Chemistry 260 Summer 2024

E1 Chemically Active Extraction

**<<Complete this report form by inputting the information indicated by red text. Delete red text instructions before submitting (there are marks associated with doing so.>>**

Name: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_Section: \_\_\_\_\_\_\_\_\_\_Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Abstract (6 marks; 3 for written portion, 3 for equations)**

In this experiment, an extraction was performed to separate three organic compounds: benzil, *m*-toluic acid, and *p*-bromoaniline. The success of the extraction was tracked by thin layer chromatography (TLC).

A mixture of the three compounds dissolved in <<solvent>> was extracted three times with aqueous <<concentration and reagent (e.g., 1.0 M NaHCO3)>>, causing <<compound>> to be <<choose: protonated OR deprotonated>> via the reaction equation shown below:

<<IMAGE>><<Delete this text and insert a balanced chemical equation showing the reaction that occurred between the aqueous acid or base solution and the active component of the stock solution. Chemical equations must use line drawings that fully represent the structure, NOT generic formulae>>

Following this extraction, the <<list two compounds>> remained in the organic solvent. <<Write a short description of the second extraction, using the one above as a template. Include a second balanced equation to show the chemical process that occurred.>>

<<IMAGE>>

After the two extractions were complete, <<name compound(s)>> remained in the organic layer, and it was recovered by removing the solvent under reduced pressure. Neutral <<compound>> was recovered by <<choose: acidification OR basification>> of the aqueous layer, and neutral <<compound>> was recovered by <<choose: acidification OR basification>>. The success of the extractions were analyzed using TLC.

**Procedure and Observations**

The procedure was followed as provided in the Chem 260 lab manual.1

**Observations (3 marks)**

<<Delete this text and insert any observations you made during the experiment. Make sure to also indicate at which stage of the experiment each observation was made. Observations worth noting include: colour changes, gas evolution, changes in solubility (such as precipitate formation), if the addition of a reagent was exo- or endothermic, etc. This section will likely be quite short>>

**Reagents and Products Tables (2 marks each)**

<<This time the compound fields have been filled out for you on the tables on the next page. In future reports you will be expected to decide what compounds need to be in these tables on your own!>>

**Table 1.** Reagents for the liquid-liquid extraction of benzil from *m*-toluic acid and *p*-bromoaniline.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | MW (g/mol) | Used | moles | Physical and Safety Data |
| Benzil |  |  |  |  |
| *m*-Toluic acid |  |  |  |  |
| *p*-Bromoaniline |  |  |  |  |
| Diethyl ether |  |  |  |  |
| 3 M NaOH |  |  |  |  |
| 6 M HCl |  |  |  |  |
| 3 M HCl |  |  |  |  |
| 6 M NaOH |  |  |  |  |

**Table 3.** Products of the liquid-liquid extraction.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Compound | Physical Description | MW (g/mol) | Isolated | moles | % recovery |
| *m*-Toluic acid |  |  |  |  |  |
| *p*-Bromoaniline |  |  |  |  |  |
| Benzil |  |  |  |  |  |

**Results**

**Percent Recovery Sample Calculation: (2 marks)**

%Recovery Calculation for <<compound>> (your choice of the three):

<<Delete this text and insert a calculation for the percent of compound you recovered relative to the amount in your original stock mixture. >>

**Thin-layer Chromatography: (8 marks)**

<<Delete this text and make a note of the conditions used to record the TLC (type of plate, solvent used and visualization method(s)). Insert your two TLC plate photos (one under UV, one after chemical development. Colour photos preferred here)>>

**Table 4.** Summary of TLC results from the liquid-liquid extraction.

<<Add or remove rows to suit your data. Shading is included to try to help divide data for each lane.>>

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Lane (number from left to right) | Number of spots | Rf | Response to stain | Identity of compound |
| **1** |  |  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
| **2** |  |  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
| **3** |  |  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |

**Rf sample calculation for benzil:**

Rf = distance travelled by spot/distance travelled by solvent front

Rf = <<Your value for spot>>/<<your value for solvent front>>

Rf =

**Discussion** (**16 marks**, 850 words max.)

<<Delete this text and insert your discussion. Explain the chemical significance of your results, and do not assume that the reader already knows the answers! Explain it as if you are trying to convince someone who has not seen this data before. Some prompts are included below but the amount of prompts and detail included in the prompts will decrease as the semester progresses. By the end of the term, we expect you to identify what needs to be included in the discussion section.

Include the following: Discuss the success (or failure!) of the liquid-liquid extraction. Do your data indicate that you were able to successfully separate the three components from the original mixture? How can you tell? Are there any data that are confusing or contradictory? Did the extraction provide pure material? (Use your observations and data). Was the percent recovery for each compound good? How might you improve the extraction, if you were to repeat it?

Which compound was separated by extracting with acidic aqueous solution? What was the chemical basis for this extraction working? Why did we basify the aqueous layer? Similarly, what compound did you isolate after extracting with basic aqueous solution? Briefly explain the chemistry, and indicate the purpose of each step. Do the relative Rf values of the three components and their response to stain make sense?>>

**Conclusion (2 marks)**

<<Delete this text and insert a conclusion. What is/are the major finding(s) or results of this experiment? Concisely summarize what data allowed you to make this conclusion.>>

**References (1 mark)**

1. <<Delete this text and insert a reference to the lab manual>>

**Additional Graded Components:**

**Prelab: 4 marks**

**Samples & Clean-up: 2 marks**

**Appropriate editing and formatting of the report: 2 marks**