LAB MAP – Safety & Orientation Activity

After familiarizing yourself with your locker equipment, find the following items in your assigned lab room and make a map in your notebook. Feel free to open any and all drawers and cabinets in your lab room to get to know your space. Check this with your TA.

1. Hotplates, lab jacks, and sand baths
2. Thermowells and rheostats - for heating RBF's
3. Cystallization dishes - for water baths
4. Vials – for product storage
5. Water lines – for water-cooled condensers
6. Rota-vaps – note the one closest to your station
7. Chemical spill mats
8. Glass pipets
9. Filter paper
10. Gloves
11. Parafilm
12. Boiling chips/stones, $MgSO_4$, pH paper, copper wire, scissors & tape
13. Foil and cotton
14. Soap refill
15. Glass waste
16. Fume hoods
17. Beakers
18. Erlenmeyer flasks
19. Test tubes
20. Larger filter flasks
21. Vacuum tubing
22. Clamps, support rings, clamp holders, ring stands
23. TLC plates, chambers, tygon tubing for columns
24. Eye wash station
25. Safety shower
26. Fire alarm, fire extinguisher
27. Evacuation route

In the adjoining stockroom, please find the following but do not touch or move anything in this room. This is a semi-restricted area. For most labs, students will only be allowed in this room with permission and in designated areas only.

28. Fume hoods – these will be designated as waste or reagent hoods
29. Flammable solvents cabinet
30. Acid cabinet
31. Refills of gloves, kim wipes, pipets, etc.
32. IR Spectrometer

KEEPING A LAB NOTEBOOK

Students are required to use a fresh, new notebook for this class.

1. Use a standard side-bound notebook (no loose leaf, spiral, or binding at the top). Number all pages ahead of time if they are not pre-numbered. Never remove pages from the notebook.

2. Start a **table of contents** on the last page. Keep this up-to-date for each new page added, starting with the LAB MAP. Otherwise, fill in with the experiment number & title and descriptive but brief subtitle every day (Ex. "Exp 2 – Distillation of Lavender Oil").

3. Use a **title** for each new experiment and begin each new experiment on a new page. Your title may use chemical structures rather than long chemical names when referring to a specific organic reaction.

4. Start a new notebook page each day. Add the date to the page where you are working as you start lab work each day. Write in the notebook throughout the lab, not just at the beginning or end. This will help you keep track of what you have done and provides a record for others to follow.

5. **When performing a reaction,**
   - **…start a new notebook page and draw the reaction scheme at the top of the page (reactants, arrow, expected products) followed by…**
   - **…a table with chemical info and properties (mmol, mg, mL, MW, bp or mp, density, and hazards). Use the reaction scheme to define abbreviations for chemicals to use in the reagent table for simplicity.**
   - **After the reagent table, give a <u>concise</u> description of what you plan to do and leave space to make changes based on your experience in the lab. With a few exceptions, you may not refer to the lab manual while performing the experiment.**

6. Don't compress multiple parts of experiments onto a single page – spread it out to a reasonable extent. **Leave space for data, observations, and reflections.** There are many pages available in the lab notebook and it should be easy to find everything based on page titles and the table of contents.

7. Write neatly enough for other people to be able to read, follow procedures, and easily find data. Don't agonize about being too neat: it just needs to be well organized and legible, not perfect!

8. There is no need to use complete, grammatically correct sentences in your lab notebook: this should be reserved for your laboratory reports. An example might be: "Isolated amine as 2.3 g off-white solid. Recrystallized from methanol (1 g amine in ~15 mL methanol; recovery: 0.85 g). mp (crude) = 123-129 °C; mp (recryst.) = 130-132 °C."

9. **Reflect** on the day's activity with a notebook / journal entry before leaving:
   - Summarize what you did that day
   - Where were you successful?
   - What was challenging?
   - What's your plan for the next day?

Community and General Cleaning Descriptions

Keeping a clean and organized lab space is a group effort.! Keep an eye out for items that need to be restocked at all times, not just at the end of lab. Either refill it yourself or ask the TA for assistance, especially in the case of reagents and solutions. Don't assume it'll be magically refilled – the lab fairies do not exist!

*** A table with community cleaning assignments will be posted on the first day of lab. On each day, we'll decide your community cleanup role. Please initial that you've done your task at the end of every lab or you get zero clean-up points for the day ☹**

*** This is in addition to cleaning your own workspace, including a pristine drawer with no extra, missing, or dirty items. Drawers will be checked without warning and points taken off for each infraction. These penalties will increase as the course progresses, if necessary.**

Reagents and Solutions – Check it is at least ¼ of the way full and in the correct place. Notify TA if empty or near empty. Do not re-make solutions.

Gloves, Pipets, Paper Towels – Check all glove boxes in the main labs (314/318). Bring in new boxes from the stockroom (322) if boxes are empty or close to it.

Soap & Sinks – Check all sinks have full dish soap dispensers. Re-fill using stock containers under the sink. Concentrated soap under sink in 322. Notify the TA if the pink hand soap is near empty – do not refill hand soap with dish soap! Remove any debris from sinks.

Waste containers – clean any spills in secondary containment (ask for help if needed, do not lift carboys), cap all containers, notify TA if full

Benchtops clean – Use a wet sponge followed by dry paper towel to clean & dry every benchtop, including fume hood space. Alternate who cleans the benchtops in 322. Bring any stray equipment to the TA or, even better, ask around to find out who it belongs to!

Floors swept – Sweep your lab room or 322.

Drying Rack – beginning or end of lab – put dishes away from the drying rack - applies to shared glassware not in lockers. Bring any stray glassware to the TA or, even better, ask around to find out who it belongs to!

