

Lab 3: Extraction

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March 10, 2019

Procedure: See laboratory manual: "Extraction" CHM 1321 Organic Chemistry Laboratory Manual, Dr. Rashmi Venkateswaran , Exp.3, p. 5-6

Observation:

-The first dyes used were methyl red and methyl blue mixed together with solvents ethyl (organic top layer) and water (aqueous bottom layer) where methyl red was at the top and methyl blue was at the bottom. The second part used crystal violet where when NaOH was added, the purple colour accumulated at the top, clear at the bottom.

1. Gas came out in between shaking of separatory funnel when stopcock opened.
2. Visible separation line was observed in between the organic and the aqueous layer. Organic layer on the top, aqueous on the bottom.
3. Aqueous solvent became cloudy white when HCl was added and eventually visible precipitate occurred on the bottom of the flask.
4. Both organic and aqueous solvents were transparent
5. Solid, powdery products started to appear in the gravitational filter

-Based on the aforementioned observations extraction is a good method to separate methyl red and blue using ether and water.

Table of Result: (unknown number, initial mass, mass obtained, composition and composition %)

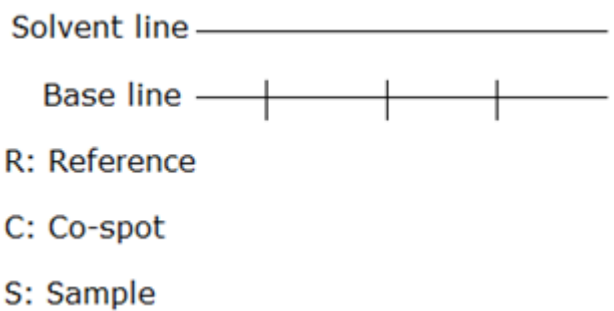
Table 1

Extraction Result

Unknown number	94
Initial mass	0.55g
Mass obtained	0.44g
Composition	biphenyl
Composition %	80%

TLC Plates:

Legend: For all figures below



Test between unknown #94 and organic solutes:

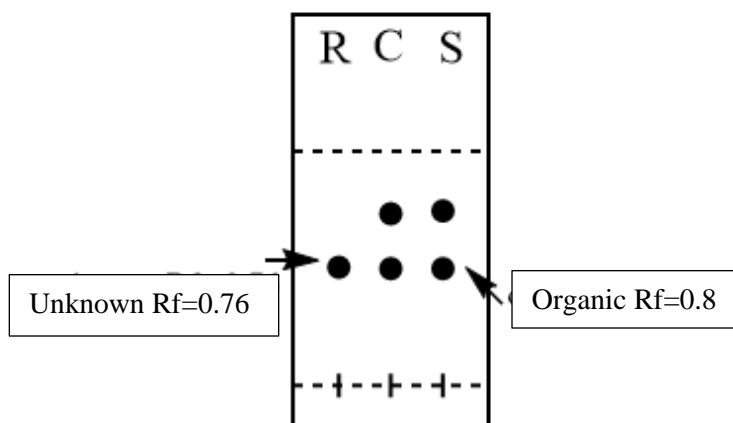


Figure 1. TLC plate with organic layer of separatory funnel on the sample spot (S), unknown #94 on the reference spot (R) and both of the spots are on co-spot(C). TLC plate was developed in 2:8 ethyl acetate and hexane.

Test between unknown #94 and dichloromethane (DCM):

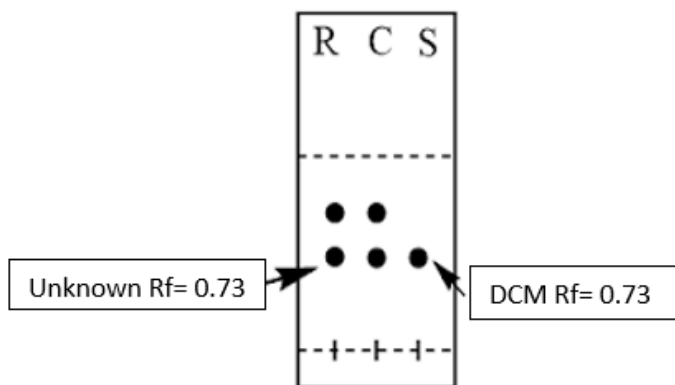


Figure 2. TLC plate with dichloromethane as sample, unknown #94 as reference and both spots on the co-spot. TLC plate developed with the solvent of 2:8 mixture of ethyl acetate and hexane.

