Suggestions for JAG Group laboratory Notebooks

The following is a list of things that all laboratory notebooks should contain. It is by no means a comprehensive list and various experiments may require different forms of record keeping. This list is mainly meant for new initiates to learn what kinds of things are expected from them in their lab notebooks.

General laboratory notebook guidelines include:

1. A Table of Contents
    a. This should include the experiment titles and the pages where they can be found.
2. A unified labeling scheme
    a. A unified labeling scheme for experiments, data and compounds is useful for figuring out where things came from in relation to the notebook. The commonly accepted format is INITIALS-BOOK #-EXPERIMENT #.
    b. For example, data labeled ALE-2-034, or ALE2034 would represent data for Alex Estrada, book 2, page 034.
3. All experiments should have page numbers along with descriptive/useful titles.
4. Experiments should include dates for ALL days an entry was made
5. Drawn reactions schemes should be as detailed as possible
6. References for experiments should be included.
7. There should be some reasoning or background information for the experiment.
8. Reagents should be in a table describing the amounts needed/used, their source, as well as any relevant safety issues.
    a. If reagents have been prepared, there should be either an explanation or a reference to an explanation of how they were prepared.
9. Experimental sections should be as detailed as possible. They should contain all observations, measurements and procedures.

a. This includes things like masses used, orders of addition, solution concentrations. Everything is important!
b. Don't forget about more subtle things like column dimensions and crystallization conditions.
c. Drawings of special setups or glassware are especially useful for reproducing an experiment.
d. TLC plates may also be put into this section. It may be useful to cover them completely in packing tape to prevent fading.
e. Experimental sections are also an appropriate place to mention safety hazards including, but not limited to, dangerous synthetic procedures or flammable/toxic compounds.

10. Data sections where all data is presented and worked up (if applicable) should also be present.
    a. For most synthetic experiments, data should include pertinent NMR signals, IR bands, etc.
    b. Instruments and their locations should also be noted if they do not belong to the group.
    c. Instrument parameters should also be noted here if different from typical values.
11. If data cannot be put in the notebook, there must be some clear indication as to where it can be found.
12. Some commentary or conclusions indicating what the results meant.
13. All notebooks should be written clearly and neatly. This includes having good penmanship as well as correct grammar.

BOOK # - PAGE #

EXPERIMENT TITLE AND CODE

REACTION
SCHEME

Reference(s)

PURPOSE

PRELIMINARY CALCULATIONS

REAGENTS	SOURCE	AMOUNT	OTHER DETAILS

REAGENTS	SAFETY ISSUES

EXPERIMENTAL, DATA AND
OBSERVATIONS

SIGNATURE

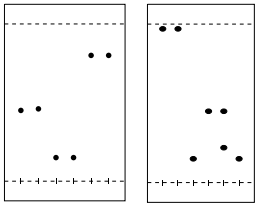
DATE

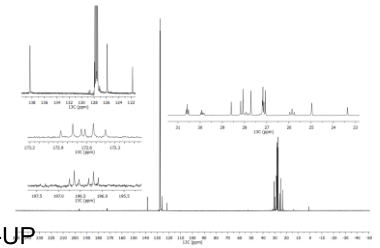
CONT. FROM PG.

BOOK # - PAGE #

EXPERIMENT TITLE AND CODE

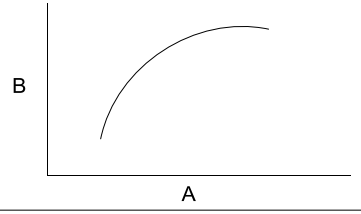
EXPERIMENTAL, DATA AND OBSERVATIONS





DATA WORK-UP
CHARTS, GRAPHS, TABLES

	A	B
1	###	###
2	###	###
3	###	###
4	###	###



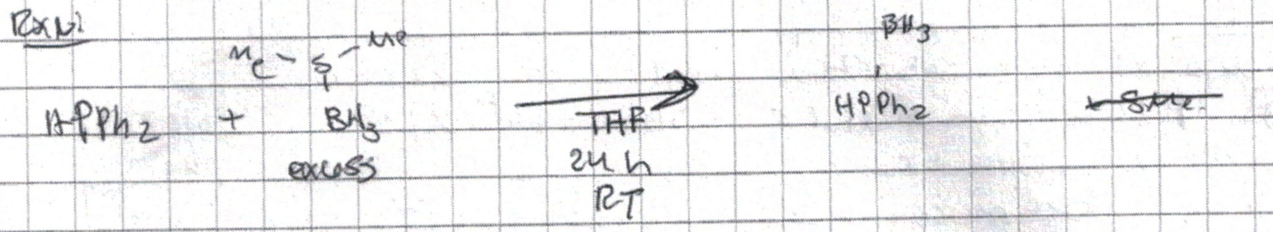
SIGNATURE

DATE

TITLE Borane Protection of Diphenylphosphine **PROJECT** ADC-2-121

Continued from page X

Rxn:

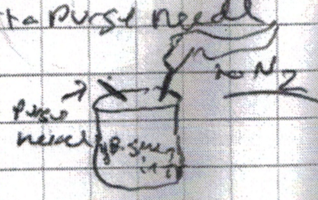


Ref: M. Van Overschelde et al. / Tetrahedron 65 (2009) 6410–6415
Purpose: Needed for the synthesis of "gearbox ligand"

SAFETY
$HPPh_2$ - Causes skin + eye irritation (wear goggles + gloves)
+ is pyrophoric + self-heating (avoid contact w/ air)
H_3B-SMe_2 - Flammable (avoid open flames), has acute oral + dermal toxicity so avoid contact
- releases flammable gases when contact H_2O
$HPPh_2$-BH_3 - releases flammable gases in contact w/ H_2O
- Store in a dry place but is air stable

Reagent	Source	MW	M	V	mass (g)	mmol
$HPPh_2$	Strem	186.19	-	-	0.4137	2.222
H_3B-SMe_2 (in THF)	Acros org.	N/A	2	11.1 mL	—	22.2

PROCEDURE
- Put hot 100 mL schlenk tube, equipped w/ stirbar in g.box antechamber to dry under vac. Then move into g.box
- in g.box weigh 0.4137 g of diphenyl phosphine. Dissolve in 4 mL of DCM + transfer to ~~flask~~ to flask. Remove flask from g.box.
- Put flask on line + flush at least 3x before opening to N_2
- Put bottle of H_3B-SMe_2 on line. Connect to N_2 + also insert a purge needle
- Flush a 24 mL syringe 3x w/ N_2.
- via syringe take 11.1 mL of BH_3SMe_2 soln + transfer to 100 mL schlenk tube.



- Let stir @ R.T. Rxn start @ 10:45 AM
- Solution is clear + colorless

Continued to page 121

SIGNATURE [signature] DATE 2/19/15

DISCLOSED TO AND UNDERSTOOD BY DATE

PROPRIETARY INFORMATION

2 - 122

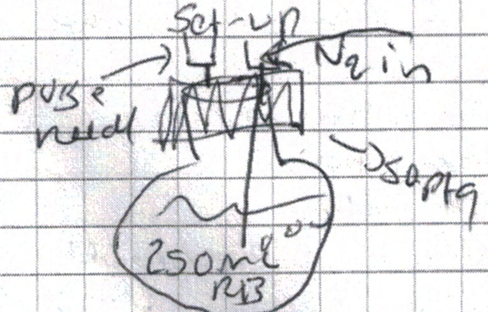
BOOK PAGE

TITLE Protection of Diarylphosphine **PROJECT** ADL-2-121

Continued from page 121

2-10-15

- degassed aq. saturated NH_4Cl soln for 1 hr.
- @ 11:30 cooled rxn mixture + slowly added 10mL of NH_4Cl solution via syringe. Placed a needle in the septa in case too much pressure builds up (gas is produced)
- quenching produces white solid + lots of bubbly
  - → This process is quite exothermic. Take caution.
  - → Finished @ 12:10 p.m. Removed ice bath. @ rt
- → @ 2:00pm dumped rxn mixture in 100mL DI H_2O
- → extracted w/ 3x90mL DCM | 250mL sep funnel.
- → Then washed the organic layer (DCM) w/ 100mL total saturated $NaHCO_3$ aq solution.
  (Did this by dividing the collected layer into 2 portions. Rinsed each portion w/ 50mL of the $NaHCO_3$.
- → Dried the DCM solution w/ $MgSO_4$
- → Filtered. used ~25mL to rinse the $MgSO_4$
- → reduced volume via vacuum removal of solvent. (Caution)
- → Filtered soln through 1in pad of silica. rinsed the pad w/ 200mL DCM. Transferred most of the soln to a 250mL RB (it all wouldn't fit ∴ but saved what wouldn't fit.)
- → evaporated soln w/ N_2. ~~the soln was low enough added the solution that didn't fit back. Let continue to evap~~
  - → let soln that wouldn't fit evaporate in open air. Label ADL-2-121B
- bubbled N_2 through the soln (This will be a test to see how air sensitive it is)



2-22-15

- Solvent from RB was finally evaporated.
- redissolved in minimal DCM + transfer to weighed vial (not labeled) mass = 13.2415g
- removed DCM via rotary evaporator (H2O bath @ ~~30°C~~ 35°C)
-

