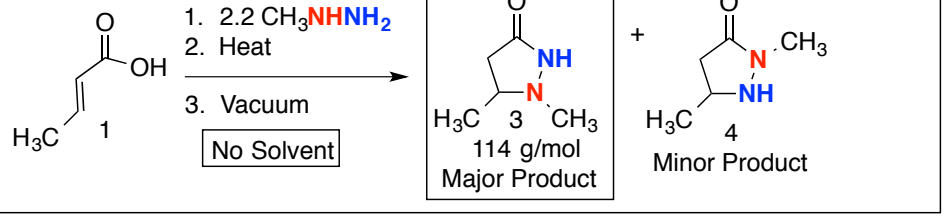
Scheme 1: Synthesis of N1,C5-Dimethyl Pyrazolidinone



- See the following page for the hypothetical mechanism.

Reagents:

1. 20.0 mmol of Crotonic acid
   - Crotonic acid: 86.0 g/mol
2. 44 mmol of Methyl Hydrazine (52.4 ml/mol)
3. 125 mL Ground-Glass Erlenmeyer flask
4. Long sized stir bar
5. Hot plate
6. Reflux Condenser
7. Vacuum Condenser and Vacuum adaptor.

Scheme 1 Procedure: Formation of Pyrazolidinone Ring Using Hydrazine

Scheme 1 Part A. Heating/Methylhydrazine Addition Phase

1. Get a 125-mL ground-glass-jointed Erlenmeyer, and add a long stir-bar. **Weigh the combination and record the mass.** Write your name(s) on the flask with your sharpie.
   - You'll need this mass to calculate your product yield, so record it somewhere!
2. Weigh out 20.0 mmols of "crotonic" (2-butenoic) acid:
   - Crotonic acid, 86.0 g/mol, is a solid. Add it using a powder funnel.
3. Add a rubber septum to the flask, and poke a syringe needle through it to vent any pressure buildup.
4. Add 44 mmol (0.044 mol) of liquid methylhydrazine (0.0524 ml/mmol) via syringe while stirring.
   - You can pull the septum out before injecting, and replace the septum following the addition.
5. Add a reflux condenser, with a gentle flow of water running through it, and place the septum-with-needle on top to prevent oxygen exposure. (Oxygen causes some oxidative decomposition.)
6. Turn your hot-plate setting to 8, and stir for an additional 30 minutes.
7. During the stirring time, prepare and run H-NMR and GC for your aryl iodide 5.
   a. If it's a liquid, use a long-stemmed pipet and draw up about ½-inch of aryl iodide into the skinny end section of the pipet; place into NMR tube; then pour in ~1.3mL of CDCl3 through the pipet.
   b. If it's a solid, just stab some into your NMR tube, and add ~1.3mL of CDCl3.
   c. Shake to mix/dissolve.
   d. Using a pipet (could be the same one you just used, if you added a liquid), reach as deep into the NMR tube as the pipet will allow, withdraw what solution the pipet can reach, and transfer that volume into a GC vial. You will still have enough solution in your NMR tube to run your NMR.
   e. As time allows, run both your NMR and GC samples.
   f. Do not include these in your Scheme 1 lab report. Include in your Scheme 2/Week 2 lab report.
8. During the stirring times, plan ahead. Prepare the following:
   a. Find your vacuum adapter, and plug it into the vacuum hose. This adapter is about 1 inch long, with a ground-glass joint on one end and a 90-degree curve. The ground-glass end will eventually plug into the reflux condenser, the tapered end into your vacuum hose.
   b. Familiarize yourself with your vacuum: With your vacuum adapter attached to the vacuum hose, put your thumb over the end, and turn the vacuum counterclockwise to get an idea of how far you have to turn it before any vacuum actually starts to work. Then turn the vacuum back off.

- You'll want to know this so that when you really need to apply the vacuum, you'll be able to get near the point where the vacuum engages, and then open it VERY cautiously and slightly.

c. Ensure that the vacuum is turned all the way off (to the right). IF SOMEBODY HAS THEIR VACUUM OPEN TO THE AIR, THAT AIR LEAK WILL COMPROMISE EVERYBODY'S VACUUM AND NOBODY WILL GET THE LOW PRESSURE THEY NEED for the next procedure. Don't let your hood's vacuum ruin everybody's experiment!

