1. EXPERIMENT # 1 GRACE KIM
7/2/2007 MIKE HALL
TWO BASE EXTRACTION

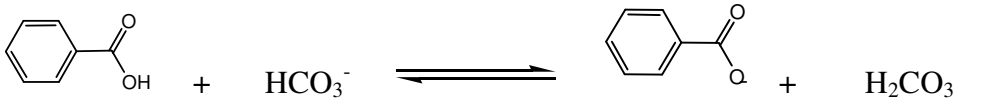
2. PURPOSE AND INTRODUCTION

A mixture of benzoic acid, 2-naphthol, and naphthalene (Mixture #156) dissolved in diethyl ether was separated using differences in acid-base reactivity and solubility.

Benzoic acid and 2-naphthol are both water-insoluble acids. When reacted with the appropriate base, these acids convert into water-soluble conjugate bases (i.e. benzoate, and 2-naphthoxide, respectively). After extraction, the aqueous layer can then be acidified to obtain the original acid.

Benzoic acid has a pK_a of 4.17, while 2-naphthol has a pK_a of 9.5. To selectively react and extract the stronger acid, benzoic acid, without reacting the 2-naphthol, a base was selected that had a conjugate acid with a pK_a greater than 4.17 but less than 9.5. After extraction of the benzoic acid, the 2-naphthol was extracted by a base with a conjugate acid with a pK_a greater than 9.5:

Reaction of benzoic acid:

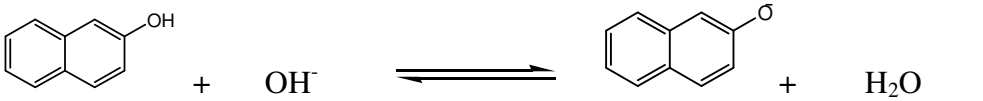


pK_a = 4.17
water-insoluble

water-soluble

pK_a = 6.37

Reaction of 2-naphthol:



pK_a = 9.5
water-insoluble

water-soluble

pK_a = 15.7

3. EXPERIMENTAL PROCEDURES

Table of Reagents:

Compound	*Molecular Weight*	*Physical Properties*		
Benzoic acid, C_6H_5COOH	122.1232 g/ mol	m.p. = 123^0C,	pK_a = 4.17	water-insoluble
2-naphthol, $C_{10}H_7OH$	144.1726 g/ mol	m.p. = 123^0C,	pK_a = 9.5	water-insoluble
Naphthalene, $C_{10}H_8$	128.1732 g/ mol	m.p. = 80^0C,	pK_a = ~35	water-insoluble
Diethyl Ether $CH_3CH_2OCH_2CH_3$	74.1224 g/ mol	b.p. = 35^0C	volatile	d = 0.7134 g/ mol
10% $NaHCO_3$ soln.	84.00687 g/ mol	K_a of H_2CO_3 = 6.37		
10% NaOH soln.	39.99707 g/ mol	K_a of H_2O = 15.7		
3 M HCl	36.4609 g/ mol			
Na_2SO_4 drying agent	142.03714 g/ mol			

Extraction of Benzoic acid with $NaHCO_3$ (Bicarbonate extraction)

A 2.08 g sample of Mixture #156 was weighed and dissolved in 30-mL diethyl ether and placed in a separatory funnel. 20-ml of aqueous 10% $NaHCO_3$ solution was added to react and extract the benzoic acid. After the first extraction, the remaining organic portion was treated with another 20-mL of aqueous 10% $NaHCO_3$ solution, and the aqueous extracts were combined.

Extraction of 2-naphthol with NaOH (Hydroxide extraction)

20-mL of cold aqueous 10% NaOH solution was added to the remaining organic ether in the separatory funnel to react and extract the 2-naphthol. The hydroxide extract was cooled in an ice bath to ensure proper crystallization of the 2-naphthol upon acidification.

