Grignard Synthesis of Triphenylmethanol

**Goal:** To synthesize triphenylmethanol using a standard Grignard addition of phenyl magnesium bromide to benzophenone.

\[
\text{benzophenone} \xrightarrow{\text{i) PhMgBr, Et}_2\text{O}} \text{triphenylmethanol} \xrightarrow{\text{ii) H}_3\text{O}^+}
\]

**Materials:**
- 18 X 150 mm test tubes (5)
- Anhydrous diethyl ether
- Bromobenzene
- Magnesium turnings
- Glass stirring rod
- Benzophenone
- 3 M HCl solution
- Saturated NaCl solution
- Calcium chloride pellets
- Ligroin (petroleum ether)
- 10 mL Erlenmeyer flask
- Boiling stick

**Procedure:**

First, it cannot be overstated that it is necessary to use glassware that has been thoroughly cleaned and dried in order for the reaction to be successful.

Using an analytical balance measure ~0.7 g of benzophenone and add the solid to a test tube. To the same test tube add 2.0 mL of anhydrous diethyl ether to dissolve the solid. Place this test tube aside for later use.

Measure ~0.15 g of magnesium turnings and add them to a second test tube. Into a third test tube weigh ~0.70 g of bromobenzene and add 2.0 mL of diethyl ether. Using a Pasteur pipette transfer a small amount of the bromobenzene/diethyl ether mixture to the test tube containing the magnesium turnings. Transfer only enough of the solution to cover the magnesium turnings. Initiate the reaction by using a glass stirring rod to crush the magnesium turnings. Be very careful to not break the test tube or the stirring rod. The best way to do this safely is to hold the test tube vertically, placing it on a laboratory notebook while gently crushing the magnesium turnings. The reaction can be considered initiated when bubbles begin to form on the surface of the magnesium turnings.

Allow the reaction to proceed while adding the bromobenzene/diethyl ether solution slowly. If the bromobenzene is added too quickly side reactions can occur. In order to prevent side reactions from occurring wait to add additional bromobenzene/diethyl ether solution until the reaction looks like it has slowed significantly. At this point add approximately 0.5 mL more bromobenzene/diethyl ether solution. After all of the bromobenzene/diethyl ether solution has been added allow the reaction to proceed to completion by waiting until the formation of bubbles stops. At this point the Grignard
reagent (phenyl magnesium bromide) has been synthesized and must be transferred into a clean test tube. Using a Pasteur pipette transfer the solution to a clean test tube making sure to leave the excess magnesium and any solids that form behind.

To the solution of phenyl magnesium bromide add the solution of benzophenone/diethyl ether drop-wise while gently swirling the test tube to mix the reagents. When all of the benzophenone/diethyl ether solution has been added to the Grignard reagent mix the reaction by swirling or by the use of a glass stirring rod. Allow five minutes for the reaction to proceed to completion.

Cool the reaction in an ice bath and slowly add 4 mL of 3M HCl solution to quench the reaction. Mix the reaction well with a glass stirring rod.

Remove the aqueous layer from the test tube and set it aside. Add approximately 4 mL of saturated sodium chloride solution to the ether layer. Shake the mixture vigorously using a stopper to seal the test tube. When the layers have separated remove the aqueous layer and set it aside. Slowly pour the ether layer into a clean dry test tube to separate it from the water that is adhered to the side of the test tube. Dry the solution by adding calcium chloride pellets and allowing the solution to sit for five minutes. Transfer the ether solution to a 10 mL Erlenmeyer flask leaving the calcium chloride pellets behind. Allow the solvent to evaporate yielding white impure crystals. (At this point the crystals can be left to dry until the following lab period.)

Remove a small amount of impure crystals for a melting point and recrystallize the remainder of the crude product. Using diethyl ether dissolve the crystals. Add 3-4 mL of ligroin to the solution and a boiling stick to aid in solvent evaporation. Place the Petri dish on a warm hotplate and evaporate the diethyl ether leaving only the dissolved solid and ligroin. Remove the Petri dish from the heat and allow the crystals to form upon cooling. After the flask has come to room temperature place it on ice to aid in crystallization. When crystallization has finished remove the solvent using a Pasteur pipette. Dry the crystals by pressing them between filter paper.

**Data:** When the crystals of alcohol have dried completely obtain the mass of the solid and record the data into the laboratory notebook. Obtain the melting point of both crude and purified triphenylmethanol and record the data into the laboratory notebook. Obtain an FTIR spectrum of the product and compare it to spectra of the starting material and possible by products.

**Calculations:** For this experiment calculate the limiting reagent (from the data that you collected) as well as the theoretical and % yields. Compare the melting points of the starting material and the product to the literature value and comment on the purity. Interpret the FTIR spectrum and state whether the expected product was formed or not.