group of the benzoate ions, producing benzoic acid and a salt. This addition of the hydrochloric acid was to the extent where the mixture became translucent and solid precipitates appeared. This indicates that at that point, the concentration of sodium and chloride ions has caused the solubility product to be exceeded and as a result (derived from the equation: $NaCl\ (s) \rightleftharpoons Na^+(aq) + Cl^-(aq)$), the ions associate together to form solid NaCl. The benzoic acid can be extracted by first filtering the aqueous mixture through suction funnel to remove the solid NaCl and then pouring the mixture into a separatory funnel along with dichloromethane. The benzoic acid will migrate to the dichloromethane layer, where it can be extract out.

The percent yield of NaCl is surprisingly low. A plausible explanation for this low yield is that during the act of pouring the aqueous mixture into the suction beaker through the funnel, some solid precipitates remained in the flask and required the addition of distilled water into the flask in order to remain the solid NaCl. It is likely that some of the NaCl dissociate into the distilled water, decreasing the moles (and hence, mass) of NaCl that can be extracted. As a result, some of the NaCl was lost into the aqueous mixture in the suction flask.

Question:

1. It is difficult to perform an extraction using ethanol and water because as a result of both water and ethanol having polarity due to their O-H bonds, the two substances are miscible to each other and produces a single layer where the distinction between the ethanol layer and the water layer is very limited.
2. As a result of the salting out effect, adding NaCl to a test tube containing water, ether and methylene blue would likely decrease the amount of dye in the aqueous layer. This is due to the dissolution of NaCl into ions, which electrolytically interact with the water molecules more effectively than the methylene blue. As a result, the decreasing number of water molecules that interact with the methylene blue causes molecules of the blue dye to no longer mix with the aqueous layer and causing the dye to migrate to another layer.
3. Solubility in water: 2.0 g/100 mL   Solubility in ether: 20.0 g/100 mL
   KD =Solubility in water/Solubility in ether = 0.1
   $0.1 = ((1.8\text{ g} - x)/(100\text{ mL}))/((x)/(100\text{ mL}))$
   $x = 1.64$ g
   In a single extraction with 100 mL of ether, 1.64 g of Compound Y was removed.
4. KD = Solubility in water/Solubility in ether = $([1.8\text{ g} - x_1]/100\text{ mL})/(([x_1])/(50\text{ mL})) =$ 0.1
   $x_1 = 1.5$
   1.5 g of Compound Y was removed, leaving 0.3 grams.
   KD = Solubility in water/Solubility in ether = $([0.3\text{ g} - x_2]/100\text{ mL})/(([x_2])/(50\text{ mL})) =$

