

Laboratory Notebooks, Spectra, and Samples

The *Laboratory Notebook* is the primary scientific record of all experiments carried out by each investigator. A well kept, orderly notebook is essential to careful scientific research. The records kept by each researcher must be understandable to any informed investigator who wishes to reconstruct the experiments which have been performed. Although many styles are acceptable, all must conform to certain strict guidelines.

1. All pages of the notebook must be numbered sequentially, with the pages either permanently bound (preferred) or kept within a suitable binder. (Research labs at most chemical companies do not allow looseleaf notebooks.)

2. The data of each experiment, including the year, must be recorded. Since a notebook may be consulted many years after the original recording of the experiments, an accurate and complete date is essential.

3. Each experimental description should contain sufficient information as to what was done. For a chemical transformation, the reaction itself should be written out at the beginning of the experiment along with the quantities (mmol, mg, dpm, ml, etc) of each reagent. (For example: 2.5 ml (4 mmol) of 1.6 M *n*-butyllithium, or 1.2 mg (3 μ mol) of [1-³H] farnesyl pyrophosphate (2 x 10 dpm/nmol).) The first time a procedure is carried out it should be described in full. Subsequent experiments can simply list quantities used and refer unambiguously back to the relevant procedure (e.g. "procedure as on p. 10", or "procedure as described by C. Abell, Res Rpt 7/83, p 22). Statements such as "same as last time" or "by method of Felix" (Felix who?) are unacceptable. Similarly, the origin of all reagents should be described (Aldrich, or obtained from R. Lattman, or DEC-I-121). The yields of isolated products (mg, dpm, etc) should be measured and the % yield calculated. Melting points should be obtained on all solids.

4. For each sample isolated, a systematic numbering scheme should be used, based on the researcher name, notebook page, and the number of entries on that page. For example the crude product might simply be CAB II-45, while the two fractions isolated from a flash column might then be CAB-II-45-a and CAB-II-45.b. Such a system is especially important when multiple preparations of certain compounds are carried out. Simply identifying a substance as "nerolidol" does not allow identification of the method of production, nor the batch. Since even frequently used procedures often go wrong, the ability to look back at data from previous experiments is crucial to sorting out the perennial mysteries surrounding the sudden appearance of an apparently previously unobserved side product. New analytical techniques, or simply greater care, will often reveal phenomena which were there all along.

5. The laboratory notebook should also contain a record of all spectra recorded. Liquid scintillation data and gas chromatograms can now simply be inserted directly in the notebook. Similarly, thin layer chromatograms can either be resketched or imaged and transferred to the notebook page, properly labeled with a record of solvent, visualization reagent, and the position of the solvent front. All NMR, IR, and Mass spectra should be referenced to the laboratory notebook, and the notebook should contain a record of which spectra were recorded for which compounds and how they can be located (Spectrum No., NMR disk file, etc.).

6. Microbial strains, plasmids, and other recombinant materials: You should develop a systematic numbering system for all microbial strains, plasmids, and other recombinant materials that you generate. A good idea is to keep a master spreadsheet or other chart that lists key details such as strain or plasmid name, storage, use, sample number as well as a hierarchical tree that shows the source of each new prep (parent strain, plasmid, etc).

Spectra themselves can be simply numbered sequentially or grouped according to compound. In either case, the data, compound identification code, operator, solvent and spectroscopic parameters must be attached to the spectrum, either as part of the peak printout, or written directly on the spectrum. It is essential that all mass spectra note the instrument, ionization mode and conditions, GC conditions, and any other essential information for documenting how the spectra are recorded. It should therefore be possible to look at a spectrum and determine the appropriate reference in the laboratory notebook, or look at the laboratory notebook and determine where the relevant spectra can be found.

Samples should be stored in suitable containers, with proper labels, including identification code, and quantity, if known. Ground glassware should not be used for long term storage of routine samples, nor should NMR tubes. Most sensitive organic compounds are best stored under refrigeration. Solids are often more stable than oils or solutions. All labels should be firmly attached to the sample container. Long-term storage in a refrigerator will often result in loss of common stickers and/or washing away of soluble inks.

Publication of results requires that all new compounds be adequately characterized for purity and identity. These means that **all new compounds** should be characterized by ^1H NMR, ^{13}C NMR, IR, and high resolution MS. For chiral compounds, the α_{D} should be recorded, and any other relevant analytical data obtained. Please remember, it is a lot easier to record and store this information when the compound is first prepared, then months or years later when you are writing your thesis or a manuscript for publication. Centrifugation data should be reported as g values, not just rpm and rotor.