The Laboratory Notebook for Chem 269.

See the grading rubric posted on the course website. Your TA will use this as a template to grade your reports.

Use only the required notebook, one that allows a copy of each page to be made and torn out. The copy is given to the TA for grading and the original is kept by the student. Entries should be dated and entered in ink. Write-ups for the technique experiments are done in the notebook. Synthetic experiments require a formal report. See the Syllabus and documentation on the course website.

Note that there are two basic types of experiments – Techniques and Synthetic. Each will have a slightly different format and a different grading rubric, both of which are posted on the website. Technique experiments are those that are done to learn a new lab technique and comprise the first five experiments. These are done in the first half of the semester. In these types of experiments, lab chemicals are used but no chemical reactions are carried out so the chemicals do not change (chemically) from beginning to end. An example of such an experiment would be the purification of a crude compound by a technique called recrystallization. Impure compound X is recrystallized to produce pure compound X. No chemical change has occurred. In a synthetic experiment, starting with compound Y, one carries out a chemical reaction to produce compound Z. In both, one would make a table of all chemicals used, along with their physical properties (melting point, boiling point, density, hazards, etc). The difference lies in the measure of loss of product during the operation. In a technique experiment, one simply calculates a % recovery. If you start with 0.060 g of compound X, and recover 0.045 g after carrying out the technique, the % recovery is simply (0.045/0.060) x 100 = 75% recovery. The loss is simply a result of mechanical losses that occur in carrying out the technique. In a synthetic experiment, the molecular weight (MW) of product is usually different that the MW of reactant, so a % yield must be calculated to measure the effectiveness of the experiment. The loss in such a case is not only the result of mechanical losses but also of side reactions occurring that would consume reactant. The first technique experiment is the melting point lab and the first synthetic experiment is the synthesis of an ether.

Prelab Entries Should Include:

- A heading which includes: your name, your TAs name, Date which work was performed, your lab section day and time and the title of experiment.
- A short description of the purpose of the experiment.
- A Balanced reaction (if applicable) using structural formulas. Include side reactions and products.
- Table of reactants, products, and side-products. This should include molecular weight (MW), relevant physical properties such as melting point (MP), boiling point (BP), density, solubility information, toxicity, and quantities used (moles, grams, milliliters). All physical properties are not always needed for all the compounds. For example, BPs would be needed if a distillation were to be carried out, but including the MP of a liquid would not be useful. Solvent and catalyst data should also be noted (e.g., BP, toxicity, density). Such information can be found in the “CRC Handbook of Chemistry and Physics”, available in the library, in the organic lab, and on the library website. Chemfinder.com and web searches in general can also be helpful here.
- Prelab outline. This is the heart of the prelab preparation. It is a short version of the lab procedure, written in outline form, in your own words. It is based on the experiment handout and information gained from the OWL prelab exercises. Read the handout carefully, do the OWL assignments and synthesize a prelab outline. The purpose of the
outline is to help you prepare for the experiment and carry it out in the lab. Trying to do the experiment directly from the handout, without having prelab outline is inefficient and ineffective. You will likely need more time to do the experiment that way and will likely have errors. It is also very unsafe to work in an unprepared manner. Work will not be allowed until the TA has checked and accepted the prelab outline. If you come to lab without having an acceptable outline, you will be asked to leave and return only after you have an acceptable outline. The prelab is worth 5 points and you will receive an automatic 1/5 for the prelab if you come to lab with no outline. Also, you will unlikely have sufficient time to complete the experiment, leading to further loss of credit. Include safety and waste disposal instructions in the outline, as well as sketches of equipment.

In‐lab Entries.

As you work in the lab, enter directly into the notebook, the actual procedure that you follow. This will differ somewhat from the prelab outline. The prelab outline is what you expect to do. The in‐lab entry is what you actually do. Measurements such as weighings, MPs, BPs, and chromatographic and spectroscopic data should be entered as the data is determined. Careful observations should be noted (“the flask contents turned deep red, smoked profusely, and exploded, blowing a two foot hole in the lab bench.”) The idea here is to provide as much detail as you can so that another worker could exactly reproduce what you did and get the same results. When you work in a research lab, this is extremely important so learn it and practice it here.

