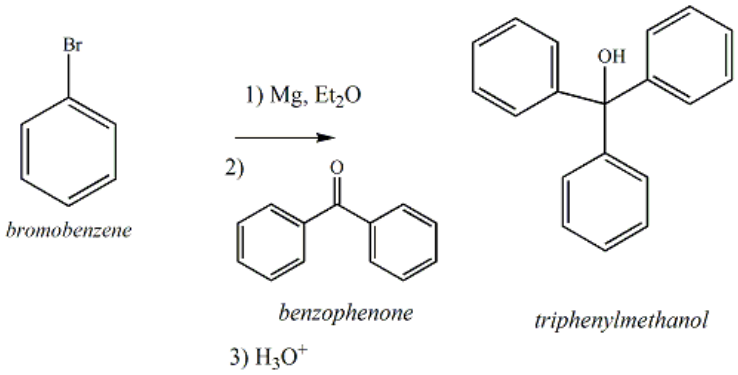
# Organic Chemistry Laboratory II
# Preparation of Triphenylmethanol (Grignard Reaction)

***Experiment Procedure*** (Printable pdf format)

## Introduction

In this two-week experiment, triphenylmethanol will be synthesized through a Grignard reaction. Students will work *individually* to prepare the Grignard reagent by reacting bromobenzene with solid magnesium in diethylether. The prepared Grignard reagent, phenylmagnesium bromide, will then be combined with bezophenone to form the desired triphenylmethanol product.



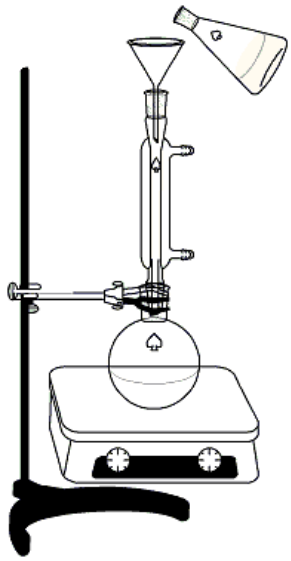

## Procedure

***NOTE: All glassware must be extremely dry! Place glassware in the drying oven for at least 10 minutes before beginning the reaction.***

***Preparation of the Grignard Reagent***

Add ~0.5g of magnesium turnings and a magnetic stir bar to a dry 100 ml round-bottomed flask, and place the flask in the drying oven for 30 minutes. Remove the flask from the oven, clamp it to a ring stand, insert a reflux condenser, fit the flask into a heating mantle and immediately attach a drying tube to the top of the reflux condenser. Position the flask in the heating mantle on a stirrer/hotplate. Set the reaction up on the side of the hood near to the faucet labeled for distillation. Allow the reaction flask to cool to room temperature completely before proceeding. While the flask is cooling, place a 150ml beaker into the drying oven.

While the flask is cooling, add 3.5 g of dry bromobenzene to a dry 50 mL Erlenmeyer flask and dissolve it in 5.0 mL of anhydrous diethyl ether. Use a glass powder funnel to dispense the bromobenze and ether to the Erlenmyer flask. Swirl the flask to completely dissolve the bromobenzene. When the 100ml rb flask is completely cool, remove the drying tube and transfer the bromobenzene solution to the rb flask (Use a funnel). If the flask is not sufficiently cool, the ether will evaporate immediately. If it evaporates, add more ether. Re-insert the drying tube and continue to stir the reaction. If the reaction does not begin immediately, carefully use a dry stirring rod to crush one of the magnesium turnings against the bottom of the flask. *Be careful not to press too hard and crack the flask!* Alternatively, a single crystal of iodine can be added to help initiate the reaction. A cloudy solution is an indication that the reaction has begun. Once the reaction has started, add an additional 10 ml of ether using a glass funnel. Connect the water hoses to the condenser and turn the water on. Use a permanent marker to mark the level of the contents of the mixture. If the reaction becomes too vigorous, cool the flask for 2-3 minutes in an ice water bath.