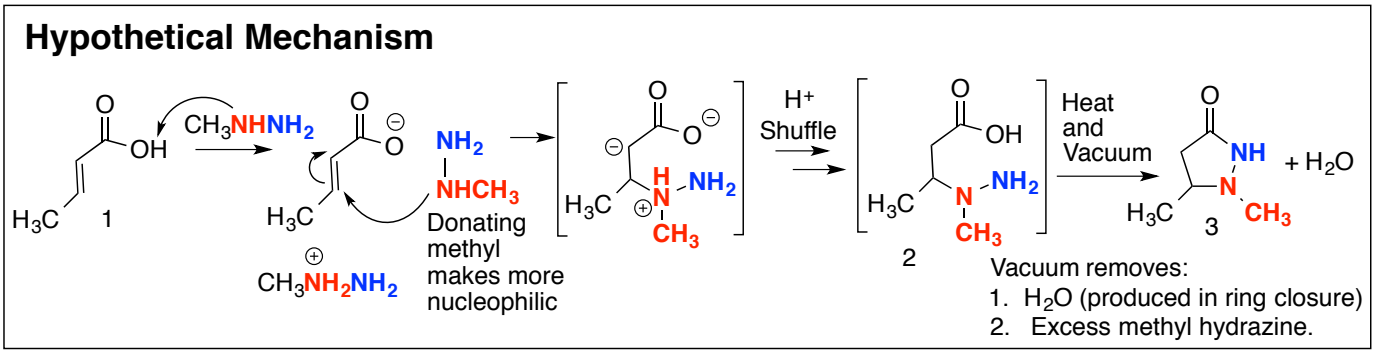
d. Calculate what the theoretical yield for your product **3** should be, given the 20.0 mmol scale.

e. Look ahead to both subsequent stages of Scheme 1, but also to Scheme 2, which you will do today and will start shortly after you complete Scheme 1. You could do all of your Scheme 2 calculations right now…

f. At this stage, you should have mostly the open form of the product with a carboxylic acid (rather than the closed ring with an amide); see the Hypothetical Mechanisms figure below).

g. At this stage you should also have, in addition to the product, at least two other chemicals:
- The excess methylhydrazine
- Water which is produced during the ring closure.
- Probably some contaminants or side products, and perhaps some unreacted acid **1**.
- The hope is to later vacuum-distill away all or most of these extra things, without decomposing the cyclic product **3** (and side product **4)** in the process.

9. After the heating is complete, a) slide the hot plate out from under your flask, b) **reduce the hot-plate setting to 5**, c) turn off your reflux condenser water, and d) detach the reflux-condenser hose from the green water-source nozzle and redirect it into the drain, so that water in the condenser can drain out.
10. Let your solution (and the hot-plate) cool for ≥ **5 minutes** before starting Scheme 1 Part B.



Hypothetical Mechanism and discussion (for your interest): The probable mechanism involves an initial proton transfer from the acid to the methylhydrazine. Amines are basic enough to rapidly and completely deprotonate acids. Amines are also modestly nucleophilic; upon extreme heating, the amine is able to add to the alkene, to produce a resonance-stabilized enolate. Without the resonance stabilization this would not be possible. It would also not be possible without high heat: in the procedure, we use a hot-plate setting of 8, which heats the mixture to ~200ºC. The nitrogen addition is irreversible; as soon as the enolate ion forms, it probably gets protonated immediately. Structure **2** in the figure is drawn in neutral form; that isn't really accurate, because it would mostly exist in an ammonium carboxylate ionic form. But picture **2** makes it easier to visualize that ring formation (**2**=>**3**) releases a water molecule. The ring formation again requires high heat, but it also requires vacuum for the removal of the water as it forms.

Excess methylhydrazine is needed because the first equivalent just deprotonates the acid, and the protonated methylhydrazine would not be nucleophilic. Using 2.2 methylhydrazines ensures that there is always neutral nucleophilic methyl hydrazine available.

When the Jasperse group first attempted this reaction, I doubted it would be effective. I thought the blue NH2 nitrogen might add preferentially for steric reasons, or else that the blue and red nitrogens would add at similar rates and give a bad mixture of structural isomers **3** and **4**. We now believe that the electron-donating effect of the methyl group makes the methylated nitrogen more electron rich and nucleophilic, resulting in useable selectivity for desired product **3** over structural isomer **4**.

