Flow Chart of Procedures for Experiment #1: Two Base Extraction

Benzoic acid (#1) naphthalene (#2) 2-naphthol (#3)

Naphthalene 2-naphthol (Organic phase)

Combine with sodium hydroxide (#8) to form Naphthoxide (#4)

2-Naphthoxide (Aqueous Phase) Naphthalene (Organic Phase)

Neutralization with acid to create again 2-Naphthol Removal of solvent through evaporation Pure Naphthalene

Benzoic acid combined with bicarbonate (H2CO3, #6) to form Benzoate (#5) (Aqueous phase)

Neutralization with Acid (HCL, #7) to create again Benzoic Acid
Two Base Extraction

**Introduction:**
Given a 2 gram sample of a mixture containing benzoic acid, 2-naphthol, and naphthalene, we are to separate each part.

**Procedure:**
1. The mixture is dissolved in 30 ml of diethyl ether.
2. 20 ml of 10% Bicarbonate is added to the solution in a separatory funnel where the aqueous layer is then removed from the organic layer.
3. 20 ml of cold 10% hydroxide solution is then added to this organic layer into the separatory funnel.
4. The new organic layer (ether) is placed into a flask containing .7 grams of Sodium Sulfate.
5. Both bases are acidified, dried and placed aside for weighing later.
6. Solvent is then removed from the ether through evaporation to be weighed later.
7. Lastly, our mass is calculated to see if we retrieved the same out we began with. Our mass for each compound is recorded to be compared with our unknown.
8. Melting points re taken ensure purity of the three separated substances.

**Please see attached flow chart and table of reagents**

**Results/Observations:**
Observations were taken and recorded during the lab procedure. These primary observations were handed in at the conclusion of the lab period. The gathered results are depicted in the following chart.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mass</th>
<th>Melting Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzoic Acid</td>
<td>.08 g**</td>
<td>120-125</td>
</tr>
<tr>
<td>2-Naphthol</td>
<td>0.51 g</td>
<td>118-124</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>1.12 g</td>
<td>81-83.5</td>
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** As noted in the primary lab observations, I spilled some of my aqueous solution after extracting with Sodium Bicarbonate, this is why my result (mass) for Benzoic Acid is extremely negligible.

Calculations are shown on the following chart.

| Unknown #121 | 1.84 g |
### Conclusions:
This lab was designed to allow us the experience and knowledge of extracting three separate compounds from one original mixture. It is important to be careful using the separatory funnel so that no spillage occurs! My results illustrate the problem of spillage during the procedure. I unfortunately lost a lot of my material during the extraction and my % recovery was probably lower than it should have been. The main point of the lab was to understand the strong acid/weak base, weak acid/strong base relationship between compounds. This will be further examined when question #11 is answered from the experimental organic text book. Overall, my % recovery wasn’t as close as I would have liked, but my procedures and observations explain the results I obtained.

### Assigned Questions:

#11. The first procedure of this lab calls for the combination of aqueous bicarbonate which acts like a weak base to draw out the strong benzoic acid. This bicarbonate will have no effect on the weak naphthol. The weaker acid, naphthol (pKa ~10) will only be removed with a strong base such as NaOH. If the NaOH was added first, it would extract both the naphthol and the benzoic into a solution from the naphthalene and diether.