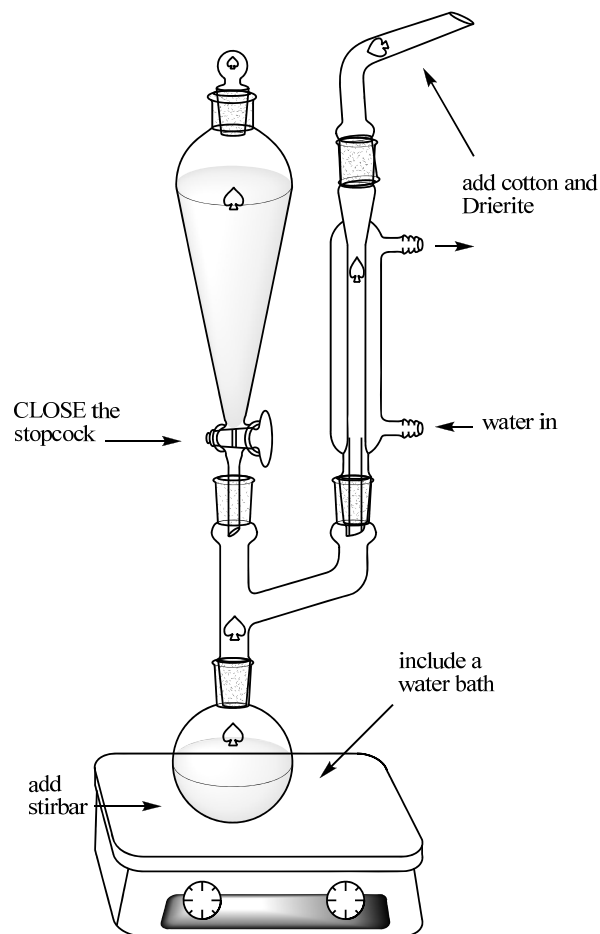


## Synthesis of Triphenylmethanol

Purpose: To synthesize and isolate a pure sample of triphenylmethanol in the highest yield possible using the Grignard reaction

Procedure:

1. Set up the following apparatus, filling the drying tube with Drierite or other drying agent provided.



2. Add about 2 mL of anhydrous ether to the roundbottom through the separatory funnel. Allow the ether to evaporate to push the oxygen atmosphere and most of the water out of the glassware.
3. Prepare a solution of 2.4 mL of bromobenzene and 5 mL of anhydrous ether in a small, dry Erlenmeyer flask and cap the flask.
4. Weigh 0.5 g of magnesium turnings that have been freshly ground into the roundbottom flask. Add 5 mL of ether to the flask, and a VERY SMALL crystal of iodine. (You only need the smallest crystal of iodine you can find—just big enough to see. DO NOT add more than this much iodine.) Reassemble the apparatus and make sure the stopcock on the separatory funnel is closed.

5. Add the bromobenzene solution into the closed separatory funnel.
6. Turn on the water to the condenser.
7. Open the stopcock on the separatory funnel CAREFULLY to add about 1 mL of the bromobenzene solution into the reaction flask, then close the separatory funnel. Watch the reaction carefully to see when it starts. You will see it start if the ether begins to bubble or the solution gets cloudy. (See the AI if your reaction doesn't start in 10 minutes.) Once the reaction starts, it is important to slowly, dropwise add the bromobenzene to the reaction. You want to maintain a gently boil in the reaction flask. If you add it too quickly, the reaction will get out of control. If you add it too slowly, the reaction will stop. The addition often takes about 5-10 minutes.
8. Once the bromobenzene has been added, heat the reaction to reflux for 15 minutes. (You should have your heat source ready at this point—don't start heating it up after you have added the bromobenzene!) If necessary, add more ether through the separatory funnel so that the total reaction volume is at least 10 mL. At this point, the reaction is often brown/gray, chalky/cloudy, with only small bits of metal remaining. Turn off the heat and allow the Grignard reagent to cool to room temperature.
9. Dissolve 1.2 mL of methyl benzoate into 5 mL of anhydrous ether in a small Erlenmeyer flask, then add the solution into the separatory funnel. (Make sure the stopcock is CLOSED!)
10. Add the benzoate solution to the Grignard reagent slowly, dropwise. The reaction is exothermic and will begin to bubble. Add the benzoate at a rate that maintains a gentle reflux. After the benzoate is all added, add 2 mL of ether to the sep funnel to wash out remaining benzoate and allow it to drip slowly into the reaction.
11. After the benzoate is added, heat the reaction to boiling for about 20 minutes, then cool to room temperature.

#### Work-up

1. Put about 10 mL of 6M sulfuric acid and 10 g of ice in a beaker. Slowly add your reaction mixture to the beaker with stirring. Add about 5 mL of ether (no longer need anhydrous ether—why?) to the reaction flask to wash out remaining material; add it to the beaker. Stir until all solids have dissolved. If it doesn't all dissolve, add ether until you have a total of 15-20 mL of ether in the beaker. Test the aqueous layer to be sure it is acidic—if not, add cold 6M sulfuric acid dropwise until the aqueous layer is acidic.
2. Pour all the mixture from the beaker into a separatory funnel. Shake and vent. Remove the aqueous layer. Wash the organic layer sequentially with a 5 mL portion of brine, two 5 mL portions of saturated sodium bicarbonate (VENT!), and a 5 mL portion of brine. Dry the organic layer with sodium sulfate or magnesium sulfate. Decant the organic layer into an appropriately sized roundbottom flask and remove the ether with a rotovap. A solid should form.
3. Recrystallize the crude product from cyclohexane (about 10 mL per gram of product.) Isolate the product by vacuum filtration, pulling air through the purified product for several minutes.
4. Obtain a melting point of the purified product.

#### General Ideas for your Discussion:

1. You should probably have a paragraph or two about the synthetic procedure. Point out the important points of the procedure. (For example, a flake of iodine was added because... Ether was allowed to evaporate to push the air out of the glassware because...)
2. Since the purpose of the experiment was to synthesize the compound in high yield and purity, you should probably discuss your yield, and use the data you collected to discuss the identity and purity of the compound you isolated.
3. You should probably have a paragraph discussing what worked, what didn't, how the reaction could be improved. (DO NOT include things like, "The reaction would work better if I didn't spill it." DO include procedural changes that would improve yield or purity.)