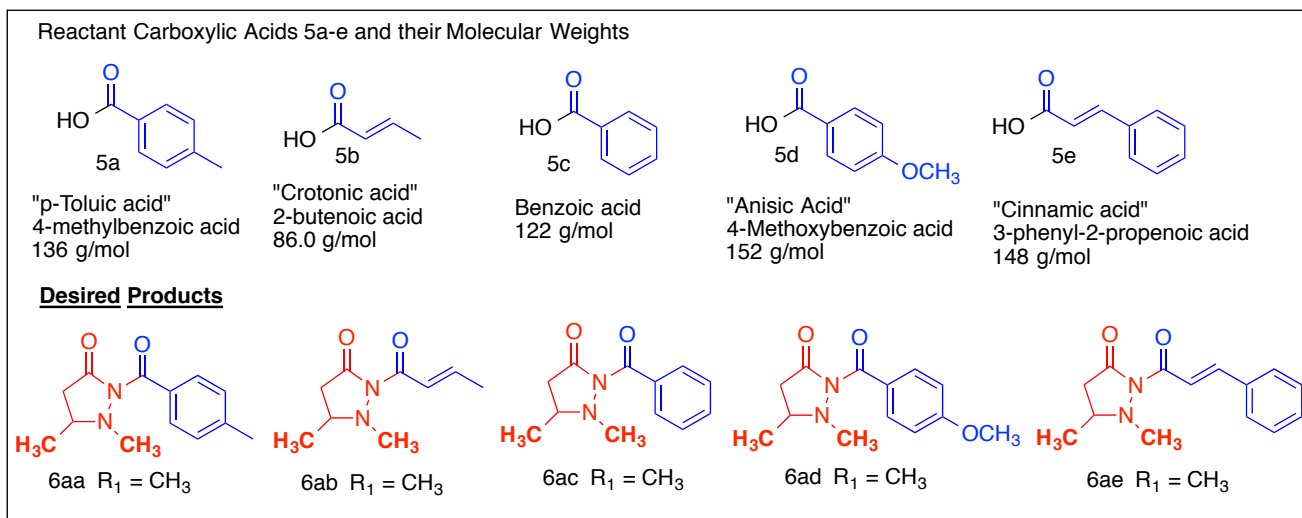
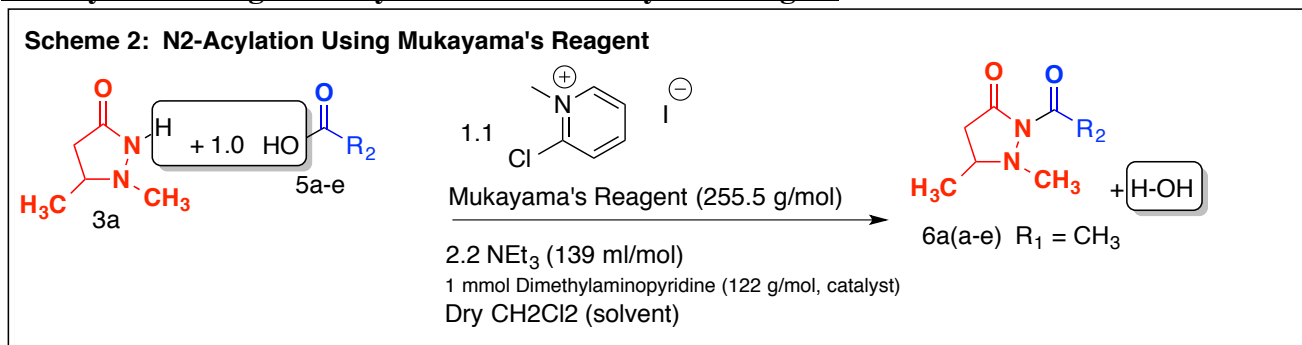


**N2-Acylation using Carboxylic Acid and Mukayama Reagent**

**Note:** See page 70 for an overview of what the lab-report requirements will be.

Or the weblink: <http://web.mnstate.edu/jasperse/Chem365/ResearchModule-2-COVID-Research-Report-Summary.pdf>

