Experiment title and number

Extraction

Date

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Name

Course

Chem 226

Section

3A

Lab partner

Objective: Use solvent extraction techniques to separate a mixture consisting of a carboxylic acid, a phenol, and a neutral compound.

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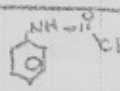
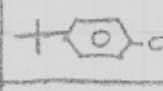
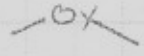
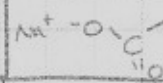
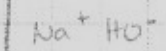
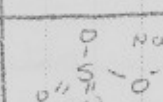
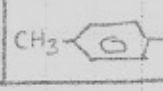
Physical Properties:

Compound Name	Structure	CAS #	BP or MP lit °C	RI	Hazards
Acetanilide		103-84-4	BP: 304 MP: 111-115	N/A	Irritating, harmful if swallowed
P-tert-butyl phenol		98-54-4	BP: 236-238 MP: 96-100	N/A	Irritant, harmful to the environment
Hydrochloric Acid		7647-01-0	BP: 57 MP: -35	N/A	Irritant, corrosive
Methyl-tert-butyl ether		1634-04-4	BP: 55-56 MP: -109	1.3675-1.3695	Highly flammable, Irritant
Sodium Bicarbonate		144-55-8	BP: N/A MP: 270	N/A	Minor irritant
Sodium Hydroxide		1310-73-2	BP: 1390 MP: 318	N/A	Corrosive, causes burns, exothermic in contact w/ H_2O
Sodium Sulfate		7757-82-6	BP: 1700 MP: 884	N/A	Irritant
P-toluic acid		99-94-5	BP: 274-275 MP: 179-182	N/A	Irritant, harmful to eyes, skin, resp. system

References:

1.) 30 Nov 2008 <https://ituweb.mst.edu/auth-cgi-rap/msdshtml/search.pl>.

2.) 30 Nov 2008 <http://www.chemexper.com/>.

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Procedures:

1.) A centerfuge tube was obtained.
2.) 5 mL of ~~the~~ kw sample was added.
3.) 2 ml of 0.5M aqueous $NaHCO_3$ was added to the ether solution in the centrifuge tube.
4.) The two layers in the centrifuge tube wee gently and thoroughly mixed
5.) The plastic cap was placed on the centrifuge tube and shaken gently.
6.) The cap was removed to allow any CO_2 gas to escape
7.) This process was repeated several times with the shaking intensity gradually increasing.
8.) The centrifuge tube was supported in a beaker and the layers were allowed to separate.
9.) The identity of the layers was confirmed by using a Pasteur pipet to introduce one or two drops of water just below the surface of the top layer. The aqueous layer was the bottom layer.
10.) A pasteur pipet was used to remove the aqueous layer and transfer it to a 50ml beaker.
11.) A second 2mL $NaHCO_3$ portion was added to the tube containing the ether mixture.
12.) The tube was shaken vigorously.
13.) The aqueous layer was removed and combined in the beaker with the first extract.
14.) A third 2mL $NaHCO_3$ portion was used to repeat the process.
15.) 1ml of distilled water was added to the centrifuge tube and mixed.
16.) The aqueous layer was removed and combined with the three $NaHCO_3$ solution extracts in the 50ml beaker.
17.) The aqueous solution was saved for later.
18.) 2ml of 0.5M NaOH was added to the ether solution remaining in the centrifuge tube. The tube was shaken vigorously.
19.) A pasteur pipet was used to remove the aqueous layer (bottom) and transfer it to a clean 50ml beaker.
20.) A second 2ml NaOH portion was used to repeat the

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extraction of the ether layer.