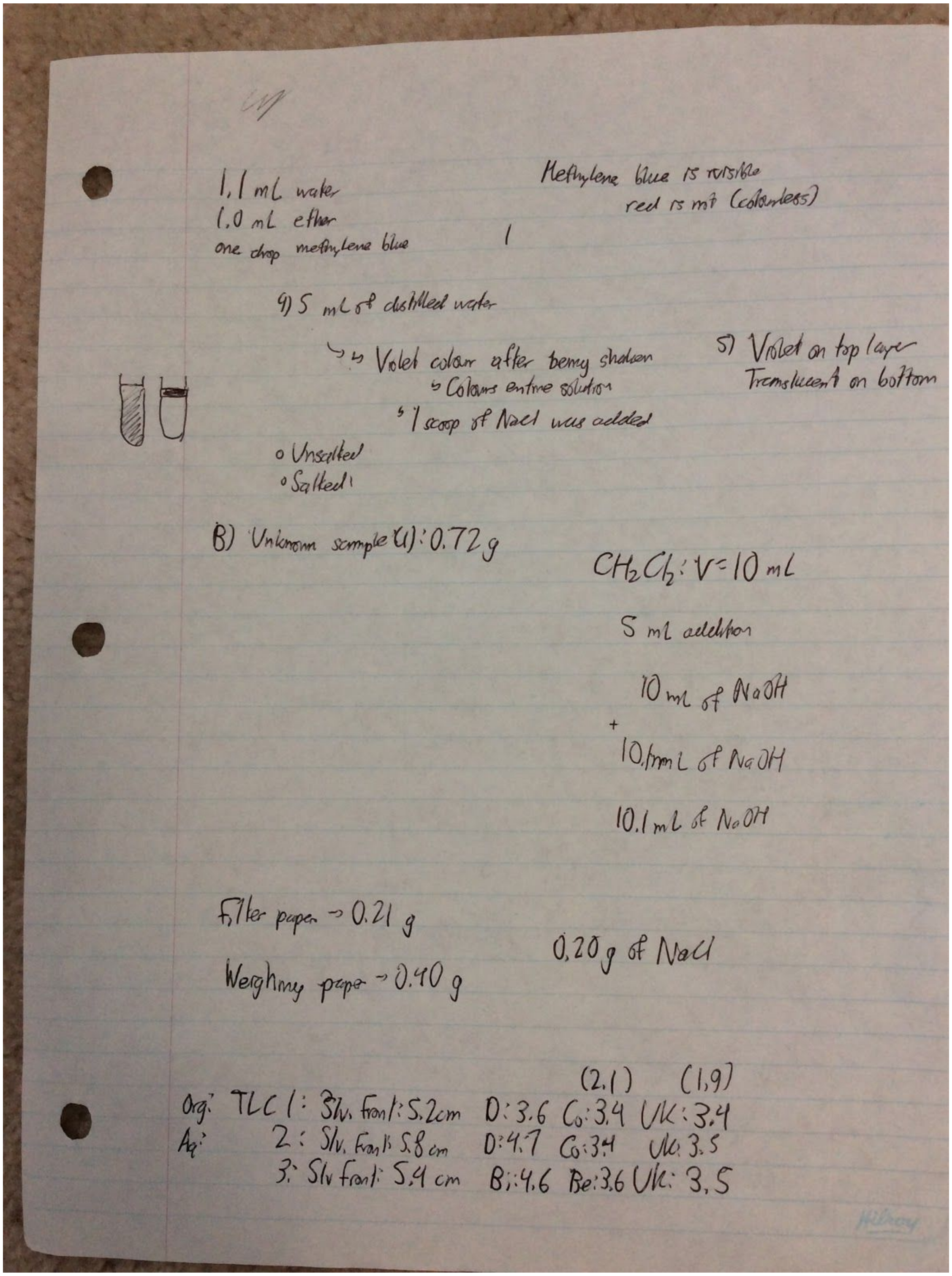
0.1

   $x_2 = 0.25$ g
   0.25 g of Compound Y was removed, leaving 0.05 grams.

Overall, 1.75 grams of Compound Y would be removed from two extractions suing 50 mL of ether.

5. One way to determine which of the layer is aqueous is to add a few drops of distilled water. If the water mixes with the layer, it is aqueous. If not, the layer is organic. Another possible way to determine which of the layers is aqueous is through the density of the solvent phases. For instance, an organic solvent such as an ether typically has less density than water. So in an extraction involving an organic solvent and an aqueous phase, the bottom layer is the aqueous layer. In addition, a chlorinated solvent like chloromethane is denser than water, so in an extraction involving chlorinated solvent and water, the aqueous layer is the top layer.
6. In order to separate a mixture of benzylamine from naphthalene, an acid such hydrochloric acid first is added to the mixture consisting of ether, benzylamine and naphthalene. The acid is interact only with the benzylamine and not the naphthalene since naphthalene is not a base. The acid donates its proton to the amine group of the benzylamine, turning the amine into an ammonium ion and hence, making the benzylamine water soluble. The naphthalene, meanwhile, remains in the organic ether layer. After the benzylamine migrates to the aqueous phase, the bottom aqueous layer (being more dense than the ether) is extracted from the separatory funnel. The aqueous phase is then treated with the addition of a base such as sodium hydroxide. This causes the ammonium group of the benzylamine to donate the hydroxide ions from the base, converting the benzylamine into a hydrophobic substance. The resulting salt is removed from the aqueous mixture through suction filtration and the benzylamine is recovered by extracting the chemical with a separatory funnel containing dichloromethane and water, in which the benzylamine migrates and mix into the dichloromethane layer.

