EXTRACTION

Experiment 3

CHM1321 Section D7

Thursday, February 25, 2016

INTRODUCTION

Extraction is a separation technique in chemistry. Different Chemical compounds are separated based on their solubilities in a mixture of two immiscible solvents commonly called phases. The process by which the desired compound moves from one phase to another it is called extracting but if it remains in one phase and the impurities move to the other phase it is known as washing. In most extractions, one phase is an organic solvent while the other is water (aqueous). The process is carried out in separatory funnel, because the solvent are immiscible they form two distinct layers. The more dense solvent is at the bottom of the funnel. When carrying out an extraction, the solution to be extracted is put into the funnel and then an immiscible solvent is added. The funnel is then shaken to disturbing compounds between the phases and then placed to stand. Each phase can then be removed carefully and the process is carried out several times to increase the efficiency of transfer of the compound.

OBSERVATIONS

Part A

Ethyl + Water - Aqueous phase below and Organic phase on top

1. 0.006M Methylene Blue - Dark blue in aqueous phase
2. 0.006M Methylene Red- Red in organic phase

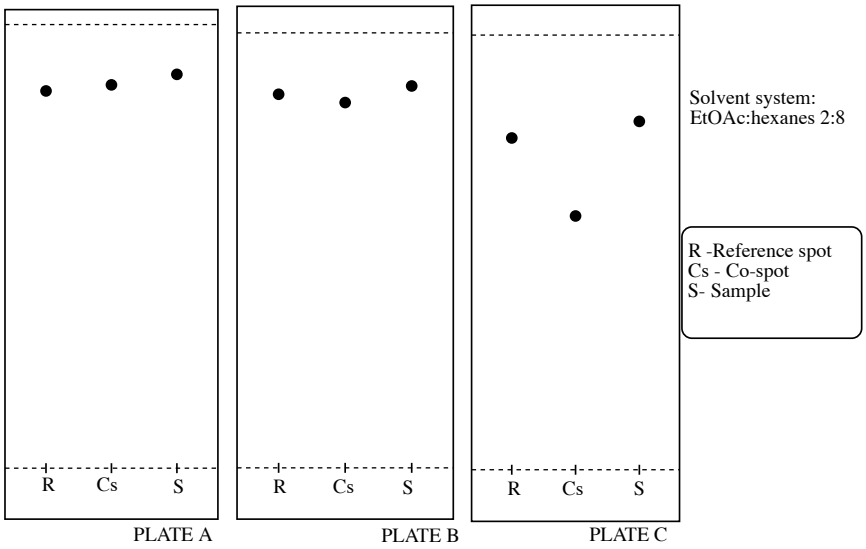
Water + 0.003M aqueous crystal violet + 1-butanol -

1. Test tube without NaCl - Purple colour distributed between both phases
2. Test tube with NaCl- Purple colour moves into organic phase

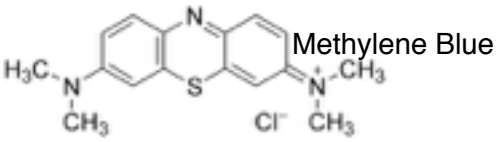
Unknown sample 2 - 0.54g Crystalline White Solid

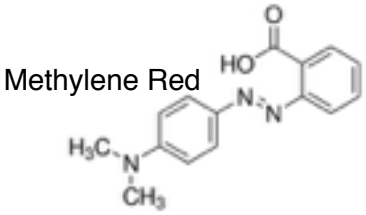
TLC PLATES

R_F Values	Reference spot	Co-spot	Sample spot
PLATE A	0.85	0.86	0.89
PLATE B	0.86	0.84	0.88
PLATE C	0.76	0.58	0.80