Scheme 1 Part B: Heat/Vacuum/Ring-Closure/Amide Formation Phase

11. After the 5-minutes, attach the vacuum adapter to your reflux condenser, and then slide your hot plate back under the reaction flask. The hot plate should be set at 5 for heat. (And maybe 3-5 for stirring?)
    - If you didn't turn your hot plate down to 5 earlier, do so now and wait five minutes.
12. Crack the vacuum open, really, really carefully and gently at first (so that it doesn't cause everything to erupt and boil/foam over). As soon as the vacuum is engaged but the bubbling isn't too wild, open the vacuum until it's wide open as soon as possible (two full revolutions will more than suffice). If the mixture splatters/bubbles a lot, it may help to lower the plate slightly.
    - **If instructor is available, it may be reassuring to request help on getting the vacuum opened**.
13. Stir/heat/vacuum for **25 minutes**. (Measure time from when the vacuum was first two-rotations open.)
    - Excess vacuum heating may result in some decomposition, so avoid excess heat and time.
    - Notice that the volume should decrease as non-product chemicals boil off with the assistance of the vacuum.
    - The hot vacuum is intended to do several things:
        a. During this time, the excess methylhydrazine, and the water produced during ring closure, should ideally boil away, leaving only the product and whatever organic contaminants and side-products remain.
        b. Each of the chemicals that get distilled away inhibits the ring formation. So, their distillative removal, in addition to being an end in itself, also enables the ring closure to proceed more effectively.
14. After the 25 minutes, slide the hot plate out from under your reaction mixture.
15. After the flask cools a little, **turn the vacuum off**, and detach the vacuum adapter.
    - **NOTE: If you forget to turn your vacuum off, it will ruin things for everybody else because their vacuum won't work well if yours is left open to the air.**
    - Pulling the vacuum adapter is easier if you grasp the hot flask with a GLOVED hand.
    - Remove the reflux condenser and replace with the septum to reduce oxygen exposure.

Critical Note: Be sure to start the next reaction as described in Scheme 2 before week one is done.

- Before week one is completed, it is urgent that you get the subsequent reaction started. See Scheme 2.
- This reaction takes at least several hours after it is begun, so you don't want to be trying to both start and finish it, and work it up, and purify it, and run GC and NMR to analyze it, all during the same lab period!
- So, if it has the whole week to complete itself, that will work out great and will allow you to waste no time next week!
- The workup will be time-consuming workup. So, I'd like to have next week completely available for that.
- If you don't get it started during the initial lab period, you will want to/need to come in sometime at least a day before the next lab period to get it started.
- **Note: The first lab report that is due next week should include only the chemistry included in Scheme 1. Even though you began the Scheme 2 Chemistry today, during week one, include ALL of the scheme 2 chemistry in a lab report that will be due the week following completion of Scheme 2 and its workup.**

Scheme 1 Part C: Product Analysis. NMR, GC/MS, and yield. And Deciding whether You Need to Heat and Vacuum Further before Starting Scheme 2.

- Hopefully everything has gone reasonably well. However, this isn't certain; perhaps the ring closure and water/hydrazine removal is not sufficiently complete, in which case you'll need to do some more vacuuming. (And in which case the yield will be too high, and the NMR will be contaminated.)
- We don't want to do additional heat/vacuum time unless absolutely necessary, though, because doing so can cause some decomposition of your product.
- To decide whether you need additional heating/vacuuming, you need a quick way to assess the situation. Both the NMR and/or the % yield can tell us. If the yield significantly exceeds 100%, that represents trouble. And if the NMR shows more complexity than expected, that too means trouble.
- You'll need both for your lab report, but either will be enough to let you get started on Scheme 2.

