

# Instructions and Guidelines

## For 3-Ring Binder

### 3-RING BINDER

Your 3-Ring Binder should be large enough to hold all of your handouts/downloads (such as this one). All handouts/downloads should be punched (there is a 3-hole punch in the lab), stapled, dated and inserted in chronological order within the appropriate section. The 3-ring binder should be divided *with* labeled tabs into the following sections:

1. Lab Safety Sheets and Course syllabus
2. Additional Notes on Experiments & Procedures (handouts and notes that you take in class must be in this section).
3. Quizzes.
4. Worksheets
5. Graded Labs Reports\*

*\* Any missing graded lab reports will result in a zero for that lab report..*

# Instructions and Guidelines

## For Laboratory Notebooks

The lab notebook will determine a significant part of a student's course grade. Students should keep it with them at all times when working in the laboratory. All of the following directions should be followed carefully; it will also be helpful to periodically review the following guidelines.

**Before the second class meeting, please complete the following directions:**

1. Purchase a bound, quadrille-ruled notebook (National 43-475 or equivalent).
2. Label the front cover of the notebook in the following format:

LABORATORY NOTEBOOK  
Orange Coast College  
*Student's Name*  
CHEM 226  
Fall, 2011

3. Label the inside front cover of the notebook in the following format:

IF FOUND, PLEASE RETURN TO:  
*Student's Name*  
*Student's Phone Number*  
*Student's E-mail Address* (optional)

4. Number all pages, starting with page 1, in the upper right-hand corner in blue or black ink.
5. On the top of page 1, prepare an Index Title. Reserve pages 1-4 for the Index. The first experiment should begin on page 5. Label the Index exactly as follows:

INDEX OF EXPERIMENTS  
CHEM 226  
Fall, 2011  
*Student's Name*  
Locker Number: ##

6. You should draw the following table below **EXACTLY** in your INDEX TITLE on the first page of your lab notebook. Be sure to maintain your INDEX during the semester.

| Date Started | Instructor Pre-lab Approval | Experiment # and Title | Date Completed | Instructor Initials | Pages |
|--------------|-----------------------------|------------------------|----------------|---------------------|-------|
|              |                             |                        |                |                     |       |
|              |                             |                        |                |                     |       |

## **General Lab Notebook Guidelines:**

The following general guidelines should be observed throughout the semester. These are presented in no particular order. All are important. Following the guidelines described here and elsewhere will help maximize points earned on the notebook. Failure to follow these guidelines will result in lost points. It is strongly recommended that students frequently review the guidelines in this handout throughout the semester.

1. **Blue or Black Ink:** Use only waterproof, indelible black or blue ink to write in the notebook. Do not use red ink. Do not use pencil except for graphs, marking TLC plates and for calculations. If you make a mistake, do not erase with white-out or write over the mistake. Instead, line through incorrect data and write correct data next to it. Cross-out whole paragraphs or sections if necessary.
2. **Recording Data:** Data must be recorded directly in your notebook as you collect data. Data must never be recorded on separate sheets of paper to be copied later into the notebook.
3. **Record Truthful Data:** The statements you make in your notebook must be truthful and accurate. Dry-labs, exaggerating yields, falsifying data, etc. will result in your receiving a grade of "F" in this course on the first occurrence.
4. **Keep Sequential Work:** Your work in your notebook must be sequential. Do not leave blank pages in your notebook to be filled in later.
5. **Fragmenting Work:** I want you to avoid, as much as possible, fragmenting your work and fragmenting your notebook. Unless the instructor gives you written permission, complete all of one lab before starting the next. Of course, waiting until the spectrometer or the gas-chromatograph is available makes this often difficult or impossible. Hence, you may leave a nearly blank page or two if you title these pages and have your instructor initial them.
6. **Index:** Keep your index current at all times. Be sure to have the instructor inspect and initial as soon as possible.
7. **Starting a New Experiment:** Each new experiment should be started on a new odd-numbered page of the notebook (right hand side). You should also cross-out any unused portion of a previous page.
8. **Start and Stop Points:** Please make it absolutely clear where you stop and start work, particularly in your data and observation section. Additionally, I want you to print STOP @ (date) & (time) at the conclusion of lab work in your data and observation section. Make it clear where you stop and start! In addition, you must obtain my signature at the stop points at the end of each lab period.
9. **Initial Work:** Initial each page as it is completed, and have your instructor initial your day's work at the end of each lab (at your stop point).
10. **Notebook Pages:** Pages must never be removed from your notebook. Additionally, the only pages that may be added to your notebook are spectra, chromatograms, separation schemes, and diagrams of apparatus.
11. **Punctuality:** Unless instructed otherwise, you are to finish one experiment before starting another.
12. **Turn in Notebooks:** Your notebook must be turned in at semester's end to receive a passing grade in the class. Furthermore, your notebook will be checked at several unannounced times during the semester.

