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**Sodium Borohydride Reduction of Ketone**

**Purpose:**

The purpose of this experiment is to utilize a reduction agent, in this case NaBH4, to reduce a compound such as an aldehyde or a ketone to an alcohol. This is achieved by the addition or removal of oxygen from the compounds.

**Reaction(s):**



**Physical Properties of Reagents**:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Reagent | Structure | Molecular Formula | Molecular Weight (g/mol) | Boiling Point (°C) | Density  (g/mL) |
| 4-t-butylcyclohexanone |  | C10H18O | 154.25 | 225.1 | 0.911 |
| Sodium borohydride |  | NaBH4 | 37.83 | 500 | 1.07 |
| Dichloromethane |  | CH2Cl2 | 84.93 | 103.3 | 1.33 |

**Procedure & Observation:**

Begin the experiment by collecting the necessary lab materials and dressing in the proper lab attire. Begin by measuring out 386mg of 4-t-butylcyclohexanone on an analytical balance and place in 25mL Erlenmeyer flask. Next use a 10ml graduated cylinder to measure 5mL of ethanol and place into the flask with a stirring rod. You will then add 4 ml of the reducing agent, NaBH4, into the flask. There will be a cloudy white presence within the flask. Use a hot plate to stir the magnetic stirring rod within the solution for 30 minutes at room temperature. Next add a couple drops of 3M HCl into the solution to completely saturate the solution and provide the necessary hydrogen atoms necessary for reduction. There will be no more bubbles that form when the necessary amount of HCl is added. Following the addition of HCl it is necessary to perform the extraction of 4-t-butylcyclohexanol. Transfer the solution into a separatory funnel. Use dichloromethane to remove any remaining solution from the flask into the funnel. Add 15 ml of dichloromethane into the flask. Place the glass stopper on the top of the funnel and using both hands, firmly and securely mix the solution in the funnel. Hold the funnel upside down and open the valve to release any pressure that has built up. Mix the solution and release the pressure a couple more times. Allow the solution to sit and notice the two layers that form within the funnel. The top layer will be the organic layer and the bottom layer will be the aqueous layer. Next, perform the extraction by removing the aqueous layer by extracting it into a 100 mL beaker. Repeat the process of adding dichloromethane into the funnel, mixing, and extraction a total of 2 more times. Remove the remaining organic layer by transferring it into a 50mL Erlenmeyer flask. Add Na2SO4 into the solution until it no longer clumps at the base of the flask. Allow it to stand for 5 minutes. Next decant the organic layer into a pre-weighed 100mL beaker with a boiling chip. Evaporate the solvent under the fume hood with a hot plate. Allow it to cool and solidify. Use the remaining product to calculate the percent yield, melting point and IR.

To perform the melting point, use a capillary tube and crush the solidified organic layer into a powder. Tap the capillary tube onto the powder until roughly 3-5 cm has entered the tube. Utilize the melting point apparatus by setting a plateau and watching as the inserted capillary tube containing the compound melts under a given temperature. Perform the IR first by creating the necessary KBr pellets used to conduct the reading. Mix a small sample of your organic mixture with the KBr salt. Next compress a small amount of the new mixture into a flat disk and conduct the IR reading.

**Data/Results:** (Include the following data from the lab)

* Theoretical yield: See notes for calculations

Start: 4-t-butylcyclohexanone= .382 g= 0.00248 mols

Excess Hydrogen from 3M HCl

Yield: 4-t-butylcyclohexanol= 0.3875 grams

* Actual yield:

Yield: 4-t-butylcyclohexanol=.255g

* Percent Yield:

= 65.81%

* Percent Error:

= 34.19%

* Melting Point:
  + Theoretical: 62-70 oC
  + Experimental: 68 oC

IR Spectrum Absorption Bands:

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

OH: 3400-3200 cm-1

**Discussion:**

Based on the results that were achieved from the experiment, we may conclude that we were able to run a successful experiment. We were able to successfully recover 65.81% of the compound after reducing it with NaBH4. This yield could have been improved and will be discussed in further detail. Additionally, upon undergoing tests for melting point, we were able to see that we received an experimental value that was in line with the theoretical value. We received a value of 68 oC while the expected value was between 62-70 oC. Additionally, the IR spectrum that we were able to achieve was able to confirm the desired structures that were created through this reduction reaction. This is prevalent through the broad peak that was present due to the hydroxyl group from 3400-3200 as well as the C(sp3)-H bonding which created an absorption band from 3000-2850 cm-1. Unfortunately, we were unable to perform the NMR reading but we were able to determine what the appropriate chemical shifts would have been given the compound.

During the lab there were some possible sources of error that may have occurred. For one, the frequent transfer of the compound may have resulted in loss of the final yield. The frequent transfer does not allow for the complete transfer of 100% of the given compound. Additionally, upon evaporating the solvent the instructor placed the temperature on too high in an effort to evaporate the solvent faster but it caused some of the compound to solidify around the opening of the beaker and may have also resulted in the loss of some product.

Unfortunately, we were unable to determine our results with the NMR. This was primarily due to the fact that the 400 MHz NMR machine was too powerful for our samples. This resulted in unreliable results to determine our samples. To improve the experiment, I would suggest performing the NMR on the appropriate machine. Additionally, this experiment would further show the effects of reducing agents if we were able to use other agents such as LiAlH4.