16. **NMR and GC analysis:** dip a long-stemmed pipet into the product and draw up about a quarter-inch (1 cm) sample (finger-nail length) of your hot mixture into the skinny tip of your pipet
    - Sample should be small, both to give sharpest NMR and to minimize yield loss.
17. Put your pipet with that small sample into an NMR tube.
18. Put a septum into the top of your reaction flask to keep air out.
    - Air can cause some oxidation and decomposition of your product, particularly when it's hot.
19. Add ~1.3 mL of $CDCl_3$ into the same pipet that has the sample in it and is in the NMR tube, and attach a pipet bulb to the pipet.
20. **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
21. **Sign up for NMR turn, and run your NMR sample**.
    - Note: Students will be using the NMR both for submitting, but also for processing (printing extra copies, doing horizontal expansions, etc.) So, you will 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. After submitting, cancel out.
22. **Submit your GC-MS sample to the GC-MS queue**.
    - This will probably take a while to run.
23. **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; you'll need it both for this week's lab report (as the product) and for next week's lab report (as the Scheme 2 reactant).
24. Calculate the percent yield of your Scheme 1 reaction (**1 → 3**)
    - **If your yield exceeds 100%, it probably means your heat/vacuum process had problems. See instructor, in that case.**
25. Calculate how many mmol of product you have.
26. If you have the NMR, or GC, or you have the mass and use that to calculate the % yield, those will provide the information needed to determine whether or not you're good to proceed with Scheme 2, or whether you need more vacuum. Consult instructor?
27. **Make an extra copy of both your NMR and your GC and put the extras in your drawer**. You'll need them for your lab report for this week, but you'll also need them for your lab report next week! ☺
    - Today's product will be Scheme 2's reactant. As you go through Scheme 2, you'll want to be able to compare your NMR and GC for your Scheme 2 product versus those of the reactants.
        - NMR: If in submit mode, click "**Cancel**" to exit Submit mode in order to enter the spectrometer/processing mode for doing additional prints or horizontal expansions.
        - In "Spectrometer" mode, must have "Zones" map displayed (96 sample nodes show). Click on little circle icon () to the upper left of the spectra-display panel, if zones map not already open.

Scheme 1/Week 1 Lab Report:

1. Write a standard synthesis style lab report for your Scheme 1 reaction (**1** → **3**).
2. Show all calculations. (Including any mole => mass for reactants, or mass => mole for products)
3. Include procedural details and observations as usual.
4. Calculate mass yields, and percent yields, etc., for product **3**.
5. Include your NMR-**3**
   - 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**.
6. Include a note about your approximate ratio of the major product **3** to the minor product **4**. Produ
7. Include your GC-MS-3, and **print and attach mass spectra**.
8. In the experiment, what was the approximate ratio of major product 3a to minor product 4a, based on GC data?
9. In the GC, what was the retention time for the major isomer, versus the minor isomer?
10. Given the major/minor **3/4** ratio measured by GC, can you see evidence in the H-NMR for a similar mixture of major-to-minor isomers?
11. What are the chemical shifts for the N-methyl signal from **3**, versus the N-methyl signal in **4**?
12. Explain very briefly why the two α-Hydrogens in the NMR have different chemical shifts, despite being connected to exactly the same carbon?
13. 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 **3**.
14. This should have already been in your main lab report, but for my grading convenience, for the major product **3**, list the chemicals shifts for the following signal sets in major product 3a:
    - N-methyl:
    - C-methyl.
    - β-H:
    - α-H (one of them):
    - α-H (the other one):
15. 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 **3** was formed successfully, and does it look reasonably clean? Or is it obviously significantly contaminated?
16. Note: Keep extra copies of your NMR and your GC-MS's. Pyrazolidinone **3** functions as the product in week one report, but it is the reactant in the week two report. So, when writing up and analyzing Scheme 2, you'll need information about mass, molecular weight, structure, and mmol of your reactant **3**. You'll also need to have NMR and GC for **3** so that you'll be able to compare your product **6** to reactant **3** and tell whether the reaction really worked.
17. See following page for some considerations that will help to understand and process the NMR and GC.