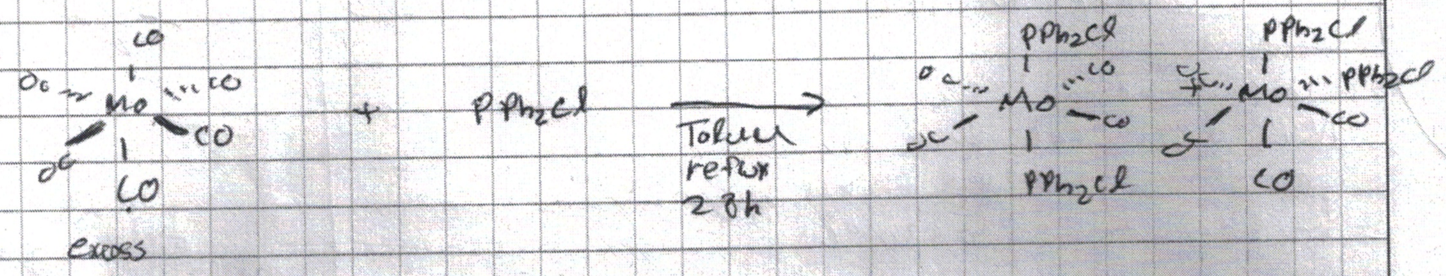
Continued to page

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE

TITLE Synthesis of trans-Mo(CO)4(PPh2Cl)2 PROJECT ADC-2-123

Continued from page ∅



Reference: J.R. Martin et al / Journal of organometallic Chemistry 751 (2014) 695–702
Purpose: To synthesize trans-Mo(CO)4(PPh2Cl)2 as a Mo(CO)4 source.

SAFETY

Mo(CO)6 – Fatal if swallowed, in contact w/ skin or inhaled
- same gloves, labcoat, do not touch
- considered to have acute toxicity (cat. 2)

PPh2Cl – Corrosive. Can cause severe skin + eye damage
Handled in box so should not be a problem!

Reagents	Source	MW	Mass (g)	mmol
Mo(CO)6 (white powder)	STREM	264.00	~~3.3222~~ 3.3221	12.58
PPh2Cl (colourless liquid)	TCI	220.63	3.9434	17.87

Procedure

- Placed a hot 250 mL 2N ~~[illegible]~~ N_2 adapter on line. Flask was equipped w/ hot stirbar, reflux condenser + N_2 adapter @ top of reflux condenser. Let cool under vac then put whole system under N_2
- weigh 3.3221g of Mo(CO)6 + place in the flask. Flush flask 3x w/ N_2 + then leave under vac.
- Tape down septa + transfer flask under vac into ~~the N_2~~ argon glovebox.
- in glovebox weigh ~~3.43~~ 3.9434g of Ph2PCl in a vial. Transfer to 250mL RB. Rinse vial w/ 4mL Tol + transfer this to RB as well.
  Remove from glovebox, place on line, flush 3x w/ N_2 before opening to N_2
- transfer ~40mL tol via syringe tech. from Straus flask + add to RB @ start 3:00pm
- heat @ 110°C - solid dissolves almost immediately. Solution turns yellow + colourless

Continued to page 124

SIGNATURE [signature] DATE 2-9-15

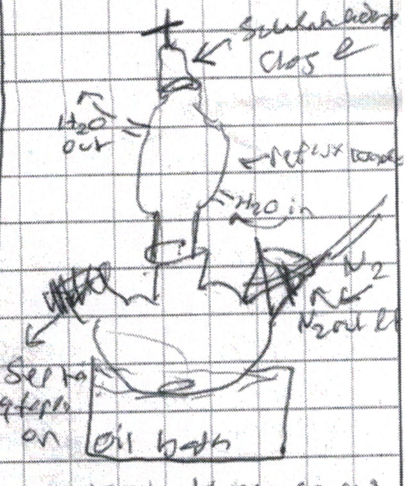
DISCLOSED TO AND UNDERSTOOD BY DATE

PROPRIETARY INFORMATION

TITLE ~~ADL-2~~ Syn? of trans -Mo(CO)4(PPh3)2 PROJECT ADL-2-123

Continued from page 123

Apparatus



- used thermocouple to control T.

- Increase temp to 129°C - reflux does not occur
~~[illegible]~~
- Increase temp to 125°C
- After heating for 10 min rxn is now slightly yellow. reflux still has not begun.
- increase temp to 130°C
- 25 min in rxn is bright yellow still not refluxing though.
- increase temp to 135°C. still not refluxing. increase to [illegible]
- Orange by 35 min in.
- hose to N2 popped off. was quickly put it back on + return temp to 135°C.
- ~~[illegible]~~ boiling profusely but does not appear to be "refluxing"
- Slow reflux is 3:45 (1 drop/2 min)
- rxn is red
- @ 4:5:03 rxn is dark red black + opaque. realized there was condensing in N2 hose.
- attached another hose to top of condenser, flush 3x w/ N2, open to N2
- close side adapter + remove hose.
- ~~[illegible]~~ continue to reflux.

2-10-15

- in the morning the rxn mixture is black and is still refluxing slowly (still 135°C.)
- the oil bath has turned yellow. It does not appear to be my fault though (no trace of popped off hose or anything like that, not sure what the contaminant is)
- at 3:30 pm remove oil bath, let rxn cool, then remove solvent via vacuum pump. ~~[illegible] under N2 [illegible]~~
- This produces a thick black residue. Put it on [illegible] under N2 before leaving.

2-11-15

- Attempted to purify mixture w/ column chrom. Used column w/ 100 g of silica, + 3:1 DCM:hex eluent
- However nothing really separated. It eluted off as a big black smudge

Continued to page 127

SIGNATURE [signature] DATE 2-11-15

DISCLOSED TO AND UNDERSTOOD BY DATE