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Experiment 3: Acid-Base Extractions

Introduction

The goal of this experiment was to separate 3 different compounds: an amine, a carboxylic acid, and a neutral compound by altering the pH of an aqueous solution to isolate and remove nonionized species in an added organic solvent. The three unknown compounds could then be identified through IR spectroscopy and melting point analysis.

Acid-base extractions are commonly used in many different applications, because it is a reliable way to isolate a desired compound, and it can be done at scale. An example is the extraction of citric acid from organics in the food industry, as it is a carboxylic acid and can be removed from a solution via acid-base extraction.

Scientific Principles – Part 1

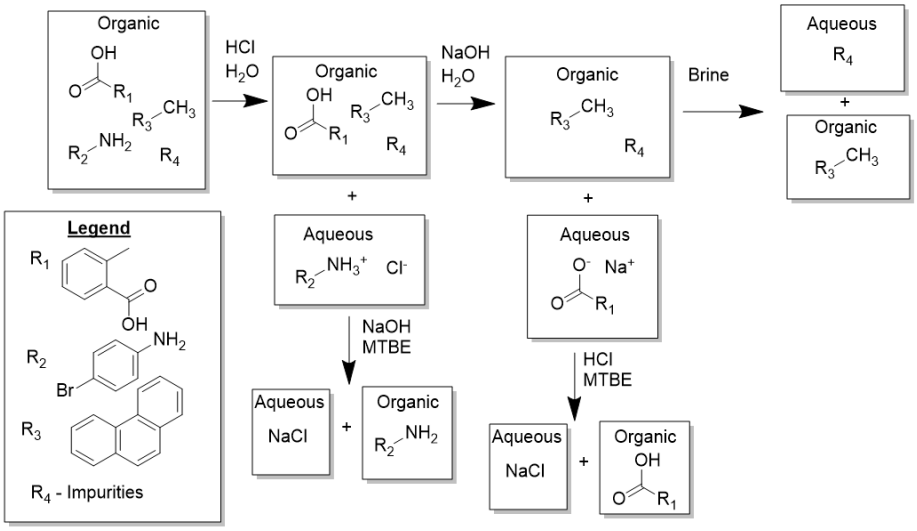

Figure 1. Flowchart of the separation of three components by acid-base extractions.

HCl was added to the initial solution to decrease it pH so the amine would transition to its ionized species and become more soluble in the aqueous solution than the organic solution. The resulting aqueous solution could then have NaOH solution and MTBE added to it for the amine to go back to its nonpolar nonionized species, and go into the organic layer to be separated from the aqueous.

The organic solution (without the amine) was then rinsed with NaOH solution, so the ionized form of the carboxylic acid would go into the aqueous solution. HCl solution and MTBE were added to the aqueous to non-ionize the carboxylic acid, making it go into the organic solution.

To separate the organic compound from impurities, the original solution was rinsed with brine, so any polar/ionized compounds go into the aqueous solution. Only the desired neutral compound would remain in the organic.

Scientific Principles – Part 2

1. Provide **one** explanation for the typical result of less than 100% recovery of the original mass. Assume complete removal of solvent from the collected solid samples. Disregard experimenter's errors such as spillage.

When shifting the pH over and under the pKa's of the amine and carboxylic acid, most of the compound would exist in its ionized form for extraction, but not all of it. Since the extraction relies on the compound existing in its ionized form, not all of the compound can be extracted, since not 100% of the compound can be in its ionized form at a given time.

2. Were the recovered products pure? Justify your conclusion with specific evidence.

Yes, the recovered products were pure, because there was a narrow melting point range for all 3 extracted compounds, and a narrow melting point range is only seen in pure compounds.

3. In the original procedure, one component from the starting mixture was *first* selectively extracted into the aqueous layer. State how you could revise the procedure to selectively extract one different component into the aqueous layer in the first step.

Instead of adding HCl to extract the amine first, NaOH could have been added first to remove the carboxylic acid first. These 2 extractions could be done in either order without altering the rest of the experiment.

4. State and explain whether any of the components in the starting mixture **cannot** be selectively extracted into the aqueous layer in the first step by any changes in procedure.

The neutral compound cannot be selectively extracted in a single step, as there is no ionized form of the neutral compound to make it soluble in an aqueous solution. The only way to isolate the neutral compound is to remove other compounds from the organic layer first.

Discussion

Yes, the objectives of the experiment were met, as all three compounds were able to be separated and identified using IR and melting point analysis. Furthermore, all the isolated compounds had narrow melting point ranges, indicating that they are pure compounds. The unknown compound number was 215.

The amine was identified as p-Bromoaniline, as its adjusted melting point range was (60-63) +/-1 degrees Celsius, which was closest to the reported melting point range of 61-64 Celsius, and when the compounds were mixed, they had a mixed melting range of 61-64 Celsius, and this narrow melting range confirmed the compound's identity. The IR spectra also indicated the presence of a primary amine attached to an aromatic ring with strong N-H and C-H peaks at 3473 and 3382 cm^{-1}, respectively. It did not indicate the presence of any oxygen or other bonds, so the possible compounds based off of the IR spectra were p-Bromoaniline or p-Chloroaniline.

The carboxylic acid was identified as o-Toluic Acid. Its IR spectra had broad peaks ranging from 2967 to 2648 cm^{-1}, which were indicative of O-H and C-H groups. Its melting point range was 100–103 Celsius, which only closely matched the reported range for o-Toluic Acid. Mixed melting point analysis of both compounds yielded a narrow melting range of 101–104 Celsius.

The neutral compound was identified as Phenanthrene. Its IR spectra had a cluster of small peaks around 3053 cm^{-1}, which were indicative of C-H groups, specifically aromatic rings. There were no additional peaks outside of the fingerprint region, meaning no O-H, N-H, or other types of bonds. The melting point range for the compound was 96-98 Celsius, which was closest to Benzil and Phenanthrene. However, Benzil contains a ketone ring (and therefore an O-H bond), meaning it was not the neutral compound. Mixed melting point analysis with Phenanthrene yielded a narrow melting range of 96-100 Celsius, confirming the compound's identity.

The initial mass of the unknown was 0.506g, and the isolated compound masses were as follows: 0.077g phenanthrene, 0.136g p-Bromoaniline, 0.088g o-Toluic acid. Adding these together, 0.301g of the initial 0.506g unknown were able to be recovered, for a percent yield of 59%.

Conclusion

All three isolated compounds had a narrow melting range, indicating that the unknown was able to be separated into its pure amine, carboxylic acid, and neutral components. Based on melting point and IR data, and confirmed with mixed melting point analysis, the amine, carboxylic, and neutral compounds were identified as p-Bromoaniline, o-Toluic acid, and Phenanthrene, respectively.