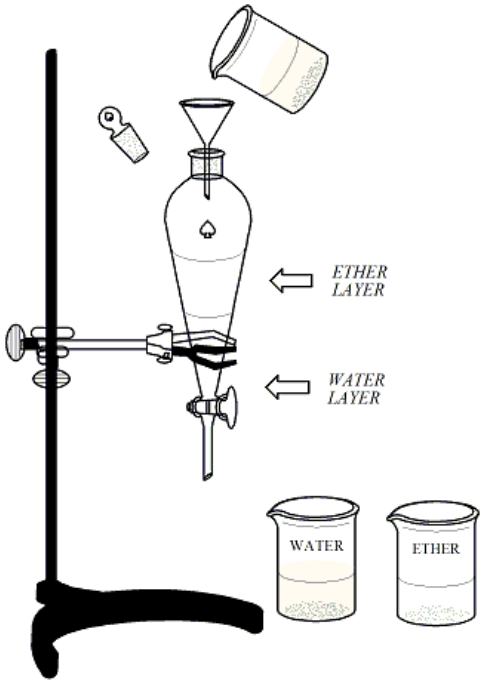
Once the reaction has become steady, warm the flask in the heating mantle on low heat for 15 minutes until the magnesium has completely dissolved/reacted and the solution has a brown or cloudy appearance. If the mixture has evaporated more than 0.5 cm below the starting line, add an additional 5ml of the ether. If loss of ether continues, turn off the heat or immerse the flask into an ice bath to cool it down. Allow the reaction mixture to cool to room temperature (if hot). Remove the condenser tube and replace it with the drying tube. Set the flask aside, keeping it clamped securely to the ring stand. *This Grignard reagent will decompose rapidly, so the next step must be started at once.*

***Preparation of Triphenylmethanol***

Remove the dry 150 ml beaker from the drying oven and allow it to cool to room temperature. Place 3.7g of benzophenone into the beaker and add 10 ml of ether. Gently swirl the beaker to completely dissolve the benzophenone. Remove the condenser tube and drying tube from the reaction flask containing the Grignard reagent, insert a funnel and begin stirring the solution. Add the benzophenone solution dropwise to the flask using a glass pipet (from the drying oven, be sure to allow it to cool to rt). (If the mixture becomes too thick to stir, add an additional 10 ml of ether.) Rinse the beaker again with 5-10ml of dry ether and add this to the reaction flask. After addition of the benzophenone solution is complete, replace the

condenser and drying tube. Turn on the water, and gently reflux the reaction mixture (with stirring) for 25 minutes. Allow the reaction mixture to cool to room temperature.

When the reaction is cooled, remove the drying tube and condenser. Continue stirring the mixture and ***slowly*** add 5 ml of distilled water dropwise using a glass pipet. This will hydrolyze the alkoxy/magnesium bromide salt to form the triphenylmethanol. The water will also react with any unreacted Grignard reagent to form benzene. After the addition of the water is complete, 15ml of 5% HCl are added with continued stirring. Continue stirring the reaction mixture until the reaction subsides. Transfer the reaction mixture to a 200ml beaker, adding additional ether (5-10ml) to the rb flask if necessary to remove any residual product. Swirl the beaker and add more ether (5-10ml) to dissolve any solids. Two layers should be apparent in the beaker and no solids should remain in the reaction flask.




Transfer the entire reaction mixture (using a funnel) to a 125ml separatory funnel (Be sure the stopcock is in the closed position). Two layers should appear. The top layer is the ether layer, containing product. The bottom layer is the aqueous layer containing the magnesium bromide salt by-product. Label two 100ml beakers or Erlenmeyer flasks as “water layer” and “ether layer". Drain the lower layer from the separatory funnel into the beaker or flask labeled “water layer”.

