Experiment #5b: Oxidation of Secondary Alcohols Part II

Make sure to monitor the boiling point of the evaporating solution to determine when the solvent has fully evaporated. If you don't check the temp, you may evaporate off product. MS

Don't bother with the 2,4-dinitrophenylhydrazine reagent test. It doesn't produce any different results- they all look the same. Many people had this problem. MS

For the second part of this experiment, I decided to use less acetic acid because the first time around, there was a significant amount of it in my overall product. Also, after reading the article " The Design of Laboratory Experiments in 1980's: A Case Study on the Oxidation of Alcohols with Household Bleach" by Mohrig, Nienhuis, Linck, Van Zoeren, Fox, and Mahaffy; they noted that there was no need to use large amounts of acetic acid as reported by Stevens and others, but rather 0.5 mL acetic acid per g of alcohol was sufficient. So this further supported the change I made. This time around, I used 2.5 mL of acetic acid for the 5 g of alcohol.

After conducting the experiment with the modification, I was successful in optimizing my reaction. I received more of my desired product and there was less acetic acid in my product. However, the only downside was that there was some of my starting alcohol in my product. Overall, successful reaction.

However, you can repeat the experiment with a slight modification or do anything you like! Both pros and cons to whatever option you choose. AG

For the second part of the experiment I decided to use 30mL portions of dichloromethane instead of 20mL portions. At the end of the experiment there was no real change with the results. Use another scientific approach rather than adding the extra 10mL of dichloromethane with each extraction. JS

If you are doing the experiment "A Solvent-Free Oxidation of Alcohols" experiment, does forget to scale up the reaction in MOLAR quantities, not just by mass. RR

I also did the solvent free reaction and i think the procedure called for way too much of the oxidation reagents. There were so much that the final product ended up being so thick and clumpy and would not come off the glass!! so my tip is to use less of the oxidation substances TM

When using "A Solvent-Free Oxidation of Alcohols" or any kind of reaction for the second part of alcohol oxidations, make sure to calculate amounts of reagents you are going to use in a ratio that is comparable to the first reaction. PO

Make sure to scale the reagents in a proportional way. You do not want to scale one reagent without doing the other because you may not get the desired product yield in the end. AJ

If you choose option B "A solvent free oxidation of alcohols in an organic laboratory" make sure you know the difference between distillation and using a reflux condenser. I did not do the reflux condenser part and I did not obtain the desired product. LC

For this second part of the lab, everyone is choosing their own experiments to do, thus it is not possible to really on your partners or classmates for much help. Make sure you know what you are supposed to do and what your experiment involves. It is important to know how to set the experiment up. For example in the solvent-free lab which I did, I had to make a reflux apparatus. I did not know how to do this at first and it took too much time trying to figure it out. Best come prepared. NT

For this experiment I chose option B (Solvent-Free Oxidation) and it worked out really well; although, you need to scale up the original reaction by a factor of about three. The procedure is very similar to the original reaction so all tips from 5a still apply.

Student Comments Spring 2009 Dominican University

This reaction is quicker and simpler than the original one and will give you a purer product; however the percent yield was much lower. BL

If you plan to oxidize methylcyclohexanol with Potassium Permanganate and copper sulfate be sure to boil off the hexane. Otherwise, it would be difficult to decide which experiment was better when comparing it to your prior experiment. BMB

Conduct the experiment using the suggestions given in "A Facile Oxidation of Alcohols using Pyridinium Chlorochromate." Lower the content of acetic acid from 15mL to about 2mL. This will retain a better product. JM

The "solvent free" procedure seemed like it would be interesting, however as mentioned already the yield was really low. I actually had a fair amount of unreacted reagent as well. Consider changing the scale of the reaction, or I would recommend trying one of the "old school" reactions from the 60's or 70's. DF

For the second part of the oxidation experiment, I increased the amount of Sodium Hypochlorite, since the sodium hypochlorite was the limiting reagent in part one of the experiment, The results where much more successful once there was more of the limiting reagent. GL

I performed the solvent-free procedure. Don't be worried that the appearance of the product is like a black charcoal solid. I thought I burned the product, however, that is supposed to be the appearance. SF

I chose to do the solvent free oxidation of alcohols. While the procedure in the recommended article suggests vacuum filtering the product, I think gravity filtering is a better method because hexane is so volatile. KB

For the first part of this experiment the starch-iodide paper was unavailable. When I used the paper for the second part of the experiment the test was negative. Just out of curiosity I tested the solution again after distillation and the test was positive. It seems that there was so much bleach in the pre-distilled solution that it was actually bleaching the starch-iodide paper and giving a false negative reading. I wanted to bring this possibility to your attention for future labs. I do not know if there is another test that can be done to confirm the presence of any unreacted hypochlorite. I believe that my experiment would have had a much higher yield if a second distillation had been done after positively neutralizing the solution a second time. NL

I altered my procedure for the second part of experiment. The change that I did was used more dichloromethane and extracted it more times, this method did not turn out very useful because there was still a lot of acetic acid left in my product. So try something different and definitely use the articles provide by the instructor to improve your percent yield. NL

I use the Solvent free experiment and I believe that it was better method in terms of the environment. BMB

Write in Bold letters so the student remembers to weigh the beaker before transferring their product. JM

I think it would be an improvement for this lab if students did research BEFORE lab to optimize their experiment. Then, the second week they can work on perfecting their technique. This way, students learn how to optimize the experiment and can also learn to optimize their lab techniques. JW