Experiment #6: Grignard Synthesis of Tertiary Alcohols

When you have to boil off the ether in one of the last steps be sure to start the air first and then bring the beaker close to the air. If you hold the beaker in front of the spout and turn it too high you'll spray your product all over the place. CA

It is also important to ensure that boiling is complete before you remove the beaker, if not left over solvent will make the product impure. KB

After the slow addition of ketone to round bottom, your solution should be clear. Otherwise, liquid-liquid extraction will be difficult to perform. You can make your solution clear by adding about 3mL of sulfuric acid. LM

To prevent this, I used a hose connected to the air. This allowed the air flow to be more controlled and while the ether is being evaporated you can begin clean up without having to hold your beaker. AJ

During the ketone solution addition to Grignard reagent I opened the separator funnel to a trickle so there was a very small steady amount of solution constantly flowing into the reagent. I monitored the trickle to be sure that not all of the solution was added before the end of 30 min. Adding the ketone in this way worked very well for me. NL

The Grignard reagent is very sensitive it is important and cannot be stressed enough that all glassware is clean and dry before adding anything to them. Also it is a very good idea to set up the apparatus prior to addition of the reagent. Remember to plug all holes! KB

Be sure not to put your Grignard reagent in the round bottom flask on a hot thermwell since some of the fumes may start to escape through the reflux even though the water might be running. The reagent starts to react very quickly, so just simply lower the amount of heat you are providing. PO

Make sure to take your time when adding the ketone solution, use the whole 30 minutes to add your ketone. Also don't let your solution boil too fast, you don't want your ether to burn. LC

You can paper napkins to dry all your equipment this works better than using the air. LV

Watch out for the extractions for this lab. Usually we keep the bottom layer of the extractions, but this time we will be keeping the top layer called the ether layer. You can pour the ether layer into a beaker and do the next extraction on it. Also, when gravity-filtering, remember to weigh the receiving beaker BEFORE you filter it. NT

A couple of tips for this lab:

-All your glassware must be dry; use air to dry out glassware quickly.

-Plug the openings of your reflux apparatus, if there are no more cotton balls use tissues.

-You must continually monitor the rate at which your ketone solution is added, at first it adds the way you set it up, but after a while it tends to stop adding any ketone and you must recalibrate the funnel.

-In this experiment you will perform the most complicated extraction(s) in your life so pay attention to what you are doing and if you are not sure, ask.

-Extract gently, flip the separatory funnel upside down and right-side-up, releasing the gas in between; don't shake the funnel or emulsion is likely to form.

-If you plan on evaporating the ether then set up the hot water bath ahead of time so it is ready to go. BL

found this link (a video) helpful when learning to set up a reflux apparatus: http://ochem.jsd.claremont.edu/lab.dir/refl1.htm JW

Remember to boil off the ether. Otherwise, your yield will be above 100%. Continue to boil until it stops so that all of the ether is boiled off. BMB

Make sure to dry out your entire equipment. There shouldn't be any amount of water. Try obtaining clean equipment. MJ

During GC analysis, there are two main peaks present; one small and one large. According to Cram's rule, the small peak represents the cis product and the large peak represents the trans product. This is so b/c the trans product's phenyl group attaches to the opposite side that the methyl group is on. SF

remember to keep a close eye on your boiling stick. Please do not just stop the evaporation if bubbles are still being produced on the stick (even if they are small) TM

I would rather not repeat this lab with Grignard reagent again since it was so volatile and explosive. When I received my reagent I needed to hold my cap with my hands the whole time until I connected it to the apparatus because otherwise it was already reacting with oxygen and popping like crazy. PO

I agree that I was a little nervous about this reaction since Grignard is so volatile. Although we learn about Grignard synthesis in class, it would be nice to use the Barbier reaction since it was noted that the reaction product can be either a secondary or tertiary alcohol. Plus, it would be in line with practicing GREEN CHEMISTRY! AG

The student should have an option of either experimenting with the Grignard Reagent or conducting the Barbier experiment. Then compare the results. MJ