Add 15 ml of aqueous 5% sodium bicarbonate to the ether layer in the separatory funnel. Stopper the funnel and shake vigorously for 1-2 minutes. Vent the funnel into the back of the hood. Set the separatory funnel back onto the ring stand and allow the two layers to separate and remove the stopper. Drain the bottom (water) layer into the beaker or flask labeled “water layer”. Add 15ml m of saturated sodium chloride to the separatory funnel and repeat the process above, combining the lower layer again into the “water layer”. Pour the remaining ether layer into the dry 100 ml beaker labeled "ether layer". Add ~100mg (tip of a spatula) of magnesium sulfate drying agent to the beaker or flask labeled “ether layer”. Swirl the flask, then allow the drying agent to settle to the bottom. Decant or filter off the magnesiun sulfate and transfer the solution into a ***pre-weighed***, clean, very dry 125 ml Erlenmeyer flask. Clamp the flask containing the ether layer to a ring stand and place it in a warm water bath to remove the ether. Store your product in the open Erlenmyer flask to allow it to finish drying over the week.

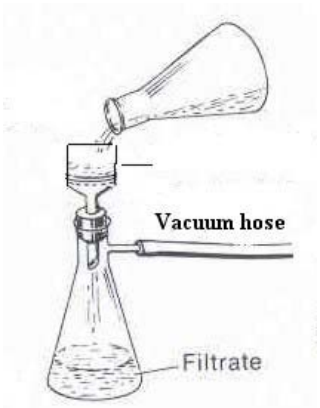
---

**End of Week 1**

---

***Trituration in Hexane of Crude Triphenylmethanol Product***

Transfer the solid to a 50ml or Erlenmeyer flask. Add 10 mL of hexane, and triturate the solid for 5-7 minutes. Triturate means to grind or pulverizea solid. In this context, trituration requires that the crude triphenylmethanol (as a suspension in hexane) is gently, but repeatedly, pressed against the sides of the beaker or flask in the hexane to promote dissolving any impurities contaminating the product. This is a form of purification. When the trituration is complete, the product is collected through vacuum filtration. Wash the solid in the Buchner funnel once with 10ml of hexane. Collect the solid from the funnel and place it on a watchglass to dry.




***TLC Analysis of Triphenylmethanol***

Prepare a TLC plate with three tick marks (labeled 1, 2, 3) to analyze your synthesized product (1) benzophenone (2) and known triphenylmethanol (3). Transfer ~10mg of the product to a vial and add 1-2ml of etherl to the vial to dissolve the product. Spot the plate with the product and use the solutions of the known compounds to spot the remaining lanes of the plate. Develop the plate using 90:10 hexane: ethyl acetate. View the plate under UV light and in the iodine chamber and mark the solvent front with a pencil. Circle the spots observed with a pencil and calculate Rf values for all the spots observed. What do the relative Rf values tell you about the success of the reaction and the purity of the product?

***IR Spectroscopic Analysis of Triphenylmethanol***

Prepare a sample of the product for IR analysis. Run a background scan if one has not yet been for the lab, and then record a spectrum of the product. Set up a table in your notebook and record the major peaks ( and frequencies in $cm^{-1}$) in the table.

***Weight and Melting Point Determination of Triphenylmethanol***

Inspect your product and if it appears to be dry, weigh it and calculate the percent yield. Calibrate the thermometer of the melting point apparatus with benzoic acid (lit mp = $122^{o}C$). Record the melting point of the product. Transfer the product to a clean vial labeled with your name, lab day, mass (in g) and experimental melting point. Hand in the labeled vial containing the product.

---

***End of Week 2***

***Waste Disposal***

Dispose of all organic waste (ether, hexane, ethanol, acetone) in the "non-halogenated" waste container. The pH of aqueous solutions should be checked. Acidic solutions should be disposed of in "acidic aqueous waste" and basic solutions in "basic aqueous waste". Used filter paper and TLC plates,$MgSO_4$ and organic solids should be disposed of in the solid waste containers. Dispose of pipets, melting point cover slips, melting point tubes in designatedwaste containers.