Test between unknown #94 and organic solvent:

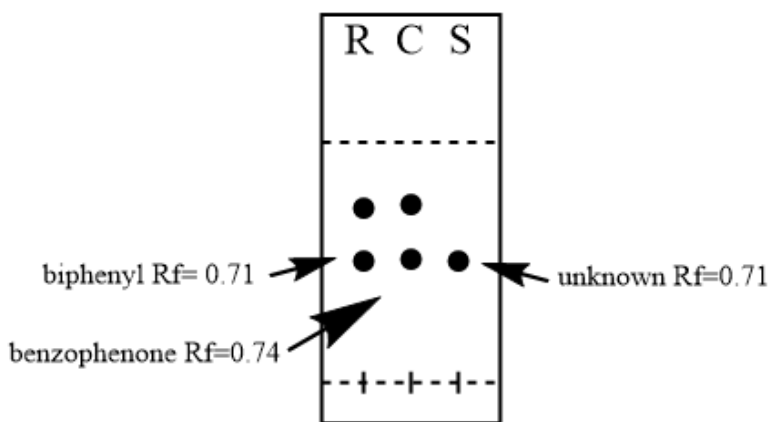


Figure 3. TLC developed in 2:8 ethyl acetate and hexane. Biphenyl on the right, benzophenone in the middle and unknown # 94 on the left.

Calculations:

Rf values are calculated individually by using $R_f = R_1/R_2$

R1 is the measurement of distance travelled by the dots from the starting line

R2 is the measurement of the solvent travelled from the starting line.

Example:

Unknown Rf- $R_1=1.9\text{cm}$, $R_2= 2.5\text{cm}$

$1.9\text{cm}/2.5\text{cm}=0.76$

Composition % = (post extraction mass / initial extraction mass) x 100

Example:

Initial mass of unknown #94 = 0.55g

Post-extraction of unknown #94 = 0.44g

$(0.44\text{g}/0.55\text{g}) \times 100 = 80\%$

Therefore, 80% of composition percentage was obtained from extraction.

Data:

Table 1. TLC test for presence of organic compound:

	R1(cm)	R2(cm)	Rf
Unknown #94 reference	1.9	2.5	0.76
Co-Spot	1.8 and 2.5	2.5	0.72 and 1
Organic Sample	2 and 2.5	2.5	0.8 and 1

Table 2. TLC test for presence of dichloromethane compound:

	R1(cm)	R2(cm)	Rf
Unknown #94 reference	1.4 and 1.9	2.2	0.73 and 0.86
Co-Spot	1.4 and 1.9	2.2	0.73 and 0.86
Dichloromethane Sample	1.4	2.2	0.73

Table 3. TLC test for presence of biphenyl and benzophenone compounds:

	R1(cm)	R2(cm)	Rf
Biphenyl	2.2 and 2.9	3.1	0.71 and 0.94
Benzophenone	2.3 and 2.8	3.1	0.74 and 0.9
Unknown #94	2.2	3.1	0.71

Discussion:

In part A, the purpose was to find if ether and water work as extraction solvents. In the experiment, when methyl red was added to the test tube, yellow colour was on the top layer. In another test tube, when methyl blue was added, the blue is on the bottom layer.

After the mixture of the two test tubes, the methyl blue remained on the bottom and methyl red remained on the top. Ether is less dense than water so it is on top of water when mixed. The organic structure of methyl red does not have any permanent charge so it dissolves in ether (top) while methyl blue has permanent charge so it dissolves with water (bottom). Crystal violet was used for the second part to see the salting out effect. Without NaCl, the test tube distributes purple colour evenly. With NaCl, the test tube has purple colour accumulated on the top and clear on the bottom.

in part B, the purpose was to determine the unknown substance (#94) using extraction and TLC plates. The TLC plates from figures 1, 2, and 3 show that the unknown substance is biphenyl, contains organic substance as well as dichloromethane. Dichloromethane was mistakenly used in place of aqueous. Due to this error, it is difficult to determine if the extraction of benzoic acid was successful. If the extraction was successful, the unknown spot should not have lined up with the aqueous layer spot on TLC plates. The unknown is determined to be biphenyl because it is lined up with the biphenyl dot, benzophenone was slightly higher. Biphenyl should be lower than benzophenone because of the polarity it has on its functional group (oxygen and hydrogen) which gives the ability to adsorb with the silica gel (TLC plate).