13. **New Notebooks:** If you start a new lab notebook, you must turn in the old one. All notebooks must be turned in at the end of the semester. If you want your notebook back, you may obtain it after one year. After two years, notebooks are destroyed.
14. **Time Management:** You are held accountable for responsible time management. Frequent absences, continuous late arrival and / or early departure from lab will seriously lower your grade. If you need to attend another lab section to catch up, you need to secure the instructor's permission and have your instructor record each occurrence.
15. **Inserted Information or Data:** Reserve a full blank page for chromatograms and spectra or handouts. Do not tape chromatograms, spectra or handouts over data or observations, instead, tape them to a blank page. Taped-in pages should never protrude beyond the edges of your notebook. Under no circumstances should there ever be loose chromatograms, spectra, TLC plates left loose in your notebook. Tape them!

### **Lab Notebook Format**

The following list details all of the possible sections which might appear in an experiment, along with some instructions for how to complete each section. **They appear in the order in which they should normally appear in an experiment.** Each of the following sections should be indicated with a heading in the lab notebook, and the heading should be in boxed.

#### **NAME**

Be sure to write your name at the top portion of your lab.

#### **DATE**

Record the date that the experiment was first entered into the lab notebook. This may be several days before the actually experimental procedures were performed.

#### **TITLE**

The title should have the following format: *Lab ## : Title.*

#### **INTRODUCTION**

This should state what you intend to do (synthesis of ...; or extraction of ...) and how you intend to do it (indicate methods of separation and analysis). If a named reaction or method is used, be sure to mention it. This section should be a quarter to a half of a page long and should not contain any theory.

#### **HAZ-MAT**

There are several sources of haz-mat data found in the lab; in addition, MSDS data can be found on the web. Present a table of all reagents along with their health (HE), flammability (FL) and reactivity (RX) ratings plus any additional safety comments. If the HE, FL, RX, cannot be found, then be sure to include additional information under comments. An example is shown below.

| REAGENT            | HE | FL | RX | Contact | SOURCE     | COMMENTS                             |
|--------------------|----|----|----|---------|------------|--------------------------------------|
| Diethyl Ether      | 2  | 4  | 1  | ----    | NFPA       | Use in hood, avoid heat              |
| Methylene Chloride | 2  | 1  | 0  | 2       | Baker p.## | Avoid contact, absorbed through skin |

### Apparatus

You should include relevant drawings of complicated glassware setups.

### CHEMICAL EQUATION

Chemical equation(s) for the main reaction(s) must be included for preparative experiments (synthesis). In general, the equations should feature structural formulas and each structure should be labeled with the appropriate name. Equations should be balanced & complete and structures of isolatable intermediates (even if the intermediates were not isolated) should be shown. Omit this section if no chemical changes occur.

### SIDE REACTIONS

If known, the chemical equations (or at least a brief description) of the side reaction, if any. Side reactions are sometimes discussed in lab text or given as lab notes. Omit this section also if no chemical reaction occurs.

### REACTANT TABLE

In preparative experiments, an important part of the planning process is determining the identity and quantities of the reactants. This is conveniently done in a table called the *Reactant Table*. The format for the reactant table will be detailed later in the semester when we begin doing preparative experiments. Also, a product table must be included when reactions are performed (product name, actual yield, theoretical yield and percent yield).

### SEPARATION SCHEME

For most experiments, a flow-diagram should be included which outlines how the product is isolated, separated, and/or purified. Clearly indicate any separation techniques & any chemical changes occurring during separation. (See your lab text.) Your separation scheme must illustrate any chemical changes with the appropriate structural formulas. Once a structure is illustrated, it may be abbreviated until changed again.

### PROCEDURE AND OBSERVATIONS

Prepare a numbered outline of the experimental procedure on the left third of one or more pages in your notebook. Include only sufficient information in each step so as to guide you through the actual experiment but sufficiently complete so as not to require frequent referral to the lab textbook. Since this is only an outline of the procedure, leave ample blank spaces between each numbered step. Note that if any procedural step is modified or changed from what is given in the text, clearly note such changes directly in your notebook. As you perform the experiment, you will then enter detailed observations (on the right two thirds of the page) made during the course of the experiment. See pages Pavia for an example of detailed observations. A sample record book page is attached on the last page of this document.

