Advanced Preparation and The Notebook

Before coming to the laboratory you will find that some advanced preparation can save significant time and effort in the laboratory. The advanced preparation will vary depending on the nature of the experiment, but some general rules will be applicable to all experiments. Read the experiment first and identify all of the reagents and substances that will be used and prepared during the course of the experiment. Next, collect the physical properties of these materials before coming to the laboratory. Physical properties that will be useful include melting point and boiling points (if available) of all the reagents and expected products, their densities (if liquid) and some general solubility properties. Molecular weights should be calculated for all the reagents. This information should be recorded in your notebook which will be described below. While this may seem like a lot of work, many of these same reagents will be used repeatedly. Once recorded in your notebook, this material can be copied or a reference made to the page on which this information is recorded. In addition to the physical property listed, some reference, including pagination, to the source of the information should be cited. Included below are a few valuable sources of physical property data. Copies of these sources of material can be found in the Thomas Jefferson Library and also in the laboratory. A variety of editions are available for most.

"The Dictionary of Organic Compounds" Buckingham, J., editor, Chapman and Hall.
"CRC Handbook of Chemistry and Physics" Weast, R. C, editor, CRC Press.
"Aldrich Catalog Handbook of Fine Chemicals", Aldrich Chemical Co.
"Merck Index", Windholz, M. editor, Rahway N.J.
"Lange's Handbook of Chemistry", Dean, J. A., editor, McGraw-Hill.

The laboratory notebook is the permanent record documenting what you did in the laboratory during a specific period of time. It should contain sufficient details and documentation that an individual with similar training could repeat your work months or years later. The laboratory notebook should contain the original data, not copies of the data. The specific requirements for entries will vary from experiment to experiment and from instructor to instructor. The following is intended to serve as a guideline of what to include in the notebook. A substantial part of the grade earned in this course will be derived from the contents of your notebook.

a. Use a bound notebook. Notebooks with a spiral binding are not acceptable nor are any that allow for insertion or removal of pages.

b. Write in ink. It is expected that you will make mistakes. Just draw a line or an X through the material you wish to delete. Neatness is always appreciated and, if someone else cannot read and understand what you have written, you have failed to properly record your results.

c. Leave the first few pages of the notebook blank for a table of contents so that each experiment can be readily located. Pages should be numbered consecutively. You may leave some blank space on a page in order to complete an experiment but if necessary, a notation instructing the reader to where the remaining portion of the experiment is to be found, is acceptable. You may wish to include supporting documentation such as spectra that you have recorded. This documentation can be attached to the notebook with staples or tape or preferably, in a separate folder. In addition you may wish to sketch your thin layer chromatography results in your notebook since the actual plates may not survive repeated handling.

d. Some notebook preparation should be completed <u>before</u> you come to the laboratory. What is required will vary from instructor to instructor. However, the following will apply to all.

Begin with a title of the experiment and a brief sentence summarizing the purpose of the experiment and specify a reference (e.g., UMSL Chemistry 263 Manual, pp. 18-19). If the experiment involves a synthesis, include a balanced chemical equation, and physical properties of all chemicals involved. Include comments on toxicity if appropriate, any side reactions and anything out of the ordinary. Prepare an outline of the procedure and what you plan to do in the laboratory. The outline should be completed before you come to the laboratory (read Zubrick, pp. 12-24 for more detailed information) *but may be on a separate sheet of paper instead of the lab notebook*. **This will enable you to work more efficiently.**

In the laboratory, you should enter directly into the notebook the masses of the reagents you used and include what you actually did and observed as you performed the experiment. Be as brief as possible. Do not simply copy the experiment from the manual to your notebook. Include a sketch of the apparatus, if one is involved. Always include the date you did the work on the outside page margin.
Finally, write your observations and conclusions which is very important. When you comment on specific tests, (eg, the ferric chloride test) do not simply say the test was negative, add that it means you do not have a phenol or that you do not have salicylic acid present.

It may not be possible to follow this format for each experiment you will be performing this semester. However, for those experiments that involve some synthetic component, following this format will suffice. It is important to remember that your notebook is the medium where you record what you did in the laboratory and what you observed. If you keep this in mind you will stay on the right track.

Notebook Calculations

The number of calculations you will need to perform in the organic laboratory is quite small but those that you will be required to perform are very important. The calculations are similar to the calculations you performed in introductory chemistry and be sure to review them if you have forgotten the details and theory behind them. What follows is a brief summary of some important definitions.

The calculations associated with conversion of the starting materials to product is based on the assumption that the reaction will follow simple ideal stoichiometry. For example, in the preparation of aspirin from salicylic acid and acetic anhydride, in calculating the theoretical and actual yields, it is assumed that all of the starting material is converted to product, even though some of the starting material actually forms a polymer as a consequence of the reaction conditions and catalyst that is used.

The first step in calculating yields is to determine the **limiting reagent**. The limiting reagent in a reaction that involves two or more reactants is simply the reagent which is present in lowest molar amount based on the stoichiometry of the reaction. This reagent will be consumed first and will limit any additional conversion to product. In the reaction of salicylic acid with acetic anhydride, the latter reagent is used in excess. There are several reasons for doing so. An important consideration whenever any reagent is used in excess, is to determine how this reagent will be separated from the product at the end of the reaction. In this case, the acetic anhydride which is not water soluble can be allowed to stand in water a while during which time it will slowly hydrolyze to acetic acid. The acetic acid which is water soluble, can be separated from the aspirin by filtration.

The salicyclic acid will be the limiting reagent. As far as the calculation of the **theoretical yield** is concerned, we assume that every mole of salicylic acid will be converted to aspirin or acetylsalicylic acid. According to the stoichiometry of the reaction, one mole of salicyclic acid will be converted to one mole of aspirin. Therefore, from the number of moles of salicylic acid we used, we evaluate the maximum amount of aspirin that can be obtained. Multiplying the number of moles of aspirin by its molecular weight results in the theoretical yield which is usually reported in grams. In all cases, the calculation is performed similarly, regardless of how many products are formed. However, we can now evaluate the **actual yield** by determining how much aspirin we have actually isolated, experimentally. The **% yield** is simply the ratio of the actual yield divided by the theoretical yield times 100.

In a multi-step synthesis, each step in the process is characterized by a limiting reagent, an actual yield and a % yield. The calculation of the overall yield of the entire process can be calculated by identifying the limiting reagent in each step. The overall yield of the process will simply be the product of the actual yield divided by the theoretical yield of the first step times the same ratio for each step in the process times 100. Thus in a two step process, if you get a 0.5 yield (50%) in the first step and the same yield in the second step, your overall yield will be 0.5 x 0.5 x 100 = 25 %. You can imagine that a multistep process operating at this efficiency is not likely to be very useful commercially.

Labels

Properly label your sample vials when you submit your products for grading.

BENZOIC ACID (*appropriate, unambiguous name of the compound***)**
mp 120-121°C (*melting point observed for this sample***)**
3.2 g (*amount in vial***)**
D.L. Green (*the name of the person submitting the sample***)**
1/15/96 (*date submitted***)**
(DLG 12-14 *which is a reference to notebook pages concerning this material***).**

Properly label your spectra. The infrared spectra you decide to save should be labelled immediately. List the name of the compound or some identifying term if the name is unknown, your name, and the method by which the spectrum was obtained. The instrument will automatically print the date but, if it doesn't, be certain that you list the date it was recorded on the spectrum.