Michael Torres

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**Williamson Ether Synthesis**

**Purpose:** The purpose of this lab is to synthesize an ether through a process known as Williamson ether synthesis. This experiment involves the reaction between an alkyl halide with an alkoxide ion. This lab will allow for a better understanding regarding the chemical synthesis and lab techniques required for such process.

**Reaction(s):**



**Physical Properties of Reagents**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Reagent | Structure | Molecular Formula | Molecular Weight (g/mol) | Boiling Point (°C) | Density  (g/mL) |
| 4-Ethyl phenol |  | C8H100 | 122.16 | 218 | 1.01 |
| Ethyl p-toluenesulfonate |  | C9H12O3S | 200.252 | 160 | 1.17 |
| Tetrabutylammonium bromide |  | C16H36BrN | 322.37 | 102 | 1.04 |
| Diethyl ether |  | (C2H5)2O | 74.12 | 94.28 | 0.71 |
| Dichloromethane |  | CH2Cl2 | 84.93 | 103.3 | 1.33 |

**Procedure & Observation:**

Begin by obtaining the necessary laboratory materials and dressing in the proper lab attire. To begin the experiment, conduct the formation of p-ethylphenoxide. Do this by measuring 440mg of 4-ethylphenol using a analytical balance and weighing paper. Place the weighted compound into a 10ml round bottom flask. Next add 3mL of 25% NaOH into the flask along with a spin vane. Place onto a hot plate and use the stir function to stir the mixture until the compound has completely dissolved at room temperature. You will use this mixture in the formation of 1-ethoxy-4-ethylbenzene. Using and analytical balance and weighing paper, add 20-30 mg of tetrabutylammonium bromide to the mixture. You will then add 720 mg of ethyl p-toluenesulfonate into the flask. Next, assemble the flask onto a water-cooled condenser and a ring-stand with an adequate amount of grease. You will heat and stir the flask over a hot plate for 1 hour.

After the time is complete, allow the flask to cool down to room temperature and make sure not to remove the condenser until it is completely cool to prevent the sample from evaporating. Dissemble when it has cooled and use a 10ml graduated cylinder to add 5mL of diethyl ether into the flask. This will then be transferred into a 125 mL separatory funnel. Use up to 10mL of additional diethyl ether to attempt to completely remove and transfer any of the remaining mixture into the funnel. Next add an additional 10mL of diethyl ether. Add the glass stopper onto the top of the funnel. Make sure that the separatory funnel is secure with the glass stopper and use both hands to carefully stir the mixture within the funnel. You will then release any pressure that has built up within the funnel by turning the valve. Repeat this step a couple times and then return the funnel to the upright position and place onto the ring-stand. Allow the mixture to separate into two layers. Perform the extraction by collecting the bottom layer (aqueous layer) into a 100mL beaker. Add 10 mL of diethyl ether and repeat the process of stirring the mixture and running the extraction. The extraction process will be repeated a total of 3 times. After the extraction of the aqueous solution has been completed, collect the organic layer in a 50mL Erlenmeyer flask. You will use a total of 10 mL of water and 10 mL of 5% NaOH to completely remove the organic layer from the funnel. Next, transfer the organic layer into a pre-weighted 50mL beaker with a boiling chip using an analytical balance. Be sure to collect as much of the organic product as possible by using additional diethyl ether to rinse as much of the product out as possible. Following this step, you will utilize a hotplate under the fume hood to evaporate the solvent. Make sure to record the mass of the beaker following the evaporation of the solvent. You will then utilize the collected sample to run IR and NMR tests.

To run the IR test, place 2 drops of the organic sample onto NaCl salt plates using a micropipette. Be sure to handle the plates by holding from the side to ensure reliable results obtained from the IR. After running the IR use the results to determine the functional groups within the product. Prepare the NMR test by adding 700mg of butylated chloroform with 30mg of the sample. Carefully utilize the 90 MHz NMR machine and use the subsequent graph to analyze your results.

**Results:**

Theoretical Yield: See notes for calculations

Start: 4-ethyphenol=0.444 g=0.0036mol

Ethyl-p-toluenesulfonate= 0.741g = 0.0037 mol

Yield: 1-ethoxy-4-ethylbenzene=0.5409g

Actual Yield:

1-ethoxy-4-ethylbenzene: 0.406 g

Percent Yield:

= 75.06%

Percent Error:

=24.93%

IR spectrum Absorption Bands: See Chart 1

C(sp2)-H: Aromatic: 3150-3050 cm-1

C(sp3)-H: 3000-2850 cm-1

C-O: 1300-1000 cm-1

Chemical Shifts: See Chart 2 for chemical shifts assigned to peaks

**Discussion:** (Provide a critical analysis of the reaction/experiment in a paragraph by summarizing and evaluating your data such as % yield, IR, and NMR signals obtained. Any possible errors that could be associated with your experiment? DO NOT just write “because of human error”. If you do so, you will receive a zero. Any suggestion to improve this experiment?)

After conducting the experiment, we were able to conlude that we were capable running a

After conducting the experiment, it is believed that there were some possible sources of error that may have altered the results of the experiment.