Sources of error:

During the gravitational filtration, some powder was clearly dropping in to water from the funnel. Although not in large quantity, it could have affected the calculated percentage composition. Another source of error is that it was difficult to tell if a capillary was completely clean, this could be improved by adding colour to the liquids. The reason for high percentage of composition may be due to the water not being completely dried out which made the actual mass heavier than it actually is.

Questions:

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

It would be difficult to perform an extraction using acetone and water because liquid to liquid extraction requires the two solvents to be immiscible of each other. If water and acetone were chosen as solvents for extraction, there will not be any layers because they are equally dense

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 will decrease the amount of dye in the aqueous layer since NaCl will only dissolve in water while the dye is less soluble in water.

3. Compound Y has a solubility of 2.0 g/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?

$K_d = \text{solubility ether(organic)} / \text{solubility water(aqueous)}$, $K_d = 10$.

$10 = [W_1/V_1] \text{ ether} / [W_2/V_2] \text{ water}$.

$10 = (X/100) / (1.4g - X/100)$. $X = 1.3g$

Therefore: The mass of compound Y removed from the solution is 1.3g of ether extracted from a single extraction.

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?

1st: $K_d = 10 = (x/50\text{ml}) / (1.4g - x/100)$. $X = 1.3g \rightarrow$ weight for second extraction: $1.4g - 1.3g = 0.1g$

2nd: $K_d = 10 = (x/50\text{ml}) / (0.1g - x/100)$. $X = 0.08g$

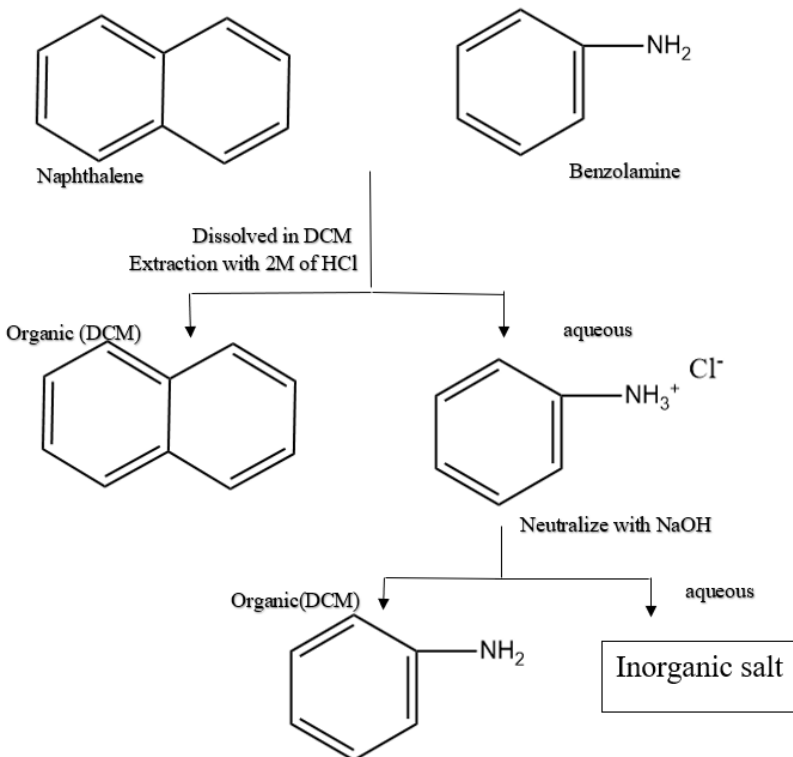
$X_{\text{total}} = 1.3 + 0.08 = 1.11g$

Therefore: The mass of compound Y removed from the original water solution is 1.11g of ether extracted from two extractions.

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?

