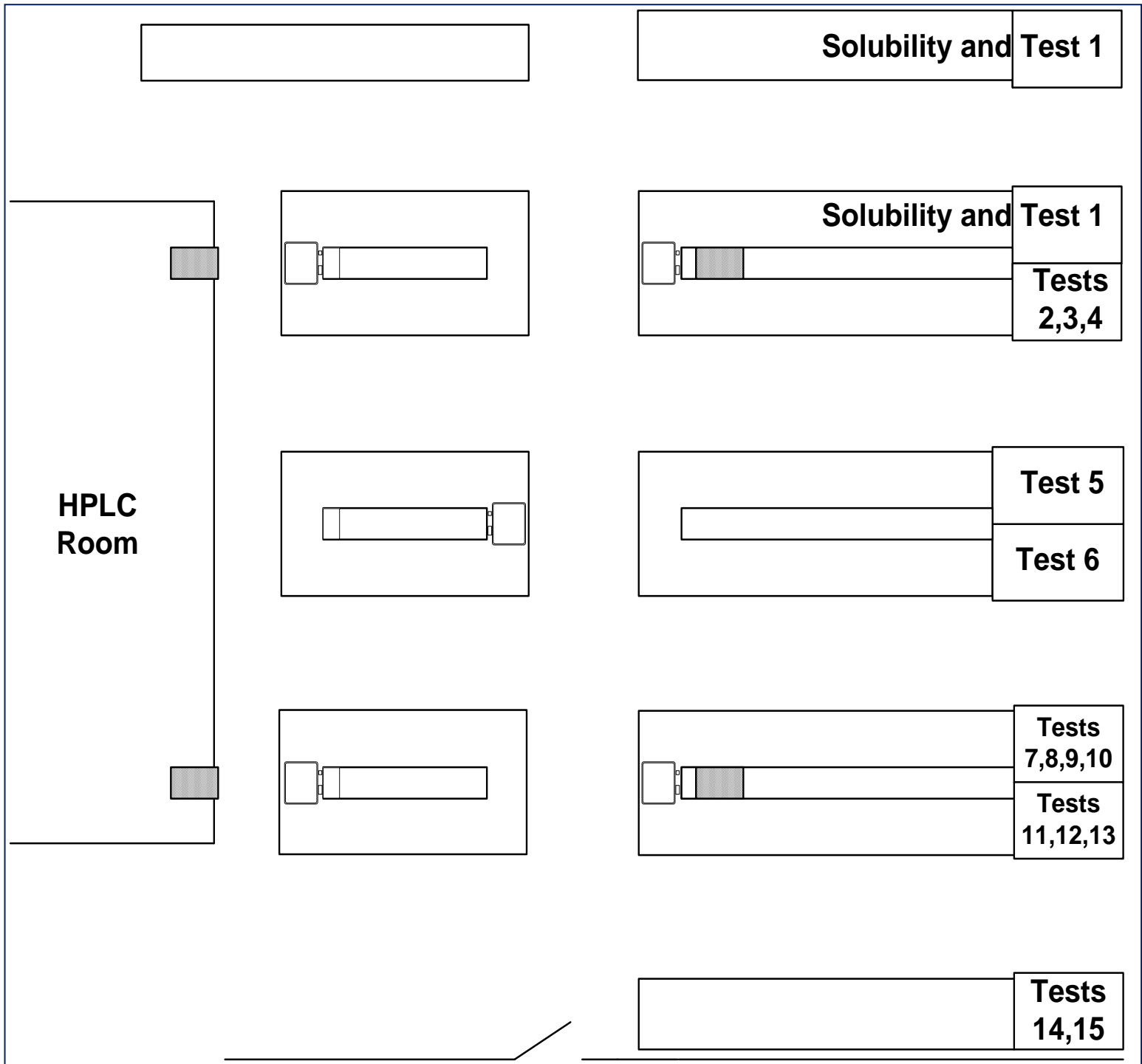


LAB EXAM LAYOUT

Chernoff Hall, Room 120/122



Lab Reports and Grades:

- ✓ Do not forget that in order to pass the course you must submit all lab reports for the experiments scheduled for your group
- ✓ Late report submission may result in significant deduction of marks (**10% of the possible total for each working day late**).