Raw Data:



1.1 mL water
1.0 mL ether
one drop methylene blue

Methylene blue is visible
red is not (colourless)

4) 5 mL of distilled water

↳ Violet colour after being shaken
↳ Colours entire solution
↳ 1 scoop of NaCl was added

5) Violet on top layer
Translucent on bottom

- Unsalted
- Salted

B) Unknown sample (4): 0.72 g

CH_2Cl_2: V = 10 mL

5 mL addition

10 mL of NaOH
+
10.1 mL of NaOH

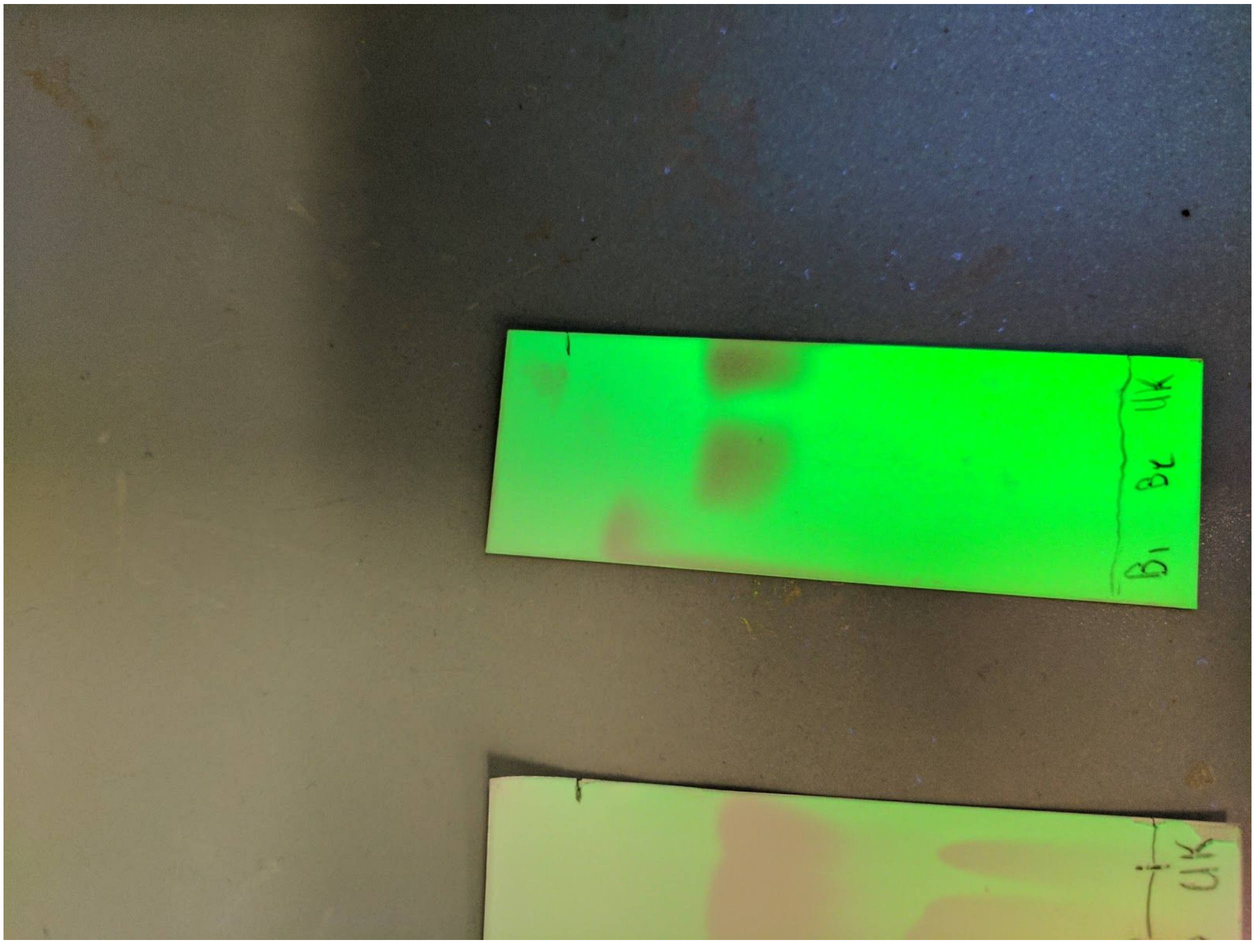
10.1 mL of NaOH

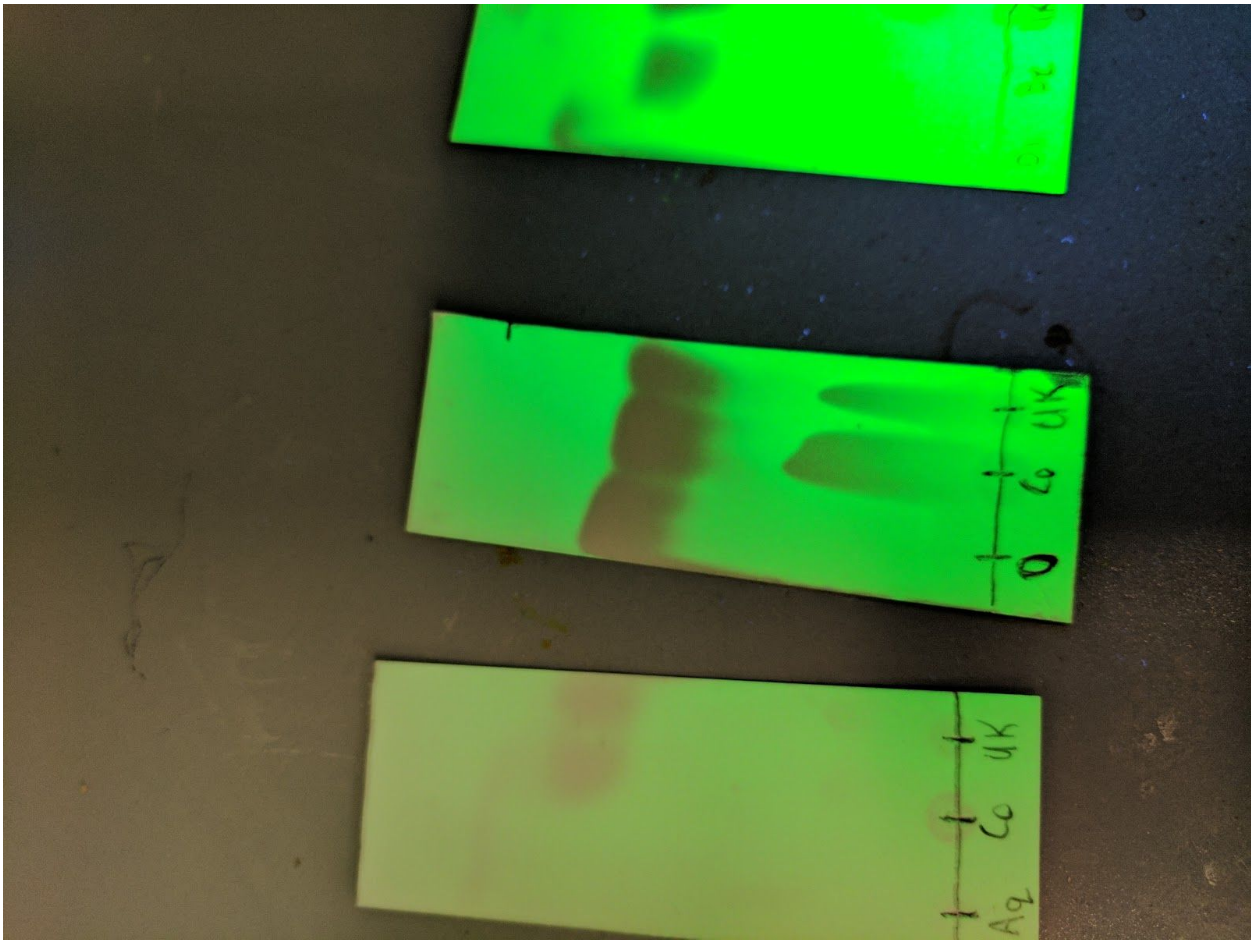
Filter paper → 0.21 g

Weighing paper → 0.40 g

0.20 g of NaCl

(2.1) (1.9)
Org: TLC 1: Slv. front: 5.2 cm D: 3.6 Co: 3.4 UK: 3.4
Aq: 2: Slv. front: 5.8 cm D: 4.7 Co: 3.4 UK: 3.5
3: Slv front: 5.4 cm Bi: 4.6 Be: 3.6 UK: 3.5