**Reagents:**

1. Pyrazolidinone **3a**: Report how many grams and mmoles were produced in Scheme 1/Week 1: That represents the starting material for this experiment.
2. Anhydrous CH<sub>2</sub>Cl<sub>2</sub>: 20 mL
3. Carboxylic acid **5**: 18 mmol
4. Dimethylaminopyridine (122g/mol): 1 mmol
5. 2-chloro-1-methylpyridinium iodide (Mukayama's Reagent, 255.6 g/mol): 22 mmol
6. Triethylamine (139 mL/mol): 44 mmol
7. Ether: 60 mL
8. Either 3:1 ether/dichloromethane: 45 mL
9. silica: 25g

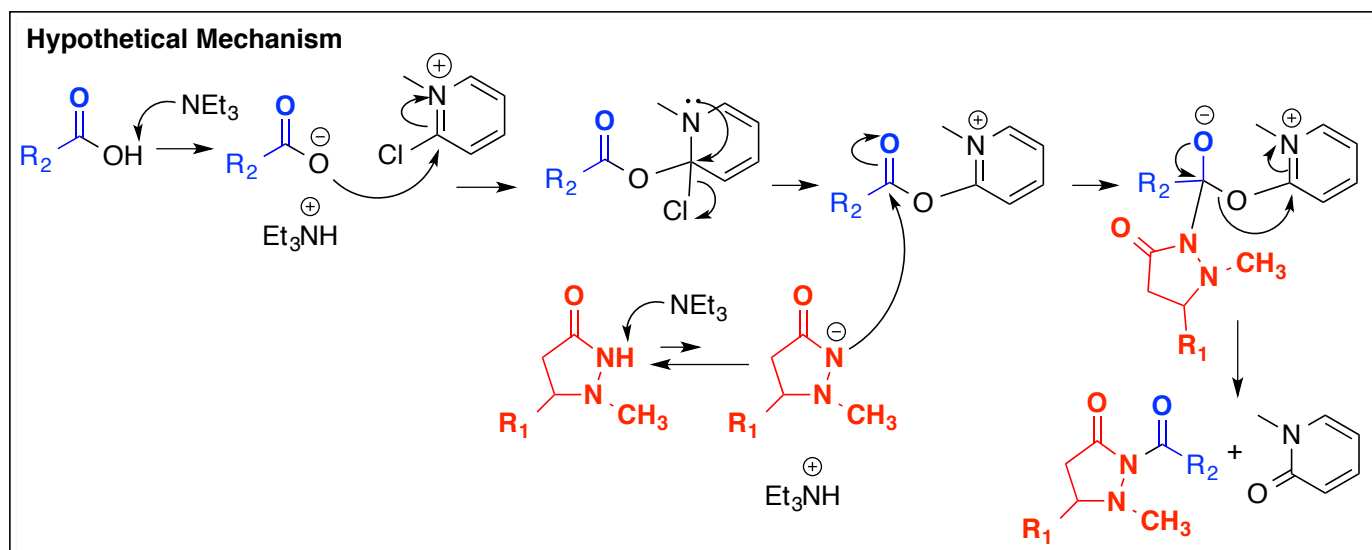
**N2-Acylation of N-Phenyl Pyrazolidinone Using Carboxylic Acid and Mukayama's Reagent (9)****Part 1: Starting the Reaction for N-Acylation**

1. Use your ≤20 mmol of the pyrazolidinone **3** that you made in Scheme 1, in the same 125-mL flask.
  - Record the mass of the flask+stir-bar that you recorded at the start of Scheme 1.
  - Record the mass of your pyrazolidinone **3** that you produced at the end of Scheme 1.
  - Record the number of mmol for pyrazolidinone **3** that you calculated at the end of Scheme 1.
  - Be sure to save NMR and GC copies for your pyrazolidinone **3**. Since that is your reactant for Scheme 2, at the end of Scheme 2 you'll want to compare the spectra for **3** versus new product **6**.
2. Add 20 mL of dry anhydrous CH<sub>2</sub>Cl<sub>2</sub> and stir. (The correct bottle will have "molecular sieves" pellets to sponge up any water, and the label should say "Anhydrous" or "dry".)

