Notebook & Data Guidelines

A. General:

1. Keeping a clear, organized, thorough and **up to date** notebook is absolutely critical for your success in the lab. This will aid you (and the group) in successfully repeating experiments, writing manuscripts, theses, *etc*. Anyone that picks up your notebook should be able to *exactly* reproduce what you had done in the lab!

2. Your lab notebook should be a *comprehensive* collection of every experiment you have performed in the lab. The definition of experiment is broad: it could be a reaction, the purification of a store-bought chemical, solubility/crystallization tests of a compound that you synthesized, *etc.*

3. Ian will carry out a lab notebook and server data check during the annual graduate student reviews, and at-will as necessary. Failure to keep your notebook up to date will result in a report to the graduate committee. Consistent failure to follow protocol could result in your dismissal from the group.

B. Instructions:

1. Start on Page 1 of your notebook. Your table of contents should be *start on the last page of your notebook*, and as you fill it out you should work backward. This will allow you to maximize the number of usable pages without having to predict how long the table of contents will be.

2. Fill out your table of contents **daily.** Not doing so will make your life miserable later on. Entries should be full reaction diagrams or descriptive of the procedure. Sometimes it is useful to indicate if a reaction is particularly successful/bad, or if it is a 'go-to' experimental for a certain procedure.

3. Use only permanent ink in your lab notebook (for a list of pen choices, as well as another perspective on laboratory notebook-keeping see: http://colinpurrington.com/tips/academic/labnotebooks).

4. Only write one experiment on a lab notebook page. **Do not skip pages!** (lab notebooks are very expensive). If you need additional pages, you can go on to the next page or if that page is already in use, on a later page. Make sure to indicate where experiments pick up, in this case.

5. **Date** each experiment at the top of the page. If you do additional work on a later date, add that within the text of the experiment.

6. Clearly write the reaction or experiment at the top of the page. If following a published procedure, make sure to cite the reference beside or underneath the experiment.

7. For reaction experiments, create a reagent table and clearly indicate the **name of each reagent**, **source** (*i.e.* Sigma Aldrich, Strem, *etc*, or the lab notebook page for a compound you synthesized), **molecular weight**, **density** (if measuring by volume), **mmol (equivalents) used**, and the **gram amount used**. This goes for solvents as well as reagents. Sometimes you may be 'eyeballing' the amount of solvent used; make sure to indicate this precision or lack of precision!

8. Write a detailed experimental, indicating ***exactly*** what you did, and in what order. Remember: you want a layperson to be able to replicate this, not just yourself.

9. All analytical data and associated with the experiment and their electronic file names should be indicated within the experimental text of your notebook (for example, NMR, TLC, IR).

10. All files associated with a particular experiment should have file names as follows: (**your initials**)(**2 digit lab notebook number**)(**page number**)-(**file number/descriptor**). For example, if I took some NMRs for a reaction performed in my first notebook, page 234, the file would be:

    IAT01234-01H
    IAT01234-01P
    IAT01234-01C

    These filenames would indicate that I took 3 NMRs of the same sample, one being a ^{1}H, one a ^{13}C and one a ^{31}P NMR. Subsequent NMR samples performed during the experiment would be labeled -02H, *etc*.

11. Make sure to **back up all of your electronic data** (NMRs, XRD Data sets, Endeavor data, kinetic data, other graphs and images you have made) on the group drive. You should make a folder under "Research Data" utilizing your three initials, and back up all data there. Filenames should clearly reference a lab notebook page (as above), and be descriptive of what is in the file.

    Our group server address is 128.101.162.164

    Username: _____ Password: _____

C. Sample Notebook Page:

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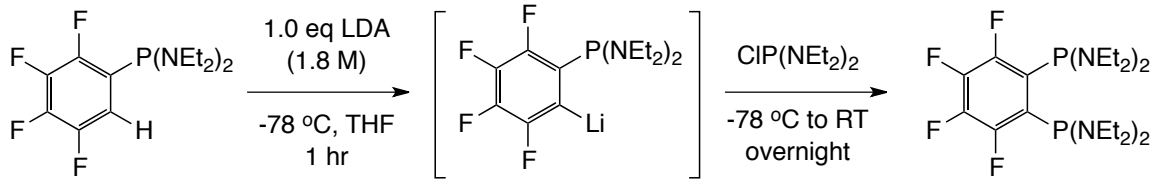

Reagent	Source	M.W. (g/mol)	Density	mmol (equiv)	Amount
$(C_6F_4H)P(NEt_2)_2$	IAT01011	324.3	N/A	4.36 mmol (1 equiv)	1.414 g
LDA	Sigma Aldrich	2.5 M in THF	N/A	4.36 mmol (1 equiv)	1.75 mL
$ClP(NEt_2)_2$	IAT01008	210.7	1.002 g/mL	4.36 mmol (1 equiv)	918.6 mg 0.916 mL

Reaction was set up on the Schlenk line.

1. In the glovebox, 1.414 g of $(C_6F_4H)P(NEt_2)_2$ was added to a 250 mL Schlenk flask, sealed, and brought out of the box. The flask was then attached to the Schlenk line and purged with N_2.
2. 100 mL of THF (dried on the solvent columns) was cannulated in to the flask, giving an amber solution of phosphine. Cooled to -78 C with a dry ice/IPA bath.
3. 1.75 mL of 2.5 M LDA in THF was then *slowly* syringed in to the solution over 15 min. Solution turned dark green.
4. Stirred for 1 hour while maintaining the bath temperature at -78 C.
5. 0.920 mL $ClP(NEt_2)_2$ was slowly syringed in to the reaction mixture over approximately 15 min. Solution turned from green back to amber.
6. Reaction was left to stir overnight (12 hours). Warmed to room temp overnight.
7. Cannula filtered reaction in to a new 250 mL Schlenk, removed solvent *in vacuo*. Yielded 1.96 g of a brown oil. (90% based on 4.36 mmol of product)

   Took NMRs in $CDCl_3$:
   IAT01012-01H
   IAT01012-01P
   IAT01012-01F
8. Crude reaction NMR indicates approx. 95% product, 5% S.M.

By signing below, you indicate that you have read and understand the content of this document.

Name: _____ Date: _____