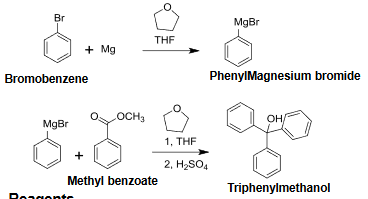
**CH 246: ORGANIC CHEMISTRY II LABORATORY (Spring 2021)**

**Title:** Preparation of Triphenylmethanol with Grignard Reaction

1. **Purpose: (1 point)**

**The purpose is to prepare the Grignard reagent phenylmagnesium bromide from bromobenzene and magnesium, and use the Grignard reagent and methyl benzoate to prepare triphenylmethanol through Grignard reaction.**

1. **Drawing of structure of the main compound or balanced chemical equation if synthesis is performed: (1 point)**



**3. Reagents and the major product (up to 5 points)**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Name** | **M.W.**  (0.5 pts) | **Density**  (0.5 pts) | **Amount (grams/mL)**  (0.5 pts) | **Moles**  (0.5 pts) | **Hazards/Precautions**  **(MSDS data) and melting point or boiling point** (2 pts) | **Role of the reagent** (1 pts)\* |
| Magnesium | 24.3 | 1.74 g/cm3 | 1 gram | 0.0412 | Flammable solid, water reactive. May cause eye and skin irritation. May cause respiratory tract irritation.  MP: 650°C  BP: 1107.2°C | Reactant |
| Bromobenzene | 157.01 | 1.5 g/cm3 | 4.2 mL/6.3 g | 0.0401 | Flammable liquid and vapor. Causes skin irritation. May cause eye and respiratory tract irritation.  MP: -31°C  BP: 155°C | Reactant |
| Methyl Benzoate | 136.0548 | 1.08 g/cm3 | 2.8 grams | 0.0206 | May cause eye, skin, and respiratory tract irritation. Combustible liquid and vapor.  MP: -12.2°C  BP: 150°C | Reactant |
| Sulfuric Acid | 98.07 | 1.84 g/cm3 | 1.5 mL | 0.0281 | Causes eye and skin burns. May be fatal if mist inhaled. Corrosive.  MP: 10°C  BP: 290-338°C | Reactant |
| Tetrahydrofuran | 72.10 | 0.89 g/cm3 | 10 mL | 0.123 | Highly flammable. Causes eye and respiratory tract irritation.  MP: -108.5°C  BP: 66°C | Solvent |
| Sodium Bicarbonate | 84.01 | 2.2 g/cm3 | 10 mL 10% solution | -- | May cause skin and eye irritation. | Solvent |
| 2-propanol | 60.09 | 0.7850 g/cm3 | 7 mL/g of product | -- | Flammable liquid and vapor. Causes respiratory tract irritation, eye irritation.  MP: -88°C  BP: 82°C | Solvent |
| Phenylmagnesium bromide | 181.31 | 1.134 g/cm3 | -- | -- | Highly flammable liquid and vapor. Contact with water releases flammable gases. Causes severe skin and eye damage. | Product (part 1)/Reactant (part 2) |
| Triphenyl methanol | 260.32 | 1.199 g/cm3 | -- | -- | May cause eye and skin irritation. May cause respiratory and digestive tract irritation.  MP: 164.2°C  BP: 380°C | Product |

**\*** Mention role as either reactant, solvent, catalyst or product

**4. Calculations: (1 point)**

Show each calculation for moles of reagents and for theoretical and actual yield. Fill in the box with the limiting reagent and theoretical yield:

Bromobenzene

The limiting reagent is

5.2 g triphenyl methanol

The theoretical yield is

**5. Procedure (up to 2 points)**

|  |  |
| --- | --- |
| **Procedure** | **Observations and Lab Data** |
| A summary of the procedure done with bullet points) | Color changes, exothermic or endothermic reactions, gas generation, etc.; tare weights for flasks, etc. |
| Part 1:   * Dry all pieces of apparatus as much as possible, and assemble apparatus as depicted at demonstration table. * Place about 1 g of magnesium turnings and 1-2 small crystals of iodine into flask. Heat for a few minutes and allow to cool to room temperature. * While heating magnesium, prepare a solution of 4.2 mL bromobenzene in 15 mL THF and place in DRY, stoppered Erlenmeyer flask or 50 mL round bottom flask. * Start water circulation in condenser. * Prepare an ice bath, and add about 5 mL of the bromobenzene solution to the magnesium through the Claisen side arm, stopper the flask, and stir gently. * If a reaction does not start spontaneously, warm the flask gently or add another iodine crystal. If it still does not start, restart reaction. * If the reaction becomes too vigorous, use ice bath to cool flask. * After the initial reaction has subsided, add the remaining solution in portions, at a rate necessary to maintain a gentle reflux. Keep adapter stoppered between additions. * When all bromobenzene has been added, apply heat to flask to gentle reflux for 15-20 minutes. * While refluxing, prepare a solution of 2.8 g methyl benzoate in 10 mL of THF. * Gradually start adding the methyl benzoate solution to the reaction mixture while it is warm but not refluxing. A reaction should be apparent. * Gradually add the methyl benzoate solution in portions over a 10 minute period, stirring gently with no added heat. * After all methyl benzoate has been added, let reaction mixture stand for 15-20 minutes. * Cool mixture over ice and pour it slowly with swirling into a 250 mL beaker containing 25 g crushed ice, 10 mL of cold water, and 1.5 mL sulfuric acid. * Leave unreacted magnesium behind but transfer reaction mixture with rest of the solid to another beaker. Mix well and warm to room temperature. * Cover the beaker tightly with parafilm and store in drawer.   Part 2:   * Take out the beaker containing reaction mixture. * If THF has evaporated during this time, add another 20 mL of ethyl acetate. * Remove some of the top layer and put in vial. * Take TLC of pure methyl benzoate, pure triphenylmethanol, and this sample. * Decant mixture from magnesium into separatory funnel. * Wash with 2 x 10 mL water and 1 x 10 mL 10% sodium bicarbonate solution. Save the top layer each time. * Pour organic layer into 50 mL Erlenmeyer flask and add 0.1 g magnesium sulfate as a drying agent. * Let stand for a few minutes and swirl occasionally. * Filter the solid using gravity filtration into 50 mL round bottom flask. * Rotavape the liquid. * Recrystallize product with 7 mL of 2-propanol for every gram of product. (Can also purify with Combiflash separation system) * Weigh dry product, record yield, % yield, and TLC. |  |

**6.** Results; include actual yield in grams and % yield.

**Results (need to get signed by instructor or TA):**