B1
B2
UK
UK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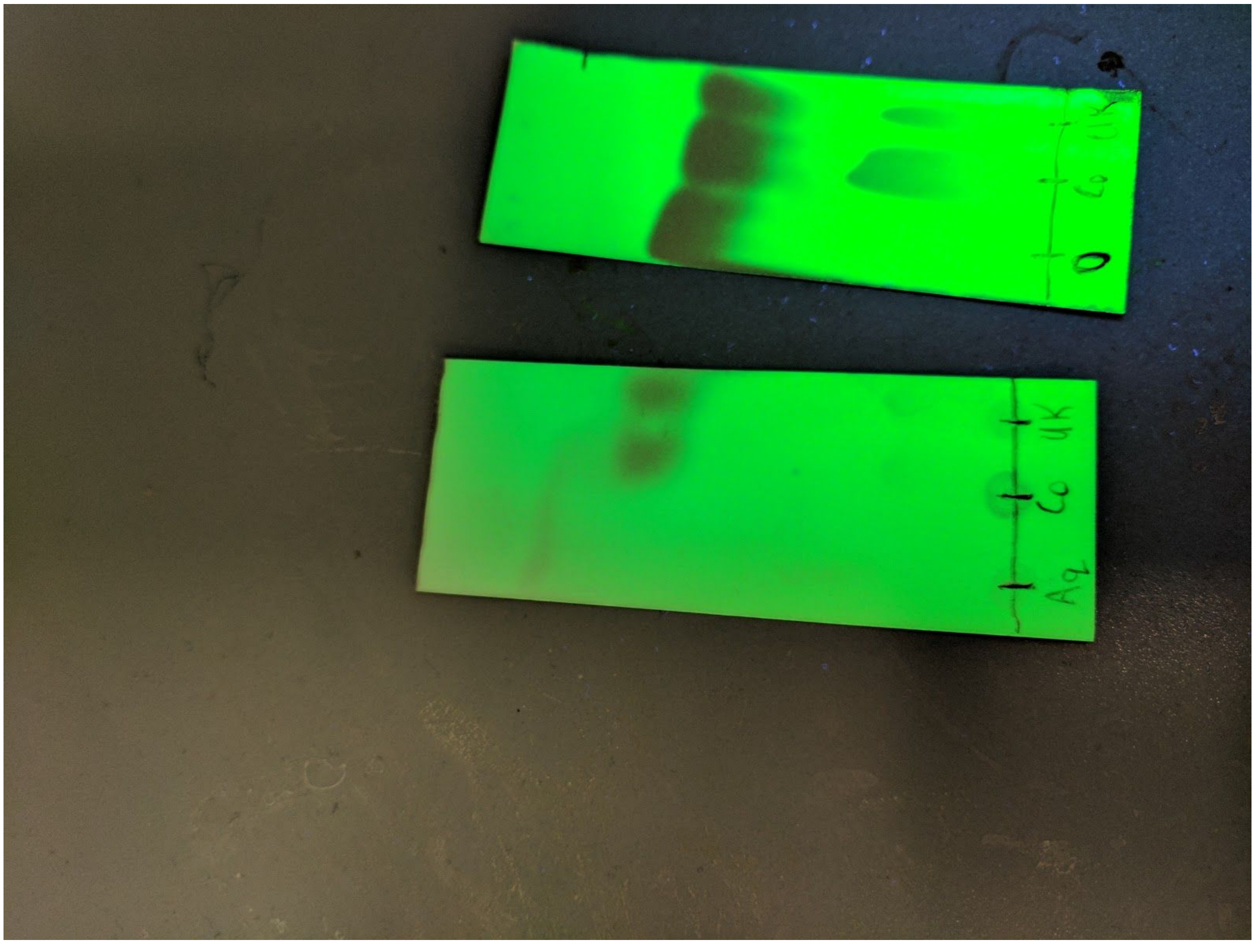
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