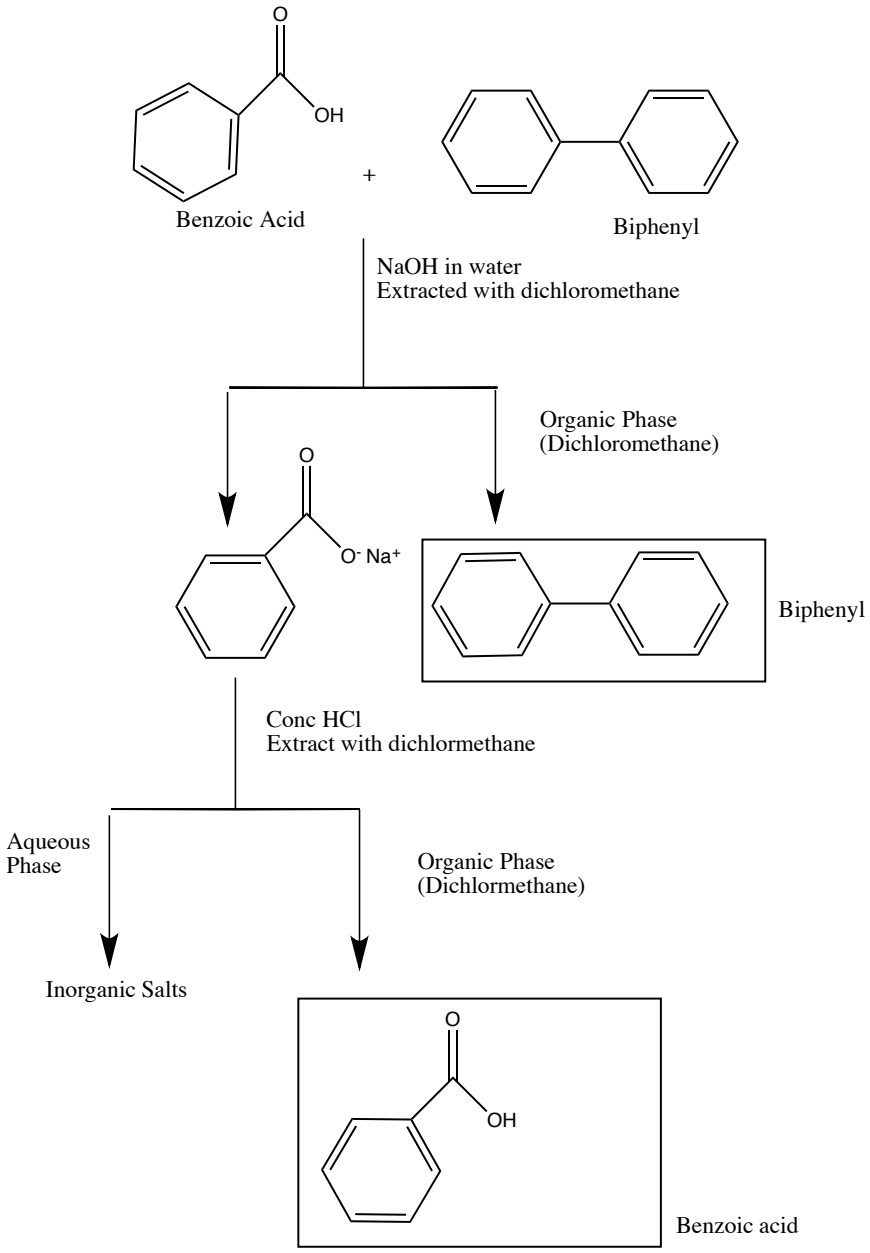
DISCUSSION





In part A, the methylene blue dissolves in the aqueous phase, looking at the structure above methylene blue contains atoms that can take part in hydrogen bonding. These atoms interact with water and these interactions allow methylene blue to dissolve in water. Methylene red on the other hand dissolves in the organic phase. In the structure above, there are atoms that can hydrogen bond present in the methylene red molecule however the non polar bonds counter the interactions between the atoms and water. Due to this reason methylene red can does not dissolve in the aqueous phase, it dissolves in the organic phase. Mixing the contents of both test tubes together, the yellowish red colour is seen on the organic phase and the blue is in the aqueous. Because methylene blue and red dissolve in different phases, ethyl and water would be a good way to separate the two compounds. Crystal Violet is soluble in both 1-butanol and water, so the contents of the test tubes were purple. When the salt NaCl was added into the test tube, it reacted with water and made crystal violet less soluble in water, thereby pushing it into the organic phase.