3. Add your septum to exclude air.
  - It should still have a syringe needle stabbed through it to release any pressure buildup.
  - Feel free to take the septum out while adding solids, but try to return it right away to exclude oxygen
4. Add 18 mmol of your carboxylic acid **5**, probably through a powder funnel assuming it's a solid.
  - Note: we're adding less than 20mmol because we're guessing not all of the 20mmol of limiting reactant in Scheme 1 converted perfectly. I'm thinking the workup and purification will be easier if the acid is limiting rather than excess. (But, who knows, I'm guessing! It's research! ☺☺)
5. Then add 1 mmol of dimethylaminopyridine (122.2 g/mol). This is a catalyst; exact amount isn't crucial.
6. Weigh and add 22 mmol of 2-chloro-1-methylpyridinium iodide (Mukaiyama's Reagent, 255.6 g/mol).
  - Immediately reclose the bottle from which you took the reagent, since it is moisture sensitive. If you leave it uncovered, it will go bad and subsequent users will have problems.
  - Mukayama's Reagent functions to stimulate loss of H-OH from reactants **3** and **5**.
  - The "excess" is to absorb any trace water on the glassware, from the air, or in the solvent.
7. Record all of your observations. (Is the mixture homogeneous or heterogeneous, etc.)
8. Syringe in 44 mmol of triethylamine (139 mL/mol), and attach bottle cap.
  - The NEt<sub>3</sub> will get the reaction started.
  - Record observations. What happens? Exotherm or anything? Color change? Solubility changes?
9. Let stir for at least ten minutes, and record observations. After ten minutes, remove the venting needle, but leave the septum in so that there will be no air exposure over the week.
10. Label your flask, and let it stir till next week!

• **THIS IS AN IDEAL PLACE TO STOP. MAKE SURE THERE IS A SEPTUM STOPPING YOUR FLASK.**

- Emergency Note: If you don't get this far during week two, be sure that you come in and get the reaction set up at least a day before your lab period.



**Week 2, Scheme 2, Part Two: Workup/Isolation**

11. Add 0.1 mL of water (The purpose is to destroy any excess, unreacted Mukayama reagent)
12. Stir vigorously for 5 minutes.
13. Add 60 mL of ether (possible labeled as either “ether” or “diethyl ether”)
14. Stir vigorously for 5 minutes.
15. Prepare a clean pre-weighed 250-ml ground-glass flask with a long stir-bar
16. Weigh the flask + stir-bar.
  - Your final yield will be determined by subtracting this mass from the flask+stir-bar+product mass.
17. Find your fritted filter column and attach it to your flask.
  - Make sure that your flask is clamped, and within reach of a vacuum hose.
18. Weigh out 25g of silica in a large boat, and then pour it into the filter funnel. Level the surface.
19. Carefully add about 10-15 grams of sand to the top. (To protect the flat surface of the silica.)
20. Carefully drain the organic solution onto the filter column, evenly so that the surface of the column doesn't get all pitted. (Pitting may compromise the effective length/function.)
21. Carefully/gradually open up the vacuum so that liquid gets pulled through without boiling out and getting sucked into the vacuum tube.
22. Add an additional 45 mL of 3:1 ether/dichloromethane, to rinse your original reaction flask, then pour this through the silica.
  - Purpose: this extra rinse of the filter column should remove more of the desired product otherwise still absorbed on the surfaces of your flask and on the silica.
23. What is happening with this silica chromatography/filtration?
  - The silica layer is meant to adsorb as many side-products as possible, while still allowing your product to pass through the column.
  - A quick and dirty chromatography like this won't do a perfect job; probably some side-products will leach through, and possibly some desired product may remain adsorbed to the silica and lost.
  - For medicinal screening at Mayo, we are more concerned with purity than with yield. So, 40-60% yield at >90% purity is much preferred to 75% yield at 75% purity, for example.
  - It's probably likely that your product **6** won't be pure enough for direct drug testing. But this is research, so I don't know! 😊😊
    - A research assistant may do flash-chromatography to further purify for drug testing at Mayo.
  - The product **6** should normally be less polar and more mobile than most contaminating side products.
  - The amount of silica and the specific solvent is selected to hopefully allow most of the more-mobile product **6** to get through, while allowing relatively little of the side products to get through. But, it's research, so we'll see how well it does!
  - Multiple side products that we are hoping to remove via the silica column:
    - a. 1-methylpyridin-2(1H)-one (what the Mukayama reagent turns into, see mechanism figure.)
    - b. Triethylammonium chloride and iodide (the two triethylamines take one proton from the carboxylic acid **5** and one from pyrazolidinone **3**, see mechanism figure).
    - c. Dimethylaminopyridine (the catalyst).
    - d. Unreacted starting material **3**, if any? (If your 20-mmol “Scheme 1” went really great, then maybe you had more mmol of pyrazolidinone **3** than the 18 mmol of carboxylic acid **4** that you added? If so, it would be nice if any trace amounts of unreacted **3** would just stick on the silica, rather than washing off and contaminating your product **6**!)
    - e. Unreacted starting carboxylic acid **5**, if any? (If your 20-mmol “Scheme 1” went not-so-great, maybe you produced less mmol of pyrazolidinone **3** than the 18 mmol of carboxylic acid **4** that you added? If so, it would be nice if any trace amounts of excess carboxylic acid **4** would just stick on the silica, rather than washing off and contaminating your product **6**!)
    - f. Other unknown side products, that were either produced in Scheme 2, or were produced in Scheme 1 but just carried along contaminating the Scheme 2 mixture!
  - As you might guess, expecting the silica to selectively bind ALL of these various side chemicals, while retaining NONE of the desired product **6**, seems somewhat unlikely!