NMR Analysis/Interpretation, General Considerations,

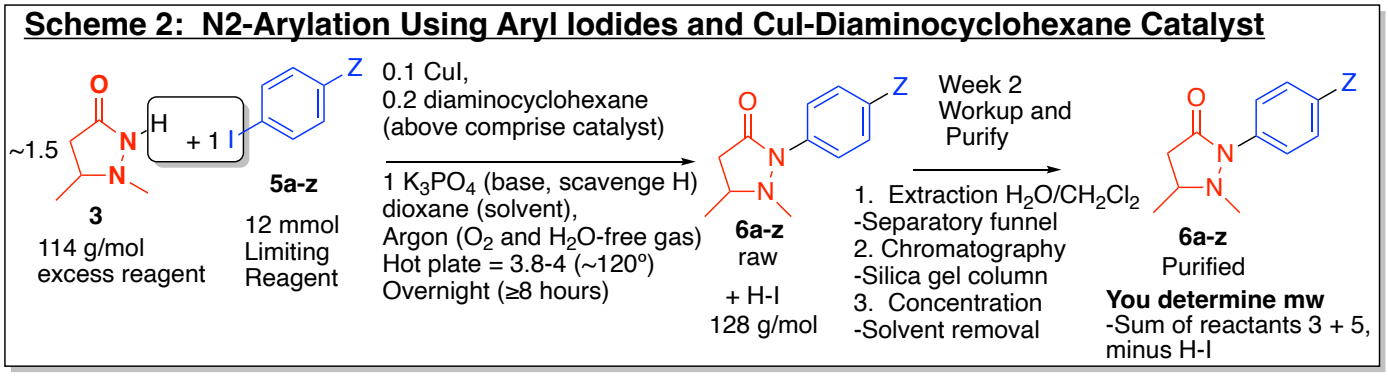
1. An **abbreviated standard summary report** will be required for 1H NMR. Draw the structure and label the different carbons. Then make a table with the actual chemical shifts, integrations, and splittings for the actual spectra, and by each one write the letter of the carbon to which hydrogens are attached. This will demonstrate that you have analyzed and understand your spectra. The two alpha-hydrogens will have difference chemical shifts.
2. **N-H hydrogens**, like OH hydrogens, are typically broad and unpredictable, and probably invisible for product **3**. Don't look for them or try to assign them in product **3**.
3. **Chirality, H-non-equivalency, and chemical shift**: In your NMR for both products **3** and **6**, the β-carbon will be chiral. When you have a chiral carbon, it makes the two α-hydrogens (on the CH2 group next to the carbonyl) unequal to each other. One α-H is cis and the other is trans to your β-H, so they are not in the same chemical environment. These unequal α-hydrogens usually come at significantly different chemical shifts, but both should appear in the 2's. The β-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.
4. **Chirality, H-non-equivalency, and splitting**: The non-equivalence of the two α-hydrogens complicates the splitting. They now are split by each other, as well as by the neighboring β-H. Plus the splitting magnitudes are different because of the differing distances. (The "other" α-H is closer than the β-H, so they don't usually provide equivalent magnetic splitting, and don't usually provide a nice triplet.) In practice, each of the two α-hydrogens will usually appear as a four-line "doublet of doublets", and the two α-hydrogens should normally combine to show eight lines. The β-H will typically look much more highly split, being split by each of the two α-H's, plus by the methyl group**.** A video talking through some of this is available.
5. **Signature signals**: Sample **3** should have a nice 3H methyl doublet in the 1's, and a nice 3H N-methyl singlet in the 2's.
6. **Nitrogen impact on chemical shift**: The impact of a nitrogen attachment on chemical shifts is 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 β-carbon (β 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 β-carbon will probably show up somewhere in the 3's.
7. **Chemical shift logic**:
   - For the α-hydrogens in each of **3** and **6**, they are next to a carbonyl. So we'd expect them to come in the 2's. They are also β to nitrogen, which further pushes them a bit downfield. So typically they should fall in the high 2's or perhaps slip into the low 3's.
   - For the β-hydrogen, the β-carbon has a nitrogen (+1.5-2) so should appear in the 3's.
8. **Impurities/contamination**: Real products often are accompanied by many impurities. The present of impurities, can complicate NMR interpretation.

