The Grignard Reaction Post. Laboratory Assignment This post-lab assignment is a formal written laboratory report. Follow the guidelines below to write your report. You must type the report. Chemical structures should be drawn in ChemDraw. Scientific reports should be written objectively, in an impersonal past tense (e.g. “the chemical was added”, not “we added the chemical”). · Title: Your report should include a descriptive title (not “The Grignard Reaction”). · Names and Date: Include the names of all group members writing the report, and the date of the report. · Purpose: The purpose should be concise (1 or 2 sentences) and state the objectives of the experiment, including the type of reaction studied, the material used/product obtained, and methods used to characterize the product or test for purity (i.e. melting point, TLC, NMR, etc.). In other words, why are you doing this experiment, and how are you doing it? · Chemical Reaction: Drawings of pertinent structures and/or reaction schemes. · Procedure/Observations: Observations (including color changes, exact masses, volumes, times, etc.) should be recorded throughout the experiment and included in the report with a summary of the procedure. · Calculations: Any necessary calculations should be shown. For this experiment, that includes determination of the limiting reagent, calculation of theoretical yield of the product, and calculation of percent yield. Write out the relevant formula or equations and clearly show your work. Use proper significant figures and indicate proper units on all numbers for which units are applicable. · Results: This section should be a presentation of your data, and should include tables of data (as many as necessary), as well as a description of your products, including yield, appearance, and other physical properties. This section should not include any discussion. · Instrumental Analysis: This section should include any spectroscopic data obtained (NMR, IR, GC/MS, etc.), as well as any interpretation of this data. · Conclusions: This section should discuss the significance of your results, any conclusions you have reached based on your data, arguments to support those conclusions, and a discussion of the methods used to obtain your results. For this experiment, you should discuss the synthesis, separation, and purification techniques used. This section should also include conclusions about the purity of your product, the possible origin of any errors that you observed or that may lead to a poor yield or quality of products (including formation of unwanted by-products), and a comparison of experimental data and spectra to literature data. Do not include excess or irrelevant procedural detail. Procedure Week 1: Assemble the reaction apparatus shown on the photo page of this notebook section. Be sure that all of the glassware used in this experiment (including graduated cylinders) is clean and dry. Lightly grease all ground glass joints. Clamp a 100-mL single neck round bottom flask fitted with a Claisen connecting tube to a ring stand. Leave room for a water or ice bath under the flask. Do not support the flask on a ring. Place a reflux condenser in the side neck of the Claisen adaptor, and place a separatory funnel in the vertical neck. Fit a drying tube containing a small amount of glass wool and calcium chloride to the top of the condenser. Use blue Keck clamps at the appropriate joints. Attach clear Tygon tubing for water and start the flow of water. Make sure there are no leaks. Have the instructor check your apparatus. Grind 2 g of magnesium turnings in a mortar for a few minutes to provide a fresh surface. Place it in the 100-mL round bottom flask with 15 mL of anhydrous diethyl ether. Add 9 mL of bromobenzene to the flask. Warm the flask gently in the palm of your hand while gently grinding the Mg against the side of the flask with a glass stirring rod. Continue to warm the flask and use the stirring rod until the reaction starts, as indicated by bubbling and turbidity. (If your reaction does not start after a few minutes, it may help to use a starter solution. Consult your instructor.) As soon as the reaction has started, reattach the flask to the connecting tube. Water should already be flowing through the condenser. Add 25 mL of anhydrous ether to the flask through the separatory funnel. Continue to reflux the solution for 20 minutes, swirling the flask occasionally and warming the flask as necessary to keep the reaction going. If the reaction is too vigorous cool the flask in an ice bath, but do not overcool as this can stop the reaction. Continue with prepping the solution for next step but do not add until after the 20 mins. Mix 5 g of methyl benzoate with 15 mL of anhydrous ether in a beaker and place the ester solution inside the separatory funnel. Once the formation of the Grignard reagent is complete (after the 20 minute reflux period), cool the flask and its contents briefly (few seconds) in an ice bath. Add the ester solution dropwise while swirling the flask to ensure mixing. If necessary, use the ice bath or adjust the rate of addition of the ester to control the reaction. When the addition is complete, label and store the flask as directed by your instructor. Week 2: Pour your reaction mixture (including all solid) from last week into a 250-mL Erlenmeyer flask containing 50 mL of 10% sulfuric acid and approximately 25 g of ice. Make sure the flask already has the ice and the acid before you pour your reaction mixture. Rinse the round bottom flask (that used to have the reaction mixture) with a small portion of diethyl ether, followed by a small portion of 10% sulfuric acid, and add these rinses to the Erlenmeyer flask. Swirl the mixture well to promote hydrolysis. Pour the solution in a separatory funnel, leaving behind any remaining Mg and ice (DO NOT ADD THE SOLIDS TO THE SEP FUNNEL). There should be two layers. Drain the bottom layer (aqueous into a medium beaker). Keep the ether layer in the sep funnel. Add 5 mL of 10% sulfuric acid, carefully extract the mixture. After the separation of layers, remove the bottom aqueous layer into the same beaker from step 5. Add 5 mL of a saturated sodium chloride solution. Extract the mixture, allow the layer to separate and drain the bottom layer in the same beaker from steps 5 and 6. In a CLEAN beaker drain the ether layer in and add small portions of magnesium sulfate (solid) using spatula until no new clumps are forming. The formation of clumps indicate water is present. Transfer just the liquid (ether layer) carefully leaving the solid behind to a small (100 mL beaker) Add 15 mL of petroleum ether to the layer, and evaporate the solution slowly on a steam bath (Large beaker half filled with water on a hot plate) just until crystallization begins. Cool the mixture on an ice bath (Large beaker with ice water) to complete crystallization. Collect, dry, and weigh the product in a clean tared watch glass. Use a spatula to transfer the solid. Make sure the you weigh the watch glass before adding the solid. Determine the melting point and IR spectrum for your product. Dispose of all waste from this experiment in the appropriate waste containers as directed by your instructor.