## CALCULATIONS

If there are any required calculations, you must include (sample) calculations with an appropriate sub-heading or label. Examples of sub-headings include "Determination of Limiting Reagent"; "Theoretical Yield"; "%-Yield"; "%-Recovered" (See Pavia).

If a GC-analysis is performed, have a subsection titled "GC-Analysis" followed by the necessary calculations. For all calculations, include an algebraic formula or word expression before numerical substitutions. For all calculations, be sure to include proper units and that the number of significant figures in your answer is appropriately determined by the number of significant figures in the measured values.

## PRODUCT TABLE

A product table must be included when reactions are performed. Product name, actual yield, theoretical yield and percent yield should be reported in a table here.

## DISPOSAL of REAGENTS and PRODUCTS

While you may have already mentioned in your procedure what you did with excess reagents and/or waste, you should explicitly state how all reagents (by name) were disposed: include information about the container and any other pertinent information.

## CONCLUSIONS

The Conclusions section provides a summary and analysis that links the Procedures and Observations to the objectives of the experiment. It should be completed as soon as possible following the completion of the experimental work. In laboratory research, the conclusion provides an opportunity to evaluate the success of a hypothesis, and it also is an appropriate place to suggest plans or further experiments.

The Conclusions should be written as much as possible in complete sentences. Each experiment will have a unique organization for its conclusion; however, there will be several reoccurring themes. Some examples might include:

- *Discussion of Percent Yield and Purity*  
A restatement of the calculated percent yield of the experiment, along with a summary of any data that supports the identity and purity of the product (MP, IR, etc..). Any physical properties measured should be explicitly compared to the literature values for those properties; cite the literature value and a source for the value. In experiments in which the desired product was not obtained or the purity and/or yield was unusually low, a reasonable suggestion or educated speculation of what may have gone wrong would be appropriate.
- *Modifications to the Planned Procedure*  
Any important modifications to the planned procedure should be described, along with a rationale for the modification. This might include any measures that were necessary because of mistakes in the execution of the procedure.
- *Suggested Improvements to the Procedure*  
Sometimes, it may be possible to suggest improvements for the procedure. These should be noted here, along with a rationale for the suggested modification.
- *Evaluation of Experimental Objectives*  
A few sentences that indicate the success of the experiment relative to the stated objectives. Examples might include "This is a successful method of making compound xxx as stated in objective one" or "In step ### of the procedure, the experiment provided additional practice in technique XXX as stated in objective two."

- *Interpretation of Spectral Data*

If an IR spectrum is obtained, the important peaks in that spectrum should be listed and analyzed. For each important peak, indicate the structural features or types of bonds that might give rise to the absorption. List any conclusions that might be drawn about the functional groups present in the molecule. Always include the reference literature spectrum for the compound, and explicitly compare the important peaks in the experimental spectrum to corresponding peaks in the literature spectrum, listing the value in wavenumbers for the absorption.

This is not an exhaustive list. Other subsections may be useful or appropriate. Try to take some time when preparing the Conclusions to think about their organization and content, as well as the significance of the experiment and its results.

### SPECTRA and CHROMATOGRAMS

Attach spectra or chromatograms (if any) directly to one or more pages of your notebook at the end of the experiment. Tape and fold spectra or chromatograms so that they do not fall out of the notebook and so that no part of the attached pages protrudes from the edges of your notebook. The part of the attached page should be taped on at three sides and no part of these taped pages should cover any other information. These pages should be folded so they may easily be examined (don't staple them shut!). Be sure that all attachments are clearly labeled and identified as well as referenced in the procedure and observation section.

### **PreLab Requirements:**

You must include the following sections in your notebook before starting the experiment. Your prelab will be checked for completeness before you are allowed to begin an experiment.

- |   |   |
|---|---|
| 1) Name                                 | 8) Side Reactions (when applicable)     |
| 2) Date                                 | 9) Reactant Table (when applicable)     |
| 3) Title                                | 10) Separation Scheme (when applicable) |
| 4) Introduction                         | 11) Procedures                          |
| 5) Haz-Mat                              |   |
| 6) Apparatus drawings (when applicable) |   |
| 7) Chemical Equations (when applicable) |   |

### **Additional Lab Notebook Guidelines**

#### 1. **Observations:**

- Procedure:** Many students spend too much time writing detailed steps of the procedure and not nearly enough time writing detailed observations. Please note that unless you deviated substantially from that given in the text, you need only outline the procedure.
- Observations:** Your observations that must be detailed and also describe not only the reactants, but also any and all changes including phase changes, color changes, temperature changes, rate of changes, etc. If the procedure instructs you to watch for certain changes, be sure to note whether or not these changes were observed.

