GRIGNARD REACTION

1. Weigh out 0.15g shiny magnesium turnings and put them into a dry, 20 mL round bottom (RB) flask with a dry, magnetic stir bar. Attach a dry condenser, clamp both firmly upright.
2. Transfer around 20 mL of anhydrous diethyl ether into a dry 50 mL Erlenmeyer flask (this will be your ether reservoir for the experiment) and put a top on it.
3. Preweigh a 5 mL conical vial and then weigh out 1.0 g bromobenzene (MW=157.0). Add 4 mL of ether to the vial and swirl gently until the bromobenzene dissolves. Then withdraw with a dry Pasteur pipette, approximately 2.0 mL of the solution and put a cap on the vial. **SAVE THE REMAINDER OF THE BROMOBENZENE/ETHER SOLUTION FOR LATER USE; RECAP THE VIAL BETWEEN USES**
4. Add the 2.0 mL of the solution to the magnesium turnings in the RB flask. Position your apparatus (RB, Condenser) right above the hotplate. **YOU DO NOT WANT THE TURNINGS TO GO TOO HIGH ON THE WALLS OF THE FLASK SO BE VERY GENTLE** Watch the bubbles begin; this is how you know the reaction is working. Alert the TA if you don't see any bubbles. After the bubbles begin, a cloudy solution should appear.
5. Remove more of the bromobenzene/ether solution from your Erlenmeyer flask with the pipette. Add this to the RB via the top of the condenser. SLOWLY OVER A PERIOD OF 10 MINUTES, ROUGHLY DROP BY DROP. Repeat additions until all of the solution is gone. Make sure that it doesn't boil too vigorously; if it does, slow the rate of addition down. If there isn't any boiling, let the TA know.
6. After all of the bromobenzene/ether has been added, put another 2.0 mL of anhydrous ether into the empty vial of the bromobenzene/ether solution. This "rinse" will make sure that all of the bromobenzene gets added to the magnesium. Add this just as you have before, slowly. You need to add more ether (your solvent) if you have boiled a lot of it off.
7. Once all of the magnesium has reacted (you don't see any more solid, or bubbling stops) cool the apparatus to room temperature.
8. Acquire 10 g of crushed dry ice in a 125 mL Erlenmeyer flask from the TA **BE CAREFUL WITH THE DRY ICE! YOU CAN GIVE YOURSELF SEVERE BURNS IF YOUR SKIN TOUCHES IT!** Slowly pour the phenylmagnesium bromide solution onto the dry ice with vigorous swirling; this will make it pretty thick. Keep swirling for a good 5 minutes.
9. Rinse the original RB reaction flask with 2-3 mL of anhydrous diethyl ether to get all of phenylmagnesium bromide out of it. After the addition is complete, cover the flask with a small watch glass or filter paper, and allow the excess carbon dioxide to sublime. You can make it go faster by swirling very slightly in warm water bath. **YOU MUST BE CAREFUL BECAUSE TOO MUCH CO_2 GAS EVOLUTION WILL SPLASH ALL THE FLASK'S CONTENTS ON YOU OR ON THE FLOOR!**
10. After the excess dry ice has completely sublimed, most of the ether will have also evaporated, so add another 20 mL of diethyl ether to the reaction flask. Combine 5g of ice and 10 mL of cold 3**M** sulfuric acid in an Erlenmeyer flask, and add the cold acid to the reaction mixture SLOWLY to

avoid excess foaming. If the ether has overly evaporated, add around 15-20mL. You can't have any more than 25 TOTAL mL in the reaction mixture.

11. Use pH paper to see if mixture is acidic. If isn't, add more of the 3M sulfuric acid drop wise until the layer is acidic.
12. Swirl the mixture and transfer it to a 100 mL beaker or small Erlenmeyer flask. Again rinse the flask with more ether to get everything and pour it into the Collect the two layers into two different flasks. The ether layer will contain your product. Label each one!
13. Extract the aqueous layer with a 10 mL portion of ether and add this to the ether layer.
14. Extract the *combined* ether fractions one after the other with 2 10 mL portions of a 1M NaOH solution. Transfer the two basic extracts to an Erlenmeyer flask and slowly add 6M HCl until precipitation of the benzoic acid is complete and the aqueous mixture is acidic.
15. Cool the solution in an ice water bath, isolate the solid benzoic acid by vacuum filtration, wash it with water, and air dry it. Recrystallize the benzoic acid with water.
16. Weigh the benzoic acid and find percent yield; find its melting point.