24. **Concentrate this solution.** Attach a reflux condenser with no water flow, and with a vacuum adaptor on top. While stirring and with no heat turned on, slowly/carefully open the vacuum. Things may bubble a lot at first. Crack open the vacuum as aggressively as you can get away with without causing the mixture to foam over.
    - Within about 2 minutes you should be able to get the vacuum fully opened.
    - Notice the condensation (and perhaps ice) that forms on the outside of the flask.
  25. Once you've been able to safely open the vacuum fully, turn the hot plate on at a setting of 5, and heat/boil/vacuum the mixture while continuing to stir (setting 6?) for 15 minutes.
    - Try to wipe off the frost from the walls as early as possible.
    - The mixture should be pretty thick and concentrated by the end, with limited bubbling.
    - In some cases, the material will foam up like cotton candy or taffy. With continued heating, though, usually the entrapped solvent does escape, and the material collapses back to a thick paste.
  26. After the vacuum heating, turn off your vacuum first, then turn off the heat, remove the flask from the heat, detach the vacuum hose, and remove the condenser.
  27. **NMR-Sample Preparation:** Immediately, while the mixture is still hot and hopefully liquid, dip in with a long-stem pipet and draw up a quarter inch of material. Immediately place the pipet into an NMR tube, and put the septum back into the flask. The material may harden/freeze as soon as it cools.
  28. Add 1.2 mL of  $\text{CDCl}_3$  as solvent to your NMR sample/pipet. **\*\*IF\*\*** the material has hardened and the solvent doesn't run through the pipet, call the instructor over and we will use a heat gun to re-melt the sample and allow the solvent to flow into the NMR tube.
  1. **GC Prep:** Using the same long-stemmed pipet, draw out what solution it can reach from your NMR tube, and transfer it directly into a GC-MS vial.
    - There will still be enough solution for the NMR.
    - Basically the same solution will feed both NMR and GC-MS analyses
  2. **Submit the NMR sample to the NMR queue.**
    - Students will be using the NMR both for submitting, but also for processing (printing extra copies, doing horizontal expansions, etc.) So you may need to be able to work your way between "submit" and "spectrometer" modes.
      - If not in submit mode, click "New Study" to get into submit mode. The correct experiment should automatically load when you do so, if the software is working right.
  3. **Submit your GC-MS sample to the GC-MS queue.**
    - This will probably take a while to run.
  4. **Mass measurement and % yield determination.** Take your flask to the balance and measure the mass.
    - Take the septum out before recording the mass; replace it immediately after recording the mass.
    - Subtract the original mass of the flask and stir bar in order to determine the mass of product.
    - Record the mass of product.
  5. Calculate the percent yield of your Scheme 2 reaction (**3 → 6**)
    - **If your yield exceeds 100%, it probably means your heat/vacuum process had issues. See instructor, in that case.**
    - If all of the solvent has been effectively removed, yields may likely fall in the 40-60% range?
    - For medicinal screening at Mayo, we are more concerned with purity than with yield. So, 40-60% yield at >90% purity is much preferred to 75% yield at 75% purity.
      - We know we're leaving some product yield on the column; but it's difficult to get it all off without also washing off increasing (and undesired) quantities of the Mukayama pyridine.
      - So, while we could increase the yield, doing so would compromise purity.
      - **\*\*IF\*\*** you wanted, you might try using an additional 50 mL 3:1 ether/dichloromethane to rinse the column into a different pre-weighed ground glass flask; then concentrate and analyze to see how much mass you recover, and how the purity compares to the original fraction?
-

