The Laboratory Notebook: Transcribed from the class-notes of S. E. Denmark

I. References

"Writing the Laboratory Notebook" H. M. Kanare, American Chemical Society; Washington D. C., 1985.
"Organic Chemistry Laboratory Techniques" Georgia Tech, 1981.
"Experimental Organic Chemistry" Harwood, Moody, and Percy Blackwell Scientific Publications, 2nd ed., 1999.

II. Why Keep a Notebook?

A. Primary source of scientific information
B. Unambiguous statements of the truth

Guiding Principle:

- The notebook is written for other scientists, not the author.
- Write with enough detail and clarity that another scientist could repeat the work and make the same observations and obtain the same results

III. Organization

A. Outside cover must have name, subject, series number
B. Table of Contents/Index: leave the first few pages of the notebook blank for constructing an index when the notebook is complete
C. Record Experiments Sequentially: never tear out or remove pages from the notebook except the carbon copies for safe keeping
D. Start each new experiment on a separate page: do not skip pages to allow for future entries
   1. If the experiment requires more pages indicate that continuation page at that bottom
   2. If the experiment ends in the middle of a page cross out the remaining blank space
E. Record all observations directly in the notebook <u>in ink</u>
   1. Do not write out equations, observations on separate sheets and copy them over later
   2. Do not erase or white out entries
   3. Cross out errors and annotate corrections nearby

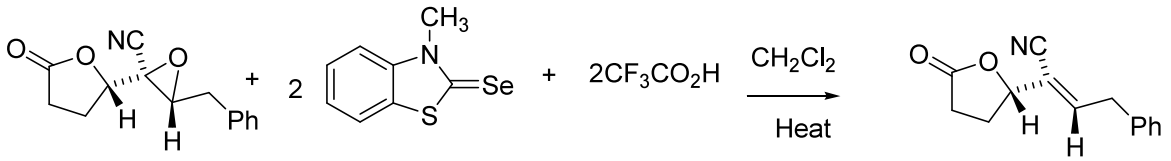
IV. Content, Layout of Notebook Page

A. The top of every new experiment page should have the date and title of the experiment
   Example:

Preparation of Phenylselenocyanate	3 March 95
Bromination of Stigmasterol	4 March 95
Deoxygenation of Epoxy Nitrile	13 Dec 78

B. Write out reaction scheme clearly with all reactants and expected products. Provide in Tabular form or underneath scheme all information regarding quantities, moles, equivalents, suppliers, physical properties, purfication

Example:



MW 243.28	MW 228.18	MW 114.03	$(CH_2Cl)_2$	MW 227.18
770mg	1.444g	484µL	Fluka Puriss	TH {720mg
3.17 mmol	6.33 mmol	6.33 mmol	Dist (P_2O_5)	TH {3.17 mmol
[from SED-XX-4]	2.0 equiv	2.0 equiv		
	[from SED-XLX-79] d=1.492			
		Fluka Puriss Dist.		

NB—Pay attention to significant figures, usually 3 for weights

C. Special Conditions, Apparatus
   1. Drying of glassware
   2. Anhydrous or oxygen-free atmosphere, exclusion of light
   3. Draw special experimental setups

D. References, Literature Citations
   1. Can be previous notebook entry
   2. Journal citation for exact or related procedure

E. Describe in concise high information content phrases the exact procedure executed
   1. Record all details whether deemed relevant or not: time, temperature, addition order
   2. Record all observations: color, solution, ppt, gas
   3. Always record <u>internal</u> temperature

   Example:

   Dissolved 770 mg of epoxynitrite (XX-4A/1) in 10.0 mL freshly dist. CH_2Cl_2 in 25 mL RBF. Fitted with condenser, N_2 inlet.
   Adding 1.444 g of selenium reagent (XLX-79) and 0.48 mL of CF_3CO_2H.
   Heated to reflux under N_2 [START 17:30]
   Solution darkens to red-orange, black precipitate deposits

F. Draw all TLC (GC, HPLC) in notebook

Insert Drawings

G. Workup Protocol

1. Extraction volumes, solvents
2. Washes
3. Drying agents

Example:

14 Dec 78 After 16.5 h filtered off black ppt, washed 1X30 mL CH_2Cl_2, air dried afford 342 mg black powder (137% theoretical Se)
Evaporated filtrate to brown oil (2.12 g). Taken up in 50mL EtOAc, washed 2X25mL 2N HCl
1X25ml brine (backwashed aqueous phases)

1X50mL EtOAc . Dried ($MgSO_4$). Filtered off drying agent.
Added ~2 spatulas of activated charcoal, stirred for 10 min, filtered through celite (yellow filtrate) evaporated in vacuo to afford 2.00 g. yellow oil SED-XX-5A---name compounds

H. Keep Track of Mass Balance

1. Include weights of crude materials, extracts, solids
2. Never discard aqueous phases, solid ppts until mass is accounted for!

I. Provide Complete Details for Purification

1. Recrystallization: Solvents, volumes, temperatures, treatment recovery at each stage
2. Distillation: apparatus, vacuum, head temperature for each fraction bath temp, weight of each fraction
3. Chromatography: adsorbent, solvents, column size, fraction size, fractions combined, weights

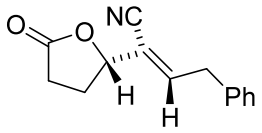
Example:

Chromatographed 5A (silica gel, 40X200mm) EtOAc/hexane, 1/1. After 120mL forerun, collected 30X30mL fractions, [checked UV, I_2]

<INSERT DRAWINGS>

Combined fractions #12-22, evap to yellow oil (670mg)
Dissolved oil in 15 mL EtOAc, stirred w/activated charcoal, filtered through celite, evaporated to give 618 mg of pale yellow crystalline solid [SED-XX-5B/1]

Crystallized XX-5B/1 from 5 mL hot EtOH, cooled to 2 °C for 2 h, collected crystals, washed with 25 mL Et_2O/pentane: 3/2. Dried in vacuo to afford 451 mg white crystals SED-XX-5B/1a.



Yield 63%
mp 79.5-87.5 °C
IR ($CHCl_3$) #162 ν_{CO} 1775 ν_{CN} 2190
NMR ($CDCl_3$) # 187 matches (XIX-32) #164
$[\alpha]^{25}_D$ +8.8 ° (c = 2.63 dioxane)

J. For Each Compound, indicate yield in g, %
1. Provide sample number
2. Provide physical data, R_f, mp, bp. n_D, $[\alpha]_D$
3. Spectroscopic data: numbers of spectra

K. Conclusion
1. Brief statement of outcome of experiments, interpretation suggestions for improvements, repeat
2. Signature and witness

L. Records of Spectra, Measurements
1. NMR, IR, MS
    a. compound number
    b. your name
    c. structure
    d. details of measurement
2. Gas, Liquid Chromatographs
    a. compound number (origin of sample, except number)
    b. column type, length
    c. temperatures (ing, det, oven-program)
    d. pressures, flow
    e. label peaks

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