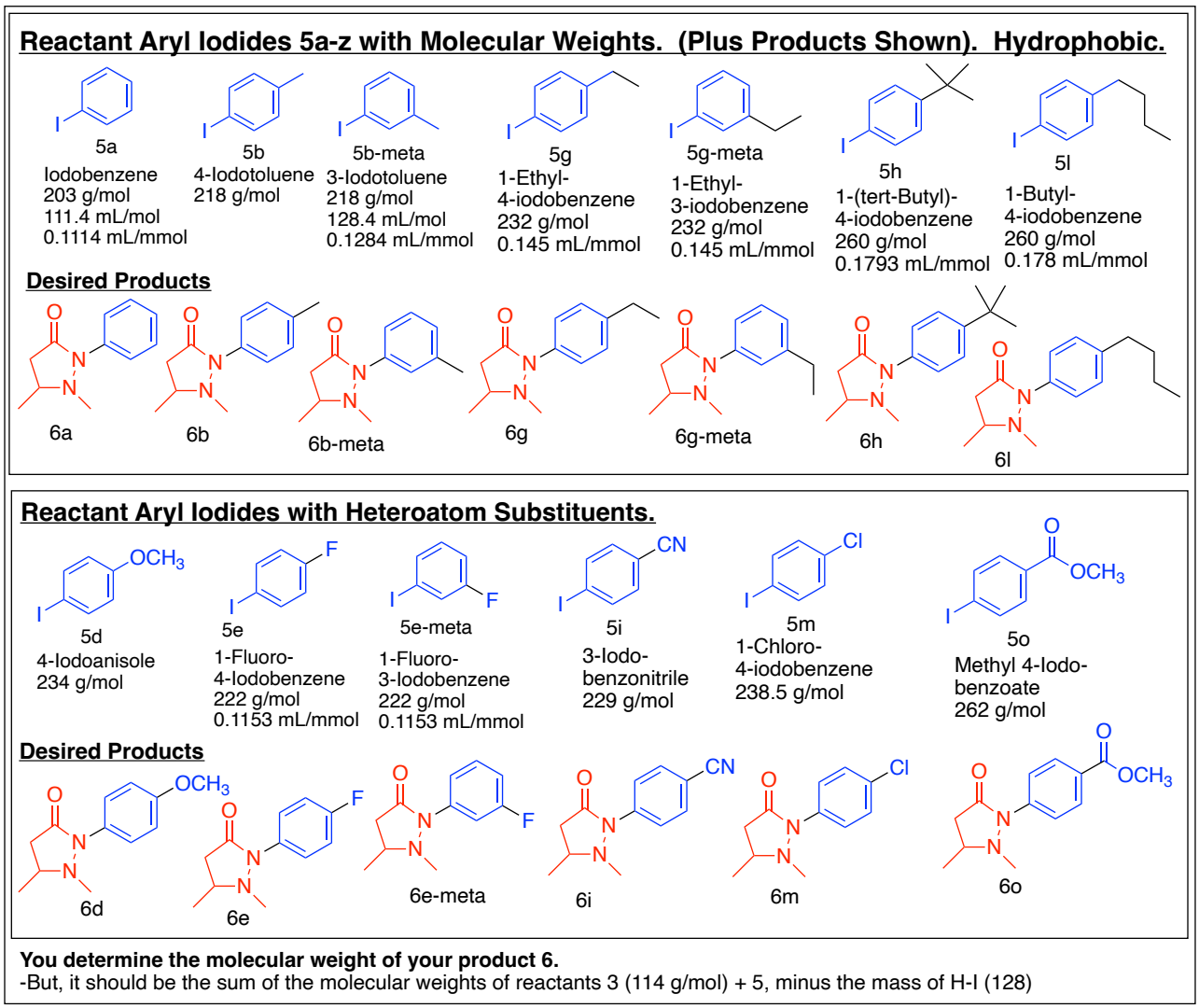
GC-MS Analysis/Interpretation

9. Draw the structure for your product on the GC-MS sheet, and write the molecular weight underneath.
10. **Retention time**? What is the retention time for your **3**? (It may be really broad, but you should still be able to get a general idea).
11. **Purity:** How pure is your **3** by GC?
    - Often nitrogen-containing molecules, because of their basicity, get badly broadened on our GC column. So we'll have to see whether the GC's look useful for **3 .**
12. **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. Both the major product **3** and the minor product **4** should weigh the same 114.
13. **Lab report**: In your lab report, make sure that you have not only attached the labeled GC-MS information, but that you also present the retention time and purity in your data/results/discussion section.

Scheme 2: N2-Arylation using Aryl Iodides and CuI-Diaminocyclohexane Catalyst

- Note: The next two pages are done week one, but should be included in Scheme Two lab report.





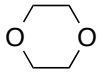
Reagents:

1. Pyrazolidinone **3**: Report how many grams and mmoles you actually produced at the end of Scheme. But it should be ≤20 mmol.
2. CuI: 2 mmol x 190 = 380mg
3. K3PO4: 20 mmol x 0.212 g/mmol = 4.2 g
4. Aryl iodide: 12 mmol. (you figure out how much of yours you need to add!
5. Dioxane (anhydrous): 20 mL
6. Diaminocyclohexane: 4 mmol x 0.120 mL/mmol = 0.48 mL
   - add only after air/argon gas replacement is completed
7. Argon atmosphere: Ask instructor for help with this.