**Scheme 3 Part Three: Cleaning Up and Collecting Products into the Collection Jars**

1. Add 10 mL of dichloromethane to your flask, and try to dissolve up all of your product **6**.
  - If that doesn't succeed, try heating the mixture on a hot plate for a few minutes to facilitate solubility, and/or perhaps add an additional 5mL of dichloromethane.
2. Once the product is dissolved, simply pour the solution into an appropriately labeled collection vial.
  - Make sure you're putting your stuff into the correct vial and that is labeled well.
3. IF POSSIBLE, AND IF YOU'VE GOTTEN AN NMR FOR YOUR PRODUCT, SHOW IT TO THE INSTRUCTOR BEFORE PUTTING YOUR PRODUCT INTO THE COLLECTION VIAL.
4. BE SURE THAT YOU'RE PUTTING YOUR PRODUCT INTO THE CORRECT CONTAINER. WE DON'T WANT ANY **6ba** GOING INTO A **6aa** CONTAINER, ETC.

**Silica Waste handling:** all of the solid silica and sodium sulfate in your filter column should go into a solid-waste container in the hood. Bulk may NOT go into the sink or garbage. (Trace amounts stuck on wall may.)

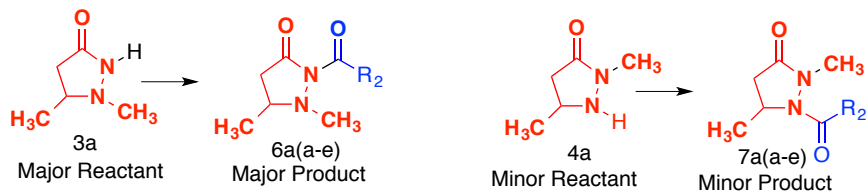
## NMR and GC-MS Data in the Research Module.

