Notebook Procedures

1. Notebooks are available from N.J. Turro. When you pick one up, immediately put your initials and notebook number on the binding; e.g. WBH-1 or WBH-2, etc.

2. Write in ink on the inside left front cover:
   Name
   Address
   Telephone
   Date of first entry
   Date of last entry

3. Save the first ten (10) pages in the front for a table of contents and the last ten pages in the back for an index.

Description of Experiments

1. Start each new experiment at the top of a new page. Leave the opposite page blank or use for jotting notes. Leave about an inch margin on the left so that comments may be inserted later.

2. The name of the game is information retrieval. If I cannot find the information in a reasonable fashion in your notebook or reports (without you standing over my shoulder explaining what that funny squiggle mark is or what the weight must have been) it cannot be published in your thesis or journal, period. This means the material must be legible, detailed and most important, easy to reproduce.

3. Paper is cheap, but time is valuable. Don't be a piker with your notebook. At the top of each page the following material should appear:
   1. the time and date the experiment was started
   2. a written description of the experiment by a title or a chemical equation
   3. if a reaction is being performed, the weights and number of moles of material should be indicated under the appropriate formula
   4. appropriate literature references should be included

   Example:
   9:00am
   12/20/67
   Preparation of n-butylbromide
   [insert image]
   Ref. Fieser, "Org. Experiments," 1964, p.76

4. Spectral data is important in the identification and characterization of reaction products. The spectra notebooks shuld be kept in binders separate frmo the lab notebooks. Spectra must be labelled in such a way as to be easily identified with relationship to the lab notebook. Thus, a spectrum must contain the page of the lab notebook on which the material being analyzed was obtained. A standard procedure to this identification is to label the spectrum in your notebook as IR, NMR, etc. followed by the page number and a letter if more than one type of spectrum is on a page.
   Example:
   suppose one takes an IR and NNMR of the purified n-butanol and b-butyl bromide in the above example. The fact that these spectra were taken should appear in the notebook with labels IR-7A, IR-7B, NMR-7A

and NMR-7B, where 7 refers to the page number, A refers to the alcohol and B to the bromide. The spectra should have the student's initials, notebook number (if not, the first notebook and more than more than one notebook is eventually used) the page of the notebook and a letter (cap.) identification. Thus, the spectra in the above example would be labelled:
IN - IR - 7A
IN - IR - 7B
IN - NMR - 7A
IN - NMR - 7B
where IN are the student's initalis. If this was an entry in your second notebook it would read:
INII - IR - 7A, etc.

5. Indicate the structure and your lab book number in the left hand margin when a new compound has been identified.

Reporting Instrumental Data in your Notebook

Keep separate three ring black notebooks (locker 26) to keep your VPC, NMR, IR and UV spectra. Use the MS sheets (locker 26) to record your MS. Keep these sheets in a black, three-ring notebook.
Report the significant features of the spectral data in your notebook. Sketch NMR spectra and MS, and important VPC traces. Required information:

NMR Data:

- On the actual spectrum, put the date, your initials, notebook number and page number, compound reference number, purity, all the significant instrumental settings, a proposed structure, solvent, standard (external or internal), approximate concentration, integration

- In your notebook record the delta (d) values and integration. Make assignments and indicate coupling constants. Report literature comparisons where appropriate.
  Hint: when you have a sample in the A60 or A60A, obtain the maximum information possible in one setting. It is frustrating to see a neat-looking spectrum for which all peaks are nicely on scale yet a complex multiplet - which may be useful for analysis of the spectrum or a finger print of a compound - is so small in height that it is a piece of information thrown away. Take a second spectrum at a higher spectrum amplitude and expanded field in such a case. High gain sometimes reveals patterns of known impurities which are not recognizable at low gain.

IR Data:

- On the spectrum, put the same information as in (a) under NMR.

- In your notebooks, report the intense and characteristic bands. Indicate the band position in m (cm^{-1} if 520 is used), shape of the band (if it is not a sharp singlet) and the band assignment.

MS Data:

- Label on the original spectrum your initials, notebook number, page number, the compound reference number, the voltage, and the temperature of th einlet and the source (you have to ask what they are). Always have a 12-10 eV spectrum taken along with the 75 eV spectrum.

- Pick up MS sheets in locker 26 to record the pertinent data in a more convenient form. On these sheets

list all m/e values greater than 5% and all m/e values that are known to be of structural significance (even if less than 5%). Record metastables and make assignments of metastables and fragmentations where possible.

- Report significant fragmentations in your notebook with equations showing your proposed cracking pattern. Give literature references where appropriate.

UV Data:

- Label spectrum with usual notebook reference. Indicate also solvent and concentration (exact).
- In your notebook record the λ_{max} and ε_{max} of each band.

VPC Data:

- Label each trace you save with the usual notebook reference. Also indicate the flow rate, the column temperature, the inlet and detector temperature, the length, diameter, % and kind of column. Draw structures of peacks on the trace, if you know what they are.
- In your notebook sketch out a typical trace and list the operating conditions in (a). Draw structures corresponding to peaks. Indicate the time scale.

Compounds Prepared:

- Must be kept in a container with the following information:
  - Name or structure of chemical
  - Approximate quantity
  - Date
  - Initials
  - Notebook reference

Analytic Samples:

- All stable new compounds must have a carbon and hydrogen analysis (sent through Neil Schore).
- Solids must be crystallized to constant melting and liquids should be collected of V.P.C., or carefully distilled.
- The following should be reported in your notebook:
  Anal. for C_3H_4O
  MW = 56
  Calc.: C, 64.30% H, 7.14%
  Found: C, 64.50% H, 7.30%
  total acceptable diff = 0.6
  total, 0.3 on carbon
- A mass spectrum of other means of determining the molecular weight must be provided.

Miscellaneous

- Custom built apparatus or apparatus not easily recognized should be drawn.
- Always check the literature for new compounds you make. An IR which is identical to that of a known

compound in the literature constitutes a structure proof. You have sheets telling you how to do this.

Xeroxing

- The amount of xeroxing done should be kept ata reasonable level. All xeroxed material charged to a research account remains with the group after you leave. Do not do extensive xeroxing without checking first with me, since I may have a loanable copy of what you want.

Signing Out

- When you have completed your research program, there are three things which you must get into the best possible shape:
    1. your notebook and associated data must be complete
    2. your chemicals must be securely bottled with clear identification and notebook reference, and
    3. your lab equipment must be checked in, especially expensive items which are not being used by other members of the group.