**Reduction of Ketone**

Material

**100 mg 4-tert-ButylCyclohexanone**

**100 μL Methanol**

**10mg of NaBH4 (Sodium borohydride)**

**1 mL HCl(aq)**

**3 mL Dichloromethane**

**Anhydrous sodium sulfate**

**Silica Gel**

**Chloroform**

Procedure

1. Add 100 mg 4-tert-ButylCyclohexanone into 3 mL micro-vial.
2. Add 300 μL Methanol.
3. Tap against until dissolved.
4. Put the needle through the top (never run reaction in close system)
5. Add 10 mg NaBH4 (1 mole NaBH4 = 4 mole product)
6. Tap the micro-vial to agitate.
7. Set up air condenser.
8. Leve the reaction at room temperature for 10 min in fume hood
9. Add 1 mL HCl(aq) to quench NaBH4.

Extraction

1. Add 1 mL dichloromethane in to the reaction vial, shake it and stand for 2 min until 2 separate layers appear.
2. Take the glass pipette, extract the bottom layer (Product) without mixing any top layer (aq. layer) in to a clean micro-vial.
3. Repeat the extraction on aq. layer twice.
4. Fill the glass pipette with cotton, silica gel, and anhydrous sodium sulfate.
5. Weight your net weight of round bottom flask with cap.
6. Place your product in the glass pipette column and collect your pure product with round bottom flask.
7. Bring it to TA for rotovap evaporation.
8. Weight the round bottom flask again. (To find out your product weight) NMR analysis.

Purification

1. Add 0.7 mL chloroform-d in to your product.
2. Transfer 0.7 mL into NMR tube.
3. Bring to TA for NMR

**Williamson Ether Synthesis**

Material:

**160 μL *p*-cresol**

**3 mL diethyl ether**

**260 μL of 25%, aq, NaOH**

**Silica gel**

**18 mg Bu4N+Br- (tetrabutylammonium bromide)**

**Anhydrous sodium sulfate**

**150 μL 1-Iodopropane**

**Carborundum granules**

Procedure:

1. Attach the sand bath mantle to power controller unit (keep the knob at 6-7) check the temperature, maintain the temperature around 100-120 oC.
2. TA use heat gun to melt p-cresol with cap vented.
3. Set up reflux condenser attached with water tubing and check the water flow. (bottom in, top out).
4. Add 2 carborundum granules.
5. Add 160 μL *p*-cresol into 5mL conical base micro-vial.
6. Add 260 μL NaOH (25% aq)
7. Shake gently, the chemical may stick to the wall.
8. Add 18 mg Bu4N+Br- (tetrabutylammonium bromide) (Bring the vial to TA).
9. Add 150 μL 1-Iodopropane.
10. Attach the vial to the reflux condenser on sand bath.
11. Maintain the temperature around 100-120 oC.
12. After 40 min turn off the heating, lift up the vial and cool to room temperature.

Extraction and Purification

1. Add 1 mL diethyl ether in to the reaction vial, shake it and stand for 2 min until 2 separate layers appear.
2. Take the glass pipette and remove the upper layer (Product) without mixing any bottom layer (aq. layer) in to a clean micro-vial.
3. Repeat the extraction on aq. layer twice.
4. Fill the glass pipette with cotton, silica gel, and anhydrous sodium sulfate.
5. Weight your net weight of round bottom flask with cap.
6. Place your product in the glass pipette column and collect your pure product with round bottom flask.
7. Bring it to TA for rotovap evaporation.
8. Weight the round bottom flask again. (To find out your product weight)
9. Transfer your product in to a vial and label it for NMR next week. (Ask TA for the vial)

\*\*\* All work needs to be done in the fume hood

\*\*\*\*\*\* do not place the thermometer in sand bath too long, check the temperature and take it out

**Grignard reaction**

Material:

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| --- | --- |
| **105 mg benzophenone**  **0.3 ml diethyl ether**  **0.25 ml Phenyl magnesium bromide** | **Anhydrous sodium sulfate**  **3ml dichloromethane**  **2 ml chloroform for GC-MS**  **(Spectroscopic Grade)** |

**Silica Gel HCl (aq)**

Procedure:

1. Take a 5mL vial, add 105 mg benzophenone.
2. Add 0.3 mL diethyl ether.
3. Allow to dissolve and shake gently.
4. Put the needle through the top
5. Bring it to TA add dropwise phenyl magnesium bromide (0.25 ml) in 30-40 sec.
6. Keep in water bath (30-40oC) for 10 min.
7. Add 3M HCl dropwise till the solution are neutralize (pH paper)
8. If solid, add 1 ml DI water.

Extraction

1. Add 1.5 mL diethyl ether in to the reaction vial, shake it and stand for 2 min until 2 separate layers appear.
2. Take the glass pipette, remove the upper layer (Product) without mixing any bottom layer (aq. layer) in to a clean micro-vial.
3. Repeat the extraction on aq. layer twice.
4. Fill the glass pipette with cotton, silica gel, and anhydrous sodium sulfate.
5. Weight your net weight of round bottom flask with cap.
6. Place your product in the glass pipette column and collect your pure product with round bottom flask.
7. Bring it to TA for rotovap evaporation.
8. Weight the round bottom flask again. (To find out your product weight) GC-MS analysis

Purification

1. TA assists in obtaining approximately 2 mg of product.
2. Dissolve in 1 ml of Chloroform (Stock Solution)
3. Dilute 5 drops of stock solution to 1 ml with chloroform.
4. Analysis

**Diels-Alder Reaction**

Material:

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| **340 mg 3-Sulfolene**  **180mg Maleic anhydride**  **200 µL Xylene** | **1 mL toluene**  **Petroleum ether**  **Chloroform-D** |

**Carborundum granules**

Procedure:

1. Attach the sand bath mantle to power controller unit check the temperature; maintain the temperature around 200 oC.
2. Set up the air condenser.
3. Add 2 carborundum granules.
4. Add 340 mg of 3-Sulfolene in 5 mL vial.
5. Add 180 mg of Maleic anhydride.
6. Once sand bath has reached 200 oC, TA will come and dispense 200 µL of Xylene in the vial. (SO2 gas will form, keep in hood)
7. Start reflux for 20 min.
8. Cool to room temperature.
9. Add 1 mL Toluene.
10. Add Petroleum ether dropwise (1 mL MAX) to precipitate the organic product.
11. Cool the vial to 0oC in Ice bath.
12. Collection product using vacuum filtration.
    1. Obtain Buchner Funnel, Buchner flask, Rubber stopper.
    2. Attach vacuum tube.
    3. Obtain filter paper.
13. Use 1 mL of petroleum ether to wash product out from the vial on the filter paper. (repeat until the vial is clean)
14. Allow the product to dry under vacuum for 5 min.
15. Scrape product to weight to obtain actual yield.
16. Perform NMR

**Nucleophilic Aromatic Substitution**

Material:

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| --- | --- |
| **100 mg 2,4-dinitrobromobenzene**  **0.6 mL toluene**  **10mg tetrabutylammonium bromide**  **300 μL of 50% aqueous potassium thiocyanate (wt/wt) solution** | **3ml dichloromethane**  **DI water**  **Anhydrous sodium sulfate**  **Silica Gel**  **Chloroform-D** |

**Carborundum Granules**

Procedure:

1. Attach the sand bath mantle to power controller unit check the temperature, maintain the temperature around 140oC.
2. Set up reflux condenser attached with water tubing and check the water flow. (Bottom in, top out).
3. Add 2 carborundum granules.
4. Add 100mg 2,4-dinitrobromobenzene followed by 0.5 mL toluene in 5 mL vial.
5. Add 5 mg of tetrabutylammonium bromide.
6. Add 300 μL of 50% aqueous potassium thiocyanate(wt/wt) solution.
7. Attached the water jacketed reflux condenser and allow the reaction run for an hour.
8. Allow the reaction mixture cool down to room temperature.

Extraction and purification

1. Transfer the top layer (Product) to a clean vial.
2. Add 1 mL DI water and 1 ml dichloromethane in to the aq. layer, extract the bottom layer.
3. Repeat step 2 twice.
4. Fill the glass pipette with cotton, silica gel, and anhydrous sodium sulfate.
5. Weight your net weight of round bottom flask with cap.
6. Place your product in the glass pipette column and collect your pure product with round bottom flask.
7. Bring it to TA for rotovap evaporation.
8. Weight the round bottom flask again. (To find out your product weight)

**Crossed Aldol Condensation**

Material:

**60 μL of benzaldehyde**

**58 μL of acetone**

**2 mL NaOH/EtOH**

**DI water**

**Chloroform-D**

Procedure:

1. Add 60 μL of benzaldehyde into 5 mL vial with a spin and air condenser.
2. Add 58 μL of acetone.
3. Add 2 mL NaOH/EtOH.
4. Stir reaction for 30 min at room temperature.
5. Collection product using vacuum filtration.
   1. Obtain Buchner Funnel, Buchner flask, Rubber stopper.
   2. Attach vacuum tube.
   3. Obtain filter paper.
6. Use DI H2O to wash product out from the vial on the filter paper. (repeat until the vial is clean)
7. Wash the solid cake with DI H2O to remove NaOH.
8. Allow the product to dry under vacuum for 15 min.
9. Scrape product to weight to obtain actual yield.

**Esterification**

Material:

**800 μL of isopentyl alcohol (3-methyl-1-butanol)**

**4 drops of sulfuric acid (98%)**

**1.5 mL of acetic acid**

**Anhydrous sodium sulfate**

**100 mg silica gel beads**

**2mL of Diethyl Ether**

**Silica Gel**

**Carborundum granules**

Procedure:

1. Attach the sand bath mantle to power controller unit check the temperature, maintain the temperature around 110-120 oC.
2. Set up reflux condenser attached with water tubing and check the water flow. (bottom in, top out)
3. Add 2 carborundum granules.
4. Add 800 μL of isopentyl alcohol (3-methyl-1-butanol) into 5 mL.
5. Add 1.5 mL of acetic acid.
6. Add 100 mg silica gel beads.
7. Add 4 drops of sulfuric acid (98%)
8. Reflux the solution for 1 hour.
9. Lift up the vial and cool to room temperature.

Extraction and Purification

1. Add 1 mL of diethyl ether.
2. Add 1 mL of sodium bicarbonate (5%) to wash organic solution.
3. Extract aqueous (BOTTOM) layer in to a new vial.
4. Repeat step 2 and 3
5. Add 1 mL DI H2O to wash the organic layer (reaction vial)
6. Extract aqueous (BOTTOM) layer in to a new vial.
7. Repeat step 5 and 6
8. Fill the glass pipette with cotton, silica gel, and anhydrous sodium sulfate.
9. Place your product in the glass pipette column and collect your pure product with round bottom flask.
10. Bring it to TA for rotovap evaporation.
11. Weight the actual yield.

\*\*\* All work needs to be done in the fume hood

\*\*\*\*\*\* do not place the thermometer in sand bath too long, check the temperature and take it out