21.) The second NaOH layer was removed and combined with the first.
22.) This was repeated using a third 2mL NaOH portion.
23.) 1 ml of water was added to the ether remaining in the centrifuge tube and mixed.
24.) The aqueous layer was removed and combined with the three NaOH extracts.
25.) The NaOH extracts in the 50ml beaker was saved for later. The remaining ether layer in the centrifuge tube for later.
26.) The 50ml beaker containing the $NaHCO_3$ extracts was selected.
27.) 3M HCl was added dropwise to the $NaHCO_3$ solution.
28.) The 3M HCl was added dropwise with stirring until no more solid was produced and the solution tested acidic.
29.) The crystals from the solution were seperated from the solution using vacuum filtration with a Hirsch funnel.
30.) The 50 ml beaker containing the NaOH extracts was selected.
31.) The NaOH solution was heated to 60°C on a hot plate.
32.) The beaker was then allowed to cool.
33.) 3M HCl was added dropwise to the cooled solution until it tested acidic.
34.) To facilitate crystallization the mixtue was cooled in an ice bath and the sides and bottom of the beaker were ~~scratched~~ kw scratched with a microspatula.
35.) The crystals were separated from the solution by vacuum filtration using a Hirsch funnel.
36.) The crystals were allowed to air dry.
37.) The centrifuge tube containing the ether layer was selected.
38.) .5g anhydrous Na_2SO_4 was added to the centrifuge tube.
39.) The tube was capped, shaken and allowed to stand for 5min
40.) A 50mL beaker was weighed 25.578g.
41.) The dried ether was decanted into the 50mL beaker, and the Na_2SO_4 was left in the centrifuge tube.
42.) The ether was evaporated on a hot plate at 50°C with air blowing gently over the solution.

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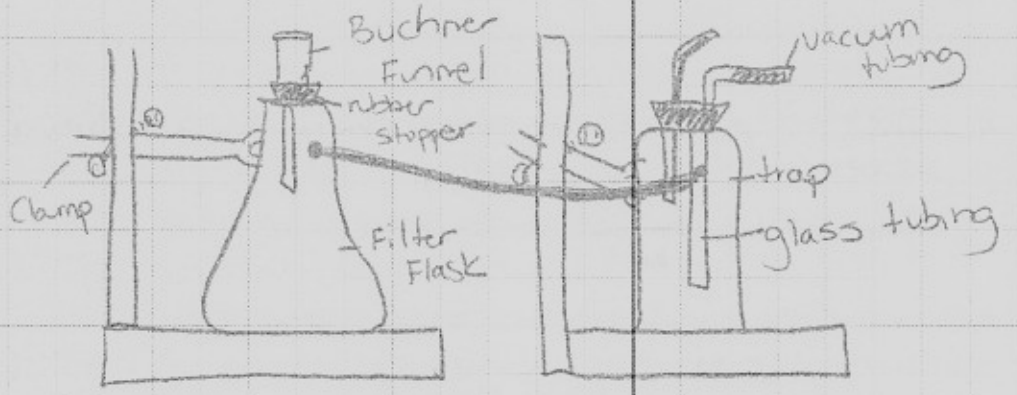
43.) The beaker was cooled in an ice bath. The bottom and sides of the beaker were scratched with a microspatula.
44.) The crystals were allowed to dry.
45.) The mass of the 1st extract was .14 grams.
46.) The mass of the 2nd extract was .264 grams.
47.) The mass of the 3rd extract was .065 grams.
48.) All materials were cleaned and put away or properly disposed of.



Vacuum filtration Apparatus

Observations:

- The first crystals obtained were white
- The second set of crystals were whitish brown
- The beaker needed to be scratched on the bottom and the sides to induce crystallization

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extract number	Yield in grams	Melting Point	Percent Yield	Melting point % error
extract 1	.14	170-172°C	2%	5.5%
extract 2	.264	95-96°C	.66%	4%
extract 3	.065	110-112°C	4%	2.6%

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Calculations: Percent Yield

extract 1: p-toluic acid $6.5g \times \frac{1mole}{136.15g/mol} = .048 mol$

$.048 mol \times \frac{136.15}{1mole} = 6.5352 grams$

$\frac{.14}{6.5352} \times 100\% = 2\%$

extract 2: p-tert butylphenol $6.5g \times \frac{1mole}{150.22} = .043 mol$

$.043 mol \times \frac{150.22}{1mol} = 6.46 grams$

$\frac{.043}{6.46} \times 100\% = .66\%$

extract 3: Acetamilide $4g \times \frac{1mol}{135.16} = .030 mol$

$.030 mol \times \frac{135.16}{1mol} = 4\%$

Melting Point Percent error

extract 1 p-toluic acid $\frac{|171 - 181|}{181} \times 100\% = 5.5\%$

extract 2 p-tert butylpanol $\frac{|95.5 - 99.5|}{99.5} \times 100\% = 4\%$

extract 3 Acetanilide $\frac{|111 - 114|}{114} \times 100\% = 2.6\%$

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