Procedure:

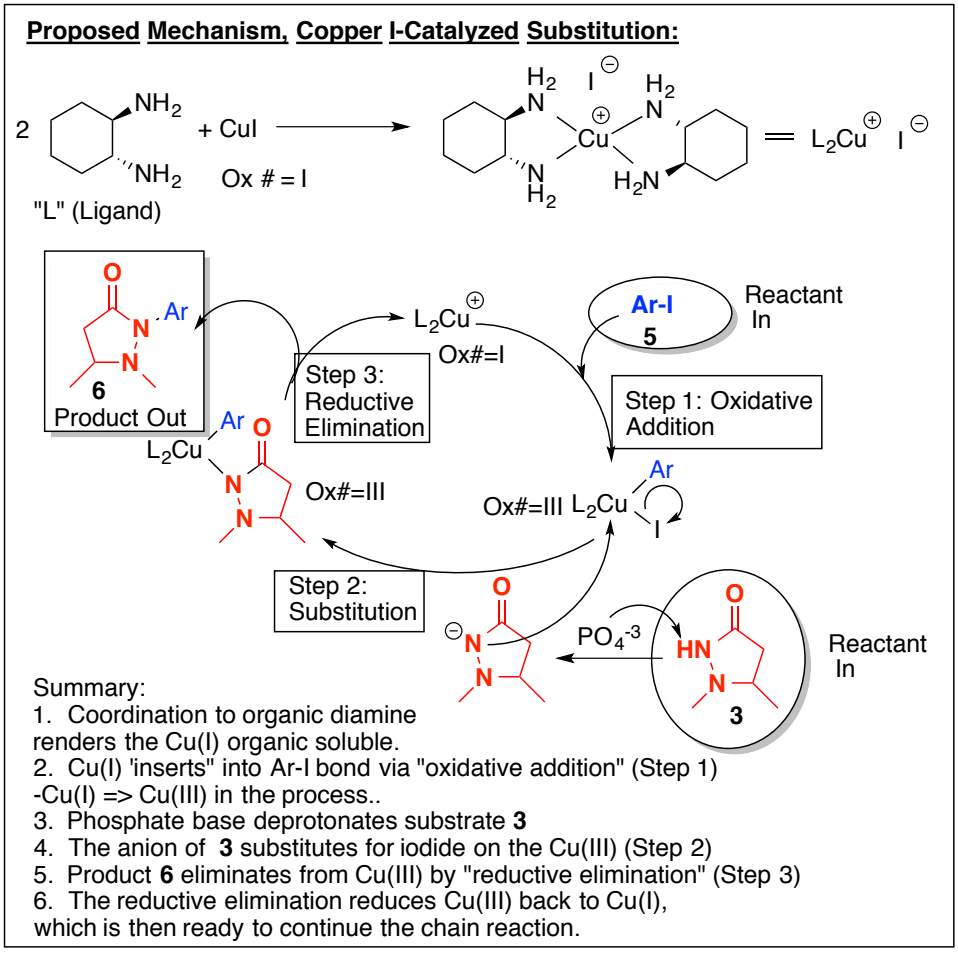
Part 1: Starting the Reaction for N-Arylation

1. Carry your flask with your reagent **3** in it, and the septum, to the balance, to weigh in any solids. Avoid air exposure, so pop the septum back in after each addition.
2. Add Copper iodide: 2 mmol x 190 = 380mg
3. Add Potasssium phosphate: 20 mmol x 0.212 g/mmol = 4.2
4. Add 12.0 mmol of your aryl iodide, whether it's a solid or a liquid.
   - Details for each one are listed on previous page.
   - For a solid, the molecular weight is used to determine how much mass to add.
   - For a liquid, use the mL/mmol value to calculate how much to add.
   - In either case, calculate for 12 mmol.
   - If uncertain, check with Dr. Jasperse.
5. Add ~20 mL of anhydrous dioxane.
   - The measurement doesn't need to be precise.
   - Get within 2 mL of 20mL, but better to act quickly than to be super precise! ☺
   - The dioxane is air/moisture sensitive. Because it a cyclic ether, it hydrogen-bonds to water, so moisture from the air can dissolve in and contaminate the solvent.
   - We want it to stay as dry as possible for future users, so screw the cap back onto the dioxane bottle first, as soon as you've poured your 20mL out, before pouring your dioxane into your flask.
   - Then pour your 20-mL into your flask, and put the septum back in to exclude further air exposure.



