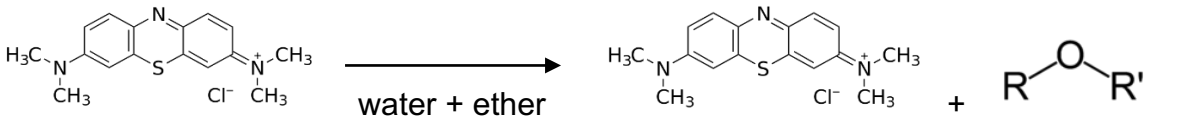
Experiment 3
Extraction
Valeriya Kohar
February 24th, 2016

Introduction:
The purpose of this experiment was to determine the unknown sample by using extraction and thin layer chromatography.
The process of extraction is used to separate different chemical compounds in immiscible solvents. In extractions, there is an aqueous phase which is usually water and the organic phase.
There is a rule that states that “like dissolves like”, henceforth polar solvents will dissolve in polar solvents and non-polar in non-polar. This separation will be seen in a faded line between the two layers. Usually the polar solvent will be aqueous and the non-polar organic.

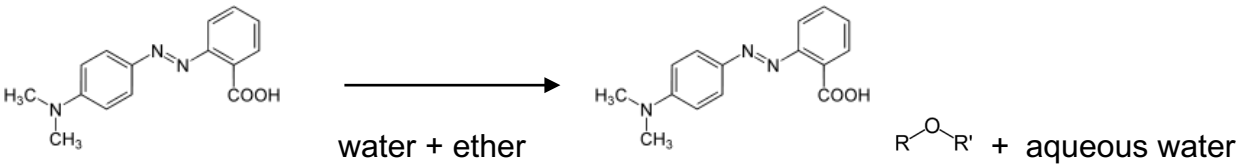
Part A- Extraction of water soluble dyes

Methylene blue separation:



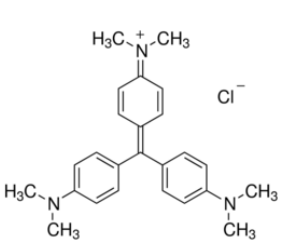
Methylene blue aqueous methylene blue organic ether

Methylene red separation:

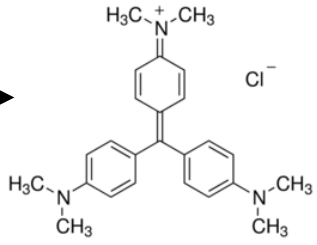


Methylene red organic methyl red and ether

Salting out Effect:



Ether+



+ H2O + NaOH

Water+
1-butanol

crystal violet

organic crystal violet and 1-butanol

*Refer to flowchart for the mechanisms for Part B.

Procedure:
Refer to:
Dr. Venkateswaran, Rashmi. CHM 1321 Organic Chemistry Laboratory. *Experiment 3: Extraction.* 2013. p.32-33. Print.

Observations:

Table 1: Qualitative observations of the solutions and compounds used during part A and B of the experiment.

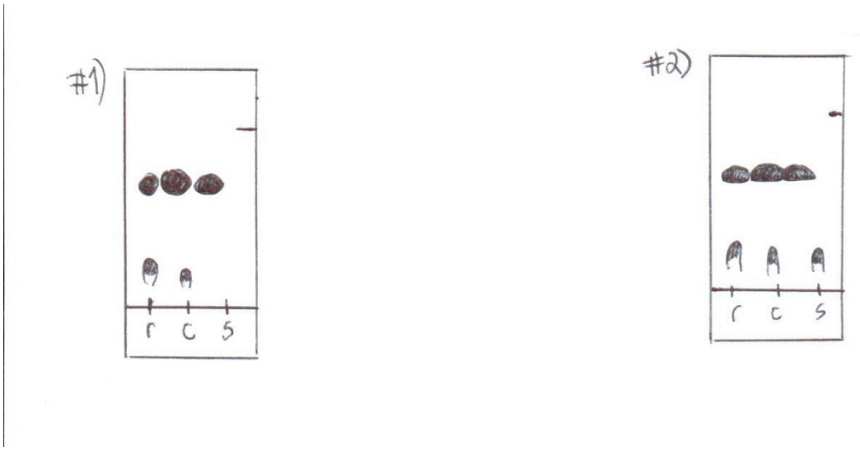
Compound	**Observation**
Ether	Clear, colourless, liquid
Methyl Blue + ether + water	Clear on the top layer and blue settled to the bottom
Methyl red + ether + water	Clear on the bottom layer and red settled at the top
Methyl Blue + Methyl Red + water + ether	Red on the top layer and blue on the bottom
Aqueous crystal violet + 1-butanol + water	Opaque purple solution
Aqueous crystal violet + 1-butanol + water + solid NaCl	The addition of NaCl makes the solution to become a dark purple at the top and almost clear on the bottom
Unknown sample # 1	Small white solid
Dichloromethane	Clear, colourless liquid
NaOH	Clear, colourless liquid
12.0M HCl	Clear, colourless liquid
Biphenyl	Small white solid
Benzophenone	Small while solid

Table 2: Quantitative observations for the unknown sample for Part B.