### NMR Analysis/Interpretation, General Considerations,

### NMR Analysis/Interpretation, General Considerations,

- An **abbreviated summary report** process will be required. Draw the structure and label the different carbons. Then make a table with the actual chemical shifts, integrations, and splittings for the actual spectra, and by each one write the letter of the carbon to which it is attached. This will demonstrate that you have analyzed and understand your spectrum. The two alpha-hydrogens will have difference chemical shifts
  - Include integration.
  - Do not analyze/report the splitting (although you may do so.)
  - Which signal is from the  $\beta$ -H, and which two signals are from the  $\alpha$ -H's?
  - Does your product look pretty pure? If so, there should be a fairly limited number of contaminant signal sets.
- Scheme 2: Does your NMR confirm that your heat-vacuum process removed the solvent? Does it look like your product is relatively clean, or is there a lot of junk present? Does it confirm that you starting material 3 converted to new product 6?**
- You should be able to recognize the presence of residual solvents.  $\text{CH}_2\text{Cl}_2$  gives a singlet at  $\sim 5.28$  ppm; diethyl ether gives a quartet in the low-to-mid 3's, and a triplet in the low 1's.
- If you compare your NMR for 2<sup>nd</sup>-week product **6** versus for reactant **3**, you should be able to see whether reactant **3** is gone or is still present at the end of the reaction.
- Chirality, H-non-equivalency, and chemical shift:** In your NMR for both week-one product **3** and after week-two product **6**, the  $\beta$ -carbon will be chiral. When you have a chiral carbon, it makes the two  $\alpha$ -hydrogens (on the  $\text{CH}_2$  group next to the carbonyl) unequal to each other. One  $\alpha$ -H is cis and the other is trans to your  $\beta$ -H, so they are not in the same chemical environment. These unequal  $\alpha$ -hydrogens usually (not always) come at significantly different chemical shifts, but both should appear in the 2's. The  $\beta$ -H, which is attached to a nitrogen-bearing carbon, should be further downfield, probably in the 3's. A video talking through some of this is available.
- Chirality, H-non-equivalency, and splitting:** The non-equivalence of the two  $\alpha$ -hydrogens also complicates the splitting. They now are split by each other, as well as by the neighboring  $\beta$ -H. Plus the splitting magnitudes are different because of the differing distances. (The "other"  $\alpha$ -H is closer than the  $\beta$ -H, so they don't usually provide equivalent magnetic splitting, and don't usually provide a nice triplet.) In practice, each of the two  $\alpha$ -hydrogens will usually appear as a four-line "doublet of doublets", and the two  $\alpha$ -hydrogens should normally combine to show eight lines. The  $\beta$ -H will typically look much more highly split, being split by each of the two  $\alpha$ -H's, plus by the methyl. A video talking through some of this is available.
- N-H hydrogens**, like OH hydrogens, are typically broad and unpredictable. In many cases you won't be able to see them at all. **Don't look for them or try to assign them**
- Signature signals:** All of the samples will have the interesting  $\alpha$ - and  $\beta$ -hydrogens. But there will be other signature peaks for other situations:
  - N-methyl singlets for all samples **6**.
  - A nice methyl doublet in the 1's for all samples **6**, for the C5-methyl attached to the  $\beta$ -carbon.
  - **6aa** should have a nice methyl attached to the new benzene, plus two aryl doublets
  - **6ab** should have a nice allylic methyl doublet, plus two alkene signals.
  - **6ad** should have a nice methoxy methyl singlet, and two aryl doublets.
  - **6ae** should have a some aryl H's, plus two alkene doubles that are probably down in the 7's.

9. **Nitrogen impact on chemical shift:** The impact of a nitrogen attachment on chemical shifts is somewhat similar to but weaker than the impact of an oxygen attachment. In other words it has an addition factor of about +1.5-2. This will impact the chemical shift for hydrogen on the  $\beta$ -carbon ( $\beta$  relative to the carbonyl) and the N-methyl signal. Typically the N-methyl should appear somewhere in the 2.6-3.3 range. The hydrogen on the  $\beta$ -carbon will probably show up somewhere in the 3's.
10. **Chemical shift logic:**
- For the  $\alpha$ -hydrogens in **6**, they are next to a carbonyl. So we'd expect them to come in the 2's. They are also  $\beta$  to nitrogen, which further pushes one or both of them variably downfield within the 2's or perhaps even into the low 3's.
  - For the  $\beta$ -hydrogen, the  $\beta$ -carbon has a nitrogen (+1.5-2) so should appear in the 3's.
11. **Impurities/contamination:** Real products often are accompanied by many impurities. The present of impurities, can complicate NMR interpretation. Recognizing which signals come from the desired product and which do not is significant. And qualitatively recognizing whether a spectrum is relatively clean or is pretty contaminated is important.
- Remember that there should be a logical integration ratio for the main H's in your actual product **6**.
  - Often there will be a variable amount of smaller signals in the baseline resulting from contaminants, side products, and product-decomposition. The less, the better.
12. **Isomer ratio:** Two structural isomers will normally form, the "major" isomer **6** derived from reactant **3a**, but also a "minor" isomer **7** derived from the structural isomer **4a** that was mixed in with your reactant **3a**. The minor isomer will be more easy to recognize in the GC, but there should also be a set of minor signals in the NMR. The product ratio should be reflected in their integrals: in other words, if the isomer ratio is 5:1, then the N-methyl signal for **7** should be only 1/5 as big as the N-methyl for **6**; and the  $\beta$ -hydrogen for **7** should be only 1/5 as big as the  $\beta$ -hydrogen for **6**, etc.