In part B, looking at plate C, biphenyl and the unknown mixture have similar R_F values meaning that unknown sample is a mixture of biphenyl and benzoic acid. While carrying out the extraction, NaOH was added into the aqueous phase. When the funnel was shaken, NaOH reacted with benzoic acid in the organic phase and converted it to sodium benzoate, a salt that can be dissolved in the aqueous phase. HCl was then added to the aqueous phase to convert it back to benzoic acid, the mixture was cooled and the solid was collected by filtration. The percentage yield of the reaction 41% meaning that the organic phase still contained a significant amount of benzoic acid, the extraction could be done several more times. The reactive separation is simplified in a flow chart below



QUESTIONS

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

It would be difficult to use ethanol and water to perform an extraction because they are both polar solvents.

2. Would adding NaCl to a test tube containing water, ether and methylene blue increase and decrease the amount of dye in the aqueous layer?

It would decrease the amount of die in the aqueous layer, this is because NaCl will react with water and the water will become saturated with NaCl. This reduces the solubility of dye in the aqueous phase and pushes it into the organic phase.

3. Compound Y has a solubility of 2.0g/100mL in water and 20.0 g/100 mL in ether. What mass of compound Y would be removed from a solution of 1.8 g of Y in 100 mL of water by a single extraction with 100 mL of ether?

$$K_D = \frac{[Y]_{Organic}}{[Y]_{Aqueous}}$$

$[Y]_{Aqueous} = 0.02$ g/mL $[Y]_{Organic} = 0.2$g/mL $K_D = \frac{0.2}{0.02} = 10$

$$10 = \frac{x \text{ g}/100\text{mL}}{(1.8-x) \text{ g}/100\text{mL}}$$

Let x be the mass of compound Y that will move from the aqueous phase to the organic phase

$$10 = \frac{x \text{ g}}{(1.8-x) \text{ g}} \qquad x = 18/11 \cong 1.6 \text{ g}$$

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? $K_D = 10$

1st Extraction: x is the mass of compound Y removed after extraction 1

$$10 = \frac{x\ g\ /50\ mL}{(1.8\text{-}x)g\ /\ 100mL} \qquad 10 = \frac{2x\ g}{(1.8\ -x)\ g} \qquad x = \frac{18}{12}\ g = 1.5g$$