LAB REPORT DEADLINE: 5:30 P.M. on April 9th (earlier submission preferred). You must hand in your reports to the TAs personally

- ✓ Check out all lab marks in the database – if any marks are missing contact your TA ASAP!
- ✓ I would be happy to address your concerns and answer your questions regarding lab/tutorial evaluation

IMPORTANT FOR YOUR LAB EXAM (4 hr duration):

- **Lab Exam Requirement:** submit a report with your analysis of the provided NMR and MS spectra and your experimental data to identify your unknown chemical species.

You will be given:

- ✓ A vial containing your compound
- ✓ ¹H-NMR spectra
- ✓ ¹³C-NMR (J-mod) spectra
- ✓ A mass-spec (may be *electrospray*, EI, CI, positive or negative mode)
- ✓ If your compound is a liquid, you will be given a boiling range.
- Take your compound to the fumehood to do the tests, **do not remove reagents from the hoods and move them around the room.**
- **If you are leaving the lab (e.g.: heading off to the quiet 'area'), do not take your sample from the lab** - leave it in the box - you can retrieve it again whenever needed.
- Place used **test tubes** in an appropriate location in the fumehood. If they need to be neutralized with **Sodium Bicarbonate**, do it and pour the solution in a beaker.

- For **functional group tests**, present your observations and argument in a clear and very concise manner. Control test with solvents and/or a known compound may be necessary. Remember that no single test should be treated as absolutely conclusive! Try to extract from your tests as much useful information as you can regarding the chemical properties of your compound and, ultimately, its identity!
- **Pace yourself:** Every group needs to do the **IR, UV, Solubility, Oxidizing ability and Unsaturation tests, and at least two Functional group tests.** Most will also need to do **melting points**. If a setup/instrument you intended to use has been taken by another group, do not wait but rather run a different test for which there is a setup available in the lab. There will be only up to 7 to 8 groups in the lab at the time, so logjams should not occur.

MOST COMMON PROBLEMS

- **Misinterpreting your Mass Spectra:**
For the determination of the **correct molecular weight**, it is crucial that you review your **Mass-Spec tutorial** and **Chem222**, especially as related to the mass-spectra obtained by different *ionization modes* (e.g.: **EI**, **ESI** in *positive* and *negative* mode). Recall the '**nitrogen rule**' but check out a possibility that you have two or more identical *hetero-atoms*.
- In some cases you may get **NMR in different solvents**. This gives you additional information. Pay attention to what solvent your NMR was run in (remember that solvents also exhibit NMR peaks).
- **NMR data** –table, assignments, predicted shifts (for $^1\text{H-NMR}$), splitting trees.
- **Misinterpreting the J-mod ^{13}C spectrum (similar to DEPT):**
It's not always even up and odd down!

^{13}C spectra may show **low-intensity impurity peaks** (especially watch out for those with odd H's) – reconcile your ^{13}C spectrum with other data (MS and ^1H -NMR)

- ***Misinterpreting UV spectra*** - make sure that you have recorded the spectrum correctly (pay attention to *solvent, concentration effects, cuvette material, etc.*). The spectrum should be scaled appropriately.
- ***Poor IR spectra and interpretation*** – watch for saturation effects, ensure adequate peak assignment (stretch/bend)
- ***Solubility tests*** – do not, forget that even if your compound appears to be non-water-soluble, it may still have some limited water solubility, and a quick pH test may give you additional data to deduce or confirm the identity of your compound.
- **Always consider alternative structures** and present your argument as based on your spectral data and qualitative tests.