- c. **Melting Points:** When reporting melting point ranges or boiling points, record the temperature to the highest precision allowed by the thermometer (1°C or 0.1°C). Additionally, when measuring melting points, be sure to include a description of the behavior of the solid on melting (softening, sweating, discoloration).
- d. **Phase Changes:** Be sure to describe any phase changes such as formation of bubbles, formation of a precipitate, dissolution of a solid, formation of a second liquid layer.
- e. **Properties of Mixtures:** Be sure to note degree of clarity of all mixtures (clear, slightly cloudy, cloudy, opaque). A clear solution that turns cloudy may be due to formation of a finely-divided precipitate, formation of tiny bubbles, or formation of a nearly immiscible liquid. Note any unusual appearance at the interface between two liquid phases. Note if an emulsion is present.
- f. **Reaction Time:** Be sure to note time/ rate of reaction / change. You must record how long you mixed, heated, observed, etc. Semi-quantitative descriptions are good (briefly, slowly, immediately, quickly, etc.) but quantitative descriptions are best (approx. 15 s, 5 min., 1.5 h).
- g. **Appearance of Solids:** Describe the appearance of all solids collected as seen under the dissecting scope (in the balance room). You are required to describe the crystal form, if any, of solids (needles, cubic, rhomboid, amorphous, etc.)
2. **Apparatus and Instruments:** Always indicate the type / brand / or model of apparatus used. For volume transfers (Pasteur pipet, syringe, calibrated pipet, etc.). For filtrations (Buchner funnel, Hirsh funnel, filter-tip pipet, etc.) Also indicate which apparatus was used for m.p., b.p. determinations, etc....
3. **Extractions:** When doing extractions, refer to both the type and position of each layer. For example, "The top hexane layer, the bottom methylene chloride layer, the top organic layer, the bottom aqueous layer, the ether layer."
4. **Distillation:** When performing a distillation, give appropriate descriptions to the separated portions. For example, "the original liquid mixture, the distillate, the first fraction, the second fraction."
5. **Gas Chromatography (GC):** Column information, flow rate, amount & identity of injected sample must be recorded directly on your chromatogram at the time the chromatogram is obtained. If you use the cut & weigh method of analysis, do not cut your original chromatogram. Instead, make a good photo-copy of your peaks, and then cut & weigh from the copy. Your original should be trimmed, taped, and fan-folded neatly onto a blank page in notebook.
6. **Thin Layer Chromatography (TLC):** In addition to noting the R<sub>f</sub> values, be certain to record the visualization methods used and the shape of the spots. You should also record on each TLC plate; the solvent system used, the identity of the spotted material, and the number of times the material was spotted at the bottom immediately below the spotted material. Organize all of the information in tabular form. Remember, that TLC plates must never be loose within your notebook: Until you are finished with them, they should be numbered and kept in a labeled container in your lab drawer. When you are finished with them they are to be taped into your notebook. If you are using both UV as well as iodine visualization methods, make a sketch of the plate after UV-visualization, making detailed notes of the appearance of each spot. Also, record the type of plate being used: silica gel, alumina.

7. **Infrared Spectra (IR):** A printed spectrum in the range of 600 to 4000  $\text{cm}^{-1}$  should be obtained. Tape the spectrum neatly into your lab book. Be sure to include a copy of a reference IR in your lab notebook. Draw the structure of the anticipated compound in each spectrum and analyze as described below:
  - a. Several peaks (all of the functional group peaks and some in the fingerprint region) of each IR spectrum should be analyzed as follows:
    - i. The wavenumber you measure for the particular peak should be printed immediately below the peak.
    - ii. Below this, indicate whenever possible the probable origin of functional group peaks (O-H stretch, C-H bend, etc.).
    - iii. See the Appendix on IR Analysis at the end of Pavia for instructions on spectrum analysis of each functional group. You will be graded on both the quality of your spectra as well as the quality of the analysis.
  
8. **Reagents:** When performing any experiment, it is standard laboratory practice to record all source information regarding a reagent used. In particular you are to record (if known) manufacturer, lot number, bottle number and purity; additionally, record any identifying features (such as size and bottle type). While this may seem a little strange, it is for a good reason. Often when a student fails to obtain the expected results, the problem may lie in the reagent used.
  
9. **Submission of Products:** You may be asked to save and submit products as the semester progresses.