Compound	**Observation**
Mass of unknown sample #1 before extraction	0.76 g
Mass of unknown sample #1 after extraction	0.2066g
Percent yield of unknown sample #1	27.2 %

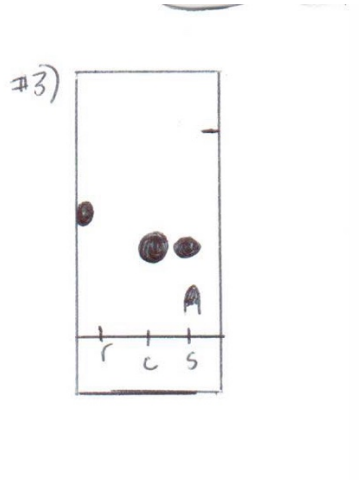
TLC Plates:

Legend:
R= Reference
R+S= Reference + Sample (co-spot)
S=Sample



organic layer Rf = 0.70
unknown mixture # 1 Rf = 0.37

Unknown mixture #1 Rf = 0.62
Aqueous layer dissolved in dichloromethane Rf = 0.62
Aqueous layer dissolved in dichloromethane Rf = 0.37
Unknown mixture #1 Rf = 0.37



Biphenyl Rf = 0.82
Benzophenone Rf = 0.65
Unknown mixture #1 Rf= 0.61 and 0.37

Questions:

1. Using ethanol and water would make the extraction difficult to perform due to the fact that ethanol is polar and so is water. Because of the rule “like dissolves like”, ethanol would just dissolve in water. This would not allow the separation to be seen.

2. The addition of NaCl to a test tube with water, ether and methylene blue would increase the amount of dye due to the fact that the salt will cause the water to become more ionic. Methylene blue is polar and henceforth it will dissolve in the water causing the ether layer to separate and the methylene blue will be in the aqueous layer.

3. KD = W1/V1 = 10
W2/V2

Ratio →water to ether is 20/2= 10.

X = mass of compound
Y = what is extracted from the ether
Z = mass of compound Y that is extracted from the water.

X+Z= 1.8g
Z= 1.8- X
KD= Xg/100ml of ether/Zg/100ml of water
10 = X/1.8-X
X= 1.64g
1.64g of compound Y would be removed by a single extraction with ether.

4. This is the same process as question 3, however instead of 100mL ether it is now 50 mL.

10= Xg/50ml of ether/ Zg/100ml of water
5 = X/1.8-X
X= 1.5g

1.8-1.5= **0.3g.**

5. The student can determine this by dropping water into the mixture. Because water is polar, it will mix with the aqueous phase. If the drop mixes with the bottom, then the aqueous layer is at the bottom and if it mixes with the top, then the aqueous layer is at the top.

6. This separation can be achieved by adding an acid. When an acid is added, it would convert the benzyl anime to a salt which would be aqueous. Benzyl amine would become an aqueous layer and naphthalene an organic layer.

Discussion

The purpose of this experiment was to determine the unknown sample #1 by using extraction and thin layer chromatography. By observing the given observations and the TLC plates, it was determined that the unknown was benzophenone and benzoic acid.

The first part of the experiment involved the mixing of methyl blue and ether and water. Ether is less dense henceforth it formed the organic layer on the top. When methyl blue was added, it mixed with the bottom layer of water since it is water-soluble. When methyl red was added, it mixed with the organic layer because it is non polar and hydrophobic. When both of the mixtures were added to one another, the blue stayed at the bottom and the red at the top.

Once a vast amount of solid NaCl was added to the aqueous crystal violet, 1-butanol and water, it caused it to become saturated henceforth the bottom was clear and the top dark purple.

