

The equipment setup for this lab will require the use of the following glassware assembled as shown:



- Attach the Claisen adapter to a 50 mL round bottom flask that contains the one inch stir bar and secure it with a Keck clamp. Make sure that the flask is in the correct hole of the aluminum heating block that is centered on the hotplate.
- Attach a metal clamp attached to the “Y” arm of Claisen adapter. Attach a condenser to the “Y” arm and secure it with a Keck clamp.
- **Do not attach the water lines to the condenser.**

- Attach the straight adapter and the rubber thermometer adapter on the straight side of the Claisen adapter.



Grignard Procedure

General steps students should follow:

1. Add 0.5 g of Mg powder to a clean and dry 50 mL round-bottom flask and a one inch stir bar.
2. Prepare a solution of 5 mL ether and approximately 2.5 mL of bromobenzene. Add 4 mL of this solution to the 50 mL round bottom flask.
3. Add 5 mL of the ether solution to the round-bottom flask.
4. Using a nylon rod, start grinding the magnesium powder to the bottom of the flask until the solution turns cloudy. Quickly connect the round bottom flask to the Claisen adapter.
5. Add the rest of the bromobenzene solution dropwise through thermometer adapter using a syringe. It should take about 15 minutes to add it all. Let the reaction continue to mix for an additional 15 minutes or until the solution has turned into a dark brown color.
6. Crush 5 or 6 pieces of dry ice and add ~ 10 g of the pieces to a 125 mL Erlenmeyer flask (crush using the aluminum heating block).
7. Pour the reaction mixture into the large beaker that contains the dry ice and swirl to mix thoroughly. Rinse the round bottom flask with 5 mL of diethyl ether and add it to the flask. Swirl the mixture in the flask for 5 minutes and then set it down and let the excess dry ice sublime.
8. While the dry ice is subliming, prepare a solution of 5 mL of sulfuric acid in ~5 mL of water. Add the sulfuric acid slowly to the water and then place on ice until it is needed.
9. Add the sulfuric acid solution to the reaction mixture slowly, there will be some bubbling as the excess magnesium dissolves. The ether volume should be about 25 mL. If below 25 mL, add more ether until it reaches 25 mL
10. Transfer the solution to the separatory funnel, you should be able to observe two clear layers (the top layer is ether the bottom is aqueous)
11. Shake the separatory funnel (and vent) then drain the bottom aqueous layer (waste).
12. Add 10 mL of sodium hydroxide solution, shake, vent, and drain the bottom aqueous layer (product!) Into a clean beaker
13. Acidify the basic solution with 6 M HCl until the pH is ~1, white precipitate will form.
14. Cool the beaker in an ice-water bath while you set up the Buchner funnel.
15. Filter the solid product and wash with cold water.
16. Save the product for the following week for product analysis and obtain a % yield, IR, and MP.