

Experiment #2: Diels Alder

I did a little bit of research and it appears that the product will readily go through hydrolysis in the presence of water. So maybe water usage during recrystallization should be eliminated and something should be used in its place. Maybe add a boiling chip or something. CA

<http://www.franklincollege.edu/pwp/lmonroe/Organic%20Chem/Diels-Alder%20Reaction.pdf>

In step 8, the lab instructs you to carefully transfer the hot liquid from the round bottom to a beaker. Do this as quickly (and carefully) as possible. I waited for it to cool down a little, and it had already started to crystallize. MS

I am not sure the recrystallization with hexane was really necessary. The product really did not seem to improve in purity, and I think I lost a lot more product that way. MS

When recrystallizing, try using the smallest amount of solvent possible so the solvent is not recrystallized. LM

This is a general suggestion, since this is the first experiment requiring reflux (or distillation) I thought this would be an appropriate place to mention it. When setting up a reflux apparatus or distillation apparatus in your half of the hood take a few moments to lay it out. The direction that it is facing in the picture/lab handout may not be the best lay out for your half of the hood. It will not cause the experiment to yield lesser results if the apparatus is rotated 180 degrees and may result in a more efficient and less clustered work space. NL

This is the first experiment of the semester that requires analysis of IR and NMR. Be sure and hand in the IR and NMR print outs with the Post-Lab. Do not be afraid to draw and write on the print outs to indicate the presence of functional groups. This will help to avoid confusion in a typed analysis and seeing functional groups indicated on actual computer readings will help when it comes time to take the tests. NL

This is also a general suggestion. When refluxing or distilling, start timing when the solution being refluxed or distilled starts to boil. Do not start timing when the solution is placed in the apparatus and heating begins. This will add several minutes to the experiment but will yield much better results. NL

In the introduction to the lab it states that the Diels Alder Reaction is useful with high yield. To increase the percent yield of your product you may want to vacuum filter the crude product through the Buchner funnel twice. I noticed that i had quite a few crystals remaining tin the funnel which when vacuumed through again increased my percent yield. JS

After the reflux process, there may be leftover product in the round bottom. Do your best to collect all of the substance in the flask. If you have already poured the liquid out of the round bottom into another container, you may pour it back into the round bottom, swirl it, then pour it back into the other container. You may also take a spatula to sweep out the product in the flask that way. RR

Make sure you keep the contents in the round bottom flask then put it directly into the ice bath for cooling instead of transferring contents into another container first. I stupidly transferred the contents in the round bottom flask to a beaker first and I had to rewarm the contents up more in order to try to dissolve the solid that already crystallized. this made me loose product. NT

When setting up your apparatus, use a clamp to hold the neck of the round bottom flask so that you can immediately remove it from the heat source and transfer it before it begins to crystallize. AJ

When working with thermwells or other heating sources, make sure to turn on the heat source immediately at the start of the lab. This way you are not wasting any time during the experiment and when your reflux or distillation apparatus is set up you can immediately start the boiling process because your heat source has been pre-heated; this trick usually saved me about 15min of lab time. BL

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it is easy if you place the heat thermwell on a ring clamp as well. When i did it in the beginning, I had the thermwell on the lab surface and it was difficult to remove the round flask because the reflux/condenser apparatus was getting in the way. It is easier just to move the thermwell down and then remove the round bottom TM

When conducting the Diels Alder, make sure your equipment is set the correct way because, one small mistake will cost you the experiment. MJ

This applies to this lab and most others as well, but when getting an IR not only does it help to immediately label the functional groups, but make notes as to the desired product, and any unreacted reagent as well. Also the date is really important to note (sounds silly) but trying to tell different IRs apart after a week or so got really involved. Do the work of labeling ahead of time and it will pay off. This was important for this lab. DF

*Take time to scrape the crystals from the bottom and sides of glassware. It takes time, but it's worth it for a better percent yield.

*Don't tip over your Buchner funnel! I lost some product with this mistake. JW

When setting up your apparatus, make sure you have a good seal on all joints. JW

Don't forget to re-weigh the sample after drying and record both weights. Use the dry weight for calculations not the weight of the sample immediately after the procedure like I did. SF

The hexane to purify the crystals was unnecessary because the crystals were clear enough. Some product was lost due to this step. But the results were still sufficient.