

Lab 05: Grignard Synthesis of Triphenylmethanol

Reading

- Pavia, et al., Expt. 38 and 38A, pp. 317-326.
- Wade, 6th ed., Sections 10-8 to 10-11, pp. 432–451.

Prelab Assignment:

Your prelab should include: Date, Introduction, Haz-Mat, Separation Scheme (attach and fill in the scheme as part of your prelab), Apparatus, Chemical Equation., Reactant Table, and Procedures.

Procedural Notes

We will follow the procedure in the lab book. Please note the following:

Preparation of Glassware

1. We will use 50 mL round bottom flasks that will be checked out to students. A special adapter that fits into the ground glass joint will also be checked out. Please clean the flask and adapter before returning them.
2. **All equipment used in formation and reaction of the Grignard must be completely dry.** Any glassware that needs to be cleaned should be washed, rinsed with a small amount of acetone, and dried in the 120 °C oven for 20 min prior to use. If you washed your glassware during the previous lab you do not need to wash your glassware again.
3. The apparatus should be assembled as shown on page 321, with the addition of the adapter for the round bottom flask. **However, the apparatus should be clamped on the neck of the flask, not on the Claisen adapter as shown in the diagram.**

Preparation of the Grignard Reagent

4. **Please keep the anhydrous ether can closed to prevent it from absorbing moisture.** Only the freshly opened can of anhydrous ether should be used for the Grignard formation and reaction. Older ether, which might not be anhydrous, will be provided for use in the workup steps after the Grignard reaction (starting with the hydrolysis using 6 M HCl solution).
5. When using the small conical vials, they should be placed in a small beaker to prevent them from tipping over and spilling.
6. When heating the reaction mixture during the formation of the Grignard reagent, try to avoid overheating and boiling away the ether. **If the volume becomes too low, add more anhydrous ether to replace the ether lost by evaporation. You can often just keep the apparatus just above the hot plate (~1-2 cm) so to avoid overheating.**
7. The most obvious sign of reaction during Grignard formation is bubbling and the appearance of a grayish-white cloudiness. Be sure to cut the Mg(s) into small pieces.

Also, you should crush the magnesium with a stirring rod once you have added the Mg (s) to your ether. Sometimes, transferring a small amount of a successful Grignard solution from another student using a Pasteur pipet will also start the reaction.

8. Keep the Grignard mixture gently boiling during and after the addition of the bromobenzene. **The mixture should be refluxed for a total of 30 min after the Grignard reaction starts.**

Preparation of TPM

9. The benzophenone should be added to the Grignard mixture in the round bottom flask. **Do not suck the Grignard mixture into the syringe** (it will form precipitates that ruin the syringe).

10. The Grignard reagent must be formed and reacted with benzophenone in the same lab day. The procedure cannot be stopped until after the step marked "You may stop here" on page 323 of the lab text.

11. Extra ether at the end of day one can be saved for use in the extractions. Seal the Erlenmeyer flask that contains the ether with a cork and parafilm.

12. Separatory funnels for the extraction will be checked out to students. Their use will be demonstrated by the instructor. **Remember to vent the funnel frequently during the extractions.**

13. The ether will be evaporated using the aspirator. The apparatus will be demonstrated by the instructor.

14. After recrystallizing the crude product, leave the crystals to dry overnight if possible. Obtain a final weight, a melting point, and IR (using a KBr pellet). The product should be submitted in a labeled sample tube.

Additional Notes

- Write the reaction in two parts, showing formation of the Grignard and then addition of benzophenone.
- Petroleum ether is a mixture of hydrocarbons. **Petroleum ether is not the same as diethyl ether (or ether).** We may use pentane instead of petroleum ether depending on what is available.

Postlab Questions

Answer these questions in your lab notebook after your conclusion is a section titled "Post-lab Questions"

1. Why is it important that all the equipment used in the Grignard formation be completely dry? Demonstrate this by drawing a sample reaction to illustrate your point.
2. Draw and name the major undesired by-product formed in the Grignard reaction in this experiment.
3. Read the procedure section on page 324 entitled *Evaporation*. What is the purpose of adding petroleum ether to the crude product obtained after evaporation of the ether?
4. Write the chemical equation for the reaction that occurs when 6 M HCl solution is added to the reaction mixture (in the procedure section *Hydrolysis* on page 323).
5. What product would be formed if acetone were added to the Grignard reagent instead of benzophenone?