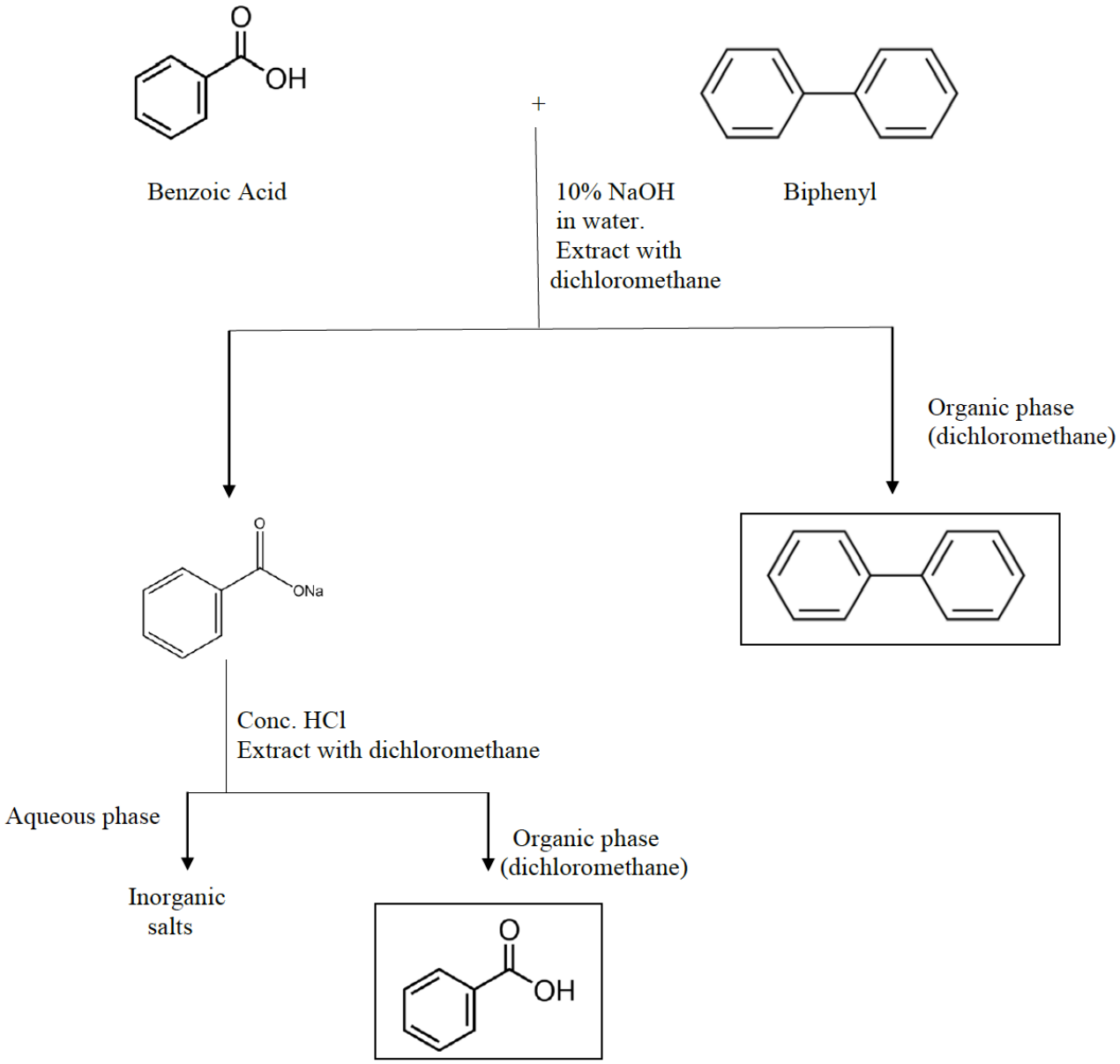
When looking at TLC plates, it can be noted that the Rf values are higher for the organic phase rather than the aqueous. This is because the non-polar organic phase moves up the plate faster than the aqueous polar phase. In the case of the uknown sample, it can be said that the benzoic acid was in the aqueous layer and the benzophenone was in the organic.

There are many errors that could have changed the results. An error that could have occurred is that not all of the precipitate was collected after it was cooled and dried. Another error that could have occurred is that the spotting on the TLC plates would be too big. To fix that error, new TLC plates would have to be prepared.

Conclusion:

The unknown sample #1 was determined to be benzophenone and benzoic acid with a percentage yield of 27.2 %.

The following flow chart exhibits the reactive separation during the extraction:



Reference:

Dr. Venkateswaran, Rashmi. CHM 1321 Organic Chemistry Laboratory. *Experiment 3: Extraction.* 2013. p.28-33. Print.