Dioxane
1. Ether-type dissolving properties (only better)
2. Larger, so higher boiling point
   => hotter + faster reactions, but not too high-boiling to distill away at the end
3. Symmetry gives it a very simple NMR, so residual dioxane doesn't confuse spectra much

6. Purge three times with argon. The instructor will do this for you, at the purging station. (Where that is will be announced.)
   - The goal is to replace the air in your flask with inert argon gas, which has no water and no oxygen in it.
   - Residual oxygen is otherwise able to oxidize and destroy the Cu(I)-oxidation-state catalyst. If all of your catalyst gets destroyed, the catalytic reaction will fail!
   - The instructor will execute the gas purge for you, but make sure both partners come to watch how this is done.
7. Bring the flask back to your hood and stir vigorously for 1 minute to get everything mixed.
8. Add diaminocyclohexane, via syringe by puncturing through the septum.
   - 4 mmol x 0.120 mL/mmol = 0.48 mL diaminocyclohexan
   - The order of addition of the other chemicals didn't really matter. But the diamine must be added last, and only after the argon purge is complete.
   - The diaminocyclohexane attaches to the copper to make the catalyst active.
   - It also serves to get the Cu(I) ion dissolved into the organic solution. (with two diamonocyclohexanes attached, the composite catalyst now has a lot of organic character.)
9. Stir vigorously on a hot plate.
   - Set hot-plate setting to ~3.5.
10. Stay for 15 minutes to watch and record color changes or other observations. After that, you are free to go! **YOU ARE NOW DONE WITH THE FIRST WEEK OF ACTIVITY**! ☺
11. But remember that your Scheme 1 Lab Report should cover through Page 9. Pages 10 and on belong in the Scheme 2 Lab Report due the week after the second week.
12. Let stir hot overnight. The instructor will come in the morning and turn the heat off.
13. The mixture will then wait till next week for workup, isolation, purification, and analysis! ☺



Proposed Hypothetical Mechanism for the Cu(I)-catalyzed arylation and discussion (for your interest): The mechanism is very interesting and is **very** different from anything you've seen before. In introduction to SN1 and SN2 reactions, for example, we couldn't use aryl iodides in either of those type mechanisms.

But something very different happens here with the Cu(I). Several low-oxidation-state transition metals [Cu(I) and Pd(0)] have a capacity to do "oxidative addition" into certain aryl-halide bonds. In Step I, Cu(I) inserts into the Ar-I bond, creating new Cu-I and Cu-Ar bonds. This is formally an oxidation-reduction reaction: copper is oxidized from Cu(I) => Cu(III). The iodide and carbon are reduced; they can be viewed as anions following Step 1. The mechanistic detail of how this oxidative addition proceeds is beyond the scope of this course!

The nitrogen then substitutes onto copper in Step 2. This can be viewed as a simple SN2-type substitution. The phosphate base is strong enough to generate the resonance-stabilized nitrogen anion under the high temperatures.

After both the nitrogen and aryl groups bond to the Cu(III), those two then hook together and detach from the copper (Step 3) to make product **6**. This is termed "reductive elimination" because the Cu(III) is reduced back to Cu(I). The aryl and nitrogen, formally anionic when coordinated to the copper, are oxidized back to neutral. The mechanistic detail of this reductive elimination is again beyond the scope of this course! ☺☺

The diaminocyclohexane serves two crucial roles. First, coordination to the Cu(I) makes the complex mostly "organic" so that it becomes soluble in the dioxane solvent. Solubility of the catalyst is essential. Second, coordination enriches the electron density of the Cu(I), which makes it more reactive as a reducing agent in Step 1. In the figure, "L" is a shorthand for "ligand", which is a general term for something coordinated to a metal. So "$L_2Cu(I)$" represents two diaminocyclohexane ligands coordinated to a Cu(I).

Notice how the $L_2Cu(I)$ catalyst, shown at the top of the loop, functions as a catalyst. Following the cycle of oxidative addition-substitution-reductive elimination (Steps 1-3), the original $L_2Cu(I)$ is regenerated and can repeat the chain. Thus, a stoichiometric amount is not required.

Aryl-substituted nitrogens are prolific in nature and in medicinal reagents. The ability to use catalytic arylation to attach aryl groups onto nitrogen is very powerful and useful.

You probably noticed some color changes. If you saw some blue, that would be some Cu(II), probably resulting from trace oxygen oxidizing the catalyst. As the reaction proceeds (or when you return next week), you'll probably see a lot of red/purple. That is the color of iodine, resulting from oxidation of iodide product, either by adventitious oxygen leakage through the septa, or else by reduction of something else in the mixture.