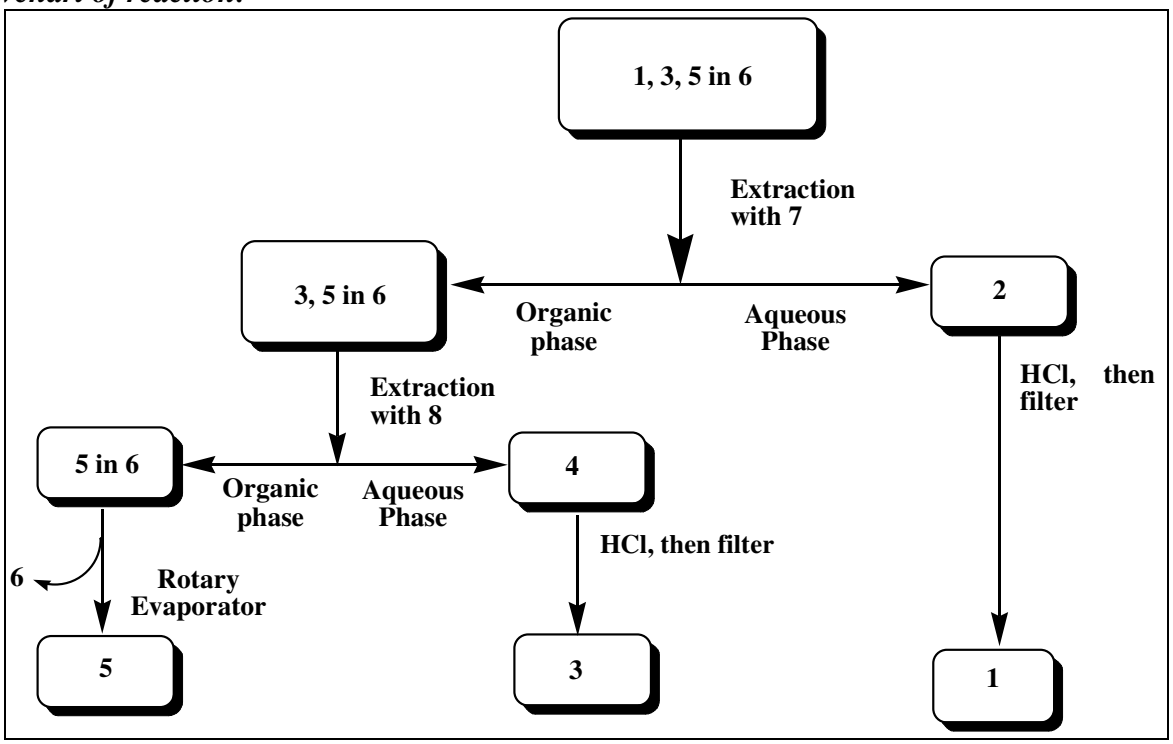
Evaporation of solvent to obtain naphthalene

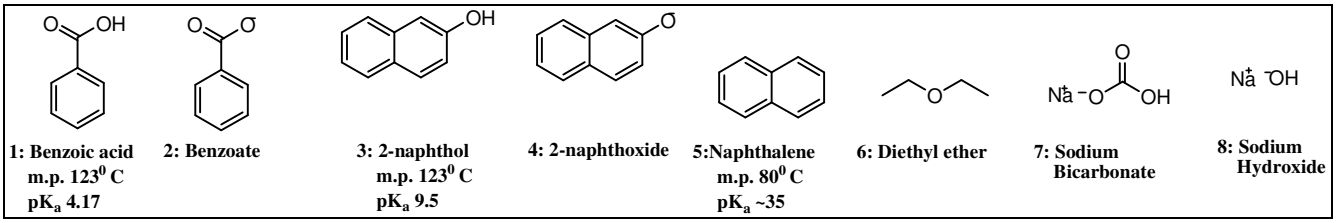
The naphthalene-ether solution was removed from the separatory funnel and dried with ~0.7 g $Na2SO_4$. The ether was decanted and evaporated off with the rotary evaporator to obtain solid naphthalene.

Acidification

3M HCl solution was added dropwise to both the bicarbonate and hydroxide extracts until litmus paper showed an acidic pH and the acids re-crystallized. The flasks were cooled in an ice bath, and the contents vacuum-filtered and air-dried to obtain solid benzoic acid and 2-naphthol.

Flowchart of reaction:





1: Benzoic acid
m.p. 123^0 C
pK_a 4.17

2: Benzoate

3: 2-naphthol
m.p. 123^0 C
pK_a 9.5

4: 2-naphthoxide

5:Naphthalene
m.p. 80^0 C
pK_a ~35

6: Diethyl ether

7: Sodium Bicarbonate

8: Sodium Hydroxide

4. RESULTS AND OBSERVATIONS

Sample # 156

mass, original sample	2.08 g
mass, recovered	1.32 g
% recovery	63.5%

Naphthalene

mass, 50-mL round bottom flask		56.90 g
mass, 50-mL round bottom flask with naphthalene		57.35 g
mass, naphthalene		0.45 g
% composition of recovered sample		34.1%
melting points, experimental	Trial 1 (fast)	75-77^{0}C
	Trial 2 (slow)	80^{0}C
melting point for pure sample		80^{0}C
light brownish white crystals		

Benzoic Acid

mass, filter paper		0.14 g
mass, filter paper with benzoic acid		0.43 g
mass, benzoic acid		0.29 g
% composition of recovered sample		22.0%
melting points, experimental	Trial 1 (fast)	114-116^{0}C
	Trial 2 (slow)	121^{0}C
melting point for pure sample		123^{0}C
white, clumpy/ chalky solid		

2-Naphthol

mass, small filter paper		0.14 g
mass, large filter paper		0.35 g
mass, small and large filter paper with 2-naphthol		1.07 g
mass, 2-naphthol		0.58 g
% composition of recovered sample		43.9%
melting points, experimental	Trial 1 (fast)	114-116^{0}C
	Trial 2 (slow)	120^{0}C
melting point for pure sample		123^{0}C
Light brownish white powdery solid		

