Scheme 2 Part F: NMR and GC-MS.

- Checking your NMR prior to starting Scheme 3 is wise.
- Each NMR and each GC/MS will again need to have a clear label that corresponds to the identity of the chemical.
- You MUST again draw actual structures of your product on both NMRs and GC/MS's.
- When discussing NMR's or GC/MS's in the reports, always do so by label. (For example, "**GC-MS-6c** was taken", or "as shown in **NMR-6C**, the product was very pure….")

NMR Analysis/Interpretation. Concepts and expectations are similar to the Scheme 1 report.

41. An **abbreviated summary report** process will again be required. Draw the structure and label the different carbons. Then make a table with the chemical shifts for the actual **non-aromatic C-H's**, 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.
    - **Also include integration**.
    - But you do not need to analyze/report the splitting (although you may do so.)
    - Which signal is from the β-H, which are from the α-H's, and which are the benzyl H's?
    - Which signals are the methyls or methoxy signals in **3b, 3c,** and **3e?**
    - Does your product look reasonably pure?
42. **Does your NMR confirm that the reaction, and the solvent-removal basically worked**?
    - Does it look like your starting reactant **3** is still present, or gone?
    - Does it look like there is significant amount of solvent left? (Dichloromethane leaves a singlet at about 5.3 ppm.)
43. **Chirality, H-non-equivalency, and chemical shift**: The chirality of the β-carbon not only makes the two β-H's non-equivalent, but also makes the two hydrogens on the newly attached CH2 carbon nonequivalent. The two benzyl hydrogens should each appear as two doublets.
44. **Chirality, H-non-equivalency, and splitting**: As in product **3**, the non-equivalence of the two α-hydrogens, and now the two benzyl hydrogens, complicates their splitting.
    - Each of the two α-hydrogens will usually appear as a four-line "doublet of doublets".
    - The β-H will typically also look like a 4-line "doublet of doublets", unless further split by the methyl group in **3e**.
    - **The two benzyl H's are each split by each other, so each should look like a doublet.**
    - **The appearance of these two new doublets is very diagnostic for product 6 formation!**
45. For your reports, **account for all and only the hydrogens connected to sp^3 carbons.** You don't need to discuss/present N-H hydrogens or aromatic hydrogens. (There are so many overlapping aromatic H's that they aren't interpretively useful in this case.)
46. **Signature signals**: All of the samples will have the interesting α- and β-hydrogens, and the benzyl hydrogens (5 hydrogens combined). But there will be other additional signature methyl peaks for **6c**; **6b**; and **6e**.
47. **Chemical shift logic**:
    - The α-hydrogens, being next to a carbonyl, but being also β to nitrogen and perhaps also an aromatic, should fall in the high 2's or perhaps the low 3's.
    - The β-hydrogen in **3a-3d** should show up around the low 4's. For the β-hydrogen in **3e**, we'd anticipate the β-hydrogen to show up in the 3's.
    - For the benzyl hydrogens, they are on a carbon that has both a nitrogen (+2) and a benzene (+1) attached, so we'd expect them to come around the low 4's or high 3's as well.
48. **Impurities/contamination**: Recognizing which signals come from the desired product and which do not is again significant.
    - Remember that there should be a logical integration ratio for the main H's in product **6**

49. **Comparison to Other NMR's**: It may be very interesting to look at how your NMR **6** looks compared to how other NMR's look.
    - How different is your **6** from the **3** that you began with in Scheme 2?
    - How different is your **6** compared to classmates who made different versions of **6**?
    - How clean is your NMR compared to that of classmates who made the same version of **6**?
50. **GC-MS:** Clearly label each page of each GC/MS printout.
51. Draw the structure and molecular weight for your specific product on each GC-MS sheet.
18. **Retention time**? What is the retention time for your **6**? How much longer is it than **3**?
19. **Purity:** How pure is your **3** by GC?
20. **Mass Spec and Molecular Ion**: For your major product **3**, check in the mass-spec whether there is a molecular ion peak that matches the molecular weight for your product.
21. **Mass Spec and Fragmentation**: The weakest break-point is at the N-benzyl bond. You should be able to see a benzyl fragment (PhCH2+ = 99) and a fragment that it molecular weight – 99. Do you see both of those fragments?
22. **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.

Scheme 2/Week 2 Lab Report:

1. Write a standard synthesis style lab report for your Scheme 1 reaction (**3** → **6**).
2. 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 your **actual** reactant **3** and product in your reaction.
   - None of your pictures should have an "R1": you should illustrate each structure with your actual R1 group drawn, whether that's methyl or phenyl or 4-methoxyphenyl or whatever.
3. Show all calculations. (Including any mole => mass for reactants or products)
4. When listing your chemicals/reactants and showing gram → mol calculations, make sure that you include your main reactant **3**!
5. Include procedural details and observations as usual.
6. Calculate mass yields, and percent yields, etc., for product **6**.
7. Include your **NMR-6** and **GC-MS-6**, with clear labels, structures drawn, **and the abbreviated summary report** for non-aromatic C-H hydrogens for **NMR-6**.
8. Print and attach mass spectra for **GC-MS-6**.
9. 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**.
10. 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 react **3** was successfully converted to product **6**, and does your product **6** look reasonably clean? Or is it obviously significantly contaminated? Was the yield respectable, or terrible?
11. Note: Keep extra copies of your NMR and your GC-MS's. Pyrazolidinone **6** functions as the product in week two report, but it is the reactant in the Schemd 3/Week 3 report. So when writing up and analyzing Scheme 3, you'll need information about mass, molecular weight, structure, and mmol of your reactant **6**. You'll also need to have NMR and GC for **6** so that you'll be able to compare your product **10** to reactant **6** and tell whether the reaction really worked. You'll also want copies of **6** for your Final Report after week 3.