2nd Extraction: t is the mass of compound Y removed after extraction 2

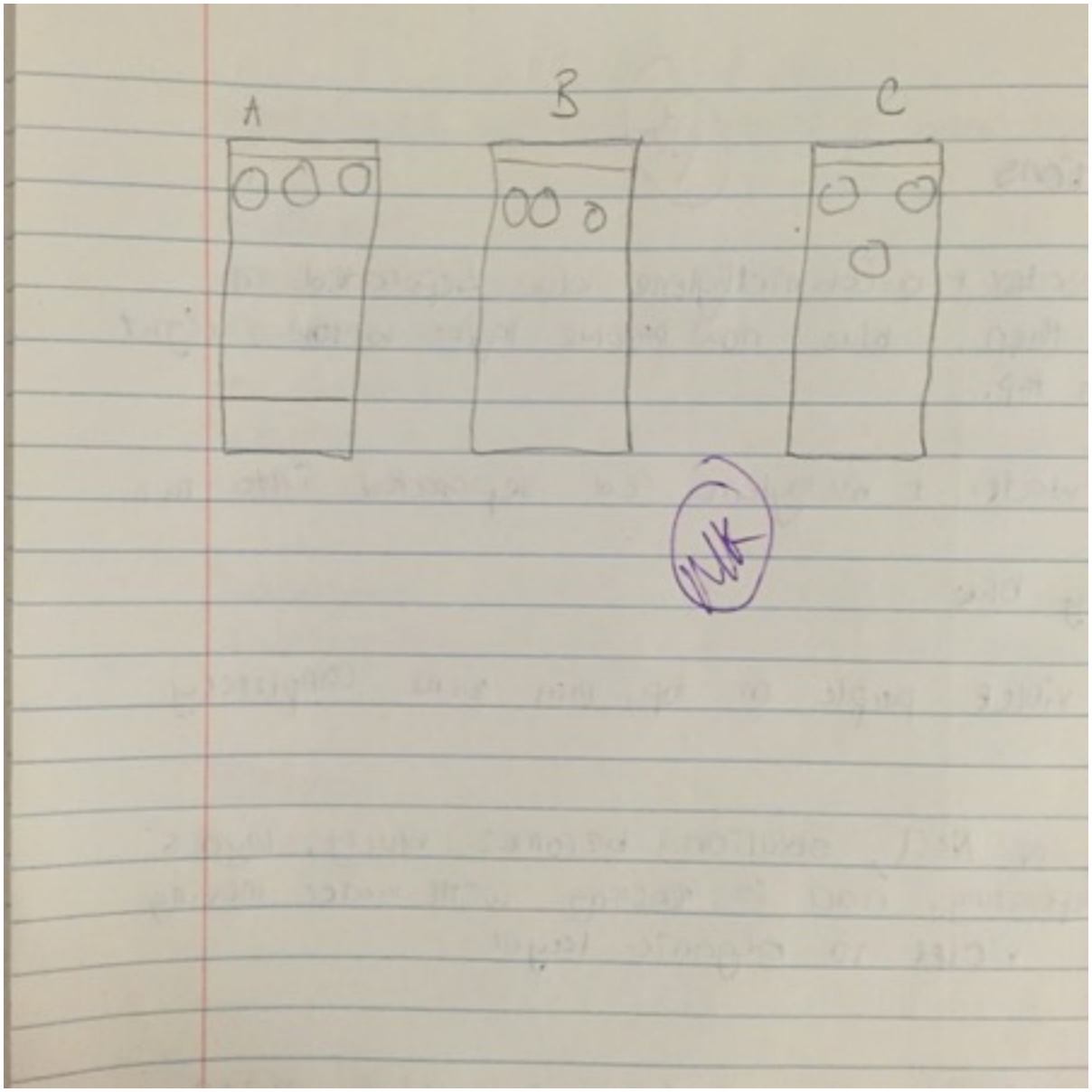
$$10 = \frac{t\ g\ /50\ mL}{(0.3\text{-}t)\ g/\ 100mL} \qquad 10 = \frac{2t\ g}{(0.3\text{-}t)\ g} \qquad x = \frac{3}{12}\ g \cong 0.25g$$

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?

By doing a water test, when drops of water are added to the separatory funnel, it would dissolve in the aqueous phase.

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.

By adding a sufficient amount of a strong acid such as HCl, the acid would react with the organic base forming a salt that would be soluble in the aqueous phase. The aqueous layer would then be removed and NaOH would be added to the solvent. The NaOH would react with the salt to produce benzyl amine, it is filtered then dried. The organic phase would be washed with water several times to ensure that all of the salt is removed a drying agent can be added to the organic solvent and naphthalene can be collected.


A
B
C

Observations

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Ethyl + water + 0.006 methylene blue seperated in purple then blue now DARKblue layer below, light blue on top.

ethyl + water + methylene red seperated into two completely blue

Crystal violet purple on top, then turns completely purple.

0.30g of NaCl, solution becomes blurry, layers start seperating. nacl is reacting with water, moving crystal violet to organic layer.

Unknown sample 2 - 0.54g crystalline white solid, ~~no smell~~

Precipitate - $\frac{0.22}{0.54} \times 100 =$

Rf Dp	R 5.3	CS 5	S 5
Plate A	4.5	4.6	4.7
PLATE B	4.3	4.2	4.4
PLATE C	3.8	2.9	4.0

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