Equipment organization in cabinets – any shared equipment (hotplates, sand baths, clamps, etc.) is neatly organized in each of these cabinets every day

Equipment turned off – rota-vaps unplugged, water pumps off, buckets empty & upside-down in the sink. All other equipment (hot plates, etc.) is unplugged and neatly put away.

Stockroom (322) – check that all benchtops, including the IR station, are clean. Notify the TA if any stock items are missing. Double check that hoods are clean and waste bottles are capped.

LOCKER EQUIPMENT
THIMANN 314, 318

Everyone is responsible for items in their assigned drawer, whether or not it is used in the quarter. At the end of each lab, check for any broken, missing, extra, or dirty items, even if it wasn't used that day. Any student with an imperfect drawer risks penalty throughout the quarter and being dropped a full letter grade at the end of the term. It's not uncommon to find dirty glassware that you swore you cleaned, but apparently not well enough!

Beakers
☐ 50mL
☐ 100mL
☐ 150mL
☐ 250mL

Bottle, wash

Bulb, small

Clamp, screw

Cylinders, graduated
☐ 10mL
☐ 25mL
☐ 100mL

Dish, Crystallizing

Filter vac

Flasks, Erlenmeyer
☐ 50mL
☐ 125mL
☐ 250mL

Flasks, filter
☐ 50mL
☐ 125mL

Forceps

Funnels, glass
☐ 25mm
☐ 65mm

Funnel, Buchner

Pluringe
☐ 1mL ☐ 3mL

Rings, cork
☐ 2" ☐ 4"

Rods, glass stirring
☐ regular (3)
☐ micro

Ruler

Scoopula

Spatula
☐ regular
☐ micro

Split stopper

Stir bar
☐ 0.5" ☐ 1"

Watch Glass
☐ 100mm ☐ 125mm

<u>Organic Chem Kit</u>

Round Bottom Flasks
☐ 500mL ☐ 250mL ☐ 100mL ☐ 50mL ☐ 25mL

Adapters
☐ Claisen ☐ 3-Way ☐ Vacuum ☐ Straight tube

Condenser, West

Separatory funnel, 125mL with 19/22 stopper

Neoprene thermometer tip

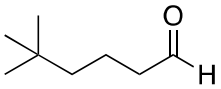
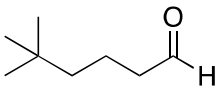
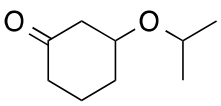
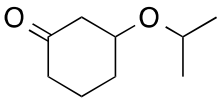
Reacquainting with NMR Activity

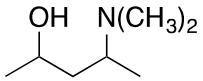
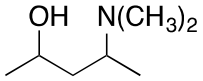
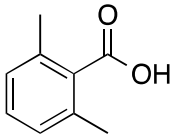
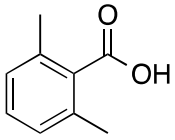
Part 1 – each room completes a collaborative google doc linked on the 146A acrochem site.

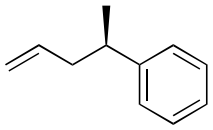
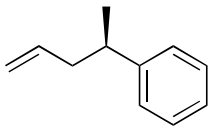
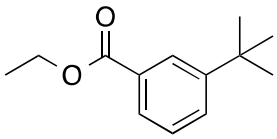
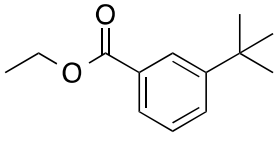
Part 2 – NMR Predictions

1. Predict the detailed ^{1}H NMR spectrum for each compound below using any resources at your disposal. Approximate chemical shifts within provided ranges and relative to other signals in the molecule. The correlation tables online and in the Mohrig or Palleros textbooks can be used to calculate more exact values, but relative assignments within the proper range is ok. Students should work independently first before working together. Work can be corrected using online NMR predicting tools (link on 146A site).

2. Sketch the ^{13}C NMR spectrum for each compound, including approximate chemical shifts and peak heights relative to other signals within the molecule. Use **Appendix VI**.

Structure (add labels)	**Assignment** (corresponds to label on structure)	**Chemical Shift** (ppm)	**Integration** (# H's per signal)	**Splitting**
 ^{1}H NMR				
 ^{13}C NMR				
 ^{1}H NMR				
 ^{13}C NMR				

Structure (add labels)	**Assignment** (corresponds to label on structure)	**Chemical Shift** (ppm)	**Integration** (# H's per signal)	**Splitting**
 ^{1}H NMR				
 ^{13}C NMR				
 ^{1}H NMR				
 ^{13}C NMR				

Structure (add labels)	**Assignment** (corresponds to label on structure)	**Chemical Shift** (ppm)	**Integration** (# H's per signal)	**Splitting**
 ^{1}H NMR				
 ^{13}C NMR				
 ^{1}H NMR				
 ^{13}C NMR				

3. Structural Elucidation – Choose any molecule on the previous pages and draw a derivative - add a functional group, change the branching or substitution pattern, etc. Predict its IR, ^{1}H NMR and ^{13}C NMR spectra. **Check this with the TA then re-write this as a structural elucidation problem (molecular formula, ^{1}H NMR signals listed, and ^{13}C NMR sketched). TAs will collect your problem and exchange with another student.**

Structure ^{1}H NMR	**Assignment** (corresponds to label on structure)	**Chemical Shift** (ppm)	**Integration** (# H's per signal)	**Splitting**
^{13}C NMR				

IR Signals – approximate wavenumbers (cm^{-1}) and intensity (weak, medium, sharp, broad), corresponding functional group and bond