Before leaving the lab, clean up your workspace and any common workspaces that your TA may ask you to clean up, and get the TA to sign the notebook right after the last entry. Unsigned work will not be given credit.

Postlab Entries. Results, Discussion, Answers to Assigned Questions.

Calculate % recoveries (starting with compound X, doing an operation on it, and recovering compound X, what % did you recover?) and/or % yields (starting with compound X, carrying out a reaction, recovering compound Y, how much did you recover and how did that compare to the theoretical amount? Moles and stoichiometry needed.) Summarize results in an easy to read format (e.g., compound X, MP 101.5-103.5° C, 0.067 g obtained, 56% yield). Interpret spectra and chromatograms, write a brief (less than 1 page) conclusion, and answer any assigned questions.

In all cases, NEVER write in the first person. Instead of "I added xxx to a Erlenmeyer flask and I swirled it . . . ," use "xxx was added to an Erlenmeyer flask and was swirled for 10 min in an ice bath."
A partial sample report for a synthetic experiment.

**Name:** John Connor  
**TAs Name:** Connor Smith  
**Experiment Date:** 3/1/2016  
**Lab Section Day and Time:** Monday, 1:25 PM  
**Title:** Synthesis of 1-Bromo-3-methylbutane.

**Purpose:** The purpose of this experiment is to synthesize 1-bromo-3-methylbutane by an SN2 reaction of 3-methylbutanol with hydrobromic acid. The HBr *in situ* by reaction of sodium bromide with sulfuric acid. The product is purified by distillation and analyzed by gas chromatography (GC).

**The Reaction:**

\[
\text{3-methylbutanol} + \text{H}_2\text{SO}_4 + \text{NaBr} \rightarrow \text{1-bromo-3-methylbutane}
\]

**Table of Reagents:**

<table>
<thead>
<tr>
<th>Compound</th>
<th>MW (g/mol)</th>
<th>Amount Needed</th>
<th>moles needed</th>
<th>BP</th>
<th>MP</th>
<th>Density</th>
<th>Hazards</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-methylbutanol</td>
<td>88.148 g/mol</td>
<td>0.90 g, 1.11 mL</td>
<td>0.0134</td>
<td>131.1 °C</td>
<td>--</td>
<td>0.8104 g/mL</td>
<td>None</td>
</tr>
<tr>
<td>NaBr</td>
<td>102.894</td>
<td>1.18 g</td>
<td>0.0115</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>98.079</td>
<td>1.07 mL</td>
<td>--</td>
<td>337 °C</td>
<td>--</td>
<td>1.84 g/mL</td>
<td>highly corrosive strong acid/oxidizer</td>
</tr>
</tbody>
</table>

**Proposed Set-up**

![Diagram of the experimental setup]
Outline:
- weigh 1.18 g NaBR into a 5 mL round-bottomed flask.
- add 0.90 g 3-methyl-1-butanol and 1 mL water to flask
(USE EXTREME CAUTION IN USING SULFURIC ACID)
- carefully add 1.07 mL (1.96 g) conc sulfuric acid with constant swirling.
- add a boiling chip
- connect distilling head and attach receiving vial.
- place receiving vial in 50 mL beaker containing ice-water. Beaker held in place with large three-pronged clamp.
- heat flask for 1 hr
- increase heat to distill crude product.
- (and so on and so on.)
- distill dried product into a clean, dry tared vial using a fractional distillation set-up (sketch set-up), collecting product over a temperature range of about 117-121°C.
- measure amount obtained
- carry out GC
- WASTE: place all liquid waste into the ORGANIC LIQUIDWASTE container in the fume hood.

In Lab Observations.
Actual mass of NaBr used: 1.180 g.
Actual amount of 3-methylbutanol used: 1.20 mL
Actual amount of sulfuric acid used: 1.10 mL

Upon addition of acid, the solution became quite warm. Solution remained colorless throughout reaction. Procedure from pre-lab/experiment handout was followed.

See "The Formal Report" handout on the website for results and discussion section and a properly written experiment procedure example.

Answers to Assigned Questions.