### GC-MS Analysis/Interpretation

13. Draw the structure for your specific product on each GC-MS sheet, and write the molecular weight underneath the picture. (No "R" groups; write the specific structures.)
14. **Retention time?** What is the retention time for your **6**?
- Bigger structures will have longer retention times. Product **6** should have a longer retention time than product **3**.
15. **Purity:** How pure is your **6** by GC?
- Many contaminants will NOT appear, since they come off fast during the solvent delay. (Certainly true for any solvents.). So your purity reading will be deceptively high. NMR, which shows everything, is qualitatively more representative.
16. **Isomer ratio:** What is the ratio of the "major" isomer **6** versus the "minor" isomer **7**?
17. **Mass Spec and Molecular Ion:** Check in the mass-spec whether there is a molecular ion peak that matches the molecular weight for your product.
18. **Lab report:** In your lab report, make sure that you have not only attached the labeled GC-MS information, but that you also discuss/present the retention time and purity in your data/results/discussion section.

## Lab Report for Research-Module Scheme 2 Lab Report: Pandemic-Modified

1. You will do theoretical yield, % yield, GC, and NMR analysis on one of the five products **6**. Your specific reaction-analysis assignment is based on the starting letter of your last name:

### Student Last Name Starting Letters:

- A-B (if your last names starts with an A or a B): Sample **6aa**
- C-F (if your last names starts with a C, D, E, or F): Sample **6ab**
- G-K (if your last names starts with a G, H, I, J, or K): Sample **6ac**
- L-O (if your last names starts with an L, M, N, or O): Sample **6ad**
- P-Z (if your last names starts with a P or later letter): Sample **6ae**

- Copy the written procedure for Scheme 1 reaction (**3** → **6**).
- Insert observations as they occur; or any changes in procedure. (A different font or a different color, so your inserts stand out from copied text, would be nice! ☺)
- Insert listing of chemicals used; show your mole calculations; identify limiting reactant; and show your theoretical yield calculation. (As per normal report.)
- Note: make sure your figures are specific to your actual carboxylic acid reactant and actual product **6**. (In other words, no generic "R"s are allowed in your pictures! ☺)
- Include final mass and % yield calculation.
- Make sure that all structures are drawn explicitly.
  - As always for a synthesis style report, you'll want to draw out the reactants and the products. In this case, be sure you draw the **actual** reactant **3a** in your reaction, your actual carboxylic acid reactant, and actual product **6**. (In other words, no generic "R"s are allowed in your pictures! ☺ None of your pictures should have an "R2": you should illustrate each structure with your actual R2 group drawn, whether that's phenyl or 4-methoxyphenyl or whatever.
- Show all calculations. (Including any mole ⇒ mass for reactants, or mass ⇒ mole for products)
- Calculate mass yields, and percent yields, etc., for product **6**.
- 10. Include your NMR-6**
  - Be sure to draw your structure, and then provide an abbreviated summary report. This should include a listing of chemical shifts, integrations, and splittings, and a matchup-assignment between signals and hydrogens in the molecule.
  - **Note: you do not need to include impurities/solvents/contaminants in the abbreviated NMR summary report.**
- Include your GC-MS-**6**, depending on what Dr. Jasperse provides you.
- 12. Include a results/data/discussion/analysis section.** The analysis/discussion section needs to address what the yield information told you, and what the NMR and GC-MS data tells you about both the success and the efficiency of your reaction, and the purity of your product **6**.
- The results/data/discussion/analysis section should summarize what the mass/yield/NMR/GC-MS data is, and what conclusions can be drawn from them. Just attaching the NMR's and GC-MS's without discussing or showing that you understand them will not be good. What is the summary for the key non-aromatic C-H hydrogens in your NMR? What is your GC-retention time? Between the NMR and the GC, did it look like the product **6** was formed successfully, and does it look reasonably clean? Or is it obviously significantly contaminated?
- Answer the post-lab questions on the following two pages, and include in your report.

