

Nerz Notes for Week of November 17, 2012

1. Due this week - you need to take the paper that is linked on the website and on this week on moodle – alkylation of 4-methylumbelliferone and outline the procedure in your notebook. You need to scale the reaction down to your scale. Note: very exciting - you are using your compound from next week. So far they look pretty good from IR an mp and I think they will be ok. Scaling down meand if the procedure is writing on a 1 molar scale and you have .1 mole you would cut all the reagents down to ten percent. If you can do this in advance it would be great. It would be wonderful if you could work with over 0.1 gram of material. It is going to be tough.

You should also read up on the moodle reading on the most recent moodle additions about TLC and Chromatography. Please try to watch the embedded videos. You will be doing a little TLC only in lab.

That is what is due.

2. I will be also loading a worksheet but this is not due until after THANKSGIVING. I and Jisun will be grading this and every student must write one. It will cover last week and this week in lab. This worksheet on the the mini-synthesis and the worksheet for the last lab after Thanksgiving and a quiz are what are left to do in this course. All relatively small assignments.

I know some people are frustrated to me, but I was trying to be flexible given the large differences in chapters covered by the two sections and the many trips and the excess stress some people seemed to be experiencing. I try to be responsive and my changes normally make things a little lighter not harder. I hope you can see this.

3. As far as the worksheets for fumarase and GCMS, I will reiterate I want one for each lab from each group with all names on it – all will receive check plus which translates into a ninety five for participating in the workshop/conference.

4. This week in lab. You will be setting up the very small scale version of the SN2 alkylation of your 4-methylumbelliferone. You must use your smallest glassware. It will be a standard reflux with a 25 mL roundbottom with a reflux condenser. Instead of doing the reaction under nitrogen we will be using drying tubes. I would recommend drying your glassware in your oven. I will have the ovens on when you get there.

5. You will reflux the reaction for two hours using your small heating mantle and a voltage of about 45 (this will require some monitoring). Refluxing means your have one drop per second dripping back into the flask from the condenser. What is the purpose of a condenser – think about this.

6. When you get the reaction started, you need to run a TLC plate of your reagents as a baseline (this will be discussed in lecture). This will be like running a GC of your starting materials.
7. Then for an hour you should go in room 180 and work on your gcms worksheet. We will work with you. Your reaction will be cooking and you should be able to leave it for an hour.
8. After an hour, you should go back in the lab and run a TLC and your starting materials to see how your reaction is going. You will actually have to lift off the condenser and remove a tiny bit of the solution to spot on the TLC plate.
9. Then you should return to the lecture hall and finish the GCMS worksheet. Try to get a handle on this before you leave. We will work with you on this.
10. Then you should go back in the lab after two hours of refluxing and run another TLC to see how your reaction is progressing. After this analysis you will stop and the most you will do with your reaction, is decant the reaction off the potassium carbonate and allow the solution to dry on a tared watch glass. If you want to see what it looks like by IR we can do that. We will see how it goes.

This is a little adventure. It is a new reaction. Be prepared for adaptations and failure :). Everything at this level does not always work. You are using your own product of course.

Hope this makes sense.

Due this week: prelab preparations as outlined above.

Due next week: nothing there is no lab, unless you need to do a makeup

Due the week after Thanksgiving, the worksheet I will be posting and prep for the last lab – oxidation of isoborneol.

Due the last week of lab - your worksheets for the conferences if you have not turned them in JUST for accounting purposes and the quiz. The quiz will be distributed the last week and will be on the two conferences. I am going to print out this last paragraph and post it on my door and give it out in printed form.