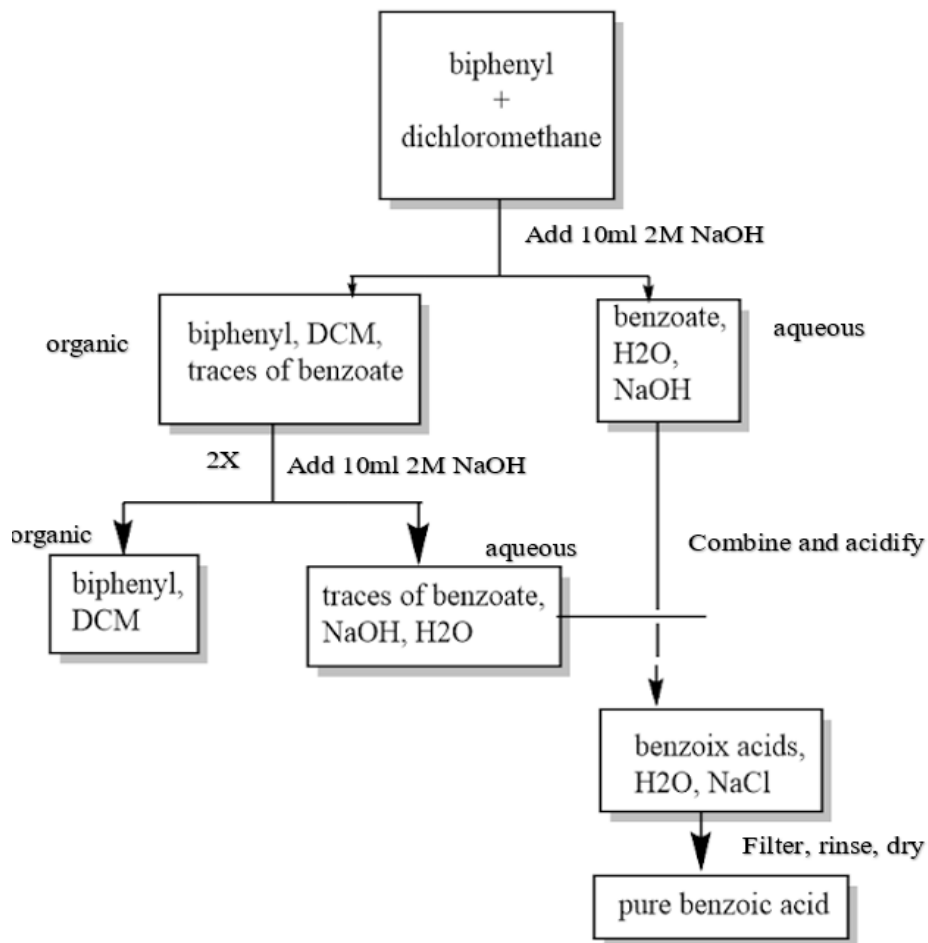
In order to identify the layer, she could add water to the layers. Because organic solvent will not mix with aqueous (water), organic phase will be either at top or at the bottom.

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



Benzylamine has a lone pair (can act as a base) will pair up with H in HCl and get a positive charge and becomes soluble in water. Naphthalene is in organic layer that is unreactive to acid or bases. Aqueous layer will have $\text{Ph-NH}_3^+ \text{Cl}^-$ (ammonium salt). Performed the extraction three more times and combine with aqueous layer. Ammonium salt neutralized with NaOH and becomes Ph-NH_2 (s). The extraction is done with an acid (HCl) which reacts with benzylamine.

Part B Flow Chart:



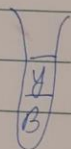
Raw Data:

Part A
Exp 3. movement of the desired compound.
Water density: 997 kg/m^3
Ether density: 713 kg/m^3 (should be on top)
Salt in aqueous \uparrow ionic strength and pushes out organic solvent
1-butanol density: 810 kg/m^3

unknown 94: 0.55g.

part A

1. blue bottom, clear at top (ether) dye in aq
2. dye in organic (red/yellow on top, aq on bottom)
3. when both solutions mixed together, they were still separated with yellow/red dye on top & blue on bottom



5. When NaCl was added to 1 of the tubes, the bottom of test tube was clear & the top has purple accumulated and the other one was uniform

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part B.

-organic (yellowish) at bottom.

-repeated twice.

original 0.55g

1. 27 sec

2. 26 sec

3. 30.14 sec

} shake time

Paper: 0.01g, product ~~+ Paper~~ = 0.44g

	Organic R ₁	R ₂
(u) R	1.9	2.5cm
C	1.8/2.5	2.5
(o) S	2cm/2.5	2.5
<hr/>		
	DMF	
(u) R	1.9 1.4/1.9	2.2
C	1.4/1.9	2.2
(o) S	1.4	2.2
(DMF)		
<hr/>		
	bi, ben, u	
bi	2.2/2.9	3.1 cm
ben	2.3/2.8	3.1
u	2.2	3.1

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