### Research Module

Student Last Name Starts with Letters:	Carboxylic Acid Used	Product Produced	Yield of Product	NMR + GC Data URL
A-B	<b>5a</b> , p-toluic acid 136 g/mol	<b>6aa</b>	3.55 g	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Toluy1.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Toluy1.pdf</a>
C-F	<b>5b</b> , Crotonic acid 86.0 g/mol	<b>6ab</b>	2.02 g	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Crotonyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Crotonyl.pdf</a>
G-K	<b>5c</b> , Benzoic acid 122 g/mol	<b>6ac</b>	3.16 g	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Benzoyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Benzoyl.pdf</a>
L-O	<b>5d</b> , Anisic acid 152 g/mol	<b>6ad</b>	3.62 g	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Methoxy.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Methoxy.pdf</a>
P-Z	<b>5e</b> , Cinnamic acid 148 g/mol	<b>6ae</b>	3.85 g	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Cinnamoyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Cinnamoyl.pdf</a>



### **Postlab Questions for Research Module Week 2**

Some of the information that addresses these questions may have come in the written prelab; in the prelab video; or perhaps in the experiment itself or the associated video.

1. Mark which sample you analyzed

Carboxylic Acid Used	Product Produced	I analyzed this one	Student Last Name Starts with Letters:
<b>5a</b> , p-toluic acid 136 g/mol	<b>6aa</b>		A-B
<b>5b</b> , Crotonic acid 86.0 g/mol	<b>6ab</b>		C-F
<b>5c</b> , Benzoic acid 122 g/mol	<b>6ac</b>		G-K
<b>5d</b> , Anisic acid 152 g/mol	<b>6ad</b>		L-O
<b>5e</b> , Cinnamic acid 148 g/mol	<b>6ae</b>		P-Z

2. List the following **mass and yield-related information**:

- How many grams of carboxylic acid should I have used?
- What is the molecular weight of my product?
- What is the theoretical yield for my product?
- What was the actual yield for my product? (see table on previous page)
- What was the % yield for my product?

3. Provide a table summarizing your GC-MS data.

- Most of you (crotonyl product **6ab** might be accepted...) will see both the major and minor isomers **6** and **7**.
- List any other retention times for contaminants with % >1%.

	Retention Time	% of total	Molecular weight	
Major Isomer <b>6</b>				
Minor Isomer (if visible) <b>7</b>				

## NMR Analysis:

4. Draw your product **6**, and label the different hydrogens or carbons in some way.
5. This should have already been in your main lab report, but for my grading convenience, for the major product **6**, list the chemical shifts for the following signal sets in major product, which will be involved in each of the different products **6**. Note: it will not be unusual at times to have overlapping signals. Perhaps both  $\alpha$ -hydrogens will overlap; or perhaps one of the  $\alpha$ -hydrogens will overlap with the  $\beta$ -hydrogen. Depending on which version of **6** you are analyzing, you will also be plagued by varying amounts of impurity signals.
- N-methyl
  - C-methyl
  - $\beta$ -H
  - $\alpha$ -H (one of them)
  - $\alpha$ -H (the other one)
6. Depending on which version of **6** you are analyzing, like the chemical shifts and identities for whatever signals are sourced from hydrogens included in the newly introduced group attached to the carbonyl on the N2-nitrogen.

## H-NMR Data:

Student Last Name Starts with Letters:	Product Produced	NMR + GC Data URL
A-B	<b>6aa</b>	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Toluyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Toluyl.pdf</a>
C-F	<b>6ab</b>	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Crotonyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Crotonyl.pdf</a>
G-K	<b>6ac</b>	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Benzoyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Benzoyl.pdf</a>
L-O	<b>6ad</b>	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Methoxy.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Methoxy.pdf</a>
P-Z	<b>6ae</b>	<a href="http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Cinnamoyl.pdf">http://web.mnstate.edu/jasperse/Chem365/NMR-GC-MS-Cinnamoyl.pdf</a>