Knight Group Policies and Procedures on Daily Research Records
(02/05/2008)

Lab Notebooks

(a) Andy will provide you with a labeled hard-bound lab notebook when you join the group or require an additional book. Print out a label for the spine (e.g. "Simmers Lab Notebook 1") and attach well with scotch tape. Ensure the label reads in the correct direction. Write your name on the cover label.

**DO NOT REMOVE YOUR LAB NOTEBOOK FROM THE CHEMISTRY DEPARTMENT UNDER ANY CIRCUMSTANCES.**

(b) Notebooks should be open at the experiment page you are working on, located on your desk or bench at all times for viewing by other group members or safety personnel.

(c) The first few pages of each lab notebook should be set aside for a table of contents. The titles and page numbers of each experiment should be entered in the table of contents upon completion of the experiment.

(d) Note books should be contemporaneous i.e. experiment entered as they are done or very shortly afterwards (2 days is an absolute lag time).

(e) All records are made directly into the lab notebook in pen - do not perform calculations or take measurements down on scraps of paper or post-its. When working with equipment and instrumentation in other labs in the building take your lab notebook with you for recording notes and data.

(f) Each experiment should be described in 5 parts:

   (i) the date followed by a descriptive title, such as "Attempted synthesis of triphenylphosphine triphosphonate" or "Low temperature 13C NMR experiment to determine binding of carbon monoxide to tungsten hexacarbonyl"

   (ii) a brief descriptor of the experiment which for a synthetic reaction may simply be a visual chemical equation with structures; provide; provide molecular weights and vendors (including purity of purchased compound) under the structures for reactants and products.

   (iii) the best literature references for the procedure (e.g. cite full papers instead of communications where possible or papers as opposed to dissertations). References are included if an experiment is being repeated. If compounds originate from your own or another group members synthetic experiments, indicate this with the correct two letter/4 digit abbreviation for the compound e.g. AK-1-156 (i.e. compound reported in Andy Knight's lab book 1, page 156).
(iv) the narrative description of the experiment, including all conditions (e.g. temperatures, amounts of solvents (method of purification), size of flask, amounts reagents reported as (g, mmol) or (g, mol) as appropriate, time, stirring etc and observations (gas evolution, fireball, precipitates etc). Normally masses will be reported to 3 dp e.g. 0.345 g. Pay attention to significant figures.

(v) a summary of the primary (raw) data collected during the experiment, such as integrals vs time for rate experiments, gc traces, drawings of TLC, list of spectral parameters or where to locate them.

(g) all compounds and spectra should be coded with the researchers initials, lab book numbers, page.

(h) color coding is encouraged (e.g. use red ink for compound numbers, blue for vendor) to aid quick location of important information.

(i) useful information such as small printouts of spectra, printed e-mails from Andy or collaborators, Fedex label tracking numbers, completed microanalysis forms etc. should be taped on all sides directly onto the lab pages.

(j) do not insert loose papers (e.g. spectra) in your notebook. Almost certainly they will fall out.

Avoid excessive narrative, the primary role of the lab notebook is to report the data in a brief and succinct way. Especially avoid wordiness and use 3rd person past tense. Detailed descriptions of experiment procedures, conclusions etc. should be reserved for research reports. Avoid describing routine lab practices and procedures e.g. the following is correct:

"compound AK1-042 was then purified using vacuum distillation at 50 °C at 0.45 mmHg (oil pump) and stored under N2 in the fridge" You should not fill up several pages of lab notebook describing how a routine vacuum distillation was set up. The assumption is that subsequent readers will know how to perform a routine distillation.

Remember, the two main uses of your lab notebook are:

A. primary source of data which you will use to write reports and dissertations (and ultimately manuscripts).

B. provide the necessary information for another researcher to follow up on, or repeat your reactions. As such, your notebook should be legible.

An extract from well written lab notebook follows:
22 February 1997

12-di-n-butylyphosphinoctadecan-1-y1 tosylate. \textit{VI},

\[
\text{(n-BuO)}_2P(CH_2)_{12} \text{OH} \xrightarrow{TSCl} \text{(n-BuO)}_2P(CH_2)_{12}O^-
\]

FW 532.72

12-di-n-butylyphosphinoctodecan-1-y1 FW 379.54 (TS4055)

p-toluene sulfonyl chloride FW 190.65 (Aldrich-redkt)

tetraethylamine FW 101.19 d. 0.726 (Fisher)

dichloromethane (Fisher)

A 500-mL RB flask, containing the alcohol (13.10 g, 34.61 mmol) and a magnetic stirring bar, was charged with dichloromethane (100 ml). The flask was capped with a septum and flushed with nitrogen. To the cooled solution was added tetraethylamine (7.2 ml, 51.9 mmol, 1.5 equiv), followed by p-toluene sulfonyl chloride (7.26 g, 39.07 mmol, 1.1 equiv). Reaction mixture stirred at room temp and followed by TLC. RXN worked up after 30 hrs. EtOH-11 vol was filtered off and solvent removed using room temp water bath and rotary evaporator. Residue taken up in toluene (100 ml) and filtered. Filtrate washed with additional toluene (50 ml) and the filtrate washed with NaHCO$_3$ soln (2 x 75 ml) followed by H$_2$O (50 ml). The organic phase was then dried (MgSO$_4$). High-Rf impurities still present so solvent was removed by rotary evaporator and the residue was chromatographed on 160 g silica gel (50 mm column) using CH$_2$Cl$_2$ for first 10% solvent followed by 5 x 100 ml 3% MeOH-CH$_2$Cl$_2$ and 5 x 100 ml 6% MeOH-CH$_2$Cl$_2$. Fractions containing compound co Rf 0.6" (5% MeOH CH$_2$Cl$_2$) combined and stripped of solvent.
13.82 g of a clear, colorless oil (75% yield).

The last fraction showed the elution of the starting alumina along with tosylate. The column was run dry and the solvents removed to give 2-3 g additional material.