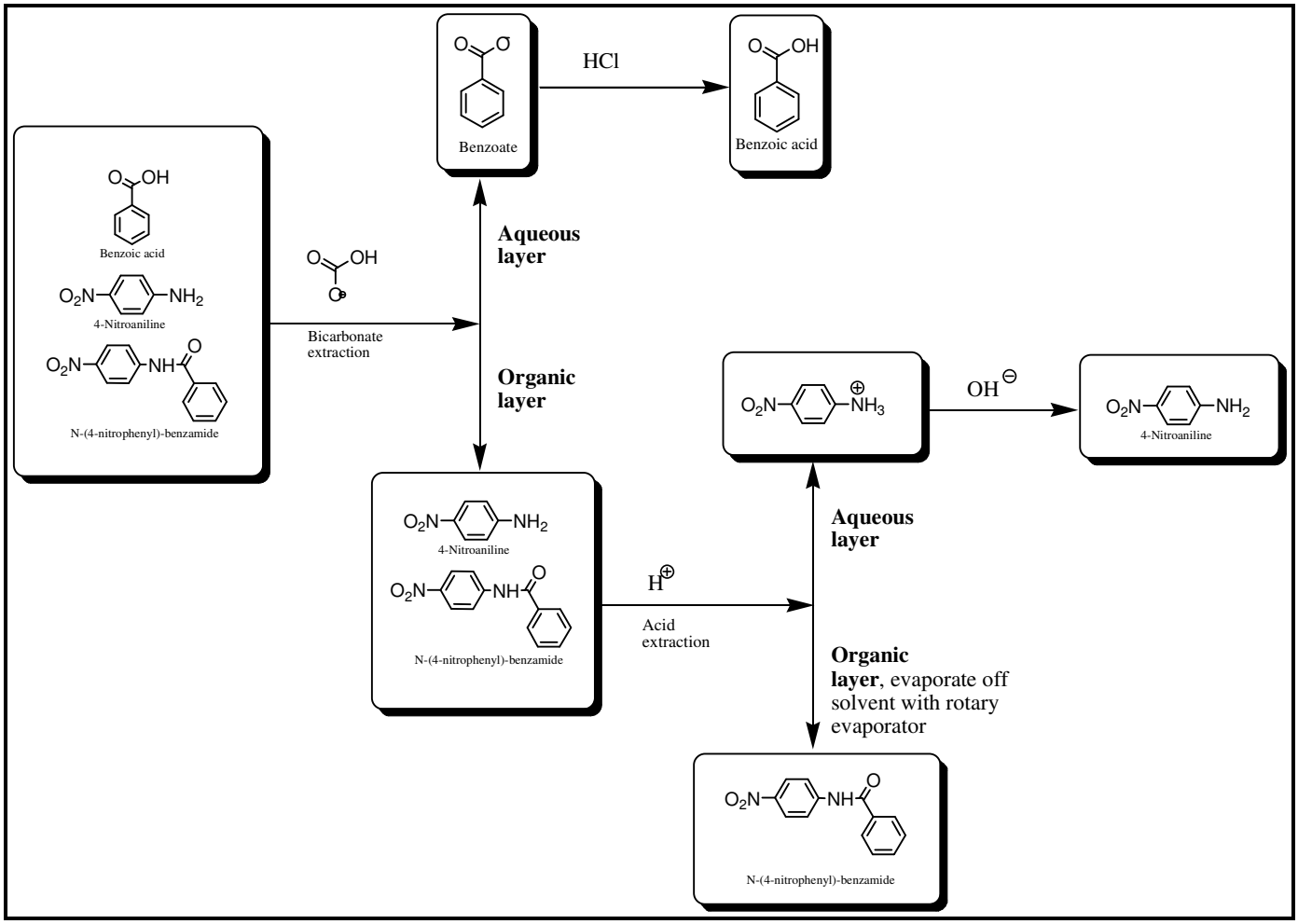
Note: Trial 1 for the m.p. was done at 60V on melting point apparatus #12.
Trial 2 for the m.p. was done at 45-50 V on melting point apparatus #8.

5. CONCLUSIONS

Since only 63.5% of the original mixture was recovered, there was some product loss. The procedural step where significant product loss most likely occurred is in the acidification and filtration of benzoic acid and 2-naphthol. Some benzoate and 2-naphthoxide may have remained dissolved even upon acidification, or the benzoic acid and 2-naphthol formed may have re-dissolved to some extent in the acidic solution. These substances would not be filtered out, resulting in product loss. Furthermore, because the experimental melting points are not consistent with the melting points for a pure sample, there is some contamination in the recovered benzoic acid and 2-naphthol. Perhaps with more thorough washing of the filtered solids, this contamination could be avoided in the future.

6. ANSWERS TO ASSIGNED PROBLEMS

#8.



#11. The use of the strong base OH^- *first* would cause the reaction of both benzoic acid and 2-naphthol, resulting in an ineffective separation of the two acids from each other.